Functional Ceramic Materials, Oxide Thin Films and Heterostructures

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Symposium D

Functional Ceramic Materials, Oxide Thin Films and Heterostructures

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Scope of Symposium
This symposium is aimed at providing a forum for participants from academic, research and development, and industrial communities worldwide to exchange latest information and progress on functional ceramic materials, oxide thin films and heterostructures. It also addresses the future development of functional ceramic materials and oxide thin films for electrical, electronic, magnetic, optical, optoelectric and functional applications. Topical areas will cover all the new and rapidly developing areas of functional ceramic materials, oxide thin films and heterostructures, fabrication, physical and functional properties as well as their applications.

Symposium Topics
- Ferroelectric and piezoelectric ceramic materials
- Electronic properties of complex oxide thin films and heterostructures
- Multiferroics
- High K and low K materials
- Transparent conducting oxides
- High-Tc superconductors
- Magnetic materials and applications
- Mesoporous materials, nanohybrids and thin films
- Materials for photovoltaics and sustainable energy
- Complex oxide thin film growth techniques with atomic control
- Mott metal-insulator transition
- Interface electronic effects
- Defects and dopants
- In situ diagnostics (e.g. RHEED at high oxygen pressures)
- New characterization techniques
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Balaji P MANDAL; Avesh K TYAGI

A02503-04300 Synthesis and Photoluminescence Properties of (Gd$_{1-x-y}$Zn$_x$Tb$_y$)PO$_4$ (0≤x≤0.06 and 0.06≤y≤0.15) Phosphors
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A02516-04329 Enhanced Dielectric Properties of Low Temperature Sintered Ferroelectric PMN-PT (65:35) Ceramics
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A02528-04326 Textured Growth of Terbium Iron Garnet Thin Films as a High Coercivity Media
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A02536-04342 Transmitting Conducting Oxide Layers for Thin Film Silicon Solar Cells
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A02539-04348 Oxide Film Assisted Dopant Diffusion in Silicon Carbide
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A02547-04619 Characterization of a Refractory Crucible for the Drawing of Silicon Ingots for Photovoltaic Applications
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Interplay of Nanoscale Phases in Some Ferroelectric Oxides
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Structure and Photoactivity of ZnO Derived from Microwave Assisted Chemical Routes with Different Precursors
Shahab KHAMENEH ASL; S.K. SADRNEZHAAD; Mansour KIANPOUR RAD

Epitaxial Growth of β-FeSi₂ Thin Film on SiC
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Tailoring Glass Surfaces via Internal Cationic Diffusion
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Electromagnons, Ferroelectric Soft Modes and Magnetoelectric Coupling in Multiferroics
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Investigations on Room Temperature Magnetoelectric Multiferros
Ram S. KATIYAR; A. KUMAR; M. K. SINGH; R. PAL; J. F. SCOTT

The Ultraviolet Electroluminescence of the ZnO Nanorods/ Organic Heterojunction Devices
Sailing ZHAO; Xu ZHENG; Pengchi KAN

Magnetoelectric Effects in Composite Thin Films of Ferroelectric and Magnetic Oxides
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Deposition Temperature Dependent Electrical Properties of BiFeO₃ Thin Films with SrRuOₓ Buffer Layer
Jiangang WU; John WANG

Synthesis of Corundum Nanocrystals
Georgy PANASYUK; Lidiya AZAROVA; Igor VOROSHILOV; Victor BELAN; Dmitry SHABALIN; Irina KOZEROZHETS

Structural and Electrical Properties of BiFeO₃ Thin Films Prepared on SRO Buffer Layers of Varied Thickness
Rongyan ZHENG; John WANG; Seeram RAMAKRISHNA

Effect of Heat Treatment on Structural and Optical Properties of Sol-gel Spin Coated Nanocrystalline TiO₂ Thin Films
T.S. SENTHIL; N. MUTHUKUMARASAMY; K.V.R. MURTHY; R. BALASUNDARAPRABHU

Fatigue Behavior of Heterolayered Lead Zirconate Titanate Thin Films
Fransiska Cecilia KARTAWIDJAJA; John WANG

Pulsed Laser Deposition (PLD) of the Gd₂O₂S:Tb⁺³ Thin Films
JJ DOLO; HC SWART

Electrical Field Control of Ferromagnets Using Multiferroics
Ramamoorthy RAMESH

A Novel and Facile Synthesis of Hybrid Silica Nanocapsules and its Application in Fluorescence Imaging
Christopher HAPPY; Nicole LIU; Beiping HE; Siew Yee WONG; Zhi-Kuan CHEN; Xu LI; Wang JOHN

Positron Annihilation Studies on Gamma-Irradiated Barium Zirconate Ceramic
Aparna SHETTY; V M JALI; B R BHAT; R GOVINDARAJ; S B KRUPANIDHI

Labile Ferroelastic Nanodomains in Bilayered Ferroelectric Thin Films
Varatharajan ANBUSAHTAIAL; Daitsuke KAN; Fransiska C. KARTAWIDJAJA; Reza MAHJOUB; Miryam A. ARREDONDO; Samantha WICKS; Ichiro TAKEUCHI; John WANG; Nagarajan VALANOOR

Co-sensitization of Porous TiO₂ by PbS Quantum Dots and Ruthenium Based Organic Dye N719
Yanqiong LIU; Zhibin XIE; John WANG

Hierarchically Nanostructured ZnO for Dye-sensitized Solar Cells
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Templated Mesoporous Titania for Dye-Sensitized Solar Cell Applications
Yu ZHANG; Zhibin XIE; John WANG
A02815-04821 Modulated Charged Defects and Their Effects on Electrical Behaviors of BiFeO$_3$-based Thin Films
Yang WANG; John WANG  

A02840-04861 Thickness Dependent Properties of Epitaxial BiFeO$_3$ Multiferroic Thin Films
Huaqin LIU; John WANG; Kui YAO  

A02853-04880 Effects of Anodization Parameters on Titania Nanotube Arrays and the Performance of Dye-sensitized Solar Cells
Zhibin XIE; Stefan ADAMS; John WANG; Daniel BLACKWOOD  

A02860-04891 Relaxor Ferroelectric Behaviors in Sr$_{(La_{x}Nd_{1-x})_{2}}$Ti$_4$Nb$_{6}$O$_{30}$ Tungsten Bronze Ceramics
Xiang Ming CHEN; Xiao Li ZHU  

A02862-04887 Dielectric Properties Bi$_{3.15}$Nd$_{0.85}$Ti$_3$O$_{12}$ Ceramic with a Core-Shell Structure
Guangqing KANG; John WANG; Kui YAO; Jiagang WU  

A02866-04904 Epitaxial Functional Oxide Films: A View From the World of Ferroelectrics
Stephen STREIFFER; Matthew HIGHLAND; Timothy FISTER; Marie-Ingrid RICHARD; Dillon FONG; Jeffrey EASTMAN; Paul FUOSS; Carol THOMPSON; Brian STEPHENSON  

A02890-04938 Engineering Exotic Phenomena at Ferroelectric Oxide Interfaces : A First-principles Perspective
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A02891-04939 High Temperature Interface Superconductivity in Copper Oxide Multilayers
Adrian GOZAR; G. LOGVENOV; A. T. BOLLINGER; I. BOZOVIC  

A02909-04974 Optimization of Thickness of Magnetron Sputtered MgO Thin Films Thickness as Protective Layer for Plasma Display Panels
Chandra Bhal SINGH; Vandana SINGH; Debjit DATTA; Surajit SARKAR; Satyendra KUMAR  

A02909-04975 Effect of Deposition Parameters on Structural and Surface Characterization of Magnetron Sputtered MgO Thin Films
Chandra Bhal SINGH; Surajit SARKAR; Vandana SINGH; Satyendra KUMAR  

A02910-04976 Characterization of Si doped MgO Thin Films for Plasma Display Panels
Surajit SARKAR; Pradeep Kumar RA; Chandra Bhal SINGH; Vandana SINGH; Satyendra KUMAR  

A02917-04985 Novel Ferroelectric Capacitor for Non-volatile Memory Storage and Biomedical Tactile Sensor Applications
Shiyang LIU; Lynn CHUA; S. E. VALAVAN; Kian Chuan TAN  

A02917-05045 Multilayered Ferroelectric Micro Sensors for Biomedical Applications
S. E. VALAVAN  

A02941-05026 Structure and Properties of Domain Walls in Multiferroic BiFeO$_3$ Thin Films
C. T. NELSON; Y. ZHANG; Y. B. CHEN; C. M. FOLKMAN; S. H. BAEK; C. B. EOM; Xiaoping PAN  

A02944-05033 An Investigation of Structural, Magnetic and Dielectric Properties of R$_2$NiMnO$_6$ (R = rare earth, Y)
K. V. RAMANUJACHARY; R. J. BOOTH; R. FILLMAN; H. WHITAKER; Abanti NAG; R. M. TIWARI; J. GOPALAKRISHNAN; S. E. LOFLAND  

A02947-05037 Fabrication and Mechanism of Large Area and Uniform Silver Dendritic Micro-structures
Hui LIU; Yang YANG; Xiaopeng ZHAO; Zhenfeng ZHU  

A02989-05108 Synthesis of ZnO Nanorods and Highly Textured Films by Hydrothermal Route
Tong LI; H. M. FANG; J. M. XUE; J. DING  

D-00001 Solid State Catalysis Of Epitaxial Oxide Thin Films
S. DHAR; F. ROCCAFORTE; K. P. LIEB; T. VENKATESAN  

D-00002 Femto-Second Laser Excitation Studies Of Oxide Thin Films And Heterostructures
X. WANG; G. YOU; Q-H. XU; J. HUIBEN; H. HILGENKAMP; T. VENKATESAN; ARIANDO  

D-00003 Ta Doped TiO$_2$: Substitutionality and Bandgap Variation
A. Roy BARMAN; S. DHAR; M. R. MOTAPOTHULA; T. K. CHAN; M. BREESE; G. OSIPOWICZ; T. VENKATESAN
Influence of A-site Modification on the Dielectric, Ferroelectric and Relaxor Behavior of Lead-Free BaBi$_4$Ti$_4$O$_{15}$ Ceramics
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Polycrystalline powders of lead-free Ba$_x$La$_{1-x}$Bi$_4$Ti$_4$O$_{15}$ ($x=0$-$0.8$) and Ba$_{1-(3/2}$$y$La$_{3/2}$$y$Bi$_4$Ti$_4$O$_{15}$ ($y=0$-$0.4$) have been synthesized via the solid-state reaction route. X-ray powder diffraction analysis confirmed the above compositions to be monophasic and belonged to $n = 4$ member of the Aurivillius family of oxides. Dielectric properties of the ceramic samples (powders sintered in 1375 -1425 K temperature range) were measured in a wide frequency range (1 kHz–1MHz) at different temperatures (300–750 K). The temperature of dielectric maximum ($T_m$) for Ba$_x$La$_{1-x}$Bi$_4$Ti$_4$O$_{15}$ ceramics was found to decrease from 696 K for sample corresponding to $x = 0$ to 395 K for the sample corresponding to $x=0.8$ accompanied by a decrease in the magnitude of dielectric maximum ($\varepsilon_m$) with the increase in lanthanum content. The temperature variation of the dielectric constant on the high temperature slope of the peak ($T > T_m$) was analyzed by the Lorentz-type quadratic law and the diffuseness of the peak was found to increase with increasing $x$. Vogel-Fulcher modelling of dielectric relaxation showed a decrease in freezing temperature ($T_f$) and an increase in activation energy from 5 to 24 meV for frequency dispersion with increase in $x$. Strength of frequency dispersion of $T_m$ increased with lanthanum content. On the contrary, substitution of La$^{3+}$ for Ba$^{2+}$ was found to decrease the frequency dispersion of $T_m$ and the compositions corresponding to $y \geq 0.3$ showed non-relaxor diffusive type phase transition. Remnant polarization ($P_r$) was improved from 5.5 μC/cm$^2$ for pure BaBi$_4$Ti$_4$O$_{15}$ to 11.1 μC/cm$^2$ for Ba$_{0.8}$$La_{0.2}$Bi$_4$Ti$_4$O$_{15}$ ceramics.

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one specific purpose such as smart cards. An additional secondary lithium microbattery provides backup power and allows the digital memory states to remain unaffected during power failure or storage. So the all-solid-state thin film lithium ion rechargeable batteries are hot topic. The electrochemical performance of the all-solid-state thin film lithium ion rechargeable batteries is largely determined by the active electrode materials, especially cathode materials. Thin-film cathode materials consisting of LiMn$_2$O$_4$, LiCoO$_2$, LiFeO$_2$ and so on have been studied by numerous researchers for theoretical study. Up to now, lithium cobalt oxide based electrodes exhibit superior properties in terms of voltage, cycle stability and energy density, no other material can substitute for LiCoO$_2$. To date, many methods have been used to fabricate thin-film LiCoO$_2$ electrodes, including electron beam evaporation, pulsed laser deposition (PLD), ratio frequency magnetism sputtering. Compared with other preparation methods, pulsed laser deposition technique has some advantages: oxidation environment, favorable stoichiometry and saving-time during preparation process.

In this paper, LiCoO$_2$ films were fabricated through PLD. The LiCoO$_2$ (rhombohedral, space group R3-m) target was mounted on a rotating holder. A KrF excimer laser ($\lambda=248$ nm, repetition rate of 2Hz) source was used to ablate the target. The laser beam was focused on the target to obtain beam intensity of 2–8 J/cm$^2$. A Si(100), Si(110) wafer or Al plate as substrate was placed parallel to the target at a distance of 50mm. At first, the vacuum chamber was evacuated down to less than 10$^{-5}$ Pa, the substrate was room temperature, deposition time was not less than 1.0h. and then, the as-deposited films was sintered at 500$^\circ$C for 0.5h. After cooling to room temperature the films were exposed to the ambient. The thickness of fabricated thin films was about 500nm, the roughness was less than 30nm by sidestep apparatus. From X-ray photoelectron spectroscopy (XPS, XSAM800 with monochromatic Al-K$\alpha$ irradiation) indicated beam intensity has an effect on the ratio of Li to Co in the films. To obtain favorable stoichiometry, the beam intensity is 3 J/cm$^2$, higher than 2 J/cm$^2$ from the reference. X-ray diffraction patterns (Philips X diffractometer with Cu-K$\alpha$ radiation source) of the as-deposited films showed substrate structure has an influence on the film orientation. The electrochemical performance of the thin films was measured using the same method as described in reference, LiCoO$_2$ film electrode exhibited better cycle behavior. Compared with the as-deposited films, the films morphology (AFM, Sit SPA300HV) after 20 circles changed from acicular to round and the roughness became bigger, but the crystal structure kept stable.

**Colossal Dielectric Response In All-Ceramic Percolative Composite PMN-PT–Pb$_2$RuO$_{6.5}$**

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Percolation theory predicts that the dielectric constant of a composite comprising a conductive filler embedded in a dielectric matrix diverges at the percolation threshold, where the insulator-metal transition occurs. The fact that the effective dielectric constant of a so-called percolative mixture is much larger than dielectric constants of individual constituents can intuitively be understood in terms that close to the percolation point there are many conducting particles isolated by thin dielectric layers. However, until very recently mainly organic percolative composites or composites with metal particles dispersed in an inorganic matrix have been developed.

After a successful development of the lead-based Pb(Zr,Ti) O$_3$–Pb$_2$RuO$_{6.5}$ and lead-free (K,Na)NbO$_3$–RuO$_3$ all-ceramic percolative composites, an exceptionally high dielectric constant was obtained by making use of the conductive percolative phenomenon in the composite, comprising Pb$_2$RuO$_{6.5}$ with high electrical conductivity denoted as the conductive phase and ferroelectric 0.65Pb(Mg$_{1/3}$Nb$_{2/3}$)$_3$O$_7$–0.35PbTiO$_3$ (PMN-PT) perovskite ceramic systems. The structural analysis revealed that there were no chemical reactions between the constituents during processing, which resulted in a uniform distribution of conductive ceramic grains within the PMN-PT matrix. Consequently, the dielectric response of the PMN-PT–Pb$_2$RuO$_{6.5}$ composite follows the predictions of the percolation theory. Thus, close to the percolation point exceptionally high values of the dielectric constant were obtained - values higher than 10$^5$ were detected at room temperature at 1 kHz. Fit of the data, obtained for samples of different compositions, revealed critical exponent and percolation point, which reasonably agree with the theoretically predicted values.

Finally, not only structural and dielectric results, i.e., a successful synthesis of lead-based and lead-free systems exhibiting a stable giant dielectric response, but also electromechanical properties demonstrate the potential of developed all-ceramic percolative composites for use as high-dielectric-constant materials in various applications.
Thermoelectric Effect in Highly Doped SrLaTiO$_3$, Quantum Wells and Heterostructure
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Thermoelectric effect has attracted much attention recently due to its potential application in cooling and power generation. The related theory (for bulk materials) is not new and the key parameter of interest, i.e., the thermoelectric power TEP or the Seebeck coefficient $S$ can be derived based on charge transport and energy/heat transfer. Over the years, many thermoelectric materials have been identified and they exhibited high conversion efficiencies. Recent studies on thermoelectric effect in heterostructure and delta-doped layers also reported large thermoelectric power and it is of interest to find out if these selectively doped and/or interface layers (sometimes down to 1-2 unit layers) also exhibit thermoelectric properties as found in bulk materials. From the perspective of maximizing the thermoelectric power, the question would be whether one ought to favor the use of a quantum well structure or heterostructure. This work provides a comparative study on the performance of thermoelectric materials built on these structures. Analyses have suggested that the thermoelectric effect in highly doped samples can be explained using conventional “metallic” model. Such model when applied to doped SrTiO$_3$, quantum wells and TiO$_2$/SrTiO$_3$ heterostructure also revealed major charge spread into the neighboring layers or the substrate. We are of the opinion this may be used to explain the “giant” thermoelectric power measured in doped SrTiO$_3$ quantum wells and TiO$_2$/SrTiO$_3$ heterostructure recently reported in the literature.

Can Aqueous Synthesized ZnO be Our Future Semicond
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Interest in ZnO has increased due to the current and potential success of GaN in lighting our world. Namely, ZnO shares many of the same properties. For example, the band gap for ZnO is 3.4 eV relative to 3.5 eV for GaN. Both have the wurtzite structure. Band gap engineering can be accomplished by substituting either Cd or Mg for Zn for production of all wavelengths for the solid state lighting industry. ZnO has an exciton binding energy that is approximately twice that of GaN. Zn is relatively abundant and ZnO, inexpensive. Although n-type ZnO can be synthesized by all vapor phase and a number of solution methods, p-type ZnO still needs to be consistently synthesized to enable light emitting diodes. Even so, ZnO is an important candidate for a wide variety of other energy related applications that include UV lasing, transparent conducting electrodes, gas sensors, solar cells, and varistors. The provocative, ‘green’ title will be addressed with our current understanding and applications of n-ZnO synthesized in water at ≤ 90°C to form epitaxial films and 3D structures.

Low Resistivity and High Mobility of p-type Li-doped ZnO Materials
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Lithium doped zinc oxide polycrystalline bulks (LiZO) were prepared by soft-chemical route process. The quantity of lithium in the sol was varied from 0.05 to 0.3 mol%. The structural characteristics studied by X-ray diffractometry were complemented resistivity measurement by Hall Effect. Prepared under tartaric acid as chelating agents and sintered at 1400 °C was employed to elucidate the LiZO formulation for the bulk process. The lowest resistivity were obtained for the LiZO bulk containing 0.05 mol% of lithium. The Li-doped ZnO bulks prepared at 1400 °C possessed the lowest resistivity of 1.734 Ωcm with a Hall mobility of 32.7 cm$^2$ V$^{-1}$ s$^{-1}$ and hole concentration of 1.1 × 10$^{17}$ cm$^{-3}$.

Electrical and Optical Properties of ZnO:Ga Thin Films Fabricated via the Sol–gel Technique
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Ga-doped ZnO (GZO) thin films were fabricated by a sol–gel process using zinc acetate dihydrate and 2-methoxyethanol as starting precursor and solvent. The morphology, electrical and optical properties of GZO films were studied. The resistivity of 1.449 x 10$^{3}$ Ω was obtained after annealing in reduced atmosphere (20 %H$_2$–80% Ar). The average transmission of the films were over 90% in the visible range.
The Temperature of Electrochromically-Colored WO₃ Under Solar Radiation

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The temperature variations of electrochromically-colored WO₃ thin films, the substrate (FTO on glass), and ambient air were investigated as a function of increasing incident solar radiation. Thin films of WO₃ were deposited using an electron beam coating unit at a substrate temperature of 250°C, and produced amorphous films with mean roughnesses of approximately 15 nm.

The electrochromically-colored WO₃ increased the visible light absorbance of the substrate by more than a factor of four. As a result, the colored WO₃ increased the maximum temperature of the substrate under solar radiation during the day by more than 10°C.

The temperature of the electrochromically-colored WO₃ layer reached 88°C after peak solar radiation during the day and 23°C during the night. The rates of temperature increase as a function of incident power density were calculated to be 0.066, 0.056, and 0.01°C/W/m² for the electrochromically-colored WO₃, substrate, and ambient air, respectively.

Studies on TIG Welded Monel 400 and Characterization of Oxide Film Formed on the Weldment Exposed Under Air Oxidation and Na₂SO₄ + NaCl (60%) at 700°C

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Monel 400 is widely used in fixtures, pumps, valves and piping systems for marine application. Because of its good corrosion resistance it is also used in power plant equipment. The strength of the weldments is reported to be poor at molten salt environment. The weldments are characterized particularly by compositional gradients and microstructural changes, which yield large variations in metallurgical and mechanical properties across the weld.

In the present investigation an attempt has been made to evaluate the mechanical properties include the microhardness, tensile strength TIG welded monel 400. The fractography of tensile fractured specimen shows the ductile fracture. It was found that the hardness was increased in weld interface as compared to base metals. High temperature corrosion tests were performed on weld, HAZ and base metal in molten salt of Na₂SO₄ + NaCl (60%) at 700°C under cyclic condition and air oxidation environment. Corrosion was extremely severe on the interface of the weldment after exposed 50 cycles. It was also observed that the corrosion across the weld interface was higher in the case of molten salt environment rather than air oxidation environment. Thin oxide film formed on the weldment was characterized using the combined techniques of optical microscopy, XRD, SEM/EDAX and EPMA analysis.

The Effect of Process Parameters on Mullite Based Ceramic Nanocomposite Coatings

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Thick mullite based ceramic nanocomposite coatings designed for corrosion protection, have been produced on aluminium under various conditions (electrolyte, processing time, temperature and current density) and their thickness, growth rate, hardness, microstructure and phase composition have been studied. The final coating thickness depends predominately on the processing time and the applied current density. A through thickness variation in the composition of the coating is observed. The concentration of silicate in the electrolyte has reasonable effect on the resulting coating thickness, Composition and porosity. The deterioration of the microhardness with increasing electrolyte temperature could mainly be attributed to the increase of the porosity in the outer region of the oxides since the rate of microhardness reduction is almost synchronous with the rate of porosity increase. Electrochemical impedance spectroscopy and potentiodynamic polarization methods are powerful techniques to investigate the corrosion protection of Al₂O₃ nanocomposite coatings. However, the oxygen atoms were not diffused into the unoxidized aluminium substrate, even near the coating/substrate interface. This facilitates maintaining mechanical properties of aluminium substrate after anodic oxidation treatment. Overall this paper highlights the important influence of the electrolyte temperature on the microstructure and mechanical properties of the anodic film and further emphasizes the significance that electrolyte convection and heat transfer of the anodizing process have in terms of influence on the mechanical properties of the anodic oxide coatings.
**Size Effect of Ferroelectric Properties for Nanoc grain Barium Titanate Ceramics**

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The bulk dense nanocrystalline BaTiO\(_3\) (BT) ceramics with the grain size range from 20 to 100 nm ceramics have been successfully prepared by the spark plasma sintering (SPS) method. The dielectric data were investigated by dielectric constant, hysteresis loop, and piezoresponse force microscopy. Raman spectra and X-ray diffraction (XRD) were used in combination with electron microscopy to study the evolution of lattice structure and phase transformation behavior with grain growth from nanoscale to micrometer scale for BT ceramics. The results show that a broad ferro-para phase transition with the decrease of the grain size. The dielectric constant decrease with the grain size going down, above 400Hz dielectric constant increase when the frequency going up and loss decrease with the frequency increase. The hysteresis loop demonstrated that 20 nm BT ceramics has a remanent polarization and is switchable by an electric field. Thus, if a critical grain size (GS) exists for ferroelectricity, it is less than 20 nm for polycrystalline BT ceramics.

**Nanoparticles of the Giant Dielectric Ceramic, CaCu\(_{3}\)Ti\(_4\)O\(_{12}\) from a Complex Oxalate Precursor Route**

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A complex oxalate precursor, CaCu\((\text{TiO})_x(\text{CO}_3)_y\)\(_3\)H\(_2\)O (CCT-OX), was synthesized and the phase singularity was confirmed by wet chemical analyses, X-ray diffraction, FT-IR and TGA/DTA analyses. The thermal decomposition of this oxalate precursor led to the formation of phase-pure calcium copper titanate, CaCu\(_3\)Ti\(_4\)O\(_{12}\) (CCTO) at 680°C. The bright-field TEM micrographs revealed that the size of the as synthesized crystallites to be in the 30-80nm range. The powders derived from the oxalate precursor have excellent sinterability resulting in high density ceramics which exhibited giant dielectric constants up to 40,000 (1 kHz) at 25°C, accompanied by low dielectric loss < 0.07.
moment data show fairly strong magnetic nature of the samples. The observed electron paramagnetic resonance (EPR) lineshapes of K1-K4 at 11, 77 and 300 K show axially symmetric features with well resolved 4-parallel lines and poorly resolved 4-perpendicular lines typical a \( g = 2.0036 \) at a \( Cu^{2+}(3d^9) \) site. From the trends \( g > g > g (g = 2.0023) \) obtained from the EPR lineshape simulations we conclude that the paramagnetic site in the specimen is \( Cu^{2+}(3d^9) \) which is in a tetragonally elongated octahedron \([O_{12}^-\text{CuO}_{12}^-\text{O}_{12}^-] \) exhibiting John–Teller distortion having \( D_{4h} \) symmetry with orbital singlet \( ^2B_{1g} \) \((d_{x^2} \geq \gamma^2) \) as the ground state. The calculated values of the molecular orbital coefficients, \( \beta, \alpha, \delta \) and \( \kappa \) with usual connotations are also found to be similar at 11, 77 and 300 K, which shows that the geometry of the octahedron containing the paramagnetic site \( Cu^{2+} \) is fairly 'rigid' within the temperature range studied. The optical absorption spectra show a band in the range 567-575 nm due to the superimposed three \( d-d \) transitions as \( ^2B_{1g} \rightarrow ^2E_g, ^2B_{1g} \rightarrow ^2B_{2g}, ^2B_{1g} \rightarrow ^2A_g \) of the octahedral splitting with tetragonal distortion of the \( Cu^2+ \) (d\(^9\)) 3d-energy levels from the \( ^2B_{1g} \) \((d_{x^2} \geq \gamma^2) \) ground state.

Sol-gel Synthesis and Structure-property Relations in Silver Nanoparticles – Dispersed on GeO\(_2\) Matrix

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Synthesis of Ag nanoparticles dispersed in polycrystalline germanium dioxide matrix was performed via sol-gel route. Analysis of the powder xrd pattern shows cubic unit cell of Ag with lattice parameters: \( a = 4.0884 \) Å, space group Fm3m and calculated density= 10.467 g/cc in the sample. EDX results show the presence of only GeO\(_2\) and Ag in the sample. The average crystallite sizes of Ag particles determined by Scherrer’s relation are found to be ~ 18-47 nm. The calculated value of the magnetic susceptibility of 1.014×10\(^{-1}\) emu/gG due to reduced Ag atoms from the observed magnetic moment data show fairly strong paramagnetic nature of the sample. Calculated value of exchange integral is found to be 8.362×10\(^{-2}\) Hz from the Weiss constant value of 200 K. Observed sharp EPR lineshapes at 8, 77 and 300 K with \( g \)-values 2.0036, 2.0036 and 1.986, respectively shows that the Ag 6s\(^1\) electrons are localized in the Ag nano particles. The unit cell structure of Ag is developed in Fm3m space group with Ag atoms localized in the Ag nano particles. The calculated density of states below Fermi energy \( (E_F) \) from -2 to -8 eV are due to valence d orbitals of Ag. The observed and the calculated optical absorption spectra show close similarity and the intense absorption around 200 nm is attributed to the charge transfer transitions. The calculated value of refractive indices less than one at some frequencies may be due to the anomalous dispersion from core bound electrons in Ag particles. The calculated electrical conductivity plots show that below ~ 11000 cm\(^{-1}\) the real part is positive and the imaginary part is negative, and at higher frequencies both are positive.

Thermoelectric Response Of Electron-doped CaMnO\(_x\) Perovskites

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Environmentally-friendly thermoelectric materials have attracted widely interests. The figure of merit \( Z=S/\rho\kappa \), where \( S \) is thermopower, \( \rho \) resistivity and \( \kappa \) thermal conductivity, is used to characterize materials for their thermoelectric performance; \( ZT>1 \) (\( T \) is absolute temperature) is required for practical application. Compared with conventional thermoelectric materials (doped semiconductors and alloys), metal oxides are more suitable for practical application because of their structural and chemical stability, oxidation resistance and low cost. Recently, the discovery of large thermoelectric response in 3\( d \) transition-metal oxides (manganites, co-baltites, titanates etc) attracted a renewed interest in studying thermoelectric oxides.

As one of the most typical 3\( d \) transition-metal oxides, perovskite manganites \( A_{x}R_{1-x}MnO_{3} \) \((A=\text{alkaline earth, } R=\text{rare earth}) \) have been studied widely because of their rich physical properties such as colossal magnetoresistance, phase separation, charge ordering, orbital ordering, and spin glass behavior. In contrast to magnetic and transport properties, systematic studies on their thermoelectric properties are quite few. Especially, due to the complex interplay between spin, charge, orbital and lattice degrees of freedom, the thermopower behavior of these manganites still keeps unclear. But thermopower is crucial to understand the transport nature and thermoelectric performance of these systems.

In this study, a series \( Ca_{1-x}R_{1-x}MnO_{3} \) perovskites in electron-doped range were prepared. Skipping the details of phase diagram and magnetic properties, we direct attention to the properties relative to thermoelectricity and particularly focus on thermopower.

Considering thermopower is a function of carrier concentration and configurational entropy, we first
investigate semi-theoretically thermopower of these perovskites based on the electronic structure of the 3d orbitals of Mn ions using metallic diffusion model, Boltzmann transport model and narrow band model. As temperature decreases, the evolution of the freedom degree of spin and orbital together with the change of phonon scattering mode can describe thermopower behavior satisfactorily. Besides, when the carrier concentration and site degeneracy are fixed, structural distortions have strong effects on thermopower. The phonon drag effects at low temperature are also discussed.

Furthermore, magnetic field has strong influence on configurational entropy and electronic effective mass, and thus induces remarkable magneto-thermopower effect. In high temperature range, configurational entropy tends to unit under magnetic field, giving rise to thermopower tends to zero, however, in low temperature range, the notable increase of electronic effective mass under field leads to the enhancement of thermopower whereas the variation of configurational entropy is slight. Such results found in manganites are in contrast to cobaltites in which spin entropy is the source of large thermopower, revealing intrinsic difference in the two electronic strongly correlated systems.

Finally, we test high temperature thermoelectric performance of these manganites and get the optimal ZT around 0.2 at 1000K. This ZT value is still too low far from the applied criterion (ZT > 1). Using the dynamical mean field theory, we demonstrate that a ZT value larger than one in electron-doped CaMnO systems seems rather unlikely. Some strategies for searching new thermoelectric materials with high performance in transition-metal oxides are also proposed.

### A00138-00291
#### Effect of Heat Treatment on the Structure and Properties of Chemical Solution Processed Multiferroic Bi$_2$CrFeO$_6$ Thin Films
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In this paper, we report on growth and characterization of polycrystalline Bi$_2$CrFeO$_6$ (BFCO) thin films deposited on indium tin oxide (ITO) coated glass substrates via chemical solution deposition technique. The coated thin films were crystallized by heat-treating the films at various temperatures for 1 h in flowing nitrogen. The structure of thin films was analyzed by grazing incidence X-ray diffraction (GIXRD) analysis. Hysteresis measurements on BFCO films showed that the films were ferroelectric in nature and ferroelectric nature was dependent on the heat treatment temperature. Magnetic hysteresis measurements suggest canted Antiferromagnet behaviour of the films.

### A00153-02511
#### Preparation of High-Activity Au/CeO$_2$ Nanoneedles for CO Oxidation
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Gold supported cerium oxide (Au/CeO$_2$) nanoneedles were synthesized by the deposition-precipitation method for the catalytic CO oxidation. Experimentally, nanocrystalline CeO$_2$ needles (length of 100-200 nm and diameter of 8-10 nm) were firstly prepared by the two-stage precipitation method proposed previously. Then, Au nanoparticles were deposited on the prepared CeO$_2$ nanoneedles starting from the HAuCl$_4$ solution followed by aging, washing, and finally calcined in air at 400°C for 4 hours. The pH value of the Au precursor solution, in the range of 3 to 12, was adjusted by adding sodium hydroxide or hydrochloric acid solution. The morphology, crystalline structure, surface area, electronic states of Au, and oxidation ability of the resulting Au/CeO$_2$ nanoneedles were characterized by using the TEM, HRTEM, XRD, BET, XPS and TPR techniques. In advance, catalytic activities of samples on CO oxidation were studied in a tubular reactor at temperatures of 303-673 K. Moreover, particulate shaped Au/CeO$_2$ nanoparticles were also synthesized for comparison.

The results showed that the pH value of the Au precursor solution played an important role on the deposited amount and dispersion of Au nanoparticles. The sample prepared at an optimal pH value of about 8, having largest deposition amount and highest dispersion of Au, exhibited a maximum catalytic activity on CO oxidation. As compare with the particulate sample, the Au/CeO$_2$ nanoneedles showed a higher catalytic activity due to the morphological effect of support. Since needle-like CeO$_2$ sample was characterized with relatively larger portion of high energy {100} and {110} facet on the exposed surface, it possessed higher activity than the particulate one. Even at room temperature, the CO conversion of the Au/CeO$_2$ nanoneedles reached 95% in the studied condition, indicating the studied samples showed a very promising development for catalytic uses.
Synthesis and Field Emission Properties of Well-Aligned ZnO Nanowires on Buffer Layer

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Transparent conducting oxides (TCOs) with a remarkable combination of high electrical conductivity and optical transparency are used in many important technological applications. These include flat panel displays, solar energy devices and optoelectronic devices. Although indium-tin-oxide (ITO) has been in practical use for most such applications, the cost and scarcity of the principal material of ITO, indium, has resulted in the research and development of other TCOs as possible substitutes to ITO. Zinc oxide (ZnO) which has a wide bandgap of 3.37 eV and large exciton binding energy of 60 meV at room temperature has attracted considerable interest due to its inexpensive and non-toxic source materials.

A simple method involving thermal evaporation of a mixture of ZnO and graphite powders using a horizontal double-tube system is presented here. Well-aligned ZnO nanowires of uniform diameter and length were grown on Si (111) substrates pre-coated with a ZnO buffer layer. The peaks in the X-Ray Diffraction (XRD) pattern have been indexed to the wurtzite hexagonal ZnO phase, showing a preferential growth in the [0001] direction. Scanning Electron Microscopy (SEM) showed perfect hexagonal-faceted surfaces on the nanowires. Structural characterization of the ZnO nanowires by Transmission Electron Microscopy (TEM) showed that the nanowires have a uniform diameter with a smooth surface topology throughout their lengths. Clear lattice fringes as seen in the High-Resolution Transmission Electron Microscopy (HRTEM) indicated the single-crystalline structure of the ZnO nanowires, and the lattice spacing confirmed the [0001] growth direction. The Selected Area Electron Diffraction (SAED) pattern also verified the growth of the nanowires along the [0001] direction. Photoluminescence measurements at room temperature produced a strong ultraviolet emission and a suppressed green emission, affirming the presence of few defects in the product and the good optical properties of the synthesized nanowires. Field emission properties of the nanowire arrays were investigated and the measurements showed a high emission current density and a low turn-on voltage. The Fowler-Nordheim (F-N) plot of the data showed that the field emission behavior can be well described by the F-N mechanism. Electrical characterization of the ZnO nanowires was carried out via current-voltage, capacitance-voltage and two-point measurements. Properties such as the resistance of the nanowires were obtained from these measurements.

A simple method to synthesize well-aligned ZnO nanowires is presented here, where the growth process is easily controlled by the buffer layer. This allows ZnO nanowires to be grown on substrates other than silicon, as long as a buffer layer is pre-coated on the substrate. This development of a TCO provides a promising alternative to ITO, and it would be highly applicable for direct integration in TCO-related device applications.

In-situ Detection of Porosity Initiation during Aluminum Anodizing by High-Resolution Curvature Measurements

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Porous anodic aluminum oxide templates have been extensively used for the fabrication of nanostructures since the first report of the achievement of a regular porous structure by Masuda in 1995. However, the mechanisms underlying the porosity nucleation, growth and self-organization are not completely understood yet. Understanding these mechanisms could potentially provide a precise control of the templates morphology, or the achievement of self-organized porous growth for other materials.

Porous anodic films on aluminum are well-known to grow in two steps: the first one is the growth of a barrier layer; the second one is the growth of a porous layer on top of the dense barrier layer. When templates are fabricated, the porous layer growth stage is relatively long compared to the one for barrier layer growth. Nonetheless, the transition between the two steps has a critical influence on the porous layer morphology. Recently, experimental evidence and theoretical calculations have suggested that the porous layer grows by diffusional flow of oxide from the barrier to the porous oxide layer under the influence of internal stresses in the oxide.
Inspired by this possible interplay between stress and porous oxide growth, we have performed in-situ stress-induced curvature measurements during aluminum anodizing. We observed a well-defined transition in the rate of curvature change when porosity appears. The onset of porosity appearance was confirmed by a quantitative analysis of the spectral density distributions of anodized surfaces, obtained by analyzing ex-situ atomic force microscopy images of surfaces anodized for different times.

The in-situ stress measurements further allowed performing a perturbation analysis of the oxide growth. The analysis consisted in evaluating the energy difference between a flat oxide layer and an oxide layer with an undulating surface, taking into account the contributions from changes in surface energy, mechanical energy and electrical energy. The analysis revealed that length scales involved in the creation of porosity are more likely to be selected from the competing effects of changes in the electrical energy stored versus surface energy, the mechanical energy changes being negligible at the length scales characteristic of porous aluminum oxide.

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### TiO\(_2\) Composite Electrodes Fabrication for Hybrid Solar Cells

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A high efficiency hybrid solar cell was prepared by coating (benzylbutyl)\(_2\)(1,2-methanofullerene C\(_{80}\))\(_{61,61}\)-dicarboxylate (BBMDC)/poly(3-hexylthiophene) (P3HT) mixed photovoltaic active layer on a composite TiO\(_2\) electrode. A dense TiO\(_2\) film was first coated on a fluorine-doped tin oxide (FTO) substrate. The high refractive TiO\(_2\) film brought photon scattering for better light absorption. A mesoporous TiO\(_2\) film was then synthesized by evaporation-induced self assembly (EISA) method on the dense TiO\(_2\) film. The photovoltaic active layer adsorbed on the large specific surface of mesoporous TiO\(_2\) films therefore increased the donor-acceptor interface of the cells. Moreover, the good electron acceptor material of TiO\(_2\) thus enhanced the photovoltaic efficiency of the solar cells. The performance of the hybrid solar cell with TiO\(_2\) composite electrode was with an open circuit voltage (V\(_{oc}\)) of 0.59 V, a short circuit current (I\(_s\)) of 4.22 mA/cm\(^2\), a fill factor (FF) of 47.2%, and an energy conversion efficiency (\(\eta\)) of 1.11%.

### Preparation and Characterization of p-type ZnO Thin Films by Ga and N Co-doping Process

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Recently, p-type ZnO thin film has been importance for the opto-electronic application and has attracted considerable attentions. In this study, we have investigated the N-doping effects in ZnO-Ga thin films using high energy proton accelerator and thermal annealing. The ZnO-Ga (3 wt.%) thin films were prepared by pulsed magnetron sputtering at the substrate temperature of 300 \(^\circ\)C and N ions were doped in ZnO-Ga thin films by proton accelerator at various conditions. The N dosing flux were varied from 10\(^{15}\) to 10\(^{19}\) cm\(^{-2}\) and the films were annealed with the temperature of 500 \(^\circ\)C in vacuum atmosphere. The structural and optical properties of the thin films were measured by X-ray diffraction, X-ray photoelectron spectroscopy, scanning electron microscopy, and photoluminescence, etc. The electrical properties of N and Ga co-doped ZnO thin films were measured by Hall effect measurements using van der Pauw method. The corresponding carrier density, mobility and resistivity of the N-dosed ZnO-Ga film with the dosing flux of 10\(^{17}\) cm\(^{-2}\) were 2.6x10\(^{17}\) cm\(^{-2}\), 2 cm\(^2\)/Vs, and 17.4 \(\Omega\)cm, respectively and thus is considered applicable to p-type ZnO thin film.

### Advanced Porous Ceramics through the Microstructure Control

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Microstructure control is very important to realize various giant properties for advanced materials. In this presentation, importance of control of porous microstructures was featured by showing three materials, which can be used for save-energy applications based on the excellent water vapor and/or water adsorbing and evaporating properties by advanced porous ceramics. To enhance such target properties, the important factors are size, distribution, arrangement and surface characters of pores. Pore size is classified into three groups, i.e. micro- (\(<52\) nm), meso- (2-50 nm) and macro-pores (>50 nm).
Micro-pores show strong interaction with adsorbents by micro-pore filling or clustering because of the very narrow pore spaces. Micro-porous materials, such as zeolites, activated carbons and silica gels, show high adsorption of water vapor at low relative pressure ($P/P_0$). Improvement of adsorption can be realized by control of pore size, pore volume and surface chemical coating. Some examples of micro-porous materials developed for chemical heat pump applications using water vapor adsorption-desorption heat energy in the range between $P/P_0=0.1$ and 0.3 are introduced.

Meso-porous materials show steep increase and decrease of water vapor at middle range of $P/P_0$ by capillary condensation and evaporation with a hysteresis. We developed a meso-porous alumina with a unique microstructure of 5-6 nm in pore size using phase separation and selective leaching method. The ceramic boards used for wall materials showed an excellent effect to keep constant relative humidity at around 60% owing to the large amount of adsorption and desorption ability of water vapor. Due to the unique microstructure, this porous alumina had an excellent thermal stability maintaining the specific surface area $>100$ m$^2$/g after heating at 1200°C.

Macro-porous materials show an excellent water absorption property because of the great pore volume. We prepared macro-porous materials with unidirectionally aligned pores by an extrusion method using flammable fibers as the pore formers. By controlling the pore size, distribution and alignment of this ceramics, microstructure similar to lotus was obtained with a pore diameter of about 15 μm. This porous ceramics show very high capillary lift ability up to 1400 mm, of which height is about 3 times higher than the ability of conventional ceramics. Due to this excellent capillary lift ability and evaporation of water vapor from the surfaces, this material shows lowering of temperature $>10°C$ and is applicable to counteract heat island effect, contributing to save energy.

In this way, advanced porous ceramics prepared by controlling of their pore sizes, distributions, arrangement and surface chemical coating show various giant properties and are applicable to various save energy technologies.

**Synthesis, Crystal Structures and High-Temperature Thermoelectric Properties of Spinel-type Cobalt-Ruthenium Oxides**

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To accumulate basic data of the thermoelectric conversion oxide material, a thermoelectric characteristic of spinel-type cobalt-ruthenium oxides, $\text{Co}_x\text{Ru}_y\text{O}_{3+x/2}$ were measured. A single phase of solid solutions of $\text{Co}_x\text{Ru}_y\text{O}_{3+x/2}$ was prepared from $\text{Co}_x\text{O}_y$ and $\text{Ru}_x\text{O}_y$ powders by solid-state reaction at a limited temperature range of 1173–1273 K in air for $0.5<\alpha<0.70$ and $0.1<\gamma<0.2$. The crystal structures of the solid solutions were refined by Rietveld analysis of their powder X-ray diffraction patterns. The solid solutions crystallized in cubic cells with temperature range of 298 to 1073 K. The thermoelectric transport property of the solid solutions exhibited semiconducting behavior and positive Seebeck coefficient in the temperature range of $0.024$ was measured for $\text{Co}_{2.18}\text{Ru}_{0.65}\text{O}_{3.86}$ at 973 K.

**Diffraction Study of Thermal Decomposition of Ti$_2$AlC in Vacuum**

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Titanium aluminum carbide (Ti$_2$AlC) demonstrates interesting combination of properties of both metals and ceramics, such as good machinability, thermally shock resistant, thermally and electrically conductive, damage tolerant, lightweight, excellent oxidation resistance and elastically stiff. In this research, the effect of high vacuum annealing on the phase stability and phase transition of Ti$_2$AlC up to 1550 °C was studied using in-situ neutron diffraction. The decomposition of Ti$_2$AlC into TiC was observed from the change of phase abundances as a function of temperature. Furthermore, the activation energy for the decomposition of Ti$_2$AlC was determined by Arrhenius’ equation to be 85.7kJ mol$^{-1}$. Moreover, depth profile of vacuum annealed Ti$_2$AlC was performed by secondary-
ions mass spectrometry (SIMS) and synchrotron radiation diffraction (SRD) experiments. The depth-profiling results of vacuum annealed Ti\textsubscript{2}AlC will also be presented and discussed.

**A00207-02854**

**Diffraction Study of Thermal Decomposition of Ti\textsubscript{3}Si\textsubscript{0.95}Al\textsubscript{0.05}C\textsubscript{2} in Vacuum**

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Ti\textsubscript{3}SiC\textsubscript{2} has been widely recognized to be an excellent functional ceramic material due to its unique combination of salient properties of both metals and ceramics. The thermal stability in various atmospheres, i.e. vacuum and argon atmosphere, of Ti\textsubscript{3}SiC\textsubscript{2} is of great concern in engineering applications. Ti\textsubscript{3}SiAl\textsubscript{1−x}C\textsubscript{x} solid solutions can be formed via doping Al in Ti\textsubscript{3}SiC\textsubscript{2}. It has been shown that even a small amount of Al dopant can significantly improve the oxidation resistance of Ti\textsubscript{3}SiC\textsubscript{2}, attributing to the formation of α-Al\textsubscript{2}O\textsubscript{3} layer during oxidation. However, the thermal stability of Ti\textsubscript{3}SiAl\textsubscript{1−x}C\textsubscript{x} has not gained too much attention from research scientists. In this paper, a comprehensive study of the thermal stability of Ti\textsubscript{3}SiAl\textsubscript{1−x}C\textsubscript{x} up to 1550 °C was carried out using in-situ neutron diffraction. The decomposition of Ti\textsubscript{3}SiAl\textsubscript{1−x}C\textsubscript{x} into TiC was observed from the change of phase abundances as a function of temperature. Furthermore, the activation energy for the decomposition of Ti\textsubscript{3}SiAl\textsubscript{1−x}C\textsubscript{x} determined from the Arrhenius’ equation was 76.7 kJ mol\textsuperscript{−1}. Moreover, depth profiling of vacuum annealed Ti\textsubscript{3}SiAl\textsubscript{1−x}C\textsubscript{x} was performed by secondary-ion mass spectrometry (SIMS) and synchrotron radiation diffraction (SRD) experiments. The depth-profiling results of vacuum annealed Ti\textsubscript{2}AlC will also be presented and discussed.

**A00212-00921**

**Disorder Enhanced Magnetoresistant Effect in Manganites**

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The effects of A-site cationic size-mismatch (A-site disorder) on the stability of charge-ordered states and phase separated phase in a series of manganites with constant A-site ionic average radii \(<r_a>=1.18\text\AA\) but different A-site ionic size mismatch \(\sigma^2\) are experimentally investigated. It is revealed that the charge/orbital ordered antiferromagnetic ground state becomes destabilized and eventually collapses into coexisting of the predominant ferromagnetic metal (FMM) state and short-rang charge/orbital ordered state with increasing \(\sigma^2\), resulting in enhanced colossal magnetoresistance. However, further increasing A-site disorder will suppress the FMM state and seem to favor a cluster-glass insulating state due to the severe electronic localization.

**A00250-00469**

**Domain Reorientation Dynamics of Sol–gel Derived Strontium Doped PLZT (8/65/35)**

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Polycrystalline samples of PLSZT with the composition Pb\textsubscript{0.92−x}La\textsubscript{x}Sr\textsubscript{x} (Zr\textsubscript{0.65}Ti\textsubscript{0.35})O\textsubscript{3} (where \(x = 0, 0.02, 0.04, 0.06, 0.08, \) and \(0.10\)) have been synthesized by sol–gel technique. DTA analysis confirms that all the organic constituents get decomposed and final PLZT is formed at 545 °C. The XRD analysis confirms the formation of single rhombohedral perovskite phase with decreasing unit cell parameter. Crystalite size calculated, using Scherrer’s equation, was found to decrease with Sr doping due to smaller ionic radii of Sr than Pb. Compact uniform grain distribution was observed from SEM micrographs. The ferroelectric to paraelectric phase transition temperature, maximum dielectric constant and remnant polarization (Pr) were found to decrease with Sr doping along with increasing diffuse nature of phase transformation. Detailed domain
reorientation dynamics study suggests that Sr doping increases the percentage backswitching and decreases the normalized coercivity by decreasing the viscous nature of composition, which facilitates ferroelectric switching and can be used for making switching devices.

**A00254-00680**

**Synthesis of Sol-gel Derived Mesoporous Silica Nanoparticles**

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The synthesis of mesoporous materials has recently attracted a lot of interest, owing to their unique and excellent properties. Much attention has been paid to the fabrication of inorganic materials such as mesoporous silica. Mesoporous silica is widely used in various optical and sensing applications. With the simple approach based on sol-gel method, silica nanoparticles with high specific surface area and controlled pore size and distribution could be obtained.

In addition, by incorporating hollow structures in silica nanoparticles, the synthesized products would exhibit properties substantially different from their solid counterparts. These include lower density, higher specific surface area and lower thermal conductivity. Several researches have been reported for the synthesis of hollow silica nanoparticles. One example is the use of sacrificial templates to form core-shell particles consisting of cores of template materials and all sorts of shells. After the removal of the templates, hollow structures could be obtained.

In this work, mesoporous silica nanoparticles were obtained through a sol-gel method, employing tetraethylorthosilicate (TEOS) as a precursor and HCl as a catalyst. Processing parameters affecting the properties of silica particles were also investigated. High specific surface area of silica particles was enhanced with the formation of hollow structures, using calcium carbonate as a template material. The samples were characterized with TEM, SEM, BET and FTIR.

**Effect of Fluorine Content in FTO Electrodes Prepared by Spray Pyrolysis on Dye Sensitized Solar Cell Efficiencies**

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FTO or fluorine-doped tin oxide (SnO₂:F) is widely used as a transparent conducting oxide (TCO) especially in dye-sensitized solar cell industries. Various deposition techniques have been used to grow SnO₂ thin films. In this investigation, thin films of fluorine-doped tin oxide were prepared on glass substrates using ultrasonic spray pyrolysis technique. Air zero gas was used as a carrier gas. Solution of tin chloride (SnCl₂) in methanol 90% and 10% of DI water were used as precursors. Fluorine doping was achieved by adding ammonium fluoride (NH₄F). The effect of fluorine doping concentration was investigated by varying F:Sn molar ratios of 0:1, 0.5:1 and 1:1, respectively. All of FTO films with the varied molar ratios were deposited at 500°C.

The effect of doping concentration and substrate temperature were related to morphologies and microstructural information characterized using glancing angle X-Ray Diffraction (GAXRD: Rigaku TTRAX III), Scanning Electron Microscopy (SEM: JEOL6301F). The 4-point probe measurement (Prometrix OmniMap model RS35) and UV-VIS spectrophotometer (JASCO V-530) were used to identify electrical and optical properties of all films, respectively.

The results indicated that the films were polycrystalline having different grain sizes and shapes. The minimum electrical resistivity of 1.86 Ohm•square was obtained. This value is comparable to commercial ones.

All of the in-house FTO electrodes prepared and a commercial FTO electrode were fabricated for dye sensitized solar cells having the same cell structure. The best dye sensitized solar cell from in-house FTO demonstrated only 0.91% efficiency compared with 3.64% from the commercial FTO due to its inferior optical transmission properties.
Luminescent enhancement with tunable emission in Sr$_2$SiO$_4$: Eu$^{2+}$ phosphor for white LEDs

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Europium doped strontium silicate (Sr$_2$SiO$_4$: Eu$^{2+}$) phosphors were synthesized via the solid-state method using different sizes of silica precursors. It shows remarkable enhancement in luminescence characteristics when nano-sized silica was used. This phenomenon is ascribed to the improving reactivity through the reaction, leading to the increase in crystallinity and the formation of enlarged particles. In addition, as nano-sized silica was used, the emission wavelength blue-shifted (from orange to yellow) with prolonged heating duration. This feature is attributed to microstructural changes, eventually leading to the decrease of the non-radiative energy transfer between Eu$^{2+}$ ions. In this study, color-tunable Sr$_2$SiO$_4$: Eu$^{2+}$ phosphors with improved emission intensity were successfully prepared via using different sizes of silica particles.

Fabrication and Characterization of Sb-doped ZnMgO Thin Films by rf magnetron reactive sputtering

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We report on the fabrication and characterization of Sb-doped ZnMgO thin films by radio frequency (rf) magnetron sputtering for the application of p-type channel transparent TFTs. The target was fabricated by high-purity MgO mixed ZnO with Sb$_2$O$_3$ as the doping agent. The films were prepared on glass substrates by rf magnetron sputtering. Effects of working gas (Ar/O$_2$) ratios and annealing conditions on the structural and electrical properties were investigated. Atomic ratio of Zn/Mg in films and accordingly the film properties depended on the post-annealing conditions as well as the oxygen partial pressure in sputtering gas during sputtering. The change in the physical properties like electrical resistivity and energy band gap with Zn/Mg composition in the films was evaluated.
Effect of Fe Diffusion in MgO/Fe Seedlayers to Attain (100) Oriented Pt Underlayer for Perovskite Films with c-axis Orientation

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Perovskite type oxide thin films with high dielectric constant have been paid much attention as a key material for functional devices. In order to apply them to capacitive devices, such as ferroelectric random access memory and tunable filter devices, bottom electrode layer for the oxide films are very important, since they play a role to affect the crystallinity of the oxide films. Pt is one of the well known bottom electrode materials for oxide films. Since dielectric properties of ferroelectric films are originated from the displacement of ions in a crystal along the c-axis direction, c-axis oriented ferroelectric thin films are required to attain better dielectric properties. In order to attain c-axis oriented Perovskite type ferroelectric films, (100) oriented Pt layers are required. However, it is often that (111) orientation of crystallite is observed in Pt thin films prepared by conventional methods. In this study, (100) oriented Pt layers can be successfully deposited on the (100) oriented MgO layer prepared on very thin seed Fe layers deposited on SiO₂/Si substrates. It has been also observed that the diffusion of Fe from the bottom seed layer to the surface of the MgO layer played an important role to attain (100) oriented Pt layer at low substrate temperature Ts around 250 °C.

All the films were prepared by facing-targets sputtering (FTS) method. Crystallographic properties were determined by X-ray diffraction (XRD) analysis. The X-ray photoelectron spectroscopy (XPS) was used to analyze the depth profile of the films.

(100)-oriented MgO thin films can be prepared on (100)-oriented Fe seed layer at Ts of room temperature. The Pt thin films deposited on the MgO(100)/Fe(100) underlayers at Ts above 400 °C revealed (100) preferential orientation. We have found that the mechanism of the growth of (100) oriented Pt layer is related not only with an epitaxial effect from the (100) oriented MgO layer, but also with the surface of the MgO layer covered with Fe-rich oxide layer, which was formed by the diffusion of the Fe atoms from the bottom Fe layer to the top of the MgO layer. The XPS depth profile of the Pt/MgO/Fe films clarified that the diffusion of Fe occurs at Ts above 350 °C. We considered that there is a possibility to attain (100) oriented Pt layer can be deposited at lower Ts, if the Pt would be piled-up to the MgO surface. We developed a new process to confirm the hypothesis. The ordinary process was that the substrates were heated to the designated temperature for the deposition. On the other hand, the new process is that the substrates are heated to 500 °C to enhance the diffusion process of the Fe bottom layer and cooled down to the designated deposition temperature. The new process causes that Pt thin films with (100) preferred orientation can be grown at Ts around 250 °C.

Influence of Annealing Temperature on the Physical Properties of ZnO Thin Film

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Zinc Oxide (ZnO) thin films were prepared on glass substrate using catalytic thermal chemical vapour deposition (CVD). The influence of annealing temperature on the surface morphology, crystal growth orientation and photoelectrical response has been investigated. A conversion on the surface morphology from ZnO islands to nanowires has been confirmed by scanning electron microscope (SEM) observation. An increased on the crystallinity with the increasing of annealing temperatures from 400°C to 600°C has been measured using x-ray diffractometer (XRD). Finally, highest quantum efficiency (QE) for sample annealed at 550°C has been measured by using solar simulator system. In this paper, details on the influence of annealing temperature on the physical properties of ZnO thin film will be discussed.
Zinc Oxide (ZnO) nanowires on ZnO microcage have been synthesized using catalytic thermal chemical vapour deposition (CVD). Gas blocker has been introduced at the end of high temperature alumina tube to control the Ar gas flow. Observation using scanning electron microscope (SEM) has found that ZnO nanowires of approximately 7 um length and 60 nm in diameter has been synthesized on ZnO microcage. The x-ray diffractometer (XRD) indicates that the ZnO nanowires were preferentially grown at (002) direction. The luminescence property indicated by Photoluminescence (PL) spectrometer shows that ZnO nanowires has very high emission at UV wavelength (<400 nm). In this paper, detail of the nucleation and formation of ZnO nanowires on ZnO microcage will be explained.

The Fabrication of an Al/MgCaTiO\textsubscript{3} /Si(100) MIS Device and its Characteristics of Photo-sensitivity

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We demonstrate the fabrication of an Al/MgCaTiO\textsubscript{3} MIS device in which using pulse laser deposition (PLD) to deposit MgCaTiO\textsubscript{3} (MCT) thin films on p-type Si (100) substrate. Deposition rate of 0.34 Å/pulse has been achieved with laser fluences of 416 mJ/cm\textsuperscript{2}. Dielectric constant of 15.6 has been achieved of a film with 100nm thickness, and it is increase with the thickness of the films. Raman spectroscopy, X-ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive spectrometer (EDS), Transmission/Reflection spectroscopy are used to study the effect of crystal properties of the deposited MCT films on process parameters; such as laser fluences and annealing temperature. It shows that the transparency is improved and dielectric constant increases after annealing the deposited MCT films at 600 C for 5 minutes. In addition, I-V and C-V measurements are also used to characterize the dielectric performance and the photosensitivity of the MIS device with various thicknesses of the MCT layers. A maximum responsivity of 2.52\times10\textsuperscript{3} A/W at 950nm is achieved with 10V bias.

Effect of Annealing Temperatures on Cerium Oxide (CeO\textsubscript{2}) Gate Based on N-Type Silicon

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This work presents the effects of annealing temperatures (600, 700, 800, 900 and 1000°C) on the quality of CeO\textsubscript{2} film. CeO\textsubscript{2} film was successfully deposited onto silicon (Si) substrate as a dielectric layer using metal organic decomposition (MOD) technique. X-ray diffraction (XRD) results indicated that (111), (200), (220) and (311) planes had been obtained from the deposited film. Electrical properties of CeO, thin film had been characterized using a semiconductor parameter analyzer. Current-voltage results showed that the lowest leakage current had been recorded by the oxide annealed at the highest temperature. This results were in agreement with the interface and near-
interface traps density as well as the effective oxide charge that had been deduced from high frequency capacitance-voltage measurement.

Mesoporous 3D Ceramic Structure Synthesis from Oxide Powder Compositions Via DMLS

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The 3D mesoporous part fabrication with wide electro physical characteristics via Direct Metal Laser Sintering process from the next exothermal oxide powder mixtures, traditionally used in Self-Propagated High-Temperature Synthesis (SHS), had been realized: ceramoplastic (PZT+ PVDF or SiO2 additives) with ferroelectric properties; biocompatible Al(A1O3) + Zr(ZrO2) ceramics; ferromagnetic Ba+M and Li+M systems (M ~ Fe, Cr) for Li0.5Fe2.52xCrxO4 and BaFe12-2xCrxO19 ferrites; high temperature superconductive ceramics YBa2Cu3O7, SrFe12O19. Such materials have promised potential to be applied to the synthesis of smart micro devices (Micro Electro Mechanical Systems, or MEMS devices): ultrasonic sensors; filters; piezoelectric detectors, drug delivery systems and pumps and etc. To describe the arrangement of the component phases within a composite could be through the concept of structure connectivity. The physical properties of the 3D parts can be tailored by changing the phase–structure connectivity (by results of SEM-EDX, X-ray analysis), volume fraction of the main phases or pores in the composite and those special distributions. Optimal regimes laser synthesis 3D parts and some their electro physical features were determined depend of a laser influence parameters.

Self-assembling Nanotechnology of Pinning Centres in Thick Superconducting Films for High-field Power Applications

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For power applications of superconducting films, the critical current density (Jc) and the thickness of the film (d) should be as high as possible. Jc decreases with both thickness (for films thicker than a few hundred nanometers) and magnetic field, so artificial pinning centres in addition to natural ones are required. The earliest cost-effective method used for introducing artificial pinning centres was the so-called substrate decoration, i.e., growing nanoscale islands (nano-dots) of certain materials on the substrate prior to the deposition of the superconducting thin film. Later on two other approaches proved to be successful: bulding up a layered distribution of a second phase using a multilayer deposition (quasi-superlattices), and, respectively, by distribution of a secondary phase as a result of a compositional change in the target. Several materials have been used till now for the creation of artificial pinning centres. Here we report on the artificial pinning centres induced in YBa2Cu3O7 (YBCO) thick films by substrate decoration and quasi-superlattices approach using Au, Ag, Pd, LaNiO3, Pr Ba2Cu3O7, and non-superconducting YBCO. In addition, we will present the properties of thick superconductor films grown from a nano-crystalline YBCO target doped with 4 wt.% BaZrO3 and from a YBCO target containing 1 mol% Gd2Ba4CuWOy nanoinclusions. The films were characterized by frequency-dependent AC susceptibility, DC magnetization, field-orientation-dependent transport measurements, XRD, SEM (EDX) and TEM. The effect of film thickness and nano-dots induced pinning centres on the critical current density, pinning force and pinning potential will be discussed.
Hydrothermal Epitaxy of ZnO Films in Water

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Epitaxial ZnO films have been hydrothermally grown in water at 90 °C on MgAl2O4 (111) substrates. A lateral epitaxial overgrowth (LEO) method was utilized to reduce threading dislocations at the boundaries of crystal mosaic in the epitaxial films. In the optimized ZnO LEO with a ratio of window to wing (3:10), dislocations arose from two regions, i.e. unmasked window and coalescence of masked wings while they were seldom present in the wing area. The average dislocation density was significantly reduced from 1.4 x 10^8 cm^-2 to 2.3 x 10^6 cm^-2. A double LEO was applied for further dislocation reduction with the 3µm-wide unmasked regions centered on the coalesced wings regions from the first LEO. The average dislocation density was found not to decrease by another order of magnitude but only to 1 x 10^7 cm^-2 because new threading dislocations are created at the coalescence of wings from the first LEO.

The Electrical and Structural Analysis of Al Doped ZnO Deposited at Low Temperature Using a MOCVD System

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Zinc oxide (ZnO) is an II–VI semiconductor oxide with a direct band gap of 3.3 eV. Besides the wide band gap, ZnO has several interesting properties, such as large exciton binding energy, high chemical stability, good piezoelectric properties and non-toxicity, which have greatly considered since a few decade years ago. Especially, ZnO has been a subject of research to acquire the low resistivity and high energy conversion efficiency of Si thin film solar cells. The optimization of electrical, optical properties of Al doped ZnO thin films has been investigated in order to make them suitable for transparent conducting oxide for Si thin film solar cells.

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Annealing Effect on the Structural and Optical Properties of SnO2 Thin Films Prepared by Pulsed Laser Ablation

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Tin oxide (SnO2) thin films were deposited on optically flat quartz substrate by pulsed laser ablation. The influence of annealing temperature on the structural and optical properties of these post annealed SnO2 films were systematically investigated by the X-ray diffraction (XRD), atomic force microscopy (AFM), Uv-Vis-NIR spectroscopy, and photoluminescence spectroscopy (PL). A gradually growth of crystal grains with the increase of annealing temperature was observed from XRD patterns and confirmed by the AFM images. The optical properties were determined with Uv-Vis-NIR transmittance spectroscopy based on the Lorentz-Drude (LD) model. It is shown that the dielectric function of the annealed SnO2 is dominated by the band-to-band transition, for which the critical point is shifted to higher energy with the annealing effect. The analysis of the dielectric function based on evolution of the LD parameters can provide an insight into the structural change in the SnO2 films. The observation of defects related PL emissions and their blue shifting with annealing temperatures suggested the evolution of electronic structure caused by the annealing.
Nano-scaled Fully Strained La0.8Sr0.2MnO3/BaTiO3 Perovskite Super-lattice

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The coexistence of ferroelectricity and magnetism in solid state has attracted great attention and generated intensive study in recent years aiming for various electronic device applications, such as memory storage, transducers, sensors, and actuators, etc., and it has obvious advantage of one more additional degree of freedom on polarization switch by applied magnetic field. However, there only are few ‘multi-ferroic’ materials in nature that simultaneously exhibit both ferroelectric (FE) and ferromagnetic (FM) properties. But the two-phase composite approach can be used to overcome the obstacle in nature and enhance ferroelectromagnetic properties in materials, with each phase independently optimized for its desired properties and performance. In this work, colossal ferromagnetic La1-xSrxFeeO3 (LSMO) and ferroelectric BaTiO3 (BTO), which are representatives of the FM and FE materials, were epitaxially grown on SrTiO3 (001) single crystal substrate into various nano-scaled LSMO/BTO super-lattices to realize the strong coherence between FE and FM properties. In-situ mono-layer growth was monitored by reflection high energy electron diffraction (RHEED) and crystal structure of super-lattices was characterized by high resolution X-ray diffraction (XRD). The fully strained epitaxial structure was obtained with evidence of asymmetric reciprocal space mapping and with the growth direction parallel to [001]. The full width at half maximum (FWHM) of X-ray rocking curve measurement around the satellite peak of super-lattice was only 0.005°, indicating the almost perfect crystal plane and sharp interface in super-lattices.

Effects of Sputtering Parameters on Optical Properties of ZnO:Mn Thin Films

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This research using the RF magnetron sputtering technique to deposited ZnO:Mn thin films on glass substrates from 1 mol % Mn-doped ZnO (ZMO) target, and study the effect of correlated sputtering parameters, include substrate temperature and O2 flow ratio, in order to achieve the optical characteristics demand. The crystalline structure, surface roughness and optical transmittance measurements were carried out using a XRD, AFM and UV-VIS spectrophotometer. From the experimental result, we confirm that the sputtering ZnO:Mn thin films prepared at higher substrate temperature of 250°C and 30% of O2 flow rate having a preferred orientation with c-axis perpendicular to the substrate were obtained; it also has a fine crystal structure, uniformity of smooth surface roughness and an optical transparency of over 85% in the visible wavelength region are achieved. So ZnO:Mn thin films can used as transparent conducting film for actual optical devices.

Analysis of Brittle Ceramics Experiencing High Velocity Impact

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In this paper, both Tate penetration theory and cavity expansion theory were used to derive a general model to predict the ballistic limit of ceramic plates experiencing high velocity impact. The general model can deal with finite thickness of ceramic plates, in stead of infinite thickness assumed in most papers. It was shown that the analytical results from the model compared well with simulation and experimental values when plates are thick. Normally, response regions of the damaged ceramic plates are divided into four zones: cavity-pulverized-cracked-elastic. However, it was also discovered that the cracked zone diminishes when the plates are thinner.

Electronic Structure and Crystallinity of HfO2-TiO2 Thin Films

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The advancement of electronic devices has resulted in the vigorous downscaling of device dimensions to the nanotechnology regime. Hafnium dioxide, or HfO2, is one of the most promising candidates to be utilized as a replacement for conventional gate dielectric materials due to its thermal stability in contact with silicon, high dielectric constant and large band offset, compared to other high-κ materials. By incorporating with TiO2 in the form of composite films, it is possible to produce equivalent oxide thickness (EOT) at nano-scale regime. In this study, the effect of the TiO2 incorporation on the band structure...
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and the crystallinity of the HfO₂ thin films have been studied by X-ray diffraction, UV-VIS spectroscopy and X-ray photoelectron spectroscopy. The bandgap energy decreases with increasing TiO₂ concentration. The shift in the valence band edge and the conduction band edge are due to the changes in electronic structures, induced by the concentration of the incorporated of TiO₂ and the microstructure. The electronic structures were also studied by first principle electronic structure calculations. Our experimental results confirmed the theoretical studies.

Deposition of Bi₄Ti₃O₁₂ Thin Film on the Etched Aluminum Foil by Self-Assembled Method to Increase Capacitance

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Bismuth titanate precursor thin films were deposited on the etched aluminum foils by self-assembled method in an aqueous solution at 40-60 °C. We have investigated the film growth rate, mechanism, and influencing factors. The film microstructure and thickness can be controlled by appropriate self-assembled method and solution conditions, such as the concentration of starting materials, pH, and deposition temperatures. Uniform, smooth, and adhesive films could be successfully prepared on the surfaces of etched aluminum foils. The films were composed of closely packed particles of 20-50 nm. After anodizing, a Bi₄Ti₃O₁₂-Al₂O₃ composite film was formed and its capacitor properties were examined. It was shown that the capacitance, the product of capacitance and withstanding voltage increased dramatically compare to those without a Bi₄Ti₃O₁₂ deposition film.

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Dependence of Water Emission Rate and Microstructure upon Flux Layer Coated on the Surface of Porous Functional Ceramics

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The various fluxing agents such as alkali and alkaline earth metal-contained precursor were coated selectively on a surface of porous ceramics composing of clay and carbon to modify the shell structure, then the bloating tendency, microstructure, water emission rate for the sintered porous specimens were analyzed. The surface coating on green ceramic body by powder type precursor was relatively easy but the coating layer was not uniform and loosely stuck while the solution type precursor offered homogeneous coating layer and resisted against peeling-off even at high temperature sintering. The fluxing precursors containing metals of larger ionic radius and higher valence showed enhanced a liquid forming ability on ceramic surface at sintering due to the stronger electrostatic ion field strength over oxygen ion. For example, the porous ceramics coated with 0.8wt% of 10 M NaOH solution and sintered at 1150°C for 10min had a uniform 30μm-thick vitreous shell, and the bloated pores ranged few hundred nm ~ few tens micrometer generated in core part of the specimen. Also the specimen showed a porosity 25% and initial water emission rate (WER) 0.21%/hr, terminal WER 0.12%/hr within one month experimental period which is lower than that of as received specimen. The water emission rate in an initial step depended on a void content of porous ceramics filled in a container rather than a shell structure. But, after 7 days where the water emission rate of the porous ceramics with a flux layer coat decreased because the vitreous dense shell of porous ceramics suppressed the water emission. It could be concluded that the uniform coating of proper fluxing agent on ceramic surface induces an adequate bloating phenomenon which increases porosity and decreases water emission rate of porous sintered body, so the porous ceramics fabricated in this study could be applied to the smart soils with slow water emission rate function.
The Preparation of Hybrid TiO₂ Electrode for the Dye-Sensitized Solar Cell Applications

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A hybrid TiO₂ electrode was prepared on ITO/PEN substrate at room temperature by using sputtering and compressing methods. In this study, the sputter-deposited TiO₂ coating acted as compact layer was found to be outstanding for improving the performance of the dye-sensitized solar cells. It attained the open-circuit voltage of 770 mV, short-circuit photocurrent of 13.1 mA/cm², fill factor of 0.32, and solar-to-electric energy conversion efficiency of 3.22% under irradiation of white light (100 mW/cm²). Compared with non compact layer, the solar-to-electric energy conversion efficiency was enhanced for about 30%. This suggested that it not only achieves a desirable thickness, but also enhances photoelectric conversion efficiency by using a hybrid TiO₂ electrode.

Effects of Electrolytic Mg(OH)₂ Coating on Corrosion Resistance of AZ91D Magnesium Alloy

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The electrolytic deposition of Mg(OH)₂ on AZ91D magnesium alloy was conducted in Mg(NO₃)₂ solution at constant applied voltage. The influence of applied voltages as well as deposition time on the corrosion resistance of coated specimens was investigated. The surface morphology and phase structure were analyzed using scanning electron microscope (SEM) and grazing angle X-ray diffraction (XRD). Polarization curves, immersion test and electrochemical impedance spectroscopy were used to evaluate the corrosion resistance of coated specimens. More OH⁻ ions were produced and the migration of Mg²⁺ was further enhanced at more negative applied voltage, leading to higher nucleation rate of Mg(OH)₂ on the specimen and resulting in the (001) preferred orientation. The results indicated that the coating film carried out at -1.8 V, and condensed into MgO with thickness 16 μm at 350°C. At more negative than -1.8 V, massive H₂ bubbles were formed, making some Mg(OH)₂ float away. The weight gain of coatings was obviously increased with increasing deposition time for the initial 40 min, and the increasing rate was slow down due to H₂ bubble formation and the increasing resistance of coating film after 40 min. The best corrosion resistance of the coating film was carried out at -1.8 V for 3600s which showed corrosion current density 0.79 μA/cm² and passive range from -1.52 to -1.25 V. Compared with AZ91D magnesium alloy substrate, the corrosion resistance was improved more than two orders by MgO ceramic coating.

Sol-Gel Preparation and Optical Properties of Rare-earth Doped Zinc Oxide Thin Films Suitable for Solar Cell Application

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Owing to the superior chemical stability, ZnO has a potential as a host lattice for doping several luminescence centers and show versatile application. In this study, the optical properties of rare-earth doped ZnO were studied. Europium doped zinc oxide (ZnO:Eu) and terbium doped zinc oxide (ZnO:Tb) thin films were prepared by sol-gel dip-coating process on glass substrates. The content of terbium and europium in the sol was varied from 5% to 15%. Synthesis parameters such as annealing temperature and
concentration of doped ions on the highly oriented crystal growth were studied. The coating was characterized by diffuse reflectance UV-visible (DRUV) and fluorescence spectrophotometer. The crystalline ZnO thin films were obtained following an annealing process at temperatures between 300 °C to 500 °C for 2 h. ZnO:Eu and ZnO:Tb thin films are transparent in near ultraviolet and visible region. From luminescence analysis, energy transfer from the ZnO host to the doped Eu3+ and Tb3+ has occurred. Band gap energy of rare-earth doped ZnO suggest that the absorption edge has moved to the longer wavelength with increasing amount of doping, hence decreasing the optical band gap of films. From the optical properties, lowering of band gaps of the rare-earth doped ZnO has high potential in solar cell applications.

**Relaxation Dynamics in Epitaxial BiFeO3 Thin Films**

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BiFeO3 is the most studied among many multiferroic thin films due to its high spontaneous polarization and anti-ferromagnetism at room temperature. There are intense discussions and researches about using BiFeO3 as non-volatile ferroelectric memory or into other applications. Hence the studies on the relaxation of BiFeO3 thin films are quite crucial to understand the switching and relaxation dynamics in nanoscale. We present some relaxation results about BiFeO3 thin films on a SrRuO3/SiTiO3 substrate and show that it may follow a different behavior with the relaxation of conventional PZT ferroelectric thin films.

**The Thickness-dependent Stripe Domains in BiFeO3 Thin Films**

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The thickness-dependent stripe domain width has been studied, taking into account the elastic energy at the film/substrate interface and the electrostatic energy on the surface in the epitaxial rhombohedral BiFeO3 films. It is shown that the classical domain width scaling law with an exponent 0.5 in ferroic materials is not applicable after the reduction of the film thickness D to a critical thickness Dcrit approximate 100 nm. In the region of D < Dcrit, the domain width W experiences an inflexion point and begins increasing dramatically inversely. The theoretical dependence of domain width W on film thickness D agrees well with experimental results and indicates that the domain equilibrium structures are determined dominantly by minimizing the elastic energy and domain wall energy in epitaxial BiFeO3 film.

**Fabrication of Nd3+:Y2O3 Transparent Ceramics for Laser Application**

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Cubic Y2O3 crystal has been investigated for a long time as a laser-host material due to its high thermal conductivity, broad spectral region, chemical stability, strong Stark-splitting and relatively low phonon energies. However, it is extremely difficult to grow large-size Y2O3 single crystal with high quality because of its very high melting point (~2430°C) and structural phase transition at ≈2280°C. In present work, transparent Nd3+:Y2O3 ceramics with different doping concentration were fabricated by using the powder synthesized from co-precipitation method using ammonia as the precipitant. After calcined at 1000°C degree for 2 h, cubic phase of Y2O3 with the crystal size around 60-80 nm was presented. The as-prepared nanocrystalline powders were cold isostatically pressed into 18-mm diameter pellets under 200 MPa pressure. After the powder compacts were sintered at 1850°C for 3 h in flowing hydrogen atmosphere, highly transparent Nd3+:Y2O3 ceramics were obtained. The sintering temperature is about 600°C lower than its melting temperature. SEM investigation revealed the average grain size of prepared ceramics was about 7-8 µm. The transmittance of 1 mm thickness samples reached 80%, which is close to the theoretical value of Y2O3. The optical spectroscopic properties of Nd3+:Y2O3 transparent ceramics with the doping concentration from 0.2-3.0at.% were also investigated. Under the excitation of a 808 nm diode laser, three main emission bands centered around 870-950 nm were presented. The as-prepared nanocrystalline powders were cold isostatically pressed into 18-mm diameter pellets under 200 MPa pressure. After the powder compacts were sintered at 1850°C for 3 h in flowing hydrogen atmosphere, highly transparent Nd3+:Y2O3 ceramics were obtained. The sintering temperature is about 600°C lower than its melting temperature. SEM investigation revealed the average grain size of prepared ceramics was about 7-8 µm. The transmittance of 1 mm thickness samples reached 80%, which is close to the theoretical value of Y2O3. The optical spectroscopic properties of Nd3+:Y2O3 transparent ceramics with the doping concentration from 0.2-3.0at.% were also investigated. Under the excitation of a 808 nm diode laser, three main emission bands centered around 870-950 nm, 1047-1137 nm and 1310-1460 nm were observed, which were corresponding to the transition from 4F9/2 to 4I15/2, 4I11/2 and 4I9/2 energy levels of Nd3+ ions, respectively. With the doping concentration increased from 0.2at.% to 3.0at.%, the fluorescence lifetimes of Nd3+:Y2O3 transparent ceramics sharply decreased from 321 µs to 65 µs. From the investigation results, it can be clear seen that the optical spectroscopic properties of Nd3+:Y2O3 is quite different than that of Nd3+:YAG.
Enhanced Multiferroic Properties in Gd-doped BiFeO$_3$ Thin Films

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We have investigated the influence of Gd substitution on the ferroelectric and magnetic properties of epitaxial BiFeO$_3$ thin films. Gd substitution can successfully suppress the remnant magnetization than the undoped samples. It was found that Gd substitution can substantially suppress the leakage current density, lower the electric coercive field and destruct the space-modulated cycloid spin structure of pure BiFeO$_3$.

Structure and Microstructure of EB-PVD yttria Thin Films Grown on Si (111) Substrate

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Yttrium oxide, Y$_2$O$_3$, belongs to a relatively large group of sesquioxides with the C-type cubic structure, which is similar to the fluorite structure. For many applications the thin film form of this material is important for which a close connection between structure and microstructure/morphology is typical. These properties are basically determined by the conditions under which the films were prepared and treated. The present study deals with investigation of the effect of preparation conditions during the electron-beam physical vapour deposition (EB-PVD), in particular the deposition temperature (T$_{dep}$), on the structure and morphology of Y$_2$O$_3$ thin films as well as with their comparison with the single crystalline and ceramics forms of this material. The X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) techniques were applied. The films exhibit polycrystalline structure for all deposition temperatures. Sequence of the XRD peaks corresponds to the most intense peaks of the Y$_2$O$_3$ body-centered cubic (bcc) phase, space group 206. Peak shifts indicate a lattice expansion by ~ 1% in comparison with the well developed crystalline phase. With increasing deposition temperature, the grains of the bcc grow to 14.1 nm for 660°C and 23.7 nm for 820°C while the lattice parameter approaches the database value of 1.0604 nm. Relative peak intensities suggest a preferential grain orientation (texture) for 660°C. In addition to the bcc phase, small traces of the Y$_2$O$_3$ base-centered monoclinic phase, space group 12, are observed for 820°C. The gradual growth of the bcc grains and improvement of their crystalline structure with the deposition temperature is in a full agreement with the surface morphology/microstructure and step coverage as well as with the columnar grain cross-sectional morphology/microstructure. The microscopic details of the cross-sections exhibit a well defined columnar growth (collinear elongated columns are actually elongated grains) for both 660°C and 820°C deposition temperatures. At room temperature, the columnar structure is developed only partly with a lower degree of perfection which affects also the surface morphology. In comparison with rotationally deposited Y$_2$O$_3$ layers, our stationary deposited layers are more dense with the less porosity between the columns. Therefore the feather-like structure usually observed for rotationally deposited layers is obscured in our case.

Electro-mechanical Properties of Sm$_{2}$O$_{3}$-doped Ceria Films

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In the field of oxygen ion conducting materials there is a special need of the compounds with a low activation enthalpy for migration, and a high mobile oxygen vacancy concentration. And from this point of view, just oxides with the fluorite-type structure doped with trivalent cations are of a technological importance due to their potential applications in the intermediate temperature solid oxide fuel cells (IT-SOFCs) and oxygen sensors. Interesting are also their applications as effective coating
layers. Especially ceria doped with rare-earth oxides, e. g. Sm₂O₃, is suitable for this kind of applications, namely its film form due to the improvement of efficiency and reduction of operating temperature. In the present study were investigated the electrical conductivity, dielectric permittivity as well as microhardnes and effective elastic modulus of CeO₂ - x . Sm₂O₃ (x = 0, 10.9 – 15.9 mol% ) films prepared by electron beam-physical vapour and ionic beam assisted (EB-PVD+ IBAD) depositions on the Si(111) and Si (100) substrates, in dependence on their structural characteristics and microstructure. The special attention was devoted to the influence of Ar⁺ ion bombardment (IBAD) on the investigated properties and to the quality of interface Sm₂O₃-CeO₂ film / Si substrate. The methods used for the investigation, such as impedance spectroscopy (IS) and isothermal charge deep level transient spectroscopy (DLTS) as well as C-V technique, classical Vickers indentation method and depth - sensing indentation (DSI) technique have shown the close connection between investigated properties and conditions under which films were prepared, treated and measured. According to the results obtained, the investigated Sm₂O₃ – doped CeO₂ films could be utilized in various applications such as IT-SOFC, planar-type SOFC and protective coatings on the metals as well as optical coatings in the optical devices depending on their phase composition, structure and microstructure.

A00489-00889

Optical Phenomena in Ferrimagnetic/Piezoelectric Composite Structures in External Electric and Magnetic Fields

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An external electric field applied to a crystal induces in it the optical phenomena, which depends of the magnetic field H. The effect of light polarization plane rotation in crystals is one of such phenomenon induced by the electric field, termed by authors as the electromagneto-optical (EMO) effect (EMOE).

The EMO effect as a magnetoelectric response for yttrium iron garnets (YIG)/lead zirconate titanate (PZT) (ferrimagnetic/piezoelectric/ceramic) composite structure by applying an external electrical field E was registered by using high-sensitive optical polarimetry method. The sample represented the hybrid structure with stuck together thin magnetostrictive and a piezoelectric plates. The measurements have been fulfilled on small areas of sample (laser probing D < 2 mm). A He-Ne laser (λ =0.63 µm) was used in the experiment. EMO characterization (changes of the magneto-optical Faraday rotation) as a magnetoelectric response for YIG film by applying an external electrical field to both YIG and PZT phases was carried out as a function of the variable electric field E (0-6 kV/cm), which was applied perpendicular to the sample plane and static magnetic field H (0-1.2 kOe), which was applied parallel to the sample plane. Piezoelectric phase in given structure carries out amplifier function for EMOE already eight than for separate ferrimagnetic phase (epitaxial YIG film) that was measured by us earlier. The value of EMO effect is expected an author to be dependent sensitively on the volumes of the magnetostriective and piezoelectric phases. EMOE can be interesting from the applied point of view as a basis for creation of devices with two-channel management, on electric and magnetic fields. We consider magnetolectric effect in an optical range as a new degree of freedom in designing magnetoelectric devices.

A00487-00898

Crystallization Behavior of SiO₂-Al₂O₃-Li₂O-CaO Glasses by Nonisothermal Analysis

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The glass-ceramics produced from coal bottom ash with flux agents 10 wt% Li₂O and 0–10wt% CaO, and nucleation agent 2–8 wt% TiO₂ has a major crystal phase β-spodumene at interior of sample but simultaneously crystallization initiates from the surface nucleation sites too which could be harmful to mechanical properties.

The mechanism for the surface nucleation and crystallization of SiO₂-Al₂O₃-Li₂O-CaO glass-ceramics was studied by examining an activation energy, an Avrami constant which were calculated as a function of composition and size of specimen and heat treatment condition.

As the added TiO₂ decreased, the activation energy for crystallization increased and Avrami constant came close to 1. For the bulk sample, on the contrary, the activation energy was low as 307 kJ/mol and Avrami constant high as 2.5.

The crystals in glass-ceramics having Avrami constant over 3 growed 3-dimensionally and showed spherolite shape while in specimens of less than 2, 1-dimensionally and acicula shape. The nucleation agent addition to specimen increased the activation energy and decreased Avrami constant, depressing a surface crystallization, in consequence, the glass-ceramics containing a nucleation agent showed a higher degree of crystallization, higher 3-point strength as 45 MPa, and lower thermal expansion as 78×10⁻⁴K⁻¹, compared to the sample without nucleation agent.
Enhancement of Ferromagnetic and Dielectric Properties in Lanthanum Doped BiFeO₃ Nanopowders

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Bismuth ferrite, BiFeO₃, is an excellent candidate for use in ferroelectric non-volatile memories and high performance electronics because of the coexistence of ferromagnetism and ferroelectric polarization also is known as multiferroic. As one of very few existing single-phase multiferroics, BiFeO₃ with a perovskite structure has high electrical polarization, high Curie temperature (~820°C), and G-type antiferromagnetic Néel temperature (Tₙ ~ 475°C).

In previous work, BiFeO₃ in the forms of bulk, thin film and particles was prepared by diverse routes. However, pure BiFeO₃ is characterized by serious current leakage problems, making it difficult to attain high resistivity due to defects as well as nonstoichiometry. In order to overcome this disadvantage, many attempts have been made including doping different elements into BiFeO₃ and depositing multi-layer thin films. It is proved that lanthanum doping is an efficient way to improve its ferroelectric properties. However, it should be noted that much of the work reported so far has usually focused on the thin films. Because the features of thin films often depend on the film thickness and strain caused by mismatch of the lattice parameters between the film and the substrates, it is hard to separate the impact of elements substitution from the interaction between the film and the substrate. Therefore, it is necessary to study the magnetic and electrical properties of lanthanum doped BiFeO₃ particles.

In this work, lanthanum substituted BiFeO₃ samples were prepared by the hydrothermal method at 200°C. X-ray diffraction (XRD) and scanning electronic microscope measurements revealed the crystal structure and morphologies of the samples. XRD refinement showed that the lattice parameters were strongly affected by lanthanum doping levels. Measurement of the zero-field cooled magnetization, as well as magnetization versus field loops, verified that the ferromagnetic properties were enhanced with increasing La doping level in the samples thus produced. It was also found that both the dielectric constant and the dielectric loss were dependent on the La content. The enhancement of the ferromagnetic and dielectric properties was attributed to lanthanum substitution in the BiFeO₃, which also agreed with the XRD refinement results.

A00510-00949

Piezoelectric Coefficient Study of Spray Dried Iron Doped Lead Zirconate Titanate Ceramics

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Lead Zirconate Titanate (PZT) material is a ferroelectric material being exploited for different sensor application. Effect of ferromagnetic dopant in varying concentration in ferroelectric PZT material has been studied and presented in present paper. Nitrate solution of Lead, Zirconium and Titanium has been taken based on stoichiometry of formula. These properly mixed transparent solution has been feed in spray drying machine and spray dried nitrate powder of PZT has been collected. DTA/TG characterization of green powder reveals the calcinations temperature as well as the lead loss during calcinations. XRD test of powder calcined at 800 degree centigrade conforms the presence of PZT phases. Iron doping from 0.5 atomic percentage to 2.5 atomic percentage has been done at Zr/Ti ratio 54/46 before spray drying the solutions. Pelletization of calcined powder is done at 250 Mpa of pressure followed by sintering of pellets at 1150 degree centigrade. Sintered pellets has been poled at different D.C poling field varying from 10 Kv/cm to 100 Kv/cm. Piezoelectric coefficient (d33) measurement of Iron doped PZT materials reveal that at low concentration of dopant d33 value increases but...
Fabrication and Luminescence Behavior of Neodymium Doped Yttrium Aluminum Garnet Transparent Ceramics

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Polycrystalline Nd:YAG transparent ceramics are currently studied to replace their single crystals as a solid-state laser host material, owing to their attractive thermal properties, excellent chemical stability, unique optical properties and high efficient laser oscillations. In our work, spherical Nd:Y2O3 nanoparticles were synthesized by coprecipitation method from a mixed solution of yttrium and neodymium nitrate using ammonium hydrogen carbonate as a precipitant. The thermal decomposition behavior of the carbonate precipitate, phase evolution, microstructure and sinterability of the powders were studied. Pure phase Nd:Y2O3 nanoparticles appeared by calcining at 700 °C for 4 h. The nanocrystalline Nd:Y2O3 particles calcined at 1100 °C were well dispersed with average diameter of about 60 nm. Transparent fully dense Nd:YAG ceramics were fabricated from as-prepared Nd:Y2O3 powders and commercial Al2O3 powders using TEOS as a sintering additive by solid-state reaction under vacuum sintering at 1750 °C for 6 h. The phase evolution, microstructures and optical properties of the transparent ceramics were investigated. The results show that the grain boundary of the sample was clean and no second phase was observed. The fluorescence lifetime decreases dramatically with an increase of Nd concentration.

Observation of High Dielectric Constant in Polymer/AlCuFe-Nanoquasicrystalline Composites

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An improvement in average dielectric constant value is expected if a low and high dielectric constant materials blended to develop a composite. But interestingly an abnormal increase in effective dielectric constant (\(\varepsilon_{\text{eff}}\)) near the percolation threshold or critical volume fraction of conductive component (\(\phi_c\)) with low dielectric loss (Tan\(\delta\)) has been recently reported in metal-polymer composites. Recent reports suggest that by reducing the filler particle dimensions \(\phi_c\) can be lowered to retain the flexibility of the polymer matrix as well as superior dielectric properties. In an effort to understand the phenomenal enhancement in \(\varepsilon_{\text{eff}}\) and to develop a low cost polymer-based composite with high conductivity or high dielectric constant at low percolation threshold a variety of ferroelectric polymers like polyvinylidene fluoride based composites with different filler materials have been investigated. New interesting results continue to appear even now with novel filler materials to understand the effect of filler materials and processing conditions on percolations threshold. In this regard thermodynamically stable quasicrystalline alloys (QC) have unique properties that include lower values of coefficient of friction, thermal and electrical conductivity and high hardness, thermal stability with a major drawback of brittle nature. On the other hand, dielectric/QC/dielectric thin film multilayers are reported to be excellent solar selective absorbers. Therefore, in the present article for the first time we report on nanocrystalline Al\(_{63}\)Cu\(_{23}\)Fe\(_{12}\) QC particles embedded in ferroelectric-PVDF matrix.

In the first step fine powders of Al\(_{63}\)Cu\(_{23}\)Fe\(_{12}\) alloy have been prepared through mechanical alloying process. Subsequently well-ordered quasicrystalline Al\(_{63}\)Cu\(_{23}\)Fe\(_{12}\) (~20 nm-30 nm) powders were obtained by annealing (800°C) the powder under inert gas atmosphere for 2 hrs. The QC phase and properties have been confirmed through XRD and temperature dependence of resistivity measurements. The PVDF and QC powders were blended together in the form of disks of diameter 13 mm and thickness 2 mm under 10 MPa. Series of polymer based composites with varying volume fractions of QC were prepared in order to reach the percolation threshold as closely as possible. The dielectric properties were measured using an Agilent precision impedance analyzer (4294A) in the frequency range of 40 Hz-10 MHz, with a 16451B dielectric text fixture. It is shown that at the percolation threshold, dielectric constant of the composite exhibits a remarkable enhancement with moderate loss. The \(\varepsilon_{\text{eff}}\) rises from ~300 to 3800 when \(\phi_{\text{QC}}\) increases from 0.22 to 0.228 at 100 Hz. Similarly \(\sigma_{\text{eff}}\) clearly demonstrates an Insulator-Metal transition (IMT) in the vicinity of \(\phi_{\text{QC}}\sim 0.22\). The large enhancement in \(\varepsilon_{\text{eff}}\) in the neighborhood of \(\phi_{\text{QC}}\) can be understood on the basis of ‘boundary layer capacitor effect’.

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In Situ X-ray Photoelectron Spectroscopy Studies of HfO\textsubscript{2} Gate Dielectric on SiC

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The integration of high dielectric constant (high-k) oxides with SiC has attracted much attention for applications in high temperature, high power, and high frequency devices because of accelerated degradation with traditional SiO\textsubscript{2} gate insulator. In this work, we studied the integration of HfO\textsubscript{2}/SiC system for nanoelectronics devices using x-ray photoelectron spectroscopy. The interface stability and band offsets at the interfaces of HfO\textsubscript{2}/SiC and the effect of nitrogen on the electronic structures and thermal stability of HfO\textsubscript{2} thin film have been investigated. It was found that the valence- and conduction- band offsets at HfO\textsubscript{2}/SiC interfaces were measured to be 1.02 eV and 1.53 eV, respectively. The atomic source nitridation improves interface thermal stability with nitrogen passivation for the oxygen vacancies in dielectric films and for the defects on SiC surface, but induces band gap reduction for the HfO\textsubscript{2} dielectric layer and band alignment shift at the interface. Furthermore, Post-nitridation annealing helps to improve the band offsets of dielectric film to have sufficient injection barrier. The results show that HfO\textsubscript{2} is a promising high-k gate dielectric integrated on high-performance SiC for high-power and high-temperature devices.

Fabrication and Photocatalytic Activities in Visible and UV light Regions of Ag@TiO\textsubscript{2} and NiAg@TiO\textsubscript{2} Nanoparticles

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In this study, we prepared the highly dispersible core-shell type of photocatalysts, with Ag nanoparticles or NiAg alloy nanoparticles as the metal cores and TiO\textsubscript{2} as the oxide shells. Ag cores may trap photogenerated electrons or holes and are expected to extend the absorption to visible light region and enhance the photocatalytic activity. NiAg alloy cores not only can achieve the same effects as Ag cores but also possess magnetic property which makes it possible to recover the photocatalysts from the treated solutions by magnetic force without further treatment. TEM analysis revealed that they were found to be completely discrete with core diameters of 6.55 ± 1.20 and 7.57 ± 1.33 nm as well as shell thicknesses of 2.59 and 2.80 nm for Ag@TiO\textsubscript{2} and NiAg@TiO\textsubscript{2}, respectively. Their core-shell structure has also been recognized by the analyses of EDX and FTIR spectra. XRD analysis indicated that Ag and NiAg cores were not oxidized after TiO\textsubscript{2} coating and the TiO\textsubscript{2} shells were amorphous. Noteworthily, the resultant Ag@TiO\textsubscript{2} and NiAg@TiO\textsubscript{2} nanoparticles not only showed significant characteristic absorption in the visible light region owing to the surface plasmon resonance of Ag as expected but also had lower zeta potentials than TiO\textsubscript{2} nanoparticles, which favored the adsorption of cationic dye Rhodamine B on the particle surface and thereby enhanced their photocatalytic activities. Under visible light illumination, Ag@TiO\textsubscript{2} and NiAg@TiO\textsubscript{2} nanoparticles exhibited significantly higher photocatalytic activities than TiO\textsubscript{2} nanoparticles. A novel mechanism based on the formation of Schottky barrier banding at the core-shell interface as well as the excitation of photogenerated electrons from the surface of Ag or NiAg cores to the conduction band of TiO\textsubscript{2} shells has been proposed. In contrast with the photocatalytic reaction under UV light illumination, Ag@TiO\textsubscript{2} and NiAg@TiO\textsubscript{2} nanoparticles showed lower photocatalytic activities than TiO\textsubscript{2} nanoparticles. This could be attributed to the lower TiO\textsubscript{2} content as well as the transfer of photogenerated electrons from TiO\textsubscript{2} shells to Ag or NiAg cores where the photoinduced electrons might recombine with holes and hence led to the decrease in the photocatalytic activity. The Ag@TiO\textsubscript{2} and NiAg@TiO\textsubscript{2} nanoparticles were expected to be useful as the effective photocatalysts in the visible light region. In particular, NiAg@TiO\textsubscript{2} nanoparticles could be recovered magnetically even though their photocatalytic activity was lower as compared to Ag@TiO\textsubscript{2} nanoparticles.

Study on Moisture Adsorption Principle of Porous Mineral Materials

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Porous mineral materials, such as diatomite, sepiolite and attapulgite, have been selected as samples, and their morphology and structures also have been detected by SEM and XRD. Then the theory of moisture sorption for porous mineral materials has been analyzed. The adsorption properties of three samples are tested in the constant temperature and humidity chamber with same temperature but different humidity. It is found that the major factors affecting the moisture adsorption properties of porous mineral materials are specific surface area, pore volume, pore size distribution and pore structure of materials. At the range of middle and low relative humidity (≤50% RH), the larger specific surface area leads to higher moisture adsorption. The moisture adsorption properties increases with the pore volume at middle and high relative humidity (≤50% RH). At the same time, the capillary condensation
is influenced by the pore size distribution of materials. When the material’s pore size distribution is at the range of 3.10nm to 5.66nm, the equilibrium humidity for moisture adsorption is in a scope of 40% - 60%. And the internal pore structure of materials is one of the important factors of moisture desorption.

A00565-01031
Influence of Oxygen Pressure on the Ferroelectric Properties of Epitaxial BiFeO₃ Thin Films by Pulsed Laser Deposition
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BiFeO₃ is the only known single phase multiferroic material that show both ferroelectric and magnetic orderings at room temperature. The growth window of BiFeO₃ thin films is very small. Both temperature and oxygen pressure will affect the film quality and phase purity significantly. We demonstrate here that even within the window where phase pure BiFeO₃ thin films can be obtained, oxygen partial pressure still leads to variation of Bi/Fe ratio in the film. As oxygen pressure decreases, Bi/Fe ratio decreases and remanant polarization also drops. Bi₂O₃ phase is observed in films deposited under high oxygen partial pressure, which leads to higher leakage current, consistent with previous report. Piezoelectric force microscopy also reveals significant difference in the domain structures of these films, possibly due to the influence of Bi and oxygen vacancy defects in film growth.

A00568-01035
Fabrication of ZnO Thin Films by Atomic Layer Deposition Using Flow-rate Interruption Method
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High quality of crystalline ZnO films has been successfully grown on c-plane sapphire substrate by atomic layer deposition (ALD) technique at extremely low temperature. In this work, we obtained the ZnO thin films by ALD method using flow-rate interruption method at extremely low temperature with diethylzinc and D. I. water as precursors. The ALD growth window was found to be 60-160°C by using flow-rate interruption method (FIM). It’s effectual to decrease the growth temperature about 100 °C for ALD with general continuous flow rate method. X-ray reflectivity and high-resolution x-ray diffraction measurements were employed to characterize the microstructure of these films. The results show the low growth temperature result higher crystalline quality and to agree with thermodynamically blocked self-compensation processes.

A00575-01103
Synthesis and Characterization of Cu₂O Thin Films Deposited by Magnetron Sputtering
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Cuprous oxide (Cu₂O) is a direct-gap semiconductor with band gap energy of around 2.1 eV and is regarded as one of the most promising materials for application in photovoltaic cells. In addition, it is known that Cu₂O is an abundantly available material and exhibits multifunctional advantages, such as non-toxicity, high absorption coefficient and low-cost producibility. Thus, Cu₂O films are becoming a multifunctional material.

In this paper, Cu₂O thin films were deposited by radio-frequency (rf) magnetron sputtering at different substrate temperatures. The crystal structure, the optical and the electrical properties were characterized. The x-ray diffraction patterns show that the Cu₂O pure phase is achieved for all films and present a preferred growth orientation [111] with increasing substrate temperatures from 500 to 800 K. The electrical conductivity measured at room temperature increases with the increase of the substrate temperature, so do the optical band energies (E_g). The biggest E_g, 2.58 eV, is achieved for the sample prepared at 800 K.

A00605-01628
Photoluminescence of Manganese-doped ZnAl₂O₄ Nanophosphors
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Manganese-doped zinc aluminate spinel (ZnAl₂O₄:Mn) phosphor nanopowders were prepared by the sol-gel process. The sol-gel transition, crystallization processes and photoluminescent properties of the powders were investigated. The manganese doping concentration had a large impact on the sol-gel transition and photoluminescence intensity of the processed gel powders. X-ray diffraction revealed that the dried powders were amorphous and converted into ZnAl₂O₄ structure followed
firing at above 600 °C. On firing at 600 °C–1000 °C, the doped ZnAl₂O₄ powders had the average crystallite sizes of 12–20 nm. SEM investigation exhibited the phosphor powders had primary particle size of around 20–25 nm and narrow size-distribution after calcining at 1000 °C. The luminescence properties of doped ZnAl₂O₄ nanophosphors were characterized by excitation and emission spectra. Photoluminescence spectra exhibited prominent green emission bands centered at 512 nm, corresponding to the typical 4T₁(4G)—6A₁(6S) transition of Mn²⁺ ions under an excitation wavelength of 458 nm. The luminescence intensity increased with increasing the Mn content up to 3.0 mol %, but decreased significantly as doping of above 4.0 mol%. The emission intensity was also highly dependent on the conditions of heat-treatment.

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A00627-01148

The Effects of Carbon Nano Tubes (CNT) and Carbon on Y-123 High Temperature Superconductors
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In this research, the effects of carbon nano tubes and carbon on the crystal structure and superconducting properties of YBa₂Cu₃Oₓ (Y-123) compound were studied. Samples were synthesized using standard solid state reaction technique by adding CNT and carbon up to 1 wt %. The x-ray diffraction data confirm the single phase orthorhombic structure for all samples.

We obtained changes in critical temperature Tc, the pinning energy Uc, and critical current density Jc by changing CNT doping value in Y-123 samples (0.0–1 wt %). We find that while Tc doesn’t change much with the CNT doping (91–92 K), both Uc and Jc increase systematically up to 0.7 wt % CNT doping in a broad magnetic field ranges between 0.1 and 9 T and Jc in the 0.7 wt % CNT doped sample is at least 10 times larger than that of the pure Y-123. The scanning electron microscope image shows that CNTs forming an electrical-network between grains. These observations suggest that the CNT addition to the Y-123 compounds improve the electrical connection between superconducting grains to result in the Jc increase.

In addition, we prepared samples with different carbon percentage doping (0.05-1 wt %) and found that superconductivity disappears in compounds containing more than 0.3 wt % carbon doped value. Increasing carbon in Y-123 samples decreases the critical temperature Tc.

A00638-01149

Tribological Behavior of Si₃N₄-hBN Ceramic Materials with Water Lubrication
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The main objective of this paper is to clarify the tribological behavior of Si₃N₄-hBN composites with different hexagonal boron nitride (hBN) volume fraction under distilled water lubrication. Water-lubricated sliding tests were carried out on a pin-on-disc tester. Si₃N₄ ceramics were used as the disc
Effect of Doping of Cobalt and Lanthanum (Independently and Simultaneous) on Nature of Ferroelectric to Paraelectric Transition

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Effect of lanthanum on barium site, cobalt on titanium site independently and simultaneously (La and Co on Ba and Ti site) respectively has been investigated on dielectric properties of technologically important BaTi1-xSn0.15O3 composition. Powder X-ray diffraction, scanning electron microscope, measurements of hysteresis loop and dielectric parameters (as a function of temperature and frequency) are used to characterize the resulting single phase solid solution samples. In all the cases the diffusion of ferroelectric (FE) to paraelectric (PE) transition increases with increase in concentration of dopants. Lanthanum doped samples i.e. Ba1-xLa2xTi0.85Sn0.15O3 (x=0.01 and 0.03) exhibit relaxor ferroelectric behavior. Variation of dielectric constant as a function of temperature show that these are suitable as ZSU category of thermally stable capacitors. In all the cases the FE to PE transition occurs below 300K. Samples in ZSU category of thermally stable capacitors. In all the cases diffuseness of ferroelectric transition temperature. Hysteresis loop has been found to be asymmetric which has been explained on the basis of internal bias present in these materials due to different charge on La at Ba ions which occupy random sites.

Immittance Analysis: A Tool to Probe Electroceramics

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Immittance analysis has been used to study electroceramics by separating the contribution of grains (bulk), grainboundaries and electrode-specimen interface to the total measured electrical and dielectric parameters. The result so obtained are utilized to control the overall electrical behavior of the materials by varying processing parameters such as cooling rate after sintering, annealing, after sintering, diffusion annealing, during electroding and by using different routes to prepare the same composition etc. This is illustrated by taking a few examples in the system Sr1-xTi1-xCo3O7 (0.20≤ x ≤ 0.40). All the above mentioned parameters have been shown to influence the electrical properties significantly.

Panoscopic Assembling of Ceria Nanoparticles using Plate-like Titanate for UV-shielding Application via Solution Processes

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Because of the damaging effects of UV-rays, many kinds of UV-shielding materials have been developed. Semiconductor nanoparticles are generally used as inorganic UV-shielding materials, however, the comfort and covering capability on skin of inorganic nanoparticles are generally modest due to agglomeration of nanoparticles. In order to improve the comfort and UV-shielding ability of calcia-doped ceria nanoparticles, the composites consisting of plate-like potassium lithium titanate (K0.8Li0.27Ti1.73O4) micro particles coated with calcia-doped ceria (Ce1-xCaOx) nano particles were prepared by solution processes such as co-precipitation process and sol-gel process and the UV-shielding performance of the products were evaluated. Co-precipitation method: after putting plate-like potassium lithium titanate particles in deionized water at 40°C, appropriate quantities of 3 M

specimens and Si3N4-hBN composite with five different hBN content, i.e. 0, 5, 10, 20 and 30 vol.%, were used as the pin specimens. The results showed that the addition of hBN to the Si3N4 matrix can result in a severe decrease of the friction coefficient, from 0.35 for Si3N4 against Si3N4 to 0.01 for Si3N4-20% hBN against Si3N4. The wear rates (K) of Si3N4-hBN composites decrease greatly with the increase of hBN content, and the lowest wear rate of 10−7 mm² N⁻¹m⁻¹ or less order of magnitude is obtained while the volume fraction of hBN is near to 20%. Subsequently, the wear rates showed an increasing tendency while the volume fraction of hBN is higher than 20%. The morphological analysis and chemical characterization of the worn surfaces, which were made by scanning electron microscopy (SEM) and X-RAY photoelectron spectroscopy (XPS), revealed that the oxidation and hydrolysis of hBN and Si3N4 occurred on the wearing surfaces during the wear tests. For Si3N4-hBN/Si3N4 pairs, the reaction products could accumulate on the wearing surface, forming a tribochemical film composed of B2O3, SiO2 and their hydrates, and hence the friction coefficients are lower compared with Si3N4/Si3N4 pair. However, the reaction products are too difficult to accumulate on the wearing surface and form an efficient tribochemical film for Si3N4/Si3N4 pair, so the friction coefficient is relatively high.

A00639-02789

A00641-01155
UV-shielding ability of the sample changed depending on the microstructure, comfort when applied on skin and the sample prepared by the sol-gel method showed superior UV-shielding performance.

NaOH aqueous solution and 0.8 M CeCl$_3$-0.2 M CaCl$_2$ mixed aqueous solution were simultaneously dropped, followed by adding desired amount of 2 M H$_2$O$_2$ solution to oxidize Ce$^{3+}$ to Ce$^{4+}$, where the solution was stirred and pH was kept at desired values through the reaction. Sol-gel process: after adding glacial acetic acid in Ce(NO$_3$)$_3$·6H$_2$O mixed absolute ethanol solution at 40°C under vigorous stirring with an HAc/(Ce+Ca) molar ratio of 10, desired amounts of plate-like K$_{0.8-1.8}$Ti$_{1.7-1.3}$O$_4$ micro particles dispersed in absolute ethanol were dropwise added, then, the solution was heated at 60°C for ca. 2 h to get gel-like substance, followed by drying in a vacuum oven at 333 K for 5 h and calcination at 1073 K for 2 h. The microstructure, comfort when applied on skin and UV-shielding ability of the sample changed depending on the solution pH, ratio of K$_{0.8-1.8}$Ti$_{1.7-1.3}$O$_4$/Ce$^{3+}$Ca$_{0.2-0.5}$O$_{4-5}$ etc. and the sample prepared by the sol-gel method showed superior UV-shielding performance.

**A00650-01234**

**Samarium (III) Oxide Pulsed Laser Deposited Thin Films: Morphological, Optical and Electrical Properties**

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Exhibiting high resistivities and high dielectric constants, wide band-gap semiconducting rare earth metal oxides (REO) have recently been extensively investigated for applications including photoelectric devices, electrical switches and optoelectronic devices. This paper reports on the preparation of samarium oxide thin films by pulsed laser deposition (PLD) and radiofrequency plasma discharge assisted PLD (RF-PLD), in oxygen atmosphere without substrate heating. The influence of the deposition parameters (substrate type, oxygen pressure, laser fluence, and RF power) on the structure and morphology of the thin films were studied; the losses, dielectric constant and the leakage currents were measured.

**A00666-01592**

**Synthesis and Luminescence of YNbO$_4$:Eu$^{3+}$ by a Flux Method**

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YNbO$_4$ powders have been recognized as x-ray phosphors that show high energy emissions around 256 nm. In this work, YNbO$_4$ powders were prepared by a flux method, and luminescent properties were investigated by doping Eu$^{3+}$ ions. The stoichiometric mixtures of starting materials were fired under air and N$_2$ atmosphere, respectively using LiCl, H$_2$BO$_3$, and NH$_4$Cl as a flux. A single phase YNbO$_4$ could be achieved with LiCl under N$_2$ atmosphere, while the other flux and air atmosphere caused the formation of a secondary phase Y$_2$NbO$_6$. PL (Photoluminescence) spectra of YNbO$_4$ powders exhibited a blue emission at 412 nm due to niobate self-excitation, while Eu$^{3+}$ doped powders emitted a strong red light at 613 nm under ultraviolet excitation due to the charge transfer from niobate group to Eu$^{3+}$ ions. The increase of a flux content contributed to the increase of particle sizes, leading to the enhancement of the emission intensity at 1 – 7 wt% LiCl, but at 10 wt% the particle size significantly enlarged, resulting in the abrupt drop of PL intensity.

**A00661-01195**

**Abrasion Behavior of Al2021 Alloys Treated by Plasma Electrolytic Oxidation in Different Electrolytes**

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Al2021 is an aluminum alloy, with copper and magnesium as the alloying elements, which is used in applications requiring high strength to weight ratio, as well as good fatigue resistance. Due to poor corrosion resistance, it is often clad with Zn for protection, although this will reduce the fatigue strength. Plasma electrolytic oxidation has been proved to be an effective surface protection technique without harm to its fatigue strength, which could significantly increase the wear resistance of Al alloys. However, the abrasion resistance of Al2021 alloy PEO-treated in different electrolytes was seldom reported. In this work, Al2021 alloys were prepared by PEO method in different electrolytes, such as aluminate, silicate and phosphate, under a hybrid voltage of AC 200V (50Hz) combined with DC 260V value. Moreover, the process was controlled in a short time span of 5-15 min. A combined composition and structure analysis of surface and cross section layer was carried out by XRD, OM, and SEM&EDS for the PEO-treated specimens. The abrasion behavior was tested by dry ball on disk wear tests were carried out by a PD-102 wear test machine. The results showed that Double-layer and pancake structural alumina coatings grew on Al2021 alloy surfaces, the phases of which were consisted of α- and γ-Al$_2$O$_3$. It is possible to generate high property ceramic coatings on Al2021 alloys in the condition of low voltage within short reaction times in all electrolytes.

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The Deposition and the Photoluminescence of SrAl\(_2\)O\(_4\):Eu\(^{2+}\) Thin Films

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M-Al\(_2\)O\(_4\):Eu\(^{2+}\) (M: alkaline earth metals) powders are well-known phosphors for lighting lamps. For example, emission spectra of SrAl\(_2\)O\(_4\):Eu\(^{2+}\) powders show a green emission under near UV excitation. In this work SrAl\(_2\)O\(_4\):Eu\(^{2+}\) thin films were prepared on c-plane sapphire substrates by rf magnetron sputtering. The effects of oxygen partial pressure in the sputtering gas, rf power, and the substrate temperatures on the structure and luminescent properties were investigated. O\(_2\) / (Ar+O\(_2\)) ratios varied in the range of 10 - 50 % at the constant working pressure of 5 mTorr. As-deposited thin films were amorphous and non-radiative, but they were recrystallized and radiative after post-annealing in the electric tube furnace under 5 % H\(_2\) (95 % N\(_2\)) atmosphere. Textured films could be achieved by annealing, of which PL spectra exhibited a green emission around 530 nm under 340 nm excitation.

Optical Emission and Absorption Spectra of Zn Embedded ZnO Nanostructure and Zn-ZnO Core-shell Nanostructure

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The Zn embedded ZnO nanostructure thin films were prepared using a cost-effective novel vacuum-carbon-arc combined with vacuum annealing technique. The photoluminescence spectra of the samples exhibit a strong near-band-edge emission around 377 nm and weak defect band in visible range accompanying with some multiphonon peaks at higher energy. The shift of optical absorption band arises due to potential fluctuation of the carrier density. The core-shell Zn-ZnO nanostructures were fabricated from carbon-encapsulated Zn-nanoparticles in thin-films form by an inexpensive vacuum arc technique followed by laser ablation. The emission characteristics of laser ablated (with different powers) samples showed a strong exciton peak at 388 nm, and a few weak peaks due to weak defect states in the visible range. Optical absorption spectra evolved from the excitonic peaks (from 344 nm to 317 nm) with decreasing the laser power. These peaks occur due to coupling between exciton absorption (from ZnO shell layer) and core metal interband absorption.

Nanostructure MgO Formation on Si Substrate by 256 nm YAG Laser Deposition

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None of functioning oxide materials usually grow on silicon (Si) substrates due to interfusion, chemical reactions or in the case of epitaxial growth of oxide films lattice mismatching between those functioning oxides and silicon substrates. Buffer layers is one of choice to grow such oxides epitaxially on Si substrates. A variety of oxide films have been explored as candidates for the buffer layer, and among those candidates, magnesium oxide (MgO) is one of most popular substrates to grow functional oxide films. Epitaxial MgO(001) film is prepared on Si(001) substrate using pulsed laser deposition (PLD), sputtering method or molecular beam epitaxy (MBE) even with a large mismatching between the MgO and Si Substrate.

Despite lattice mismatching of ~10% between MgO[110] and Si[100] (45° rotation), MgO film epitaxially grows with cubic on cubic relation with the mismatching of more than 25%. Epitaxial growth with large lattice mismatching is often explained by domain mismatching, which consists of several unit cells rather than one unit cell defining lattice mismatching; the domain of (m x n) lattices in film grow on domain of (k x l) lattices in substrate as Coincident Site Lattices (CSL), and the domain mismatching is defined as difference of domain size rather than lattice size defining lattice mismatching. The CSL is usually shown on a table with some of combinations of (k x l) and (m x n). To visualize all the combinations of k, l, m and n, the domain mismatch was expressed on polar coordinates as a “polar CSL figure”, which can well describe the epitaxial MgO film with cubic on cubic growth. The lattice constants of MgO film was contracted in deposition of high oxygen atmosphere, and the contraction of lattice increased the domain matching for cubic on cubic growth. Interestingly deposition in higher oxygen atmosphere grow literally cubic structure, and cubic size was estimated less than 100 nm by Atomic Force Microscopy (AFM). After a post-annealing the samples, the crystallinity showed broad in-plane symmetry on an X-ray pole figure. To investigate the effect of defect on crystal structure, a method of ab initio was employed with varying oxygen content in the crystal. The code used in this study was the abinit project [URL http://www.abinit.org].
ZnO quantum dots are promising candidates for high efficiency UV light emitting applications. Due to quantum confinement effect, the exciton binding energy is increased while exciton-longitudinal-optical phonon coupling strength is reduced. Besides, exciton radiative lifetime is shortened due to exciton superradiance effect. Zinc acetate is a convenient metal organic source that it has been widely used in the deposition ZnO thin films and nanostructures. ZnO thin films or nanostructures prepared by sublimation of zinc acetate usually exhibit weak UV emission or multiple UV emission peaks, and strong defect related deep level emission. Improvement of the optical properties of ZnO is essential toward the realization of ZnO QD based light emitting devices.

We have prepared ZnO by sublimation of zinc acetate in a vertical cold wall reactor using oxygen and water as the source of oxygen. \(0.5 \text{ g of zinc acetate dihydrate (Zn(CH_3COO)_2} \cdot \text{2H}_2\text{O), Aldrich, 99.999\%)}\) was positioned in a quartz crucible at 90 mm downstream of a Si substrate. Zinc acetate dihydrate was first baked at 120°C for thirty minutes to remove water. The crucible was subsequently raised to 180°C while the Si substrate was maintained at 400°C. Water or oxygen was released into the reactor via a mass flow controller to maintain a growth pressure of 100 mTorr. A shutter was positioned between the crucible and the substrate to control deposition time.

ZnO deposited in oxygen ambient exhibit a weak UV emission band centered at 3.444 eV and a strong visible emission band centered at 2.254 eV at room temperature. However, ZnO deposited in water ambient exhibit a strong UV emission band also centered at 3.444 eV and negligible defect related visible emission. Low temperature 10 K PL emission of ZnO deposited in water ambient show UV band tail emission up to 3.594 eV, which is higher than free exciton emission of bulk ZnO at 3.377 eV. This indicates that the UV emission band is due to quantum confinement effect and confirming the formation of ZnO QDs. The free exciton energy difference of ZnO QDs deposited in water ambient between 10 and 300 K is 25 meV, suggesting reduced exciton-longitudinal-optical phonon coupling strength. Raman spectroscopy analysis results indicate that the improved PL quality of ZnO QDs deposited in water ambient is related to reduced carbon contamination. Analysis results using FTIR and TEM will be presented.

Bismuth ferrite (BiFeO\(_3\)) is a prototype multiferroic material with high ferroelectric Curie temperature (1100K) and antiferromagnetic Neel (630K) temperature. The smart ferroelectromagnetic bismuth layered materials of Aurivillius family can be derived by combining Bi\(_6\)La\(_3\)Ti\(_3\)O\(_{27}\) with \(n\) mole \((n = \text{integer})\) of BiFeO\(_3\). In view of this, we have synthesized and studied the electrical properties of an 8 layered complex Aurivillius oxide, Bi\(_6\)La\(_3\)Fe\(_3\)Ti\(_3\)O\(_{27}\). The compound was prepared by a high-temperature solid-state reaction technique. The phase formation and microstructure of the sample were examined by X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The electrical properties of the compound were studied by complex impedance spectroscopy. The impedance spectroscopy data of the compounds provide the contribution of both grain and grain boundary on the electrical properties of the materials. A general feature of the impedance pattern suggests a decrease in bulk resistance with rise in temperature. Negative temperature coefficient of resistance (NTCR) behavior of the compound exhibits that Bi\(_6\)La\(_3\)Fe\(_3\)Ti\(_3\)O\(_{27}\) has semiconducting nature. The bulk response, in terms of localized, i.e., defect relaxation or non-localized conduction, i.e., ionic or electronic conductivity was determined by the impedance and modulus spectral analysis. The combined analysis of impedance and modulus spectroscopy explains about the contribution to the relaxation process of different microregions in the polycrystalline ceramics, such as grains, grain boundaries, and surface-electrode interfaces. The appreciable deviation of the activation energy calculated from \(Z''\) and \(M''\) plots indicated both localized and non-localized conduction in Bi\(_6\)La\(_3\)Fe\(_3\)Ti\(_3\)O\(_{27}\).
specific properties, increasing knowledge of fabrication processes gives a better understanding of materials behavior. The development of the enormous potential of functional ceramics based on unique dielectric, ferroelectric, piezoelectric, pyroelectric, ferromagnetic, magnetoresistive, superconducting, and electro-optical properties etc have been going on for several decades. Various attempts have been made to increase the knowledge of economical and ecological production processes for materials, components, devices, miniaturization and integration, and modeling with numerical simulation. A combined approach of different functional properties in a single material has become the subject of our research program, which focuses on the multifunctional behavior of ceramics. In order to achieve multifunctionality in the ceramic materials, we have synthesized and characterized (structural, microstructural, electrical and magnetic) some Pb/Bi based multiferroic materials like Pb(Fe\textsubscript{0.5}Nb\textsubscript{0.5})O\textsubscript{3} (PFN), BiFeO\textsubscript{3} (BFO) etc. However, for device application, we need stable properties in a single phase with large magnetoelectric coupling coefficient, low leakage current at room temperature. On modifying the structure of PFN and BFO by suitable substitution at different atomic sites, many interesting dielectric and magnetic results were obtained. In addition to this, some composites with BFO and PFN have been synthesized and characterized to induce the multifunctionality in these materials. In this lecture, the design, development or research activities of some Pb/Bi based functional materials and their possible device applications will be discussed.

**Synthesis and Structural Characteristics of PbZr\textsubscript{x}Ti\textsubscript{1-x}O\textsubscript{3} Ceramics**

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Polycrystalline samples of PbZr\textsubscript{x}Ti\textsubscript{1-x}O\textsubscript{3} [referred as PZT] where \( x = 0.0, 0.2, 0.4, 0.6, 0.8 \) and 1.0 have been synthesized by a high-temperatures solid-state reaction technique. One of the objective of this work was to optimize carefully the process variables which influence the structural and microstructural properties of the samples prepared. Structural phase evolution studies of PbZr\textsubscript{x}Ti\textsubscript{1-x}O\textsubscript{3} (PZT) system have been carried out as function of Zr/Ti ratio using room temperature X-ray powder diffraction studies. The analysis of the X-ray diffraction patterns for the sample with 0.0 \( \leq x \leq 0.4 \) confirmed the tetragonal phase formation and no impurity peaks were observed. With increase in Zr content i.e. for \( x = 0.6 \) and 0.8 the observed diffraction peaks indicate the presence of rhombohedral phase in this composition range. With further increase in Zr content i.e. \( x = 1.0 \) the observed diffraction pattern confirmed the presence of single phase orthorhombic structure. The average linear particle sizes for all the compositions estimated using Scherrer’s formula lie in the range 400-500 nm. Microstructural studies for all the samples have been carried out using SEM. The SEM analysis of the samples also showed nearly uniform grain distribution i.e. uniform microstructure with no abnormal grain growth. The values of average grain size were obtained using mean intercept method and the value of grain size lie between 1-2 \( \mu \text{m} \). Vibrational spectroscopy data obtained in the wavenumber region 350 to 800 cm\(^{-1}\) for the samples showed characteristic peaks corresponding to the vibrational modes of perovskite structure and a shift in the frequency of the peaks towards the lowered side with the increase of Zr content has been observed.

**Upgrading the Efficiency and Reliability of Piezoelectric Sensors Made of Ultradispersion (nano-) Powders of ZTL-19 Piezoceramics Produced by Mechanoactivation**

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Many instruments and devices of various purposes are based on implementation of the piezoelectric effect created by piezoceramic converters. Small sizes of piezoceramic elements make it possible to solve successfully the tasks of miniaturization of electronic components. Piezoceramics on the basis of solid solutions of a zirconate – titanate of lead (ZTL) are widely used lately.

It is found out that piezoceramics is a structurally sensitive material – with the reduction of the grain size the piezoelectric properties improve. To reduce the sizes of the grain in the finished ceramics ultra dispersion powders of piezoceramic materials are necessary. Such powders can be made by mechanoactivation of commercial powders of ZTL piezoceramics in mechanical activators - planetary vapart mills (PVM).

The drawback of mechanoactivation is a considerable amount of impurities in the powder being activated from the material of the grinding garniture. Piezoceramics is a material, which is very sensitive to the metal admixtures, that is why implementation of metal grinding garniture here is completely prohibited. There were worked out technological techniques that allow avoiding impurities during mechanoactivation process for the commercial ZTL-19 powder in «Pulverisette 6» planetary vapart mill. A technology to make piezoelectric cells using mechanoactivated commercial ZTL-19 powder was worked out; basic properties of piezoelectric cells were determined.
It was found out that implementation of mechanoactivation made it possible to reduce the grain size in piezoceramics by 19%, and enlarge $d_{31}$ piezomodules by 20.7%, and $d_{33}$ by 44.4%, it also allowed increasing the limit of the working temperature of piezoceramics by 50.4°C. The strength of piezoceramics under compression has grown by 30%.

The results produced show that implementation of mechanoactivation makes it possible to upgrade considerably the piezoelectric properties and the strength of ZTL-19 material, and that will have a positive effect on the reliability of piezoelectric cells and electronic equipment where they are used. The method of mechanoactivation can be also used for other types of piezoceramic materials as it allows producing low-porosity ceramics with a fine uniform grain.

A00728-01291

Hydrothermal Synthesis of Alumina Microfibers with Mesoporous Structures and its Adsorbing Ability

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The abatement of nitrogen oxides generated by diesel engines is a matter of both scientific and technological. Since Iwamoto and Hamada shown independently that hydrocarbon could be used as reducing agents to reduce NOx in oxidizing atmosphere, many possible catalyst formulations have been extensively studied to find suitable catalysts and reductants, which are effective for the selective catalytic reduction (SCR) of NOx in the exhaust gases from numerous applications, working under oxygen excess and lower temperatures. Although the NOx reduction efficiency by means of SCR technology depends on both catalysts and reductant agents, the properties of the catalysts support, which gives the shape and most of the mechanical resistance properties and provides high surface area, and the adsorption efficiency between the reductant agents and catalysts support is also important.

In this paper, the alumina microfibers with mesoporous structures were prepared by using the surfactant directed hydrothermal method and then the thermal decomposition of the as-synthesized precursor at 773K. The experimental results show that the morphology of the obtained alumina microfibers with mesoporous structures is the same as as-synthesized precursor after thermal decomposition, the length and diameter is about 10μm and 300-500nm respectively. These alumina microfibers have a high surface area more than 350 m²/g, and the SCR experiment results show that these alumina microfibers have stronger absorbing ability to NH₃, which is used as the reductant agent to reduce NO in oxidizing atmosphere, compared with the commercial alumina materials.

TiO₂ is an ideal photocatalyst due to their chemical stability, nontoxicity, and high photocatalytic reaction decomposing various toxic gases and oxidizing organic compounds such as harmful dioxins into harmless compounds under the UV-irradiation. The addition or doping of small amounts of noble (Pt, Rd, Ag, Au, etc.) and transition metals (V, Cr, Mn, Fe, Ni, etc.) enhanced the photocatalytic reactivity of TiO₂. Also, the coupling of TiO₂ with other inorganic oxides such as SiO₂, SnO₂, WO₃, In₂O₃, (Sr,La)TiO₃, and ZnFe₂O₄ can change the photocatalytic efficiency and the energy range of photo-excitation. This study has investigated on the photocatalytic behavior of the TiO₂-coated phosphorescent materials. Nanocrystalline titanium dioxide layers were directly deposited on the alkaline earth aluminate phosphors of CaAl₂O₄:Eu²⁺,Nd³⁺ and SrAl₂O₄:Eu²⁺,Dy³⁺ by a sol-gel processing and chemical vapor deposition (CVD) and their photocatalytic behaviors were examined with the degradation of methylene blue (MB) aqueous solution under UV and visible light irradiations. TiO₂-coated phosphorescent materials showed different photocatalytic mechanism, compared with pure TiO₂. At UV-irradiation, TiO₂-coated phosphorescent materials showed slow photocatalytic reactivity in the early stage and fast in the latter, compared with that of pure TiO₂. However, at visible light irradiation TiO₂-coated phosphorescent materials showed much faster photocatalytic reactivity under visible irradiation than that of pure TiO₂, which is almost negligible. The mechanism of photocatalytic reactivity for the hetero-junction of TiO₂-phosphors will be discussed in points of energy band structure and phosphorescence. In addition, the characterization of the TiO₂-coated phosphor composites was done by X-ray diffractometer (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM) and energy dispersive spectroscopy (EDS).

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Photocatalytic Behavior for the Composite of TiO₂-Coated Phosphorescent Material

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In this work, we report on the effects of contact resistance in a-IGZO thin film transistor with ZrO$_2$ gate dielectric. The a-IGZO TFTs have been fabricated with various channel widths at a fixed channel length. In the real a-IGZO TFTs, source/drain contact on a-IGZO channel is non-ideal ohmic contact and source/drain contact resistance ($R_{\text{SD}}$) exists which is varied by changing the contact area. The contact area of source/drain electrode on a-IGZO channel with $W/L=450 \mu m$, $300 \mu m$ and $150 \mu m$ is relatively small compared with $W/L=450 \mu m/30 \mu m$. The channel widths of $450$, $300$ and $150 \mu m$, respectively. The reduction of on-current for narrow channel width is mainly due to the increase of $R_{\text{SD}}$. The field effect mobility is calculated 61.44, 64.27 and 79.64 cm$^2$/V$\cdot$m/s respectively. As the channel widths decrease, the estimated field effect mobility increases. Because, in the field effect mobility equation, the field effect mobility is inversely proportional to channel width. For a high on-current, it is necessary to increase the channel width, whereas for high field effect mobility, it is necessary to decrease the channel width. These results show that on-current and field effect mobility have trade-off and very important for the consideration of the applications.

Recently, the multiferroic materials, in which the ferroelectric (FE) and magnetic orders coexist and are intimately coupled, have attracted much attention due to their technological relevance and fundamental science challenges. Among the single phase multiferroic materials, the undoped ABO$_3$-type manganites with small size R (rare-earth) cations (like Tb, Dy, Ho and so on) are one of the most fascinating family. The physics and materials science issues of multiferroics manganites are twofold interesting: 1) various mechanisms are involved; 2) these mechanisms can be applicable to other analog system.

In this report, we overview our recent work on the origin of multiferroicity in rare-earth manganites of spiral-spin order using Monte Carlo technique. We address two substantial issues: one is how the microscopic Hamiltonians with the Dzyaloshinskii-Moriya (DM) interaction can explain related experiments in the quantititative sense, and the other is what is the microscopic origin of spiral-spin order in those ABO$_3$-type rare-earth manganites. The two issues allow us to have a comprehensive estimation of the role of DM interaction in determining the ferroelectricity, and a comprehensive understanding of the multifold interactions which eventually have impact on the magnitude of ferroelectric polarization.

In this work, we report on the effects of contact resistance in a-IGZO thin film transistor with ZrO$_2$ gate dielectric. The a-IGZO TFTs have been fabricated with various channel widths at a fixed channel length. In the real a-IGZO TFTs, source/drain contact on a-IGZO channel is non-ideal ohmic contact and source/drain contact resistance ($R_{\text{SD}}$) exists which is varied by changing the contact area. The contact area of source/drain electrode on a-IGZO channel with $W/L=450 \mu m$, $300 \mu m$ and $150 \mu m$ is relatively small compared with $W/L=450 \mu m/30 \mu m$. The on-currents of a-IGZO TFTs are 5.12, 3.49 and 2.02 mA at the channel widths of 450, 300 and 150 $\mu m$, respectively. The reduction of on-current for narrow channel width is mainly due to the increase of $R_{\text{SD}}$. The field effect mobility is calculated 61.44, 64.27 and 79.64 cm$^2$/V$\cdot$m/s respectively. As the channel widths decrease, the estimated field effect mobility increases. Because, in the field effect mobility equation, the field effect mobility is inversely proportional to channel width. For a high on-current, it is necessary to increase the channel width, whereas for high field effect mobility, it is necessary to decrease the channel width. These results show that on-current and field effect mobility have trade-off and very important for the consideration of the applications.
during synthesis resulted in a highly porous structure. The effect of MWCNT addition on sensor response towards ammonia and nitrogen dioxide gas was investigated. A high surface area and small crystallites present in the sol gel synthesized W-SnO$_2$ films were attributed to this high sensitivity. Low concentrations of NO$_2$ could be detected even at room temperature using these composite films which was not possible without CNT addition.

A00812-01649
Synthesis and Characterization of Piezo Ceramics Based Materials for Sensor Applications
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Among many techniques used for evaluation and testing, ultrasonic technique (UT) has gained much importance as it does not have any environmental or health risk and can be designed to test complex geometries. Ferroelectric materials are the material of choice and interest for UT as they exhibit highest piezoelectric constant and electromechanical coupling factors necessary to achieve large force and displacement efficiently. Among all piezoceramics, lead zirconate titanate (PZT) based materials have played an important role till date but it has poor figure of merit and efficiency in lower frequency range, which is required for applications in specific device applications such as sonar and hydrophone. This problem has been overcome by the advent of piezocomposites. They are considered to be important as they show low acoustic impedance, better compliance and echo mode. Few groups have already studied the properties of PZT-PVDF, PZT-PVA, PZT-PVDF/TrFe systems. Research activities are going on globally but optimization at the required level and understanding is yet not achieved. Pioneer work has been done by R.E Newhmann on polymer-PZT composite. Although much work has been focused on PZT ceramics but recently solid solution of lead magnesium niobate and lead titanate (PbMg$_{0.33}$Nb$_{0.67}$O$_3$-PbTiO$_3$) abbreviated as PMN-PT has gained attention as they the oxygen vacancies present in the perovskite structure of the ceramic phase has played a dominant role.

A00820-01428
Crystal Structure and Electrical Property of Ferroelectric Thin Film Deposited under Magnetic Field by Pulsed Laser Deposition
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Pulsed laser deposition (PLD) is widely used as one of promising method to prepare functional ceramic thin films. In PLD, plume, i.e., a kind of plasma generates by the irradiation of focused laser beam on the target. We developed a unique PLD apparatus that in-situ magnetic field can be applied for the plume. Application of magnetic field during deposition brings about electron temperature in the plasma and may enhance nonequilibrium state.

We applied this technique for the deposition of ferroelectric materials such as Pb(Zr,Ti)O$_3$ (PZT), BaTiO$_3$ (BT) and SrTiO$_3$ (ST). PZT thin film deposited at 400°C without application of magnetic field showed low remanent polarization of 12.7 $\mu$C/cm$^2$. On the other hand, the film deposited under magnetic field of 2kG showed high remanent polarization of 30.5 $\mu$C/cm$^2$.

ST is known as a quantum paraelectrics and it does not show ferroelectric property. The film deposited without application of magnetic field showed paraelectric properties. On the other hand the film, deposited under magnetic field of 2kG showed ferroelectricity. However, opposite phenomenon was observed in case of BT. The ferroelectricity of BT was decreased by the application of magnetic field during deposition. These results suggest
that the property of dielectric thin film can be modified by the application of magnetic field. The reason why such phenomenon was brought about was discussed.

A00822-01438

Growth and Characterization of ZnO Thin Films Prepared by Reactive Ion Beam Sputtering Deposition with External Electric field

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ZnO is a promising semiconductor material that has found various applications in UV light emitting, surface acoustic wave (SAW) and transparent conducting thin film applications. Ion beam sputtering deposition (IBSD) is a convenient deposition method that large area ZnO thin film with atomically flat terrace surface quality can be achieved which make it suitable for the fabrication of SAW and quantum well devices. Capillaritron is a versatile and economic ion source that does not require sophisticated power supply systems. Argon and oxygen can be passed through the same capillary nozzle to produce ions and atoms for sputtering and reactive deposition, respectively. Due to the asymmetric structure of wurzite ZnO, a strong electric field exists along the c-axis so that the polarity of ZnO can be controlled via external electric field. ZnO deposited by capillaritron IBSD with externally applied electric field may produce high quality ZnO thin films at reduced temperatures.

We have deposited ZnO thin films on Si (100) substrate at 300°C by reactive IBSD utilizing a capillaritron ion source. A metallic zinc target (99.99%) was positioned at 30 mm downstream of the capillaritron ion source. A Si substrate was positioned at 45 mm away from the target. The relative angle of the Si substrate and the target were adjusted so that the deposition rate is optimized. The base pressure and growth pressure were $3\times10^{-6}$ and $2\times10^{-3}$ Torr, respectively. ZnO thin films were deposited with a capillaritron anode voltage of 10 kV and an argon/oxygen ratio of 3:2. Films deposited under these conditions show lowest integrated photoluminescence intensity ratio of defect related deep level emission to that of near band edge emission, while the film is almost strain free (-0.3×10^10 dyne/cm^2) and the grain size reaches 10 mm. The crystalline quality of the ZnO film is greatly improved as a DC bias voltage of $+20$ V is applied to the substrate. Under identical deposition conditions and the applied field, the grain size increases to ~16 nm and the film exhibits a columnar structure. No improvement was observed as a negative bias of -20 V is applied to the substrate. These results indicate that the substrate biased with positive voltage can enhance the polarity along the c-axis to improve the crystalline quality of the film. Micro Raman spectroscopy analysis shows strong $E_{2g}$ (high) mode of ZnO at 436 cm$^{-1}$, indicating high quality wurzite ZnO structure. XPS analysis shows that the film is close to stoichiometry, while 75 % of oxygen ions are located at fully oxidized ZnO matrixes.

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A00825-01435

Solution-based Synthesis of Functional ZnO-based Radial Heterostructure Nanowires

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Radial core/shell nanowires (NWs) consisting of two important functional materials represent an important class of one-dimensional (1D) nanostructures for developing potential nanoelectronic and optoelectronic devices with enhanced performances. The one or more shells can passivate surface states, enable new interface properties, and create unique electronic and photonic functionalities. As a wide direct band gap semiconductor material, ZnO has been studied extensively due to its promising applications in many technologically important areas. Although there has been notable progress in preparation for ZnO 1D homogeneous system, to form desired 1D heterostructures with well-defined interfaces using facile and controllable approaches is still an enormous challenge.

Herein, we report an effective solution-based chemical-bath deposition (CBD) route to prepare ZnO-based radial core/shell heterostructured NWs. The heterostructures can be obtained in two steps comprising nanowire preparation, solution-based coating and post-synthesis calcinations. Compared with vapor-based physical routes, this synthesis approach is a low temperature process ($\leq100^\circ$C), and can realize large scale uniform coating on NWs. We have successfully coated several transition metal oxides (like NiO, Co$_3$O$_4$) and MgO on ZnO NWs to produce core/shell heterostructures. The shell thicknesses along whole nanowires (>30 μm) are uniform and can be easily controlled by reaction solution concentrations. After HCl vapor etching or post-heat treatment by 10% H$_2$, MgO or NiO nanotubes were obtained, and the shells outside ZnO NWs are homogeneous. Transition metals or Mg doping into ZnO lattice introduced by subsequent annealing core/shell heterostructures can help to modulate their electrical, optical, and magnetic properties.
Nitrogen Doped ZnO Prepared by Capillaritron Ion Beam Sputtering Deposition

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ZnO is a wide band gap semiconductor material that has found various applications in UV light emitting, surface acoustic wave (SAW) devices and chemical sensing. Due to intrinsic defects (oxygen vacancy and zinc interstitial) and impurities, the development of p-type ZnO is hindered by low reproducibility, low hole mobility and low hole concentration. Nitrogen is one of the most promising candidates as a shallow acceptor in ZnO. The ionic radius of nitrogen is close to that of oxygen, besides, unlike P or As, nitrogen does not form Zn antisite defect. Ion beam sputtering deposition (IBSD) is a convenient deposition method that large area ZnO thin film with atomically flat terrace surface quality can be achieved which make it suitable for the fabrication of SAW and quantum well devices. Capillaritron is a versatile and economic ion source that does not require sophisticated power supply systems. Argon and nitrogen can be passed through the same capillary nozzle to produce ions and atoms for sputtering and doping, respectively. By using high purity gases, the presence of carbon and hydrogen can be controlled. Controlling these impurity concentrations is essential for the production of high quality p-type ZnO since both hydrogen and C-N complex are known to form shallow donors in ZnO.

We have deposited nitrogen doped ZnO thin films by capillaritron IBSD at 300°C on Si substrates. A ZnO target (99.99%) was positioned at 30 mm downstream of the capillaritron ion source. A Si substrate was positioned at 45 mm away from the target. The relative angle of the Si substrate and the target were adjusted so that the deposition rate is optimized. The base pressure and growth pressure were 3×10⁻⁶ and 1×10⁻³ Torr, respectively. Nitrogen doped ZnO thin films were deposited with a capillaritron anode voltage of 10 kV and an argon/nitrogen ratio of 8:0.1 and 8:5. The as-deposited nitrogen doped ZnO thin film exhibits a smooth surface with a root-mean-square roughness less than 3 nm. Micro Raman spectroscopy analysis of the as-deposited film shows strong nitrogen related local vibration mode at 275, 582 and 640 cm⁻¹, while the E₁ mode of ZnO at 436 cm⁻¹ can barely be identified. Annealing at 500 ~ 800°C causes decrease of 275, 582 and 640 cm⁻¹ and increase of 436 cm⁻¹ intensity, indicating out diffusion of nitrogen and improvement of ZnO crystalline quality. Unlike undoped samples, the surface roughness of nitrogen doped ZnO deteriorates after annealing, which is also attributed to the out diffusion of nitrogen. Low temperature photoluminescence and Hall Effect measurement results will be presented.

This research work was supported by the National Science Council of Republic of China under contract number NSC 97-2112-M-011-002.
In the present study we report the formation of inorganic/organic heterojunction by depositing p-type polymer PEDOT:PSS on n-type ZnO films.

ZnO and Al-doped (3wt%) ZnO films have been deposited on patterned ITO coated glass substrate at 450°C. The as deposited pure ZnO film is annealed in vacuum (≈4×10⁻⁴ torr) at 450°C using ultrasonically assisted chemical vapor deposition technique. Both the films have been characterized by x-ray diffraction, photoluminescence, optical absorption and atomic force microscopy. The p-type polymer PEDOT:PSS has been spin coated (2000 rpm, 60 s) on vacuum annealed ZnO and Al-doped (3wt%) ZnO films. The film was then slowly heated at 80°C for one hour and then left over night to cool gradually to avoid craks. Gold was deposited on top of the PEDOT:PSS by thermal evaporation technique to promote the anatase crystallinity for a more in-depth discussion. The current-voltage (I-V) characteristics of both heterojunctions: vacuum annealed ZnO/PEDOT:PSS and Al-doped ZnO/PEDOT:PSS showed diode like behavior. The I-V characteristics of both the devices have been examined in the frame work of thermionic emission model. A comparative studies of the formation of interfaces of PEDOT:PSS with vacuum annealed ZnO and Al-doped ZnO have been carried out.

Materials with zero band gap, namely gapless semiconductors, have unique physical properties compared to conventional semiconductors, insulators and metals. Their band structures are extremely sensitive to external influences such temperature, pressure, electric or magnetic field. An overview on the physical properties will be given for some typical zero gap materials with either quadratic or linear dispersion between energy and momentum. A concept of the spin gapless semiconductor in which both electron and hole can be fully spin polarized with tuneable carrier density is proposed, and its possibility is presented on the basis of first-principles electronic structure calculations. A new class of gapless materials in which the spin polarization can be easily achieved will be also presented. Possible new physics and potential applications in spintronics, electronics and optics based on the spin gapless semiconductors and the new class of zero gap materials will be addressed. Some experimental data on the observation of colossal electromagnetoresistance and giant magnetoresistance in some zero gap materials will be presented in this talk.

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### Investigation of the Super-hydrophilic Property and the Self-cleaning Function of Amorphous and Anatase Titanium Dioxide (TiO₂)

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Self-cleaning glasses can be produced by utilizing the super-hydrophilic property and the oxidation ability of titanium dioxide (TiO₂) coatings. This project aims to compare the super-hydrophilic property and also the self-cleaning function of two types of TiO₂: amorphous and anatase. Some amorphous TiO₂-coated samples were annealed in oven at 450°C to promote the anatase crystallinity for a more in-depth discussion. The effect of the different thicknesses of the TiO₂ coatings was also investigated.

Spray pyrolysis deposition (SPD method) was used to deposit water based TiO₂ onto glass substrates and silicon wafers. All samples were located strategically outdoor for 6 months under rainfall and sunlight exposure.
Measurements and characterization of samples were done periodically every 3 months using water contact angle goniometer, haze gard plus system, X-ray Diffraction (XRD) and Atomic Force Microscope (AFM).

The results show that contact angles close to super-hydrophilic range were successfully achieved on anatase coatings throughout 6 months while high contact angles were observed on amorphous coatings. It is also found out that increasing the thickness of coatings enhances the super-hydrophilic property of TiO$_2$ coating.

**A00871-02707**

**Superconductivity, Critical Current and Flux Pinning in LaFeAsO$_1$-$x$F$_x$ Compounds**

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The discovery of the Fe-based new superconductors ReFeAsO with critical temperature ($T_c$) up to 56 K has attracted great interests due to their exotic unconventional superconductivity. Here we report our studies on the crystal structures, morphologies and superconductivity in the LaFeAsO$_1$-$x$F$_x$ compounds which were fabricated by solid state reaction. The crystal structures are refined using Rietveld refinement. Superconducting properties such as $T_c$, critical current density $J_c$ and upper critical field $H_{c2}$ were determined using magneto-transport and magnetic measurement over a wide range of temperature below and above $T_c$, and in magnetic fields up to 14 T. Our results show that the $J_c$ is sensitive to the fluorine concentration. The $J_c$ of the $x=0.15$ are almost three times higher than that of the $x=0.05$ sample. A peak effect in the $J_c$ versus field is observed at $T\leq 15$ K for both samples. The LaFeAsO$_1$-$x$F$_x$ samples exhibit a superior $J_c$ field performance in high field compared to MgB$_2$ superconductors. The $H_{c2}$ of both samples could be as high as 100 T. Flux pinning behaviour is also discussed in terms of thermal activation and mean free path fluctuation pinning models.

**A00872-01519**

**Fabrication of Aluminum-Sheathed Bi$_2$Sr$_2$CaCu$_2$O$_8$, Superconducting Tape**

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Polycrystalline Bi$_2$Sr$_2$CaCu$_2$O$_8$ (Bi-2212) superconductor was synthesized via solid-state reaction method. This Bi-2212 precursor was fabricated into tapes using calendering method. In this technique, the superconducting material is subjected to rolling and annealing in order to promote the connectivity of the Bi-2212 grains. Aluminum was the used cladding material for the superconducting tape. The magnetic susceptibility test showed the superconducting property of the Bi-2212 tape. The critical temperature ($T_c$) of the sample is 76 K. The existence of Bi-2212 in the sample was verified using x-ray diffraction. Scanning electron microscopy showed the microstructure of the fabricated tape.

**A00883-01535**

**Modeling and Simulation of Ultrasonic Piezoelectric Transducers**

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A one-dimensional modeling of piezoelectric transducer was developed and compared with ANSYS simulation. The transducer is a composite sandwich structure with piezoelectric stacks clamped by metal blocks. It has many applications from cleaning, semiconductor to the medical devices. The transducer performs one-dimensional longitudinal vibration in the ultrasonic range. The method of equivalent circuit was used for the modeling. In this method, the piezoelectric stacks and metal blocks were considered to be lossless and represented using a two-port network circuit individually. The circuits were then connected in series. The resonant frequency, mode shape, mechanical quality factor, impedance and vibration velocity of the transducer were solved from the circuit. The results were also compared with the 2D ANSYS finite element simulation and consistent results were obtained.
Mechanical Strength of Nanosize MgO Added Dip-Coated Bi$_2$Sr$_2$CaCu$_2$O$_{8+}$ Superconductor Tape at Cryogenic Temperature

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In our initial study, 3% to 8% weight percentage of nanosize MgO particles was added to Bi$_2$Sr$_2$CaCu$_2$O$_{8+}$ (Bi-2212) high-temperature superconductor in view of fabricating Bi-2212 superconductor elements with superior mechanical strength. The Bi-2212/MgO compounds were palletized and heat treated through partial-melting and slow-cooling processes. The mechanical strength of the samples was studied by conducting the compression test at room temperature, and the addition of 5% MgO particles produced the highest strength when compared with the other samples. The Bi-2212 powder with 5% nanosize MgO addition was used to fabricate Bi-2212 tapes through the dip-coating-then-stacking process. In the process, the tapes were prepared by stacking layers of single side dip-coated silver strips, and then wrapping them with silver foil. The tape samples were rolled and pressed to increase the packing density of the superconductor core, and heat-treated with partial-melting temperature of 865°C for about 6 minutes. At cryogenic-temperature (77 K) and zero fields, the transport critical current density ($J_c$) of more than 10,000 A/cm$^2$ was obtained for each sample. Tensile tests for the tapes at 77 K showed that the Bi-2212 tapes with nanosize MgO addition have significantly higher strength compared with the non-added samples. As such, a strengthening effect was observed in the nanosize MgO added Bi-2212 superconductor tapes.

Reducing Gas Response Kinetics of Nanostructured Indium Oxide Thin Films

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Indium oxide is a wide band gap n-type semiconductor. It has been widely exploited as an ultra-sensitive gas sensor for the detection of variety of gases. In our laboratory, we have synthesized nanostructured, semiconducting, transparent In$_2$O$_3$ thin films on glass substrates using indium chloride as precursor. Scanning electron microscopic image of the thin film showed that the surface of the prepared sample was found to contain nano-sized sphere-like grains. The structure of the prepared sample was body centered cubic and its average particle size is found to be 12 nm (from the XRD pattern using Scherrer’s formula). In$_2$O$_3$ thin films also showed nearly 90% of transparency to visible and ultraviolet lights. Gas sensing property of nanostructured In$_2$O$_3$ thin films was tested by depositing aluminium electrodes over the surface and observing changes in the resistance of the material to the incoming ammonia and ethanol vapours. It was found that the nanostructured In$_2$O$_3$ thin films show a good response to both of the reducing gases even at ambient conditions. In particular, sensor response for ammonia and ethanol at room temperature for indium oxide thin films was found to be around 1.5 and 1.3 respectively.

Synthesis and Characterizations Studies on Y$_{1−x}$Ag$_x$Ba$_2$Cu$_3$O$_{7−δ}$ Composite High T$_c$ Superconducting

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The preparation, processing and characterization of the composite materials on high T$_c$ superconductor (HTS) basis, YBa$_2$Cu$_3$O$_{7−δ}$ and Y$_{1-x}$Ag$_x$Ba$_2$Cu$_3$O$_{7−δ}$, where $x = (0.10, 0.30, 0.50, 0.70, 0.90)$ and $y = (0.12, 0.36, 0.60, 0.84, 1.09)$ is reported. The initial component YBa$_2$Cu$_3$O$_{7−δ}$ was prepared by the standard solid state reaction technique. The microstructures of the samples were observed by scanning electron microscope (SEM) whilst the XRD patterns of the composite HTS + Ag revealed the Y123 superconducting phase and the Ag structure. The chemical compositions of the powders were found by employing energy dispersive x-ray (EDX).

Preparation and Characterization of Al-doped ZnO by Radio Frequency Magnetron Sputtering

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Aluminum doped Zinc Oxide (AZO) films were deposited on silicon and quartz substrates by radio frequency magnetron sputtering method in an argon/oxygen mixture. The surface morphology was characterized by atomic force microscopy (AFM) and scanning electron microscopy (SEM). The crystallinity was examined by X-ray diffraction (XRD) and the optical properties and electrical properties were examined using UV-VIS spectrometer, Hall measurement and photoluminescence (PL) measurement. Microscopic analysis and composition analysis was investigated by transmission electron microscopy (TEM) and scanning
transmission electron microscopy (STEM). From our AFM results, it is shown that there is dependence on the RF power, whereby at lower power deposition, the root-mean-square roughness of the films is lower. Based on our cross-section TEM, we also observed our AZO layer has lower dislocation defect density. The resistivity of these AZO thin films is measured to be as low as 6.0e x 10^4 ohm cm and its transmittance is measured to be greater than 80% in the visible range.

A00924-01622

Efficient Photocatalytic Hybrid Low-Dimensional TiO$_2$ Nanostructures Utilizing Cooperative Self-Assembly

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Titanium dioxide (TiO$_2$) photocatalysis has been widely utilized in recent years for environmental issues such as photodegradation and complete mineralization of organic pollutants. TiO$_2$ nanostructures have high catalytic performance due to the large specific surface area onto which numerous reactions take place. Along the line of this application, the large band gap (3.2eV) of TiO$_2$ has been recognized as one of the crucial limitations to be resolved since such an inherent property significantly restricts the broad applications of the TiO$_2$ nanostructures under visible light. Thus, the development of TiO$_2$ photocatalyst with visible light activity has attracted much attention over the past several years. As facile attempts to make visible light active photocatalytic nanostructures, we have prepared noble metal/TiO$_2$ quantum dot/TiO$_2$ and Ag decorated SiO$_2$/TiO$_2$ mesoporous materials via self-assembly of block copolymers and sol-gel chemistry.

Ag/TiO$_2$ hybrid nanodot array: Well defined, ordered arrays of hybrid Ag/TiO$_2$ hetero nanodots were fabricated on solid substrates using amphiphilic poly(styrene-block-ethylene oxide) diblock copolymer (PS-b-PEO) micelles loaded with AgNO$_3$ and TiO$_2$ sol-gel precursors as templates. The inorganic precursors were selectively incorporated into PEO domains due to specific chemical affinity. The conversion of AgNO$_3$ to metallic Ag was induced by UV irradiation and confirmed by the presence of surface plasmon band in the UV-Vis absorbance spectra. The organic matrix has been removed by deep UV irradiation, leading to arrays of Ag/TiO$_2$ composite NPs. The morphology and photocatalytic activities of the resulting hybrid nanoparticle (NP) arrays were studied. Markedly enhanced photocatalytic degradation of methylene blue has been observed for Ag/TiO$_2$ nanodot arrays compared with pure TiO$_2$ NP arrays.

CdS/TiO$_2$ nanostructures: We prepared CdS and TiO$_2$ NPs embedded PS-b-PEO block copolymer thin-films on silicon substrate by self-assembly technique. The addition of CdS quantum dot into BCP/TiO$_2$ sol-gel mixture changes the morphology from mixed wire/hexagonal packing to well ordered hexagonal packing arrangement. PS-b-PEO/TiO$_2$/CdS showed enhanced and red shifted absorption in the presence of TiO$_2$. An enhanced photocatalytic degradation of methylene blue has been observed in the hybrid PS-b-PEO/TiO$_2$/CdS thin film.

SiO$_2$/TiO$_2$/Ag mesoporous materials: Titania-silica mixed oxide materials with highly ordered mesoporous structure have been prepared by sol-gel process involving simultaneous hydrolysis of TTIP and TEOS, using P123 as structure directing agent. The structure of the prepared materials was characterized by TEM, XRD, and FTIR. Regular ordered hexagonally packed mesoporous arrangement was confirmed from TEM analysis and no separate TiO$_2$ domains were found in the silica matrix. FTIR analysis reveals the presence of Ti-O-Si and Ti-O-Ti vibration bands, evidencing that TiO$_2$ was incorporated in the framework of SiO$_2$. Further, silica/titania mesoporous materials were decorated with Ag NPs and its enhanced photocatalytic properties are explored.

A00930-01646

Study of Ferroelectric Properties of Spray Pyrolysis Deposited Cesium Nitrate Films

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Cesium nitrate (CsNO$_3$) films were prepared by ultrasonic spray pyrolysis technique at different substrate temperatures (T$_s$) and their structural and ferroelectric properties were examined. The crystal structure of the film was analyzed by X-ray diffraction method. The morphology of the deposited films was studied using FESEM. The ferroelectric properties were optimized based on remanent polarization as a function of substrate temperature. The butterfly curves of capacitance-voltage (C-V) characteristics were observed and attributed to the ferroelectric polarization switching. The switching response was studied using pulse width technique. The switching parameter such as effective dimensionality, switching time, nucleation rate, and domain wall velocity were extracted using finite grain model (FGM). The width of reading pulse shows significant effect on switching transients.

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Energy Transfer and Luminescent Properties of Er\textsuperscript{3+}-doped YBO\textsubscript{3} and (Y,Gd)BO\textsubscript{3} Phosphors

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Recently, Yttrium and lanthanide (Ln) orthoborate phosphors have attracted much attention due to their high stability, low synthesis temperature, high VUV absorption and exceptional optical damage threshold. Among Ln ions, Er\textsuperscript{3+} is one of the most popular and efficient ions to obtain infrared eye-safe emission at 1.5 µm as a result of \(^{4}I_{13/2} \rightarrow ^{4}I_{15/2}\) transition of Er\textsuperscript{3+} ions, which is useful for optical amplification at the third telecommunication window, in addition to the possible NIR to VIS upconversion emission. Also, it has strong absorption bands in the UV–VIS–NIR regions where efficient pumping sources are available.

In our present work, Er\textsuperscript{3+}-doped YBO\textsubscript{3} and (Y,Gd)BO\textsubscript{3} phosphors have been prepared by solid state reaction method with 3 mol % of Er\textsuperscript{3+} and 0, 1, 3, 5 and 7 mol % of Gd\textsuperscript{3+} ions using high purity chemicals of Y\textsubscript{2}O\textsubscript{3}, H\textsubscript{3}BO\textsubscript{3}, Gd\textsubscript{2}O\textsubscript{3} and Er\textsubscript{2}O\textsubscript{3}. The synthesized materials were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), excitation, emission and lifetime measurements. The dependence of various optical, morphological and energy transfer properties of the prepared materials on the Ln ion concentration has been discussed and the results are compared with those reported in earlier literature.

Photoluminescence Properties of Li\textsuperscript{+}-doped Knbo\textsubscript{3}:Eu\textsuperscript{3+} Phosphors

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Research interest on lanthanide (Ln)-doped materials is rapidly growing in the coming new generation due to their potential applications in the fields such as nonlinear optical (NLO) materials that are widely useful in the development of plasma display panels (PDP), field emission displays (FED) and electro-luminescent panels. Potassium niobate (KnBO\textsubscript{3}) is recognized to be one of the best host matrix for Ln ions as it is a useful candidate for ferroelectric materials with excellent electro-optic and nonlinear optical coefficients which have attracted a great amount of interest for applications in optical waveguides, frequency doublers and holographic storage systems. Eu\textsuperscript{3+} is one of the most interesting Ln ions due to its simple lower energy levels scheme as well as applications in the fields such as red emitting phosphor because of its intense, narrow, monochromatic and hypersensitive red emission around 610 nm as a result of \(^{5}D_{0} \rightarrow ^{7}F_{2}\) transition. Li\textsuperscript{+} ion having small ionic radius is found to be one of the best modifier ions as it affects the luminescence properties of various phosphor materials to a better interesting extent.

In our present work, Li\textsuperscript{+}-doped KnBO\textsubscript{3}:Eu\textsuperscript{3+} phosphors have been prepared by solid state reaction method with 0.05, 0.10, 0.15 and 0.20 mol concentration of Li\textsuperscript{+} ions and 0.03 mol % of Eu\textsuperscript{3+} ions using high purity chemicals of Li\textsubscript{2}CO\textsubscript{3}, K\textsubscript{2}CO\textsubscript{3}, Nb\textsubscript{2}O\textsubscript{5} and Eu\textsubscript{2}O\textsubscript{3}. The synthesized materials were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), excitation, emission and lifetime measurements. The dependence of various optical and morphological properties of the prepared materials on Li\textsuperscript{+} ion concentration has been discussed and the results are compared with those reported in earlier literature.
Characterization of Glass-clay Lightweight Aggregates with CaCO$_3$ Coating
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The surface characteristics of artificial lightweight aggregates coated with CaCO$_3$ powder have been investigated. The aggregates made of clay and glass powder were coated with CaCO$_3$ powder by pelletizer. Slagging and fouling indices calculated from various compositions and surface melting temperatures were compared for each artificial lightweight aggregates. The specific gravity, absorption rate, percent of black core area were measured at various sintering temperatures. By observing porosity and microstructure of sintered bodies, it was found that percent of black core area depended on the contents of CaCO$_3$. When artificial lightweight aggregates (clay : glass powder = 1:1) coated with CaCO$_3$ were sintered at 1100°C for 10 min, a new phase between aggregate surface and coating materials seemed to be formed by crystal growth and this phase was further studied by using EDS, SEM, XRD. Consequently, CaCO$_3$ was considered as a governing coating material on aggregates surface melting temperature.

Lead Hydroxide Single-Crystalline Nanorods Structure Precipitates from Lead Nitrate Solution
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In this paper, we report the synthesis of lead hydroxide nanorods by solution-phase reaction of Pb(II) ions and hydroxide ions. The typical precipitation procedure to obtain lead oxide nanorods is as follows. A 50ml of 0.01M lead nitrate aqueous solution precursor (0.166g of lead nitrate hydrate with a purity 99.5% dissolved in 50ml distilled water in a 50ml glass beaker) was heated to about 80°C. When about 0.084g of KOH pellets was added at the same time with vigorously magnetic stirring, yellow or red flocculent precipitate separated out in less than a minute. The precipitate was washed many times (more than 8 times) with distilled water, filtered and dried naturally for overnight. We have characterized the precipitate by employing scanning electron microscopy, transmission electron microscopy, high-resolution transmission electron microscopy, energy dispersive X-ray spectroscopy and XRD.

Our results indicate that, by adding chloride ions into lead nitrates aqueous solution, the lead hydroxide nanorods can be obtained when an alkali is put into the precursor solution. The chloride ions are the key factor for the formation of rod-like morphology. The as-prepared lead hydroxide nanorods are single crystals with hexagonally crystalline structure.

Lead Oxide Plate Precipitates from Lead Nitrate Solution
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In this paper, we report the synthesis of lead oxide plate by solution-phase reaction of Pb(II) ions and hydroxide ions. The typical precipitation procedure to obtain lead oxide nanorods is as follows. A 50ml of 0.01M lead nitrate aqueous solution precursor (0.166g of lead nitrate hydrate with a purity 99.5% dissolved in 50ml distilled water in a 50ml glass beaker) was heated to about 80°C. When about 0.084g of KOH pellets was added at the same time with vigorously magnetic stirring, yellow or red flocculent precipitate separated out in less than a minute. The precipitate was washed many times (more than 8 times) with distilled water, filtered and dried naturally for overnight. We have characterized the precipitate by employing scanning electron microscopy, transmission electron microscopy, high-resolution transmission electron microscopy, energy dispersive X-ray spectroscopy and XRD.

Our results indicate that the yellow precipitates are beta-phase lead oxide and the red precipitates are alpha-phase lead oxide. The crystal structure of the precipitates is dramatically affected by the temperature of the precursor solution and the additive.
A00965-01699

Synthesis of β-PbO Plate Films by Electrochemical Deposition

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It is well known that physical and chemical properties of materials are related with their shapes and structures, especially for micro/nano-materials. Synthesis of various shapes and structures of micro/nano-materials is very interesting and important for various applications. Lead oxide as a metal oxide has various applications in industry and technology, such as a potential application in developing new-style electrode materials. It is reported that the microstructure and morphology of lead oxide have an important affection on its electronic properties.

We developed a simple and effective method to fabricate lead oxide plate by electrochemical deposition. The electrolyte was lead nitrate aqueous solution. Stainless steel plate was employed as both cathode and substrate, and graphite plate as anode. The controlled current that was supplied by a direct current power supply passed through the electrolyte to deposit lead oxide plate on the surface of stainless steel to form a film at room temperature. The obtained deposits on cathodic substrate are β-phase lead oxide plates, which morphology and structure are confirmed by scanning electron microscopy, transmission electron microscopy and X-ray diffraction.

A00997-02601

Thermal Effects on the Percolative Behavior of Polyvinylidene Fluoride/Nickel Composites

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It is well known that insulator/conductor composites (ICC) undergo a metal insulator transition (MIT) at a critical concentration of the metal in the composite. While the percolation theories predict the ideal percolation threshold (P_c) i.e. the critical volume fraction for any 3D random ICC to be 0.16, experimentally the P_c value is found to be at variance. It appears that the value of the P_c depends on various parameters, such as, size, shape, spatial distribution, adhesiveness, viscosity, wetting of the polymer and also on the process conditions. However, recently a P_c of 0.16 has been reported for hot molded (200ºC) PVDF/
Synthesis and Characterization of Nano-cupric Oxide by Using a High Gravity Process

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In order to find the operating conditions to prepare desired nano-cupric oxide particles, synthesis of CuO was carried out by feeding of CuCl₂ and NaOH solutions simultaneously into a high gravity apparatus. Precursors obtained here were Cu(OH)₂ and Cu₂Cl(OH)₃, depending on the concentration of CuCl₂. The decomposition temperature of precursor was determined by TGA instrument analysis. The precursor was obtained first and later it was put into an oven for three-hours calcinations at temperature of 100°C and 350°C, respectively, to obtain CuO. Subsequently, the cupric oxide was characterized by XRD, SEM, and BET instruments. The results showed that specific surface areas obtained at the decomposition temperature of 100°C were in the range of 11.31 to 50.36 m²/g, while at the temperature of 350°C the specific surface areas were in the range of 7.49 to 18.78 m²/g. Therefore, the average diameters of CuO estimated here by specific surface areas were between 18.9 to 127.1 nm. Due to this, we can adjust operating conditions more flexible to synthesis desired nano-cupric oxide.

Post-annealing Influence on Conductivity Behavior of ZnO:Li Thin Films Prepared by r.f. Magnetron Sputtering

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In this work, Li-doped ZnO films were prepared on glass substrates by r.f. magnetron sputtering. The influence of post-annealing temperature on the structure, electrical, and optical properties of ZnO:Li was investigated. The results show that the ZnO:Li films show (002) preferred orientation and high average transmittance about 85% in the visible range after temperature at 550°C. A conversion from n-type conduction to p-type in a range of temperature was confirmed by Hall measurement. The as-grown ZnO:Li film was insulating but behaves as p-type conduction with lowest resistivity of 0.12 ohm-cm, a carrier concentration of 2.48x10¹⁸ cm⁻³ and Hall mobility of 0.22 cm²/Vs after annealing at 450°C for 3 hr.

Synthesis and Characterization of Carbon Doped Molybdenum Oxide Nanoneedles by Reactive RF Sputtering

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Molybdenum oxide (MoOₓ) is an interesting metal oxide with promising properties for applications in the field of catalysts and chemical sensors. Its characteristics could be significantly enhanced by forming nanostructures. Recently, MoOₓ nanostructures prepared by sputtering, thermal evaporation and chemical syntheses have been reported. Sputtering is a practical method for MoOₓ nanostructure synthesis because it is a low temperature, well-controlled and IC-compatible process. MoOₓ nanostructures should be incorporated with foreign atoms to modify and improve its properties. However, there have been few reports on doping study of MoOₓ nanostructures. In this work, molybdenum oxide nanoneedles is prepared by reactive sputtering under suitable conditions and the structure is doped with carbon by acetylene (C₂H₂) gas addition while sputtering. The carbon doped chromium oxide was deposited on silicon substrates by reactive sputtering under a mixture of Ar, O₂, and C₂H₂ gases. The carbon content is controlled by varying acetylene/oxygen flow ratio from 0.05 to 0.25 while sputtering pressure and radio frequency (rf) power are varied in the range of 4 x 10⁻³ - 8 x 10⁻³ mbar, 150-200 W, respectively. The surface and chemical structure of the materials are characterized by means of scanning electron microscopy, energy dispersive X-ray spectroscopy, transmission electron microscopy, and X-ray diffraction (XRD). Structural characterization shows that MoOₓ nanoneedles are formed with different length and width ranging from 2-5 um and 50-200 nm, respectively. In addition, as-deposited MoOₓ nanoneedles are highly crystalline with MoO₃ Orthorhombic crystal phase and carbon dopant is successfully incorporated in the structure with controllable concentration. Moreover, carbon doping considerably affects crystal quality and crystallographic orientation. Optical characterizations including ellipsometry and photoluminescence have been performed. It was found that carbon doping significantly modifies optical constants and photoluminescence spectra.
Preparation and Characteristics of Y$_2$O$_3$-doped SrTiO$_3$ by Reaction-Sintering Process

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We prepared Y$_2$O$_3$-doped SrTiO$_3$ ceramics (YST) by reaction-sintering process. YST is a promising candidate as the anode for solid oxide fuel cell. The preparation began with mixing raw powders, followed by uniaxially pressed into bars, and sintering in air at 1250°C~1400°C for 2 h, 4 h, 6 h and 8 h, respectively without calcination process. X-ray diffraction patterns of the sintered YST ceramics show a main perovskite structure. The YST sample exhibited a higher density with a higher sintering temperature. A maximum density of 4.9 g/cm$^3$ is observed for samples sintered at 1300°C for 8h. The average grain size is around 0.9 to 1.2 μm. A longer sintering duration does not change the surface morphology of the YST samples but the grain size slightly increased with temperature. The samples do not show an expected conductivity. Mostly, it is below 1 S/cm, which may be attributed to the sintering atmosphere and the minor second phase in the bulk.

The Effect of Mn and Co Doping on the Structures, Optical and Magnetic Properties of SnO$_2$ Nanoparticles

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Mn or Co doped SnO$_2$ nanoparticles were synthesized at low temperature using hydrothermal method with SnCl$_2$·5H$_2$O and MnCl$_2$·4H$_2$O, CoCl$_2$·6H$_2$O as starting materials, respectively. Samples are characterized by X-ray diffraction (XRD), high-resolution transmission electron microscopy (HRTEM), diffuse reflectance spectra (DRS), UV-Visible spectra and magnetic measurements. Well dispersed spherical particles with narrow size distribution are observed for both doped SnO$_2$ samples. The grain size decreases from 6 nm to 2 nm when the doping level increases 0 to 20%. A considerable red shift in the absorbing band edge was observed for the doped samples. Magnetic measurements indicate that both Mn and Co doping leads to paramagnetism in the nano-SnO$_2$. The absence of ferromagnetism is discussed in terms of first-principles calculations and the particle size effect. We will also show our results on the water splitting performance of the doped and undoped SnO$_2$ nano-particles.

Influence of Potential Barrier Height of the Interface Glass on Electrical Performance of Screen Printed Ag Thick-film Contacts of Si Solar Cells

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Screen printed Ag thick-film contacts are widely used for the front side metallization of crystalline Si solar cells. Although glass holds the minimum weight percentage in Ag thick-film pastes (1-5 wt %), it plays important role during etching through the antireflection coating (ARC) and for ensuring a stable mechanical contact. It serves as a transport medium for Ag to re-crystallize on the Si emitter surface and guarantees the formation of Ag/Si ohmic contacts. In addition, it also dissolves Ag powders and affects the Ag powder sintering kinetics. Therefore, glass is a key factor that determines the Si surface etching, contact resistance, and ultimately the overall performance of cells.

We have attempted to investigate the influence of potential barrier height on the electrical performance of screen printed Ag thick-film contacts by indirectly determining it from the effective bandgap of glass. The glass samples in the PbO-ZnO-B$_2$O$_3$-SiO$_2$ system with 5, 10, 15 and 20 mol% ZnO, designated as PZ-1, PZ-2, PZ-3 and PZ-4 were prepared by conventional melt quench technique. Screen printed patterns were fabricated on polycrystalline Si substrates using carefully controlled Ag pastes and by rapid thermal processing (RTP). The thermal properties of powdered glass samples were measured using a differential scanning calorimeter, and the crystalline phases of powdered glasses after RTP were identified by X-ray diffraction. Transmission line model (TLM) was employed to measure the electrical performance of the electrodes. The linear I-V curves indicate ohmic property of contacts. With the increase of potential barrier height of glasses from PZ-1 to PZ-3, we find that specific contact resistance ($\rho_c$) of Ag electrodes stays almost constant at around 0.94 Ω·cm$^2$, but

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The electrical properties of Ag thick-film consisting of PbO is essential for the sintering of Ag powders. The decrease in conductivity from PZ-1 to PZ-4 is due to the decrease of PbO concentration since PbO is essential for the sintering of Ag powders.

The electrical properties of Ag thick-film consisting of PbO-Al2O3-B2O3-SiO2, PbO-Sb2O3-B2O3-SiO2 and Bi2O3-Sb2O3-B2O3-SiO2 systems have also been investigated and will be presented at the conference.

**A01062-01843**

*Formation and Characterisation of Cuprous Oxide Film via Oxygen Plasma Treatment of Metallic Copper*

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Cuprous oxide is a direct bandgap semiconductor material (bandgap 2.0eV), that has been studied for a long period of time relative to other semiconductor materials. It has garnered increased attention recently for its potential in photovoltaic cells, as it is non-toxic and has a high absorption coefficient in the visible region, with relatively inexpensive constituents. There have been numerous reports on devices incorporating cuprous oxide, such as heterojunction solar cells. The synthesis methods reported thus far have usually involved the more expensive and complicated methods like electrodeposition, reactive radio frequency magnetron sputtering and anodic or chemical oxidation. A relatively simpler method that is commonly used is thermal oxidation. However, high temperatures are required for pure cuprous oxide formation, with lower temperatures often yielding a mixture of cuprous and cupric oxide, or purely cupric oxide.

Here, we present a means of obtaining a layer of cuprous oxide film via a relatively simpler method. Copper was converted to cuprous oxide after being exposed to oxygen plasma treatment in a Trion Reactive Ion Etching (RIE) machine. This thin copper film was first deposited onto a cleaned glass substrate via DC magnetron sputtering, and subsequently characterised with X-ray diffraction, and a scanning electron microscope. XRD results suggested the formation of a film of cuprous oxide with a strong (111) orientation, and the absence of cupric oxide. Electrical and optical characterisations obtained by using a UV/Visible Spectrometer such as the optical bandgap and absorption coefficient will also be presented here. The method presented here is a potentially simpler and faster means of obtaining cuprous oxide film that could be of use in further enhancing the ease of incorporation of cuprous oxide into various semiconductor devices such as the aforementioned solar cells.

**A01068-01968**

*Physical and Electrical Properties of Hafnium Oxide (HfO2) Films Deposited on AlGaN/GaN Heterostructure by Pulsed Laser Deposition*

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Good quality insulators with high dielectric constant (high-k) are desirable as the gate dielectric in AlGaN/GaN metal-insulator-semiconductor heterostructure field effect transistors (MIS-HFETs), as the high-k property helps alleviate the reduction in transconductance and negative shift in threshold voltage in comparison to Schottky gate HFETs (SG-HFETs). Hafnium oxide (HfO2), which has been extensively investigated in the Si MOSFET technology, has also attracted interest to be used in AlGaN/GaN MIS-HFETs owing to its high-k property. In this report, we investigate the physical and electrical properties of HfO2 films grown by pulsed laser deposition (PLD) technique and the device performance of PLD-grown HfO2/AlGaN/GaN MIS-HFETs, where HfO2 is used not only as the gate dielectric, but also to passivate the device surface. X-ray diffraction spectra confirm that the HfO2 film grown at 50°C in a 100 mTorr, O2 ambient, using HfO2 target (99.95% purity), is amorphous. This is important because polycrystalline HfO2 has increased grain boundary leakage that can pose as reliability issues. X-ray photoelectron spectroscopy results have shown the stoichiometric atomic ratio of Hf and O (~1:2) in the film. Moreover, secondary ions mass spectroscopy reveals the uniformity of the HfO2 film component from the surface to the HfO2/GaN interface. The dielectric constant of the amorphous HfO2 is ~20, as determined by the capacitance-voltage measurement. The resulting interface state density of HfO2/GaN is estimated to be ~1.0×10^12 eV^-1cm^-2. Room-temperature Hall effect measurements have revealed a 5 % increase in the 2DEG conductivity (nσ×μ) of the PLD-grown HfO2 passivated AlGaN/GaN heterostructure compared to that of the reference unpassivated one. As a result, the fabricated MIS-HFETs exhibit better device performance than the reference unpassivated SG-HFETs. The maximum drain current and gate voltage swing have increased by ~31.5 % and 8.5 %, respectively, over that of the reference devices. Both the off-state drain current and the gate leakage current have reduced by more than 1 order of magnitude relative to that of the SG-HFETs, indicative of the higher breakdown voltage of the PLD-grown HfO2.
MIS-HFETs. In addition, the dc characteristics of the two types of devices were studied at elevated temperature up to 200°C. MIS-HFETs exhibit a smaller degradation rate in device performance upon the increase of operation temperature, compared to the SG-HFETs, suggesting a lower temperature sensitivity of the former. It is worth mentioning that even at 200°C, the gate leakage current ($V_g = -5$ V) of HfO$_2$ MIS-HFETs remains at $\sim 42\%$ lower than that of the reference SG-HFETs. All these results manifest the good quality of PLD-grown HfO$_2$ film and its potential as the gate dielectric in MIS-HFETs for high power and high temperature operations.

**A01074-01856**

**Mechanochemical Synthesis of MoSi$_2$ for Applications in Ultra High Temperature and Oxidizing Conditions**

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Molybdenum disilicide (MoSi$_2$) has a high melting point (2030°C) coupled with extremely high resistance to oxidation and corrosion, high electrical and thermal conductivities, and moderate density. Such a combination of properties enables its extensive applications in high temperature and highly oxidizing conditions. However, its conventional synthesis involves high temperatures and simultaneous formation of glassy SiO$_2$ that make the synthesis considerably difficult with deteriorating properties coupled with the extremely brittle product. An alternate synthesis technique involving mechanochemical processing route is thus proposed towards the synthesis of MoSi$_2$ with the required properties. The mechanical activation of powders of Mo, and Si were done in a planetary ball mill with ball-to-powder ratio (BPR) of 10:1 and 20:1 at a speed of 400rpm. Milling time was varied from 8 to 32 hours with intermittent sampling. The as milled powders were characterized with XRD, SEM-EDX, BET, and density measurements. The powders were then heat treated at various temperatures from 500 to 600°C and characterized in the same way as that with the as milled powders. The as milled powders milled for 8 hours indicated the formation of MoSi$_2$, clearly showing the possibility of its synthesis at a temperature far lower than that used in conventional practice. Extended milling, however, showed decreasing surface area of the powders as a result of agglomeration as confirmed by the SEM images even though individual particles of size 400nm or less were noticed. The formation of MoSi$_2$ was found more pronounced with extended milling time. The study systematically presents the effects of various process parameters on the formation of MoSi$_2$, through mechanical activation. Thus the mechanochemical route has been used as a successful alternate towards the synthesis of MoSi$_2$.

**A01082-02100**

**Synthesis, Characterization and Electric Properties of Sol-gel Derived CaCu$_3$Ti$_4$O$_{12}$ Ceramics**

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CaCu$_3$Ti$_4$O$_{12}$ (CCTO), an unusual perovskite-like ceramic, is known for its extraordinarily high (often reaching $10^5$) and relatively frequency independent dielectric constant. Recently, it has drawn a lot of attention, because of its potential applications in microelectronics and microwave devices.

In this investigation, CCTO powders were synthesized by a sol-gel route from calcium nitrate tetrahydrate, copper nitrate hemipentahydrate and titanium isopropanoxide. This route allows very fine (tens of nanometers) and uniform grains of CCTO to be obtained in considerably shorter times and at much lower temperatures than the conventional solid state route. It also allows us to study the correlation between the micro-structural evolution and dielectric properties of CCTO systematically and in an extended range of grain sizes. The structure, composition and morphology of CCTO samples sintered at different temperatures were characterized by X-ray diffraction, energy dispersive X-ray analysis and scanning electron microscopy. Their dielectric properties were measured by impedance spectroscopy.

In general, we found that the dielectric constant tends to increase with average grain size. In particular, we found that it can still be as high as 31,000 for 300 nm grains and reaches $10^5$ for 30 µm grains at low frequency 40 Hz. Furthermore, there is a maximum size of several tens of micrometers above which it does not increase significantly anymore. We also found that both the dielectric loss and conductivity depend on grain size. Because the loss shows strong dependence on frequency, lowest loss must be optimized according to desired applications. For large grains, the lowest dielectric loss occurs at frequencies lower than $10^3$ Hz, but for small grains, it occurs at frequencies higher than $10^3$ Hz. On the other hand, the conductivity is always small for small grains, regardless of frequency. These observations may be explained by the internal-barrier-layer capacitor (IBLC) model with Maxwell-Wagner (MW) relaxation. The internal barriers were revealed to be grain boundaries and domain/sub-grain boundaries.

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In this study, the optical and structural properties of Fe-TiO2 sol were investigated. TEOT\([\text{Ti(OC}_2\text{H}_5)_4]\) and Ferric nitrate\([\text{Fe(NO}_3)_3\cdot9\text{H}_2\text{O}]\) were used as precursors to make Fe-TiO2 sol. Samples for measurement were prepared on a glass substrate by using sol-gel dip coating method. The annealing temperature (Ta) was varied in the range of 400–600°C in intervals of 50°C.

The cut-off points of the samples were red-shifted with increasing Fe contents. Simultaneously, the optical band gap decreased from 4.03 to 3.64eV. A change of the crystalline phases as the Fe contents increased was observed from X-ray diffraction measurements.

Combined with the X-ray diffraction analysis, the observed variations in both the cut-off points and optical band gap have been directly correlated with the structural change of the composite films.

Properties and Structure of ZnO-Bi2O3-P2O5 Glass

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Thermal and chemical properties were studied on ternary zinc bismuth phosphate glasses (ZBP glasses). Glasses along four composition tie line, I: \(x\text{Bi}(\text{PO}_3)_2\cdot(1-x)\text{Zn}(\text{PO}_4)_2\), II: \(x\text{BiPO}_3\cdot(1-x)\text{Zn}(\text{PO}_4)_2\), III: \(x\text{Bi}_2\text{O}_5\cdot(1-x)\text{Zn}(\text{PO}_4)_2\) and IV: \(x\text{Zn}(\text{BiO}_2)_3\cdot(1-x)\text{Zn}(\text{PO}_4)_2\), have been prepared. Particularly to examine the effect of structure and properties in phosphate glass system by change of \(\text{Bi}_2\text{O}_3\) composition, the present work studied about ZnO-Bi2O3-P2O5 glass. As a method to expect the relation between structural change and compositional change, the structure was measured by FT-IR analysis. And the glasses were respectively confirmed to density, glass transition temperature \(T_g\) and chemical properties through structural change of glasses. \(T_g\) and chemical property (water-resisting qualities) increases with increase in metaphosphate phase in glass due to increase in the number of bridging oxygens. The glass properties are dependent on the O/P ratio. Compositional variations that change the dominant phosphate structure from a metaphosphate to a pyrophosphate, or from a pyrophosphate to an orthophosphate, are accompanied by a transformation in the dominant Bi coordination, complicating the effect that \(\text{Bi}_2\text{O}_3\) has on glass properties. \(\text{Bi}^{3+}\) cations incorporated in the glass network and suggested difference in \(\text{Bi}-\text{O}\) bonding state in the glasses.

Structure and DC Conductivity Studies of Sm\(^{3+}\) Substituted Ni-Zn Ferrites

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Polycrystalline ferrites having the general formula \(\text{Ni}_x\text{Zn}_{1-x}\) \(\text{Fe}_{2y}\) \(\text{Sm}_y\) \(\text{O}_4\) \((x = 0.00, 0.25, 0.50, 0.75, 1.00; y = 0.00, 0.05, 0.10)\) were prepared by standard ceramic method. The samples were characterized by XRD, IR, SEM, and EDAX techniques. The single-phase spinel formation of ferrites was confirmed by X-ray diffraction technique. IR spectrum of ferrite samples has shown the two strong bands. The band \(\nu_1\) near 6m\(^{-1}\) arises due to tetrahedral complexes and band \(\nu_2\) around 4m\(^{-1}\), due to octahedral complexes. The difference in the two strong bands \(\nu_1\) and \(\nu_2\) could be related to difference in \(\text{Fe}^{3+}-\text{o}^2-\) distances for A and B sites. From the scanning electron micrographs average grain size was measured by counting the number of grain boundaries intercepted by a measured length of a random straight line drawn on the micrographs and it is observed that the grains are spherical in shape smaller in size and no exaggerated grain growth has taken place in the sample during sintering. The grain growth mechanism is compromised between driving force for grain boundary movement and retarding force of pores and inclusions during the sintering process. The strength of the driving force generated due to thermal energy, depends upon sintering temperature and diffusivity of constituent ions. The DC electrical conductivity of the palletized samples was measured by two-probe method in the temperature range 300K to 840K. The electrical conductivity in ferrites can be explained on the basis of exchange of electrons between ions of the same element that are present in more than one valence state, distributed randomly over equidistant crystallographic lattice sites. The electrical conductivity in ferrites to be due to the simultaneous presence of both \(\text{Fe}^{2+}\) and \(\text{Fe}^{3+}\) ions and due to electron hopping between \(\text{Fe}^{2+}\) and \(\text{Fe}^{3+}\) ions on octahedral sites. The electrical properties of ferrites are affected by the distribution of cations in sites by magnetic and non magnetic substitutions, the amount of \(\text{Fe}^{2+}\) ions present, sintering conditions, grain size and grain growth. The experimental results reveal that the DC conductivity increases as temperature increases and as the \(\text{Zn}^{2+}\) and \(\text{Sm}^{3+}\) ion content decreases. The electrical
conduction mechanism in these ferrites is on octahedral sites. The activation energy for ferrimagnetic region is lower than in paramagnetic region. The addition of Sm$^{3+}$ impedes conduction in samples. Thus, the activation energy is higher for Sm$^{3+}$ substituted samples than those for the corresponding undoped samples in both the regions.

Preparation and Characterization of (Ba,Sr)TiO$_3$/Ba(Zr,Ti)O$_3$ Heterostructures Grown on (LaAlO$_3$)$_{0.75}$(Sr$_{0.3}$TaO$_{0.7}$)$_{0.25}$ (001) Single Crystal Substrates

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BaTiO$_3$ is one of the most important perovskite-type ferroelectric oxides. The introduction of substitutional elements to the perovskite lattice sites (Sr as A-site and Zr as B-site) can lead to significant changes in the properties of the materials. For example, in bulk form (Ba, Sr)TiO$_3$, the Curie temperature is linear to the content ratio of Ba and Sr (Ba/Sr). For thin films, due to substrate effects, the relationship between physical properties and structure is more complicated. In this work, we investigated the structural, morphological, and dielectric properties of (Ba,Sr)TiO$_3$/Ba(Zr,Ti)O$_3$ heterostructures grown on (LaAlO$_3$)$_{0.75}$(Sr$_{0.3}$TaO$_{0.7}$)$_{0.25}$ single crystal substrates. The compositions we chose are (Ba$_{0.7}$Sr$_{0.3}$)TiO$_3$ (BST) and Ba(Zr$_{0.2}$Ti$_{0.8}$)O$_3$ (BZT) respectively, both of them have Curie temperature around room temperature. BST/LSAT and BZT/LSAT are also studied for comparison.

The thin film samples were prepared by pulsed laser deposition and the film thicknesses were controlled by growth time. X-ray diffraction was used to characterize the structures of the heterostructures. The morphology of the sample was measured by atomic force microscopy. The ferroelectric properties of all the samples were examined using a TF analyzer 2000 (aix ACCT). Comparing the heterostructures, we found that the structure and morphology of the BZT/BST/LSAT(001) has improved significantly, which may due to the good lattice-match between BZT, BST and LSAT(001) substrate. This may have a positive effect on the dielectric, ferroelectric properties of the multilayered heterostructures which will be investigated in detailed later.

Electronic Band Structures, Thermoelectric Properties and Magnetism in the Newly Discovered Fe-based REFeAsO Superconductor

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We have performed ab initio LDA and LSDA+U calculations of electronic structure of newly discovered high-temperature superconducting series ReO1–xFxFeAs (FeAs-1111, Re=rare earth) and ReTmSbO (TmSb 1111, Tm=transition metals). For FeAs-1111, electronic spectrum is very similar in both energy dispersions and the densities of states, being unable to account for the large difference in Tc for various Re elements. It is proposed that magnetic interactions with rare-earth spins may lead to possible changes in spin-fluctuation of FeAs layers. We also found that the intralayer chemical bonding present a significant part of covalency, whereas the interlayer bonding is almost completely ionic. Negative and positive U values have been used to calculate the magnetic moment of both systems. We will discuss the effect of the U on electronic structures and magnetic moments in both systems. Some exotic electronic structures will be addressed for some of the TmSb-1111 compounds. Experimental results on the valences of Fe, Seebeck coefficient, thermo conductivity and figure of merit on some ReAs1111 compounds will be presented.

This work is supported by the Australian Research Council.

Electronic Structure, Phase Formation and Thermoelectric Properties of Doped Mg$_2$Si and Bi$_2$Te$_3$

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High temperature thermoelectric material Mg$_2$Si doped with Al or Sn are studied in terms of structures, phase formations, thermoelectric properties and first-principles calculations. It is found that a short time sintering is enough to form a highly pure phase sample with small particle sizes. Highly dense samples can be easily achieved in Sn added Mg$_2$Si under ambient pressure. The crystal structures are refined using Rietveld refinement. We will report our results on the phase formation, surface morphology, Seebeck coefficients, thermal conductivities, resistivity and figure of merit of these materials. The experimental data will be discussed in comparison with band structure calculations. The difference in the thermoelectric performance between Mg$_2$Si and Bi$_2$Te$_3$ will also be addressed.

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Luminescent Enhancement in Ba\textsuperscript{2+}-Mg\textsuperscript{2+} Co-doped Sr\textsubscript{2}SiO\textsubscript{4}:Eu Yellow Phosphors Coated with Titania

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In recent years, surface modification, i.e., the fabrication of core-shell structure, has attracted extensive attention which a suitable bandgap material was grown to increase the quantum efficiency by suppressing the energy-loss processes at the particles surface. This strategy has been extended to the inorganic luminescent systems which indicate that the formation of core-shell structure is an effective way to improve the photoluminescence (PL) efficiency. In this study, in order to improve the PL efficiency of yellow emission of the phosphors, Ba\textsuperscript{2+}-Mg\textsuperscript{2+} co-doped Sr\textsubscript{2}SiO\textsubscript{4}:Eu phosphors were synthesized and were coated with TiO\textsubscript{2}. The TiO\textsubscript{2} layer with 20 nm was uniformly coated over the phosphor surface. The PL properties of the TiO\textsubscript{2}-coated phosphors showed improved yellow-emission intensity compared to the uncoated phosphors. This seems to be due to the increase of the excitation light which occur the effective absorption of the phosphor surface via TiO\textsubscript{2}.

Enhancement for UV-shielding and Hydrophilicity for Protective Film

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In this study, surface modification of TiO\textsubscript{2} particles with silane coupling agents, hydrophilicity effect of resin, UV-resin/TiO\textsubscript{2} ratio were investigated. UV-shielding ability is enhanced up to 98% as increasing TiO\textsubscript{2} concentration. Hydrophilicity measured by contact angle show various trend depending upon UV-resin type and UV-resin/TiO\textsubscript{2} ratio. Contact angle was observed below 10\textdegree when using very hydrophilic resin or 3/7 ratio of UV-resin/TiO\textsubscript{2}.

Effect of N-incorporation on the Electronic Structure of Rutile TiO\textsubscript{2}

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N-doped TiO\textsubscript{2} for photocatalysis has gained great interests in these years because it can extend catalytic activity of TiO\textsubscript{2} from ultraviolet light (\lambda< 387nm) to visible light, and hence more energy from radiation of solar light can be utilized in photocatalytic and photoelectrochemical applications. X-ray photoelectron spectroscopy (XPS) has been employed to investigate the effects of high concentration N incorporation into TiO\textsubscript{2} by direct N atom source doping. High resolution core-level spectra indicate that N intends to substitute O in Ti-O bonds to form Ti-N bonds. Ti cations shows different state when N concentration is different. The valence band structure shows that band narrowing effect is weak although the shape of the valence spectra varies significantly. First-principle calculation further proved that Ti 3d gap state can be formed when the N concentration exceeds 1 at.% (atomic percent), which also leads to a pining of N 2p state in the band gap.

Novel Nanostructured Vanadium Phosphate Phases

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Catalytic selective oxidation, particularly of hydrocarbons is an area of tremendous research and industrial importance. Vanadium phosphates (VPO) are used commercially for the selective oxidation of butane to maleic anhydride and are also promising catalysts for the selective oxidation of propane to acrylic acid, pentane to maleic and phthalic anhydride and in the oxidative dehydrogenation of ethane, propane etc.

The catalytic activity of the vanadium phosphates is known to be very sensitive to its structural and morphological characteristics and consequently there has been great interest in the preparation of modified VPO phases for their potential use as improved or novel catalysts.
In this context, novel nanostructured vanadium phosphate phases ranging from lamellar to tubular have been synthesized using long chain alkyl amines as templating agents. Different synthetic approaches such as intercalation, organization from solution and exfoliation of a VPO solid followed by reorganization from its solution, using different surfactants have been adopted. These phases have been characterized using a combination of XRD, SEM, TEM, NMR, FTIR and thermal studies. In particular a nanotubular vanadium phosphate has been synthesized for the first time in which the inorganic walls are crystalline and are made up of a catalytically important VPO phase. It may be pointed out here that all the mesostructured VPO phases reported so far have amorphous inorganic walls and this is the first instance of the synthesis of a nanostructured phase in which the inorganic matrix is not only crystalline but corresponds to one of the well known and catalytically important VPO phases.

**Microwave-Hydrothermal Synthesis and Sintering Behavior of Lead Titanate Fine Powders**

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The effects of acoustic wave and microwave on hydrothermal synthesis and characteristics of lead titanate powders were investigated. The application of ultrasonic wave apparently favors the formation of the other oxide phases rather than the desired PT, while the microwave on the other hand results in a reduction in both hydrothermal temperature and time providing phase pure lead titanate powders. The phase pure lead titanate powders can be achieved at water boiling temperature in a very short reaction time depending on the employed microwave power. The composing lead titanate particles are of 0.1-1.7 micron in size, and mostly present as hard aggregates. The densification of the obtained powders is rather poor, and can be rationalized by the particle morphologies.

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of single crystalline \( \text{Ga}_2\text{O}_3 \) polymorphs on selected c- and r-plane sapphire substrates by plasma-assisted molecular beam epitaxy (PAMBE) is presented. Detailed information on the dependence between \( \text{Ga}_2\text{O}_3 \) films and varied growth parameters is also reported.

Heteroepitaxial \( \text{Ga}_2\text{O}_3 \) thin films were grown in a Varian/Veeco 620 MBE system. The film was grown by controlling the impinging gallium or oxygen flux as well as the substrate temperature to determine optimized growth parameters. Co-loading c- and r-plane sapphire substrates facilitated the comparison of \( \text{Ga}_2\text{O}_3 \) film properties on various substrates grown under the same conditions. The \( \text{Ga}_2\text{O}_3 \) surface morphology and film roughness on both substrates was studied by a tapping-mode atomic force microscope (AFM). The in-plane epitaxial relationships of single crystalline (201) \( \beta\text{-Ga}_2\text{O}_3 \) on c-plane sapphire and (102) \( \alpha\text{-Ga}_2\text{O}_3 \) on r-plane sapphire were establish using high-resolution x-ray diffraction (HRXRD). A correlation between \( \text{Ga}_2\text{O}_3 \) film quality and substrate temperature was determined. The films were transparent and colorless independent of the substrate temperature. By applying various impinging gallium fluxes at a fixed oxygen pressure, two growth regimes were identified on which the growth rate and film quality depended. Beginning in a low gallium flux regime, the \( \text{Ga}_2\text{O}_3 \) growth rate increased as the gallium flux increased. Reductions in growth rate and film quality were observed at high gallium fluxes. We associate this behavior with phase transitions and gallium sub-oxide (i.e. \( \text{Ga}_2\text{O} \)) formation.

This current study has shown plasma-assisted MBE as a remarkable tool for providing phase-pure single crystalline \( \text{Ga}_2\text{O}_3 \) layers on various sapphire substrates. Understanding material properties at different growth regimes will also benefit future investigation for high quality \( \text{Ga}_2\text{O}_3 \) homoepitaxy.

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Optimised Deposition Processes for Doped Lead Zirconate Titanate Thin Films

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New deposition regimes for strontium-doped lead zirconate titanate (PSZT) thin films on silicon substrates are outlined in this presentation. Optimised conditions for RF magnetron sputtering have been identified, which has enabled three significant results: (i) crystal structure control of films with unit cell parameter modification, (ii) low temperature deposition at 300°C, and (iii) direct deposition on thermal silicon dioxide without intermediary (seed) layers.

PSZT belongs to the family of doped lead zirconate titanate (PZT) compounds, and is reputed to exhibit relatively high levels of piezoelectric response. To maximise the level of piezoelectric response from PSZT (or PZT) thin films, preferential perovskite orientation is desired. This presentation discusses the results from detailed characterisation of PSZT thin films deposited on a variety of single-layer/multi-layer coatings on silicon. The films were deposited by RF magnetron sputtering and have been analysed using a suite of materials characterisation tools such as scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM), X-ray diffraction (XRD), and secondary ion mass spectrometry (SIMS). This work highlighted the influence that the bottom electrode architecture and temperature of deposition exerts on the microstructure and of the deposited thin films.

Sputtering of PZT thin films is most commonly carried out at temperatures above 650°C on platinitised silicon substrates. This presentation reports on results obtained from deposition carried on two common bottom electrode configurations using platinum, and discusses the presence of an elongated rhombohedral unit cell. This presentation will also discuss an alternative choice of deposition temperature and bottom electrode architecture to obtain crystalline and perovskite PSZT thin films. Gold, with lattice spacings close to that of PSZT, was found to have a guiding effect of orientation of PSZT thin films and this was achieved for the first time at a much lower temperature of 300°C. The study of PSZT thin films on two different bottom electrode architectures (incorporating gold) has been carried out using TEM, XRD, and SIMS. The ability of gold to exert a guiding effect on PSZT was verified by crystal structure simulations.

The presentation will also discuss the first instance of deposition of preferentially oriented PSZT thin films directly on thermal silicon dioxide (SiO\(_2\)). No intermediate seed or activation layers were used between PSZT and SiO\(_2\). Microstructure analysis confirms the presence of a nanocolumnar microstructure with preferential c-axis orientation.
Localised Field Enhancement in Nanocolumnar Piezoelectric Thin Films

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This presentation highlights the presence of localised electric field enhancement in nanocolumnar piezoelectric thin films. Failure to account for field enhancement, in the case of polycrystalline thin films with high aspect ratio columnar nanostructure, will result in significant errors in piezoelectric response coefficient measurements. The influence of field enhancement is experimentally verified for thin films with a columnar structure, showing that not accounting for this contribution overestimates the piezoresponse coefficient.

Piezoelectric materials are used in multidisciplinary applications, and in the quest for miniaturisation, piezoelectric crystals are being replaced with thin films. While in special cases, these films can be epitaxial, they are predominantly polycrystalline with a preferential orientation. To maximise the piezoelectric response, films with a preferential c-axis orientation are desired, often resulting in a columnar grain structure. The grain widths can range from tens of nanometers to a few microns.

It is experimentally shown that anomalous results are obtained during piezoelectric response of polycrystalline thin films, with a nanocolumnar grain structure. Large variations in film thickness (or strain percentages) were obtained for low electric fields. This can be effectively explained using localised electric field enhancement phenomena. Using field enhancement factor calculations used for electron emission and the ‘hemisphere on a post’ model, the dependence of piezoresponse measurements on grain size in nanocolumnar films can be established. For a 1200 nm thick film with grain widths of 100 nm, this enhancement factor is shown to be between 22 and 26.

Island-Structured Piezoelectric Thin Films by Scalable Self-Assembly

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Piezoelectric and ferroelectric thin films have been shown to exhibit increased response in the form of islands, due to reduced clamping. This work presents a novel and fully scalable process for the formation of island-structured thin films.

Platinised silicon samples incorporating a reactive adhesion layer of titanium were used as substrates for deposition. On being subject to a temperature ramp, grain growth in platinum occurs with the creation of boundaries and voids, enabling the out-diffusion of titanium. Platinum reacts with silicon to form silicide islands. The silicide islands serve as a template for subsequently deposited piezoelectric films, acting as preferential deposition sites.

The self-assembly process to form silicide islands and the existence of island structure in the films was studied using transmission electron microscopy (with in situ heating in some cases). These micrographs, in combination with energy dispersive X-ray analysis, were used to identify the mechanism of island formation. The process is scalable as the size and distribution of the islands depends on a combination of process variables (primarily deposition duration and platinum thickness).

This self-assembly process to synthesise island-structured films will enable the study of size dependent physical, electronic, and electrochemical parameters on one sample. It also has the ability to serve as a process for the development of piezoelectric sensors with increased surface area.
The role of a non-planar surface geometry on the polarization switching process in ferroelectric thin films is investigated using a real-space time-dependent Ginzburg-Landau model that incorporates electrostatic interactions. We consider a two dimensional uni-axial ferroelectric film with a thickness that varies sinusoidally. Polarization switching, starting from a single-domain remnant state is simulated for several amplitudes and wavelengths of the surface modulation. It is found that the non-planar surface introduces inhomogeneous electric fields inside the film. These inhomogeneities are responsible for nucleating reverse domains, even without defects and thermal noise, indicating the importance of surface geometry in the switching process. This domain nucleation results in a reduction of the coercive field compared to the ideal flat film, even for very small amplitude modulations in the thickness. For high amplitudes and small wavelengths of the surface modulations, it is observed that complete switching is not possible as the domains get pinned in regions with larger thickness.

Mechanical Properties of Si$_3$N$_4$/SiC Composites by Spark Plasma Sintering

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Si$_3$N$_4$/SiC are ceramic materials that exhibit excellent performance for technical and engineering applications. In this study, a novel sintering method, spark plasma sintering was applied to produce Si$_3$N$_4$/SiC composites. 0 wt %, 10 wt %, 20 wt %, 30 wt % SiC particulate reinforced Si$_3$N$_4$ matrix composite were successfully prepared by spark plasma sintering. Y$_2$O$_3$ and AlN were added as sintering additives. The open porosity was completely eliminated by SPS in 15 min heating to 1650°C and 5 min dwell time. The effects of sintering temperature and pressure on the mechanical properties and morphology of Si$_3$N$_4$ ceramic composite were determined. Microstructures of the sintered samples were observed by SEM images and phase compositions were analysed by XRD.

Preparation and Characterization of Nanoferroelectrics by High Energy Ball Milling

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The control of physical properties and structure / microstructure by restricting the particle size at the nanoscale is one of the novel approaches that has evolved as the new trend setter for achieving novelty in the performance characteristics of a material system and the devices based on it. The present work aims at the study of the effect of crystallite size on properties having perovskite structure ABO$_3$ based on lead zirconate titanate Pb(Zr$_{0.5}$Ti$_{0.5}$)O$_3$ (PZT) and its various modified forms by different dopants (Sm$^{3+}$, Gd$^{3+}$, and Zn$^{2+}$) at the A site.

The observed change in physical properties of nanosystems, when compared to their bulk counterparts, is a phenomenon that has been attributed primarily to size effect. The basic reason for an overall change in physical properties of nanosystems, on reduction of crystallite dimension, is attributed to the increase in the ratio of surface area to volume. This change brings significant modification in the arrangement of atoms, ions and density of states in the system that in turn modulates the material structure / microstructure. This modification ultimately manifests in significant changes in the system’s physical properties predominately governed by crystallite size lying below a specific critical length (i.e., crystallite size ≤ 50 nm).

The size effect mechanism developed here gives insight into the complex behavior of ferroelectrics (dielectrics, complex impedance spectroscopy, electrical conduction, mechanical etc.) and provides a starting-point for the development of different functional materials for high dielectric materials and device applications.

Monodomain to Multidomain Transitions Induced by Depletion Layers in Ferroelectric Thin Films

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The stability of the remnant state is a key issue in the development of ferroelectric thin films for memory device applications. For films below a certain critical thickness, the electrical boundary conditions can strongly influence the stability of this monodomain state, and this has often been explored through the introduction of non-ferroelectric “dead” layers at the film surface. However, in this model
we show that depletion layers in ferroelectric thin films can play an equally important role in inducing a mono to multidomain transition in ferroelectric thin films. In these films, charge-depleted layers form due to the difference in work functions between the ferroelectric and the metallic electrode. This results in a built-in electric field, which, if strong enough, will cause the system to split into a multi-domain state. Here, we apply the Ginzburg-Landau-Devonshire framework in a phase field model to investigate the stability of a remnant mono-domain state in fully and partially-depleted films of BaTiO$_3$ using grid sizes of up to 128x128x128. Our results show the reduction in the remnant polarization as a function of space charge and film thickness, as well as the formation of cone-like domain structures in the depletion layers and 180° striped cylindrical domain structures in the bulk. We show that during the switching process, domains preferentially “nucleate” from such regions, and that the presence of these domains lowers the nucleation barrier for switching.

A01263-02183

Effect of Sr Substitution on Transport Properties of Ba$_5$DyTi$_3$V$_{5-o}$O$_{19}$ Ceramics
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The polycrystalline samples of Ba$_5$-$_x$Sr$_x$SmTi$_3$V$_{5-o}$O$_{19}$($x=0-2$) were prepared using a high-temperature solid-state reaction technique with AR (Analytical Reagent) grade precursors (i.e., oxides and carbonates). Analysis of structural data (i.e., crystal system, cell parameters, particle size etc.), using room temperature X-ray diffraction patterns taken over a wide range of Bragg’s angles (20°≤2θ≤80°) with CuK$_\alpha$ (λ=1.5405Å) radiation, exhibits the formation of single-phase orthorhombic structures. The surface morphology of the compounds obtained by scanning electron microscopy shows that the grains are distributed more or less uniformly throughout the surface of the samples. The electrical properties of the materials are tailored by substituting Sr at the Ba-site. Detailed studies of electrical properties using impedance analysis technique exhibit, (i) the presence of negative temperature coefficients of resistance behavior, (ii) presence of temperature dependent polydispersive and non-Debye type of electrical relaxation phenomena and (iii) an enhancement in the barrier to the mobility of charge carriers on Sr-substitution. A significant change in the transport properties (ac conductivity) of the materials was observed on temperature and frequency.

A01267-04492

Interesting Dielectric and Magnetic Characteristics of Sr$_2$TiMn$_6$O$_{12}$ Ceramics
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Manganite-like double perovskite Sr$_2$TiMn$_6$O$_{12}$ (STMO) ceramics have been studied in detail for its dielectric and magnetic properties. Well sintered STMO pellets exhibited dielectric constants as high as ~10$^4$ in the low frequency range (100 Hz - 10 kHz) at room temperature. The Maxwell-Wagner type of relaxation mechanism was found to be more appropriate to rationalize such high dielectric
constant values akin to that observed in CaCu3Ti4O12. The impedance studies (100 Hz – 10 MHz) in the 180 – 300 K temperature range revealed the presence of two dielectric relaxations corresponding to grain boundary and electrode. However the low temperature dielectric data (< 180 K) reflects the intrinsic response of STMO. The DC conductivity at low temperatures (100 K-190 K) followed a variable range hopping (VRH) behavior owing to the polaronic conduction.

DC Magnetization (M) measurements on STMO powder samples were carried out from 5 K to 300 K under both Zero-Field cooled (ZFC) and Field-cooled (FC) conditions in an applied field (H) of 100 Oe. The ZFC curve exhibits a cusp at 70 K and coalesces with FC curve at slightly higher temperature (~ 80 K). Such features in DC magnetization could be attributed to a typical spin glass behavior. Hysteresis loop (M Vs H) (-30 kOe to 30 kOe) of STMO exhibited a coercive field of 1 kOe and M (remnant) value of 0.199 emu/g at 5 K. X-ray photoemission spectrum (XPS) of STMO was studied in order to establish the oxidation state of cations especially Mn which can exist in both Mn3+ and Mn4+ states depending on the synthesis conditions of STMO. The dielectric and magnetic characteristics exhibited by STMO make it a potential candidate for magnetocapacitance applications where in the capacitance can be tuned with respect to magnetic field.

Structural and Electrical Properties of La3+/Mn4+ Modified Lead Titanate

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A plenty of attempts are being made to obtain multifunctional materials with optimum properties for device applications. With the purpose of fabricating ceramics where ferroelectric and magnetic order coexist, nanoceramics of Pb0.88La0.12MnTi14O32 (PLMT) where x = 0, 0.04, 0.07, 0.10 have been prepared by high-energy ball-milling followed by heat treatment. Optimised value of lanthanum has also been substituted to increase the density and stability of system. A preliminary structural analysis of XRD data indicates a tetragonal crystal structure as the best fit for both pure and modified lead titanate. The surface morphology investigated by field effect scanning electron micrographs (FESEM) show homogeneous granular microstructure separated by grain boundaries. Detailed studies of electrical (dielectric and impedance properties) properties of the compounds in a wide range of temperature (35°C to 500°C) and frequency range (1 kHz - 1 MHz) exhibit that (i) the Curie temperature (Tc) and tetragonality (c/a) were found to decrease with increase in La/Mn content. and (ii) presence of negative temperature coefficient of resistance (NTCR) behaviour that is also retained on doping (iii) the temperature dependent plots reveal the presence of both bulk and grain boundary effects at high temperatures. Conductivity spectrum analysis suggests the hopping of charge carriers among localized site as the possible mechanism or electrical conduction. Modulus analysis also indicates the possibility of hopping mechanism for electrical processes in the system with a nonexponential type of conductivity relaxation.
Symposium D - Functional Ceramic Materials, Oxide Thin Films and Heterostructures

A01284-02279 Influence of Post-annealing Temperature on the Properties Exhibited by In doped ZnO Thin Films for Anti-reflecting Coating Application
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In this work we present a study on the effect of annealing temperatures on the structural, morphological and optical characteristics of indium doped zinc oxide films. Indium zinc oxide films were deposited at room temperature by sol-gel method. As a starting material, zinc acetate dehydrate was used. 2-methoxyethanol and monoethanolamine were used as the solvent and stabilizer, respectively. The dopant source was Indium chloride. The atomic percentage of dopant in solution were Zn1-xInxO (x = 0, 5 and 10 at.%). All films were annealed at temperatures of 400, 500 and 600°C in nitrogen gas for 1 h. Structural investigation including surface morphology and microstructure was carried out by XRD and SEM measurements. From the results of these experiments it was established that the In built into the crystal lattice of the ZnO and the surface of the particles enriched in In ions. It was found in the morphology that the primary particle size decreased with the increasing In³⁺/Zn²⁺ molar ratio and with the increasing zinc precursor concentration and the primary particles formed aggregates. Also, optical properties were determined by photoluminescence and UV-VIS spectrum analyses. Due to the presence of the In³⁺ ions the optical properties of the nanocrystals also changed. Blue shift was found in the absorbance spectra of the nanoparticles compared to the spectrum of the pure ZnO, and a new degraded organic on the protective film because the TiO2 particles with silane coupling agents was performed with different silane coupling agents and loading amounts between TiO2 and silane coupling agents. And UV-shielding effect and color change for protective film is measured using UV-VIS and color spectrometer.

A01287-02242 The Effect of Surface Modification TiO2 with Silane Coupling Agent on Physical Properties for Protective Film
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Colors for advertisement pictures exposed by sun light are quickly degraded due to UV light. To enhance lifetime and clearness for advertisement picture, protective film is very effective, which has dual functions of UV-shielding ability and hydrophilicity. Titanium dioxide (TiO2) has been considered as good photocatalyst and absorbent of UV. However, strong photocatalytic TiO2 gives negative effect on the protective film because the TiO2 degraded organic components including resin used in protective film. Finally, colors for protective film gradually changed to yellow. So, controlling photocatalytic activities for TiO2 are need. In this study, surface modification of TiO2 particles with silane coupling agents was performed with different silane coupling agents and loading amounts and characterized the modified TiO2 samples by FT-IR, TGA, TEM, and BET to understand bonding and loading amounts between TiO2 and silane coupling agents. And UV-shielding effect and color change for protective film is measured using UV-VIS and color spectrometer.

A01310-02504 Synthesis and Photoluminescence Properties of Continuous Freestanding SiC(Al) Films Derived from Aluminum-containing Polycarbosilane
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Continuous freestanding SiC(Al) films with high performance were fabricated by the technique of melt spinning the aluminum-containing polycarbosilane (AlPCS) precursor. An experimental apparatus including spinneret, mandril, tank, seal cover and seal groove was set up for melt spinning of films under heating condition. The spinneret which was made of carbon steel contained four adjustment screws, spinneret mouth and double special-shaped spinneret blocks. The thickness of freestanding films was controlled by adjusting the spout size of the
spinnert mouth and spinning speed. Al-PCS samples were deaerated, melt spun, cross-linked for 3 h and pre-pyrolyzed at 900°C, then the films were continuously pyrolyzed in an argon up to 1600°C, 1700°C and 1800°C to convert the initial Al-PCS into SiC(Al) ceramic films, respectively. The as-received films could avoid mismatches of thermal expansion coefficient and lattice constant at the interface between SiC coatings and substrate. The morphology, microstructure and composition of the films were characterized to provide a detailed understanding of technique and products. And the possible origin of the photoluminescence (PL) of the samples was also investigated.

The X-ray diffraction, Raman spectroscopy, X-ray photoelectron spectroscopy and transmission electron microscopy measurements provided the evidences that continuous freestanding SiC(Al) films contained β-SiC crystals, α-SiC crystals, C clusters and small amount of AlO4 and AlSi4, which might appear in the grain boundaries and enter the crystal lattice, respectively. The Al in the SiC(Al) films mainly existed in two manners: Al–C bonds connected with the surfaces of the β-SiC grains and Al–O bonds. The AlO4 and AlSi4 could greatly inhibit the SiC grain growth. The electron probe microanalysis displayed that the films pyrolyzed at 1800°C with nearly stoichiometric composition (C:Si = 1.03) were confirmed as chemical composition of SiC1.03O0.013Al0.024.

And the scanning electron microscopy showed that the films pyrolyzed at 1800°C were also smoother and denser than others. The PL spectrum showed a wide luminescence band from 320 nm to 440 nm, and PL intensity increased with increasing sintering temperature. The origin of PL centered at 385 nm might be related to the α-SiC nanocrystals using quantum size effects, whereas the peak at 412 nm was ascribed to the C clusters, and there was an optimal size for efficient luminescence. The obtained results are expected to have important applications such as microelectromechanical systems (MEMS) for the environment of high temperature, advanced optoelectronic devices and such complex shaped-materials.

Electron Microscopy (TEM) data concerning LCMO and PCMO film grown on SrTiO3 (STO). From the structural point of view the different thickness films show relevant differences: thin films (7 nm) are fully strained and no dislocations appear; thicker films of LCMO (50 nm) are again fully strained. On the other hand PCMO films, 80 nm, are relaxed as misfit dislocations appear at the interface. Using Nano-Beam Electron Diffraction (NBED), the structure of the film is carefully analysed, with particular attention to the interface. As the double exchange is influenced by the Mn valence, Electron Energy Loss Spectroscopy (EELS) is carried out to retrieve information on the Mn Valence across the films: the data reveal clear differences between interface, middle and surface; instead, at the interface results indicate a valence close to 3.25 for all the films, while moving away it changes to 3.5. At the surface, it points always towards 3 as it is possible for oxygen atoms to be easily removed, with Mn valence changing accordingly.


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Calcium silicate (CaSiO₃) phosphor has excellent chemical and thermal stability. It is one of the promising host materials for Photoluminescence and Cathode ray luminescence phosphors. In the present studies. We synthesized orange phosphor of CaSiO₃, Pb, Mn by simple, low-temperature initiated, self propagating and gas producing solution combustion process. The properties of the phosphors were characterized by powder X-ray diffraction method (PXRD) and confirmed the single phase of β-CaSiO₃. The particle size was calculated using Scherrer’s formula and found in the range 30-50nm. The microstructure and morphology was studied using Scanning electron microscopy (SEM). It is observed that, the product is foamy with large voids. The IR spectroscopic data confirms that the peaks at ~ 471, 508, 680, 904 and 964cm⁻¹ are due to β-CaSiO₃. The absorption at 1640 and 3450cm⁻¹ is due to –OH. Photoluminescence (PL) spectra of β-CaSiO₃: Pb, Mn phosphor excited by UV light. The spectrum shows two well resolved emission peaks at 350 and 650nm corresponds to Pb²⁺ and Mn²⁺ (⁴T₁ → ⁴A₂) transitions.

Properties of La0.7Ca0.3MnO₃ and Pr0.5Ca0.5MnO3 Thin Films Deposited on SrTiO3: An Electron Microscopy Study

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Physical properties of La0.7Ca0.3MnO₃ (LCMO) and Pr0.5Ca0.5MnO3 (PCMO) can be altered by growing the film on misfit substrates. In this work we show Transmission
Fabrication and Unique Properties of Ordered Multifunctional Oxide Nanodot Arrays Derived from Template-Assisted PLD

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Well-ordered nanodot arrays of the magnetic spinel ferrite CoFe2O4 (CFO) and the ferroelectric perovskite PbZr0.4Ti0.6O3 (PZT) as well as specifically designed multiferroic CFO/PZT composites have been deposited by pulsed laser deposition (PLD) through porous anodizing alumina (AAO) shallow mask membranes. Using AAO masks, we are able to deposit nanostructures directly by pulsed laser deposition at high temperature up to 550°C, hence enable the further in-situ control of nanodot crystallinity, e.g. epitaxial growth. After deposition and mask removal, well-ordered nanodot arrays of ~30 nm to 300 nm diameter with interdot distances of ~60 nm to 600 nm were obtained.

Magnetic properties were confirmed by well-defined magnetic domain-like contrasts in MFM micrographs as well as by hysteresis loops measured by SQUID. More interestingly, unique complex magnetic domains such as bubble and stripe domains which usually are not found in polycrystalline dots were observed inside the epitaxial CFO spinel oxide dots. They are switchable by applying a perpendicular external field. For PZT, the polarization configurations were investigated using in- and out-of-plane piezoresponse force microscopy (PFM). The presence of an in-plane polarization component in nominally (001)-oriented PZT dots allows the formation of complex core-polarization states, probably facilitated by the vicinity of the morphotropic phase boundary. Core-polarization states may indicate the presence of quasi-toroidal polarization ordering. The experimental observations are compared with a theoretical model, and the fingerprint of a vortex polarization state is found.

When the two materials are combined to form nanostructured composites, they exhibit both magnetic and ferroelectric properties, thus confirming their multiferroicity. For the CFO dots covered by PZT film, the MFM image shows apparent magnetic domains which can be reoriented by applying a external magnetic filed. PFM also clearly shows an out-of-plane piezoelectric response, and the polarization can well be switched by applying an electric field. Further multiferroicity and magnetoelectric couplings are still under investigation.

Surprisingly, we also observe the formation of nanoring structures of PZT in annealed samples deposited through an AAO mask, which may be a result of a surface reconstruction process. If the fabrication parameters are controlled carefully, the ring structures can also be obtained through a direct deposition through the AAO mask without a further post-annealing, likely due to a different deposition and diffusion mechanism.

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A cheaper and faster method has been developed to prepare NiO-YSZ composite from the precursor prepared by mixed oxide method by mixing NiO, Y2O3 and monoclinic ZrO2. The precursors of mNiO-(1-m)YSZ composites of compositions m = 0.2, 0.3, 0.4, 0.5 and 0.6 have been prepared by the mixed oxide method. The YSZ component has Zr0.9Y0.1O1.95 (x = 0.10) composition. The precursor of each composition have been microwave processed at 1400°C for 20 minutes and also conventionally processed at 1400°C for 4 hrs. Subsequently, the microwave processed and the conventionally processed composites have been reduced in 5% H2 + 95% Ar atmosphere to produce NiO-YSZ cermets. Microwave processing leads to the formation of cubic zirconia. This has been confirmed by EDAX.

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Comparison of Electrical Properties of Microwave and Conventionally Processed NiO-YSZ

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The non-existence of monoclinic phase of zirconia in the microwave processed composites of the same composition suggests that during the microwave processing an additional driving force (due to anisothermal heating) works which leads to the faster diffusion of ions and also higher dissolution of NiO in YSZ matrix resulting in the formation of cubic zirconia. This has been confirmed by EDAX.

The study of variations in the bulk electrical conductivity of microwave processed and conventionally processed mNiO-(1-m)YSZ show that two types of conduction mechanism are working in these composites i.e. one due to YSZ phase and another due to NiO phase. The value of the activation energy of the microwave processed and the conventionally processed mNiO-(1-m)YSZ calculated in the temperature range of 400 to 700°C found to be in the range of 0.97 eV to 1.21 eV. The observed value of the activation energy for the microwave processed mNiO-(1-m)YSZ composites in higher temperature range (700 to 900°C) is lower than YSZ and decreases with the increase in the concentration of NiO. The minimum value of the activation energy has been found to be 0.37 eV for mNiO-(1-m)YSZ composite having m=0.6. The decrease in the value of the activation energy indicates that the NiO-YSZ composites are behaving like mixed conductors i.e. ionic and p-type in the higher temperature range. The values of the activation energy of the conventionally processed mNiO-(1-m)YSZ in the higher temperature range are higher than those of the microwave processed composites. Further, the concentration of NiO does not have any pronounced effect on the activation energy in the conventionally processed mNiO-(1-m)YSZ composites, as it has been found in the microwave processed composites.

A01360-02373

Fabrication of PLZT Dielectrics on Base-Metal Foils for Embedded Capacitors

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Development of power electronic devices with higher performance, improved reliability, and smaller size and weight requires the passive components to be embedded within a printed wire board (PWB). This technology could free up surface space, increase device reliability, and minimize electromagnetic interference and inductance loss. Although the technology has primarily received attention for decoupling capacitors in microelectronic applications, it can also be extended to high-power applications at higher voltages, such as plug-in hybrid electric vehicles. However, the integration of high-
permittivity films into PWBs is a difficult task because of the incompatibility in the processing conditions for the different materials involved. Polymer layers in a PWB cannot withstand the high temperatures (600-800°C) required for processing the ceramic film dielectrics to obtain the desired crystalline structures. Development of these crystalline structures becomes extremely challenging at reduced processing temperatures. However, success has been demonstrated through a film-on-foil approach where the ceramic dielectrics are first coated on a thin base metal foil and then crystallized at high temperature. These coated foils can subsequently be embedded into a PWB for power electronic applications. We have deposited high-permittivity lead lanthanum zirconate titanate (PLZT) ceramic dielectric films on base metal foils by a chemical solution deposition technique. Among the many challenges in fabricating PLZT on base metal foils is avoiding a reduction in capacitance density due to formation of a deleterious interfacial oxide and act as a parasitic, low-permittivity, interfacial base metal oxide layer during thermal processing of the PLZT. Two approaches were taken to overcome this problem. In the first approach, a conductive oxide buffer layer was inserted between the PLZT film and the metal foil to hinder formation of the deleterious interfacial oxide and act as a bottom electrode. The second approach employed high-temperature processing under a controlled atmosphere that prevented formation of a deleterious interfacial oxide. By these approaches, we have grown high-quality PLZT films on Ni and Cu foils. We have measured a dielectric constant ≈1300 (at 25°C) and ≈1800 (at 150°C), leakage current density of 6.6 x 10^{-9} A/cm² (at 25°C) and 1.4 x 10^{-8} A/cm² (at 150°C), and breakdown field strength of 2.5 MV/cm on PLZT films deposited on conductive lanthanum nickel oxide-buffered Ni foils. With PLZT deposited directly on Cu foils, we observed dielectric constant ≈1100, dielectric loss ≈0.06, and leakage current density of 7.3 x 10^{-9} A/cm² when measured at room temperature. In this talk, we will discuss the effects of fabrication method, film structure, and film thickness on the dielectric properties of film-on-foil PLZT samples.

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Transparent Thin Film Transistors Using Co-sputtered Amorphous Zn-In-Sn-O Channel

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There is an emerging interest in the field of transparent electronics, wherein electrical devices comprising optically transparent materials. Among these, transparent thin film transistors (TTFTs) are getting more attention since it is a key device for realizing transparent circuits. Transparent amorphous oxide semiconductors (TAOxS) are preferred over polycrystalline ones for active layers in TTFTs because of a low processing temperature and uniformity of device characteristics. Transparent thin film transistors were fabricated by room temperature co-sputtered amorphous zinc indium tin oxide (ZITO) channel layer. ZITO thin films were deposited via a DC–RF co-sputtering technique. A glass substrate coated with 200 nm thick layer of sputtered ITO and 220 nm thick layer of aluminium-titanium oxide (ATO) was used as the substrate. High purity ZnO powder was used as the target for RF source and commercial In2O3:Sn (10 wt% Sn doped, ITO) target was used for DC source in a sputter up configuration. Targets were two inch in diameter and the sputtering power of RF and DC sources were kept at 25 and 60 W respectively. For source and drain contacts, RF sputtered ITO layer was deposited over the channel layer using a shadow mask. The channel width and length were 1200 and 1000 μm, respectively.

X-ray diffraction analysis of the channel layer material shows only halos of amorphous structures. The compositional analysis using energy dispersive x-ray spectroscopy shows Zn/(In+Sn) atomic ratio 0.59 in the channel layer. Optical transmission studies of device shows greater than 70% transmission in the visible region. Devices show typical n-channel transistor action with field effect mobility ~6 cm² V⁻¹ s⁻¹ and on-off ratio 10⁵ without any kind of thermal treatment. Threshold voltage of the device is around 6 V with a subthreshold voltage swing of 0.3 V/dec.

Laser Induced Thermoelectric Voltage from ZnO Thin Films

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Laser induced thermoelectric voltage (LITV) were measured from Zinc oxide epilayer films grown on vicinal cut sapphire and silicon single crystal substrates. Pulsed laser deposition was used to prepare the thin films. The high-crystal-quality ZnO thin films on the lattice-mismatched substrates were achieved by adjusting substrate temperature and the annealing oxygen pressure during the deposition, and proved by Russerford backscattering, X-ray diffraction (XRD), photoluminescence (PL), and Raman Spectroscopy.

Laser induced voltage were observed when irradiating the pulsed KrF excimer laser of 248nm on the films.
The observed signals are identified as the laser induced thermoelectric voltage (LITV), since the signal intensities are directly related to the tilting angle of the substrate orientation. It was found that the highest signals were measured from films grown at a proper annealing oxygen pressure, and substrate temperature. It corresponds to the best crystalline quality of the grown films. All the measurements show that there are induced voltages, which proved that ZnO films grown on vicinal cut substrates are an atomic layer thermopile material. The LITV properties makes ZnO films promising light detector materials.

According to measured LITV signals, Seebeck anisotropy were evaluated, which ranging from 3~12µv/k for ZnO films annealed at different oxygen pressure from 2 to 1×104 Pa. Experimental results indicate that Zinc oxide films can be used at ultraviolet wavelength with higher resistance of laser irradiation damage.

One of the most useful semiconductors, titanium dioxide (TiO$_2$) is known as promising and extensively applicable candidate in photocatalysis and solar cells due to its high photosensitivity, non-toxicity, low cost and stability. In spite of positive attributes, the large band gap energy (3.2 eV) of TiO$_2$ has been raised as a crucial problem since such an inherent property significantly limits more versatile applications under visible light. In order to overcome this specific issue and improve the visible light absorption property, a number of approaches have been proposed, e.g., metal ion loading, covering the surface with dyes, doping nitrogen or sulfur and deposition of noble metals.

Nanostructured noble metals such as Au, Ag, Pt, exhibiting surface plasmon resonance (SPR) properties have been received considerable attention in the past few years. According to recent investigations, SPR properties of noble metals deposited on TiO$_2$ act as electron traps, resulting in retardation of the rate of hole-electron recombination and mediation of band gap of semiconductor materials in hybrid metal/semiconductor nanomaterials. Therefore, noble metal doped TiO$_2$ may show enhanced absorption of visible sunlight and improve the photocatalytic activity under both UV and visible light irradiation.

Among the numerous bottom-up strategies to nanostructures, block copolymer (BCP) self-assembly attracted growing attention as versatile platforms for fabricating functional nanostructures with tailored inner arrangements and composition in terms of structural and dimensional views. By varying volume fraction of components or molecular weights of BCPs, tailored, programmed, and various of self-assembled morphologies can be obtained, ranging from spheres to cylinders. The ability to control the morphologies of BCPs makes these materials suitable for wide application in nanotechnology.

In this presentation, we suggest simple strategies to fabricate visible light active hybrid TiO$_2$ photocatalysts based on self-assembly processes. To generate mesoporous or nanostructures TiO$_2$ containing Pt nanoparticles using BCP self-assembly combined with sol-gel process, we prepared mixture solution of poly(styrene)-block-poly(ethylene oxide) (PS-b-PEO) block copolymer, metal precursor and sol-gel precursor. As metal precursor and sol-gel precursor, platinum(IV) chloride and titanium(IV) isopropoxide (TTIP) were used, respectively. By controlling the mixing protocol, ordered and distinctive arrays of hybrid organic/inorganic nanostructures could be obtained. Along with morphology study of hybrid nanooarrays using atomic force microscopy (AFM) and transmission electron microscope, we investigate into the optical properties of hybrid Pt/TiO$_2$ by UV-visible absorption and photoluminescence spectroscopy and also discuss their localized surface plasmonic resonance enhanced visible light active photocatalytic activities.
their performances as photocatalysts, optical sensors, and elements in organic thin film transistors (OTFTs) owing to localized surface plasmon resonance (LSPR) assisted modification of their inherent properties. Here, we provide various hybrid nanostructures with controlled architecture and composition via coassembly of block copolymers and inorganic precursors, and investigate their optical and photocatalytic properties by atomic force microscopy, transmission electron microscopy, and UV-vis spectroscopy.

Overall procedure for the preparation of metal/semiconductor thin films: We used asymmetric poly(styrene)-block-poly(ethylene oxide) (PS-b-PEO) and poly(styrene)-block-poly(vinyl pyridine) (PS-b-PVP) as templating materials. These diblock copolymers form micelles composed of PS corona and PEO (or PVP) core in toluene. Hydrophilic titania or silica sol-gel precursor containing titanium(IV) isopropoxide (TTIP) or tetraethyl orthosilicate (TEOS) selectively infiltrates into hydrophilic PEO domains of PS-b-PEO and forms titania or silica domains by gelation. By varying the ratio of sol-gel precursor to polymer solution, various surface morphologies were induced. Ag and Au precursors, AgNO₃ and HAuCl₄, are incorporated into PEO block. After the removal of polymer and reduction of metal precursor via UV irradiation, hybrid nanostructures such as Ag/TiO₂, Au/TiO₂ were obtained.

Hierarchical Ag/TiO₂ and Au/TiO₂ nanostructures: Hexagonally packed arrays of Ag or Au nanoparticles (NPs) were fabricated on TiO₂ substrates. Using amphiphilic PS-b-P2VP and PS-b-P4VP as templates, well-ordered Ag and Au nanodots with different areal density could be defined. Increasing density of ordered nanodots, improved photocatalytic degradation of methylene blue (MB) and optical sensing properties were observed.

Mesoporous Ag/TiO₂ thin films: We prepared hybrid mesoporous thin film generated by one-step spin-coating of common solution composed of AgNO₃, titania sol-gel precursor, and PS-b-PEO, followed by UV etching. By increasing the relative ratio of sol-gel precursor to polymer over 40 vol%, mesoporous TiO₂ film was prepared. The molar ratio of AgNO₃ was varied (Ag/EO = 0.5 and 0.7), with fixed amount of sol-gel precursor at 40 vol%. Enhanced efficiency of MB degradation was observed from the film containing 0.7 molar ratio of AgNO₃ compared with one having 0.5.

Effects of dc bias, Uniaxial Compressive Stress, and Annealing on Giant Dielectric Properties of (Li, V)-doped NiO Ceramics

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This work reported the effects of dc bias, uniaxial compressive stress, and annealing on the giant dielectric properties of (Li, V)-doped NiO (LVNO) ceramics prepared by a polymer pyrolysis route. X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive x-ray spectrometer (EDS) were used to characterize the phase composition and microstructure of the LVNO ceramics, respectively. The dielectric measurement was carried out over wide ranges of frequency (10²-10⁶ Hz) and temperature (-50-150°C). Analysis of microstructure and phase composition proved that a core/shell structure could be produced in the LVNO ceramic samples and Ni₃V₂O₈ phase existed as the grain boundary. Through the analyses of complex impedance and SEM-EDS, it was strongly suggested that the LVNO ceramics were electrically inhomogeneous, consisting of semiconducting grains and insulating grain boundaries. Therefore, the observed giant dielectric response in the LVNO ceramics can be ascribed based on an internal barrier layer capacitance (IBLC) effect. Our results illustrated that the dc bias, compressive stress, and annealing had great impacts on the dielectric and related electrical properties of the LVNO ceramics. The resistance of grain boundary decreased with an increase in the applied voltage, whereas the resistance of grain remained constant. Interestingly, we found that the increase in the applied compressive stress caused an increase in the dielectric constant and dielectric loss, indicating to the great influence of compressive stress on the dielectric properties of IBLC ceramic materials. The effect of annealing might be attributed to the variation of the concentration of oxygen vacancies in the bulk ceramics before and after heat treatment.

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Comparison of H$_2$S Sensing Response of Heterostructure Sensor (CuO-SnO$_2$) Prepared by rf sputtering and Pulsed Laser Deposition

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In the present study two sensor structures (i) bare SnO$_2$ film (90nm), and (ii) SnO$_2$ film (90nm) loaded with nanolayer (10nm) of CuO catalyst have been prepared using both rf sputtering and pulsed laser deposition. The response characteristics of sensors for trace level (20 ppm) detection of H$_2$S gas have been investigated in terms of response speed, sensitivity factor and recovery times. Response characteristics for the four sensor devices are compared and the underlying sensing mechanism has been studied in the light of Fermi-level energy control and spillover mechanisms.

The sensor hetero-structure (CuO nanolayer on SnO$_2$ film) prepared by both the techniques exhibit enhanced sensing response in comparison to bare SnO$_2$ film sensor. It is interesting to note that gas response is dependent on the microstructure of the deposited sensing film besides presence of catalytic layer which in turn depends on the growth kinetics. PLD grown hetero-structure sensor exhibits good sensing response characteristics with higher sensitivity ($\sim 2.3 \times 10^3$) and low operating temperature of 100°C. The enhanced response of PLD grown samples is attributed to the growth of sensing layer at relatively higher oxygen pressure (50 mTorr). AFM studies further indicate a rough and porous microstructure of PLD deposited films that in turn offers a greater surface to volume ratio. In general, the obtained results show the potential application of PLD grown samples for trace level detection of H$_2$S gas with lesser power consumption.

Sintering and Electrical Properties of Ni-doped ZnO

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Polycrystalline ZnO has numerous applications in such diverse areas as UV light-emitters, piezoelectric transducers, varistors, gas sensors, phosphors, and transparent conducting films. It is important to evaluate the defect levels and grain boundary characteristics in ZnO for a number of applications.

In this study, we investigated the effects of Ni dopants on the sintering properties and the bulk trap and interface state levels of ZnO using admittance and impedance-modulus spectroscopy (AS, IS and MS). For the identification of the bulk trap levels, it is useful to examine the zero-biased admittance spectroscopy as a function of frequency and temperature. Impedance and electric modulus spectroscopy is a powerful technique to characterize grain boundaries of ceramic materials as well. We prepared ZnO doped with 0.1~3.0 at% NiO sintered at 1000~1300°C and measured its electrical characteristics. The roles of Ni dopant on the ZnO have been discussed in relation to interfacial and bulk traps.

As a result, three kinds of bulk defect trap levels were found below the conduction band edge of ZnO in Ni-doped ZnO and automotive sectors. Electrochromic doping in thermochromic coating increases the transmission of solar energy which find applications in smart windows, sensors and photonic devices. In the present study attempts were made to fabricate nanocomposite thin films of VO$_2$-WO$_3$ by pulsed laser deposition (PLD) technique. The influence of varying VO$_2$ composition in the range from x = 0.0 to x = 0.5 on structural, optical and electrical properties of the films has been systematically investigated.

X-ray diffraction studies reveal the single crystalline monoclinic VO$_2$ phase (m-VO$_2$) up to 10% of VO$_2$ composition whereas hexagonal WO$_3$ phase (h- WO$_3$) was observed at higher WO$_3$ compositions (x = 0.5). Both m-VO$_2$ as well as h- WO$_3$ phases were present at the intermediate VO$_2$ compositions (0.2≤x≤0.4). Optical transmittance spectra of the films showed blue shift in the absorption edge with increase in WO$_3$ compositions. The optical transmittance of the films was found to increase with increase in WO$_3$ compositions. Electrical studies indicate significant variation in transition temperature, width of the hysteresis, and shape of the hysteresis loop. Cyclic Voltammetry measurements were performed on pure WO$_3$ and VO$_2$-WO$_3$ thin films. A direct correlation between V/W ratio and structure-property relationship was established.
(ZN). ZN system did not form definite double Schottky barrier due to the p-type nature of NiO. Low-level doping of Ni (≤1.0 at%) decreased the conductance, which was increased with high-level doping over 1.0 at%. Three kinds of bulk trap level were 0.10~0.14 eV, 0.21~0.29 eV, and 0.31~0.35 eV, and one interface state level 1.03 eV. Also we measured the resistance and capacitance of grain boundaries with temperature using impedance-modulus spectroscopy. It will be discussed about the stability and homogeneity of grain boundaries using distribution parameter (α) simulated with the $Z(T)^{logf}$ plots in ZN system.

A01430-02507

Synthesis of ZnO/α-NPD:F\textsubscript{4}-TCNQ based Inorganic-organic Hybrid Junction: Effect of Doping of Organic Layer on the Diode like Characteristics

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The realization of ZnO based p-n homojunction is hampered due to lack of efficient and high quality p-type ZnO. An alternative approach can be the preparation of heterojunction by using n-type ZnO and organic (π-conjugated) p-type semiconducting materials. The choice of organic over inorganic is due to the fact that devices and sensors based on the use of π-conjugated conducting organic materials are considered to be the backbone of the next generation of cheap and disposable electronic devices.

In the present work, we report the synthesis and characterization of ZnO/N,N’-diphenyl-N,N’-bis(1-naphthyl)-1,1’biphenyl-4,4diamine (α-NPD) based inorganic-organic hybrid junction. We have investigated the effect of organic layer doping on the diode like characteristics of the prepared device. The ZnO thin film was deposited on the ITO coated glass substrate by rf magnetron sputtering. The ZnO thin film was characterized by X-ray diffraction and atomic force microscopy. The pure and 2,3,5,6-tetrafluoro-7,7’,8,8’-tetracyano-quinodimethane (F\textsubscript{4}-TCNQ) doped α-NPD (~ 300nm) films were deposited on the ZnO film by vacuum thermal evaporation followed by the deposition of Au contacts. Following the same procedure, we have prepared three devices with pure α-NPD, α-NPD:0.4% F\textsubscript{4}-TCQ and α-NPD:0.8% F\textsubscript{4}-TCQ organic layer. The electrical characterization was carried out by measuring current-voltage (I-V) characteristics of three devices. The diode parameters viz. barrier height, ideality factor and rectification ratio were calculated from the I-V plots. The improvement of diode parameters i.e. increase in rectification ratio and the decrease in barrier height as well as in ideality factor has been observed with doping. The improvement of the diode parameters has been attributed to the modification of interface. The current is also found to increase with the increase in doping concentration. This increase has been attributed to the lowering of interface barrier by interface dipole manipulation.

A01450-02526

Physics of Functional Oxide Interfaces

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Complex oxides with the perovskite structure can be found in almost every possible solid physical ground state such as superconductors, metals, insulators, ferromagnets, ferroelectrics, multiferroics and more. Many of them are composed of transition metal oxides showing strong electron correlations. This can lead to a strong competition between lattice-, orbital-, charge- and spin ordering, all of them at similar energy scales. A special topic in this field is to combine materials with different ground states (functionalities) such as superconductivity and ferromagnetism in the form of thin films and investigate their mutual interaction. At interfaces (substrate/film and filmA/filmB) the growth properties of complex oxides are governed by the constraint of preserving charge neutrality and stoichiometry imposed by the ionic character of the constituents. In this context the self-doping of interfaces - resulting from the modified charge transfer - could turn the interface between two insulators into a metal. Changes in the bonding characteristics at the interface may affect the spin properties because of the strong interaction between orbital and spin degrees of freedom. Furthermore, electronic reconstruction at the interface and broken lattice symmetry modifies orbital physics at the interface. Consequently the physical properties of an interface dominated sample will be different from those of its constituents. This concept opens a possibility for interface engineering and unexpected phenomena, which cannot be understood in terms of conventional band pictures, may thus appear. The combination of a ferromagnet and a superconductor is regarded as a prototype for this concept since these two ordering principles are antagonistic by nature and can not coexist in a homogeneous system. In this contribution the field will briefly be reviewed and recent results for charge transfer, orbital reconstruction and the different length scales for the interaction for YBCO/LCMO heterostructures will be given. In the contribution special emphasis is given to the open problems of oxide interface physics such as oxygen distribution, band bending issues, interfacial stoichiometry control as well as the relevant measurement techniques.
Growth and Characterization of ZnMgO Films and ZnO/ZnMgO Multi Layers by RF Magnetron Sputtering

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Single crystal ZnMgO films were grown on Al₂O₃ and ZnO substrates by r.f. magnetron sputtering at 850°C in pure oxygen ambient, when the Mg concentration was Zn₀.₈₇Mg₀.₁₃O or lower in the case of ZnMgO/Al₂O₃ and when that was Zn₀.₆₅Mg₀.₃₅O or lower in the case of ZnMgO/ZnO.

Energy of PL UV peak of the ZnMgO films increased with addition of Mg, from 3.₃₂eV (ZnO) to 3.₅₁eV (Zn₀.₈₇Mg₀.₁₃O) and 3.₇₀eV (Zn₀.₆₅Mg₀.₃₅O).

FWHM values of high resolution X-ray diffraction (0002) peak were 620 arcsec and 203 arcsec for ZnMgO/Al₂O₃ and for ZnMgO/ZnO, respectively, FWHM of the PL UV peaks of the films were 140~150 meV, and the surface roughness of the films ranged between 0.₂₈ nm and 0.₄₇ nm.

ZnO/ZnMgO multi layers were also grown. Initial results indicated that the PL UV intensity of the layer and the EL intensity of the ZnO/ZnMgO/p-GaN p-n diode are much higher than the ZnO counterpart. Detailed results of the characterization of the multilayers will be discussed in the presentation.

Effect of Lithium Doping on Zinc Oxide Thin Films Properties Prepared by Ultrasonic Spray Pyrolysis

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Lithium-doped ZnO thin films were prepared by low cost ultrasonic spray pyrolysis technique. Lithium nitrate and zinc acetate dehydrate were used as Li and Zn precursor respectively with Li to Zn atomic ratio ranging from 2% to 15%. Processing parameters were optimized by controlling precursor pH and substrate temperature at around 350°C. A very thin layer of un-doped ZnO was used as buffer layer on glass and silicon substrates. Other Li doped ZnO films were also grown without buffer layer under identical deposition condition for comparison purpose.

XPS, XRD, UV-VIS characterization and Hall measurement were used to understand the effects of doping on the structural, optical and electrical properties of ZnO thin films. SEM was employed to examine film morphology and determine its thickness. Post deposition annealing in air at 300°C and 400°C for 2 hours each were attempted to realize high p-type conductivity. Hall Effect measurement was conducted after each annealing treatment. To study Li distribution on ZnO layer, XPS depth profiling was performed both on as deposited and selected annealed sample. Furthermore, observation on the energy shift was conducted to investigate bonding configuration difference between Li atoms on the surface and those that incorporated into the bulk.

Most of the Li doped ZnO thin films deposited on glass substrates showed high transparency over visible spectrum region. XRD analysis revealed that as deposited films with and without buffer layer on Si substrate showed (002) reflection different from those prepared on glass substrates, suggesting that the buffer layer enhanced the degree of c-axis orientation which is of importance for piezoelectric and ferroelectric of the devices applications. Despite the fact that lithium is theoretically good shallow acceptors, series of post deposition air annealing were proven to play significant role in activation of Li acceptors resulting in higher p-type conductivity.

Nanoscale Modification of Oxide and Nitride Film Surface by Energetic Ions

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We report here nanoscale modification of copper oxide (CuO), zinc oxide (ZnO) and copper nitride (Cu₃N) films surfaces irradiated by 200 MeV Au⁴⁺ ions at various fluences. The surface morphology of all the films is examined by atomic force microscope (AFM) in detail. The results emerge out from these studies are (i) enhancement of average grain size associated with significant change in resistivity in CuO films, (ii) morphological changes and mass flow in ZnO film surface and (iii) nanoscale line-like features in Cu₃N film surface. The results are understood on the basis of thermal spike mechanism and viscoelastic model of ion-solid interaction.
Effect of SnO₂ Addition on Structural and Optical Properties of ZnO Thin Films

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Zinc-Tin-Oxide (ZTO) thin films were deposited on glass substrate with varying concentration at room temperature by flash evaporation technique. XRD spectra show the polycrystalline cubic nature. The uniformity of the deposited films was checked by optical micrograph. The surface composition and oxidation state are analyzed by X-ray photoelectron spectroscopy. The core level and valance band spectra were monitored to observe the chemical and electronic changes as a function of nitrogen ion beam energy. It is observed that Sn (3d) core level peak shift towards the higher binding energy as the percentage composition of tin oxide increases. Further the optical characteristics of films were also studied by taking absorption spectra. It was found that as the concentration of tin oxide increases, the optical band gap decreases.

Hydrogen Sensors based on Self-Organized Titania Nanotubes Fabricated by Anodization Technique

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In this work, nanotubular titania hydrogen sensors were fabricated by anodization technique. Experimentally, a titanium thin film was firstly deposited on quartz by the RF sputtering at room temperature and followed by anodizing in a NH₄F/ethylene glycol electrolyte. Subsequently, the sample was annealed in air at 500°C for 2 hour. In the anodization process, a proper amount of phosphoric acid is added for adjusting the pH value of electrolyte. Effects of anodization conditions such as the applied voltage, anodizing time and pH value of electrolyte on the properties of resulting TiO₂ nanotubes were investigated. Moreover, the hydrogen sensing characteristics of TiO₂ nanotubes were studied under temperature ranges of 303-473 K and hydrogen concentrations from 5 ppm to 1% H₂/N₂. The experimental results found that highly ordered TiO₂ nanotube arrays could be obtained at an optimal pH of 3.92. The length of nanotubes is from 600 nm to 1100 nm, and the pore diameter is about several tens nanometer, depending on the applied voltage and anodizing time. From current-voltage characteristics, the TiO₂ nanotubes showed the behavior of resistive-type sensor. From the result of sensing measurements, TiO₂ nanotubes demonstrated excellent sensing performances with high sensitivity, low detection limit and wide detection range. It was found that the sensitivity of the prepared TiO₂ nanotubes reached to 10⁴ at 303 K and 1% H₂/N₂. Even at extremely low concentration, e.g., 5 ppm H₂/N₂, an obvious variation could be found. At small applied bias, the sensitivity increased with the increase of operating temperature.

VO₂ Thin Films: Solution Processing and Thermochromic Properties

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Energy saving has become a hot topic and a great challenge in recent years due to the world-wide energy crisis and environmental problems. Thermochromic windows show advantages over electrochromic windows because of their simple structures, visible transparency even at an infrared switched state and direct response to environmental temperature. Among all thermochromic materials, monoclinic/rutile (M/R) phase vanadium dioxide (VO₂) undergoes a fully reversible metal-semiconductor transition (MST) at 68°C that is the closest to room temperature. Moreover, the phase transition temperature can be further reduced to near room temperature by doping. The above features make VO₂ the most promising material for thermochromic applications.

VO₂ films are usually produced by vapor-based processes. We present in this talk the synthesis and property study of VO₂ by a solution process. Thermochromic VO₂ films with high visible transmittance and significant infrared switching efficiencies have been synthesized via the newly developed polymer-assisted deposition method. The design and optimization details as well as the key issues of the method are described and discussed. It is shown that the VO₂ films are consisted of VO₂ nanoparticles which were single monoclinic (M) phases and strongly oriented with the (011) planes parallel to the substrate. The spectrophotometer analysis indicated that different optical property films can be prepared by tailoring the film thickness via changing polymers and vanadium concentration, along with the metal-semiconductor transition (MST) temperature, which was reduced to ambient temperature by W doping. The current research results show that the developed solution-based process is a key step to achieve practical applications of this material.
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A01515-02638

Fabrication and Properties of Hybrid ZnO/Au Nanoparticle Superstructures by Stepwise Buildup of Self-Assembled Block Copolymer Templates
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Nanostructured zinc oxide (ZnO) materials having direct band gap (3.3 eV) and large exciton binding energy (60 meV) have recently attracted high interest for versatile functions. Among these nanostructures, ZnO-based heterostructures with desired composition and morphology are promising candidates for potential applications in optoelectronic devices, ultraviolet (UV) laser devices, chemical sensors, solar cells, and photocatalysts. Especially ZnO nanostructures incorporated with noble metals such as gold (Au) or silver (Ag) has being attracting great attention owing to their enhanced, unprecedented properties coupled with localized surface plasmon resonance (LSPR) of metals. Numerous routes have been proposed to generate ZnO/metal hybrid nanostructures. In particular, self-assembled block copolymer (BCP), consisted of two chemically different polymers covalently attached at one end, offers the potential to form nanoscale periodic patterns and has been used as templates to fabricate highly ordered arrays of inorganic materials.

Herein we introduce a new class of 3-D superstructures composed of multilayer hybrid ZnO/Au nanoparticles with hexagonally packed arrays using polystyrene-block-poly(4-vinyl pyridine) (PS-b-P4VP) inverse micelles as templates. Molar equivalents of the inorganic precursors per pyridine unit were added to the BCP micellar solution. ZnCl₂ and HAuCl₄ were used as ZnO and Au precursors, respectively. The BCP micellar film loaded with inorganic precursor was produced simply by spin coating. Based on the first monolayer film, 3-D stacked multilayer was constructed by consecutive adsorption of BCP micelle containing inorganic precursor by spin coating onto the underlying layer. In between the deposition of each layer, the film was treated with O₂ plasma to eliminate organic polymer, leaving behind well-ordered NP arrays. In this way, multilayer thin films with various inner structures and components were obtained. Such multilayer nanostructure may provide high performance of inorganic material compared with monolayer analog. Utilizing such 3-D periodic nanostructures, we primarily investigated their photocatalytic activity and optical sensing. The multilayer nanostructure of hybrid ZnO/Au system showed advanced photocatalytic efficiency by facilitating charge separation of photogenerated electron-hole pair compared with neat ZnO. LSPR based sensing properties of these hybrid structures were explored using NaCl aqueous solutions with varying concentrations. When the multilayer thin film was immersed in NaCl aqueous solution with systematically...
controlled refractive indices, enhanced sensitivity of the hybrid ZnO/Au nanostructure was clearly observed in terms of the shift of LSPR band in response to the change of the refractive index of surrounding medium. The overall process for the formation of multilayer nanostructure was studied by X-ray refractivity, UV-Vis. spectroscopy, and atomic force microscopy.

A01518-02646
Charge Order and Its Destruction Effects on Magnetocaloric Properties of Manganites
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Colossal magnetoresistance (CMR) manganites also exhibit a large magnetic entropy change (ΔSm) or magnetocaloric effect (MCE) around magnetic phase transitions. While extensive studies have been reported on the MCE effect in ferromagnetic manganites near the Curie temperature, little work have been done on the MCE effect in half-doped manganites which are charge-orbital ordered antiferromagnetic insulators at low temperature because the magnetic entropy is expected to be smaller in the later compounds. The purpose of the present is to investigate how magnetic and magnetocaloric properties of the charge-orbital ordered antiferromagnetic La0.5Ca0.5MnO3 is affected by Ni substitution at the Mn sites. We will show that the antiferromagnetic charge ordered state collapses with only 2% of Ni substitution and transforms into long range ferromagnetic for x= 0.04 in La0.5Ca0.5-xMnxNi0.5O3. Further increase in Ni reduces the saturation magnetization. While the parent compound shows both negative (ΔSm = -3 J/kgK) and positive magnetocaloric (ΔSm = +6.5 J/kgK) effects at the ferromagnetic and antiferromagnetic magnetic transitions, respectively, the Ni-doped compounds show only negative magnetic caloric effect. The ΔSm is maximum for x= 0.04 (ΔSm = -3.9 J/kgK) and decreases with further increase in x. The parent and 4% Ni doped samples show relative cooling power (RCP) of -126 J/kg at TcO and 235 J/kg at TC, under 5 T field, respectively. These ΔSm and RCP values at TcO are much larger than other 50% Ca-doped manganites. The present studies indicate that 4% Ni doping is optimal for La0.5Ca0.5-xMnxNi0.5O3 compound to obtain maximum ferromagnetic phase. The results are interpreted due to destruction of charge ordering and induction of ferromagnetic order at low Ni doping (x< 0.04) and weakening of ferromagnetic interactions and formation of glass cluster behavior for x > 0.04.

A01531-02680
Quantum Effects in Group IV Nanostructures Embedded into Crystalline Rare Earth Oxides on Silicon Substrates
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The unprecedented progress in nanoscale research in semiconductor materials has opened up many fascinating phenomena, which could have never been realized in macroscopic materials. Among many promising applications, such low dimensional semiconductor structures may be used in next generation thin film solar cells application. Especially, quantum dots (QDs) have the potential to greatly increase the photon conversion efficiency via production of multiple excitons from single photon. This is possible because the quantization of energy levels in quantum dots produces different effects; among them are enhanced Auger processes and coulomb coupling between the charge carriers confined into QDs. In general, these low dimensional quantum structures including QDs and quantum well (QW) are constrained in all three and one dimension due to the small length scale, thus the physical properties of such systems are dominated by quantum effect.

In this presentation, we will demonstrate a novel approach to grow Si and Ge nanostructures embedded into crystalline rare earth oxides using molecular beam epitaxy (MBE). By efficiently exploiting the growth kinetics during MBE one could create nanostructures exhibiting multiple dimensions. The nanostructures could either be quantum dots (QDs) that are confined in three dimensions or quantum wells (QWs) of Si (Ge) (confined in one dimension) with epitaxial Gd2O3 as barrier layers. Such QDs having varying sizes and QWs of different width embedded into crystalline rare earth oxide could be a smart way to engineer the band structure of Si(Ge) and could serve as multiple band gap materials in a single device, akin to the multijunction solar cell.

The growth and physical properties of Si(Ge) QDs and QWs were investigated by various characterization techniques such as reflection high-energy electron diffraction, x-ray diffraction, atomic force and transmission electron microscopy, room temperature photoluminescence, capacitance-voltage (C-V) and current-voltage (I-V) measurements. Stacks of oxide layers with embedded QDs of different sizes were successfully grown on Si substrates. The spatial (both vertical and horizontal) distribution of Si-
QDs was controlled by carefully monitoring the deposition parameters during MBE growth.

The photoluminescence measurements on QDs demonstrate an intense visible peak even at room temperature. The estimated band gap was found to be enlarged with respect to bulk material due to quantum confinement effect, which was established by comparing the experimental results with theoretical values. As an example, the maximum band gap of Si-QD with size about 3.4 nm was found be 1.9 eV. In further studies, the samples containing QDs of different sizes displayed PL peaks at different position inferring that luminescence is due to confinement in different sized-QDs. Such interesting properties of QDs make them a strong candidate for next generation solar cell application where the size dependent spectral response could be useful for absorption of the wider solar spectrum.

Furthermore, C-V and I-V characteristics were measured at temperature ranging from 300K to 77K. The low temperature I-V from QW exhibited negative differential resistance. Therefore, the present results could make such low dimensional oxide/semiconductor heterostructure also an attractive candidate for resonant tunneling devices and hot carrier solar cell contacts.

A01549-02699

An In-situ Study of Phase Development and the Properties of BiFeO₃ at High Pressure

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The phase development and the structure and electrical properties of multiferroic BiFeO₃ have been measured in-situ at high pressures and temperatures using (i) a diamond anvil cell and (ii) a multi-anvil high volume press at the Bayreuth Geoinstitute and Daresbury Laboratory respectively. BiFeO₃ powders and samples were prepared by the mixed oxide route and sintered in a tube furnace at 800°C for four hours. For the phase development work the powders were placed in a diamond anvil cell and heated to temperatures in excess of 800°C as a function of temperature. For the phase development work the powders were placed in a diamond anvil cell and heated to temperatures in excess of 800°C as a function of temperature. The complete assembly was subjected to pressures up to 6 GPa and temperatures up to 950°C. A synchrotron beam passing through the cell enabled the structure to be monitored as a function of temperature and pressure. Electrical conductivity and dielectric measurements were performed in-situ.

The refined unit cell parameter a of the rhombohedral BiFeO₃ structure decreased from 5.63 Å to 5.41 Å with increasing pressure. With increasing pressure to 3GPa there was a uniform reduction in electrical resistance and gradual increase in capacitance. At 3GPa, the initiation of a phase transition was identified from rhombohedral to primitive tetragonal. This coincided with large increases in the capacitance and quality factor. This transition continued gradually with increasing pressure until at least 5GPa. Upon reduction of the applied pressure, the rhombohedral to tetragonal transition was found to reverse.

A01578-03360

Process, Phase Stability and Ionic Conductivity of Multidoped Lanthanum Strontium Ferrite Perovskite Oxides

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Mixed ionic-electronic conductors (MIECs) with the ABO₃ formula have been intensively investigated as they are indispensable to solid oxide fuel cell, air separation and catalytic partial oxidation of natural gas. The oxygen permeability, the chemical/phase stabilities and mechanical properties of the membrane under the environment of a reduced pressure or high oxygen partial pressure gradients are the major features that are concerned by practical applications. All of these properties are strongly dependent on the membrane material and composite and microstructure of the membrane.

In the present work, two kinds of multi-doped lanthanum strontium ferrite oxides that have perovskite structures La₀.₈Sr₀.₁Mg₀.₀₁O₃₋δ (LSFCCMO) and La₀.₈Sr₀.₁Fe₀.₆Co₀.₂₅Cr₀.₂₅O₃₋δ (LSFCCO) have been synthesized by pechini method. The densification and microstructure of both oxides were studied to examine the effects of calcination temperature and Mg doping on ionic conductivity. The reduction behavior of LSFCCMO in different reduction atmosphere (H₂/He and CH₄/He) was investigated. The oxygen ionic conductivity was also measured by electrochemical impedance spectroscopy (EIS).
Pure cubic perovskite structure is formed when both oxides are calcined at 900 °C. Crystal grains of perovskite phase grow with increasing calcination temperatures. The perovskite structure was stable after calcined at 1350 °C for 4 h without any second phase or decomposition. High density membranes (> 92% of the theoretical density) were obtained for both materials when the calcination temperature was above 1200 °C. In addition, the Mg doped material gains a higher density and lower grain size compared with the Mg free sample. The phase stability of doped material gains a higher density and lower grain size clearly shows multiple phase transitions at 212°C phase pure. Study of temperature dependent dielectric X-ray diffraction pattern reveals that the samples are ceramics were prepared by chemical sol-gel technique. The 282.

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Effect of V Doping on the Dielectric Properties of ZnO
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In recent years, transition-metal (TM) ions doped ZnO have been intensively studied for the growth of diluted magnetic semiconductors (DMS) and many groups have reported room temperature ferromagnetic behavior in TM ion doped ZnO. ZnO has a wurtzite hexagonal crystal structure, in which both Zn and O atoms are tetrahedrally coordinated to each other and are equivalent in position. Interestingly, structural phase transition was observed in doped IV-VI binary semiconductors such as Pb, Ge, Te, Sb, Ge, Te, Ge, Zn, Te, etc. This ferroelectric behavior was attributed to the off-centered position of dopant, due to large difference in ionic radii between host and dopant ions. In other words, the dopant atoms can occupy off-centered positions, forming permanent electric dipoles and thereby resulting in ferroelectric transitions. Hence, ferroelectric transitions were reported for Li and Mg doped ZnO. Here we present synthesis and dielectric studies of V doped ZnO ceramics. V doped ZnO (Zn, V, O, x=0.01 and 0.03) ceramics were prepared by chemical sol-gel technique. X-ray diffraction pattern reveals that the samples are phase pure. Study of temperature dependent dielectric constant clearly shows multiple phase transitions at 212°C and 343°C for 1 mol% V doped ZnO and at 266°C and 407°C for 3 mol % doped ZnO. This structural transition can be explained as follows: ionic radii of dopant V ion (0.54Å) is less than that of host Zn ion (0.74 Å), hence the V ion moves to an off-centered position, there by creating permanent electric dipoles, which leads to the ferroelectric transitions. Frequency dependent dielectric constant of the samples was also studied. All the above results will be discussed in detail.

Effect of Sm Doping on the Ionic Conductivity of Ceria, CeO₂
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Ceria, CeO₂ is a potential candidate for intermediate and low temperature solid electrolyte fuel cells (ITSEFC and LTSEFC) because of its high ionic conductivity. Effect of Sm doping in CeO₂ in a typical composition Ce₀.₉₅ Sm₀.₁₅ O₁.₉₃₅ has been studied on the ionic conductivity as a function of temperature by impedance measurements. Formation of single phase has been confirmed using powder X-ray diffraction. Total conductivity as a function of temperature has been studied in the temperature range 200-600°C. Activation energy of conduction has been found to be 0.92 eV.

Modeling of Imprint in Hysteresis Loop of Ferroelectric Thin Films with Top and Bottom Interface Layers
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The imprint of a ferroelectric thin film capacitor is studied using an improved model consisting of two nonswitching thin interface layers near the top and bottom electrodes. The difference in electrical conductivity between the two interface layers induces voltage offset and deformation behaviors in hysteresis loops. Size dependence of shift effect of Bi₁₅ Nd₅ Ti₆ O₁₂ thin film is explained qualitatively by taking into account the thickness ratio of the interface layer and the bulk film. Various shifts and anomorphic shapes with different electrodes and processes have
been effectively reproduced through changing the layer conductivities. The simulated shifted hysteresis loops agree well with the experiment. Theoretical prediction based on this approach may provide a method to reduce imprint failure.

**Band Offset Control at Manganite-titanate Heterojunctions**

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With the rapidly increasing number of studies demonstrating unique device operations in oxide heterojunctions, understanding of the band alignment formation mechanisms and techniques to control band offsets have become more important. Band offset defined as the energy level discontinuity between two materials at their juncture is one of the crucial parameters governing the electronic structure across a heterointerface. Given the additional degrees of freedom in valence, magnetic order, and lattice polarizability in perovskites, understanding of the band alignment offsets have become more important. Band offset defined as the energy level discontinuity between two materials at their juncture is one of the crucial parameters governing the electronic structure across a heterointerface. Given the additional degrees of freedom in valence, magnetic order, and lattice polarizability in perovskites, understanding of the band alignment offsets have become more important. Band offset defined as the energy level discontinuity between two materials at their juncture is one of the crucial parameters governing the electronic structure across a heterointerface.

In this talk, the main focus of our study is on the band offsets at Schottky interfaces between \( \text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3 \) (LSMO) and Nb-doped \( \text{SrTiO}_3 \) (100) (NSTO), in which two different ways to manipulate the barrier heights are presented. The first is the barrier height tuning by external magnetic field in \( \text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3 /\text{NSTO} \) heterojunctions, reflecting strong magneto-orbital coupling in LSMO in proximity to the metal-insulator transition. By application of magnetic field, we have observed evidence indicating a reduction in the barrier height by \( \sim 10 \text{ meV} / \text{T} \) which is much larger in magnitude than expected from the Zeeman effect. The second is the control of barrier heights by microscopic control of the interface termination, and hence the interface dipole arising from screening of the polarity mismatch at the interface. This technique enables tuning of the barrier height without modifying the physical properties of the metal overlayer, expanding the capabilities to engineer interface band alignments.

These two cases demonstrate the significant role of the interface chemical bonding on the macroscopic behavior of the rectifying junction. In addition to conventional electrical probes, we have used internal photoemission spectroscopy to study the barrier heights. This technique allows direct measurement of the barrier height under applied magnetic field, and enables equilibrium measurements without application of a bias voltage, suitable for exploring heterojunctions between magnetic materials or semiconductors with non-linear dielectric response.

**A 3D Model to Describe Structural Transition in \( \text{YBa}_2\text{Cu}_3\text{O}_{7-\delta} \): Temperature Dependence Elastic Moduli**

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A 3D ‘spring-defect’ model for \( \text{YBa}_2\text{Cu}_3\text{O}_{7-\delta} \) until cell has been developed by us earlier for describing connection between elastic distortion and oxygen ordering; and the concomitant structural phase transition from tetragonal to orthorhombic at \( T_c \) (\( \sim 900\text{K} \)), in the high – \( T_c \) (\( \sim 90\text{K} \)), superconductor YBCO. The presence or absence of the oxygen is modeled in terms of a pseudo spin. The interaction between oxygen atoms is recovered after eliminating the lattice degrees of freedom, which is long ranged. The proposed model provides a microscopic basis to describe ferroelasticity exhibited in the orthorhombic phase of the superconducting system.

In this work the effective Ising interaction between defects mediated by host Cu atoms derived earlier recasted in equivalent form of strain-strain interactions, which imminently suitable to describe paraelastic (tetragonal) to ferroelastic (orthorhombic) phase transition and investigate the spontaneous development of strain as the system undergoes a structural phase transition. The coupling between spin and lattice degree of freedom allows for systematic study of ferroelasticity.

Ferroelasticity is shown to be a consequence of interaction between elastic (Zenor) dipoles formed around each oxygen defect and the interaction between the strain fields around interstitial oxygen defects is found to favor a situation in which the latter preferentially occupy one of the three possible sub-lattices in the orthorhombic phase.

Mean field approximation, in the statistical mechanical frame work, of the underlying Ising model yields sub-lattice magnetizations. A suitable coordination of these is the relevant order parameter staggered magnetization and also provides a reasonable estimate of the transition temperature and static compliance of the system.

The significant and original feature of our treatment is not only a derivation of strain-strain interaction, but also an explicit calculation of every term of the strain-strain...
interaction including the strain dipole tensor (the so called $\lambda$-tensor) associated with each defect and all the phase transformation properties associated with the paraelastic to ferroelastic transition from the basic microscopic model parameters.

The question of kinetics is also of great importance, especially in relation to the hopping diffusion of oxygen in YBa$_2$Cu$_3$O$_{7-\delta}$ high–$T_c$ superconductor. The study of all relaxational characteristics associated with frequency dependent compliance, internal frictions etc are left for future publication.

**A01635-02833**

**Seebeck and Magnetoresistive Effects of In-doped ZnO Thin Films**

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In-doped ZnO (IZO) films with 0~5at% In content were deposited on glass substrates by RF magnetron sputtering. The Seebeck (thermoelectric) and magnetoresistive properties of the IZO films were investigated. It shows that there is a striking Seebeck effect in the IZO thin films, and the absolute value of thermo-electromotive force increase linearly with increasing differential temperature. The thermoelectromotive forces are negative, the Seebeck coefficient is about $-256$, $-176$, $-127$ and $-75$ $\mu V/K$ for samples deposited from targets with In content 0, 1at%, 2at%, 5at%, respectively. The highest value of power factor ($1.92 \times 10^{-5} W/K^2 m$) was obtained at room temperature for the film prepared from the target of 2at% In content. The electrical properties had been investigated by Van der Pauw method. The results show that GZO thin films are polycrystalline with a preferred (002) orientation.

Highly conductive and transparent Ga-doped ZnO (GZO) thin films with Ga doping concentration (Ga/(Ga+Zn)=3at%) were prepared on glass substrate by RF magnetron sputtering at different substrate temperature. Then the films are annealed in N$_2$ or air for 1 hr at at 200, 300 and 400°C, respectively. The structural, optical and thermoelectric properties of as-deposited and annealed GZO films were characterized and analyzed by XRD, SEM and ultraviolet – visible and infrared light spectrophotometer. The electrical properties had been investigated by Van der Pauw method. The results show that GZO thin films are polycrystalline with a preferred (002) orientation. The crystallinity of ZnO films improves after annealing treatment. The resistivities of films annealed in both N$_2$ and the air are lager than the as-deposited, but the resistivity increase in N$_2$ is smaller that that in air atmosphere, while in the same atmosphere(N$_2$), the resistance of annealed samples increase with the annealing temperature. The measurements show that there is a striking Seebeck effect in the GZO thin films, and the thermoelectromotive force is linearly increased with temperature difference, and the absolute vale of Seebeck coefficient of annealed samples are larger than the as-doped, and increase with the annealing temperature. The largest Seebeck coefficient is $-101.15 \mu V/K$ for the samples deposited at the substrate temperature of 200°C and annealed at N$_2$400°C,1h. Then, the influence of annealing on the electrical and thermoelectric properties of GZO thin films was analyzed theoretically.
High-temperature Oxidation of Ti$_3$Al$_{0.7}$Si$_{0.3}$C$_2$ Compounds at 900 and 1000°C in Air

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The nanolaminated ternary carbides, Ti$_3$AlC$_2$ and Ti$_3$SiC$_2$, have a hexagonal structure, and similarly exhibit the attractive metallic and ceramic properties. Like metals, they have excellent electrical and thermal conductivities, high toughness, high fatigue-crack growth threshold, low hardness, and high-thermal shock resistance. Like ceramics, they display excellent chemical resistance, high Young’s modulus, high temperature strength and high melting points. In addition, they are machineable with conventional tools without lubricants, unlike conventional carbides. In order to utilize them as high-temperature structural components, it is necessary to study their high-temperature oxidation resistance.

The oxidation behavior of Ti$_3$SiC$_2$ and Ti$_3$AlC$_2$ was extensively studied before. However, the oxidation behavior of Ti$_3$Al$_{0.7}$Si$_{0.3}$C$_2$, which is the solid solution of Ti$_3$AlC$_2$ and Ti$_3$SiC$_2$, was not adequately studied. In this study, Ti$_3$Al$_{0.7}$Si$_{0.3}$C$_2$ was synthesized via the powder metallurgical route by partially substituting Si atoms into the Al sites in Ti$_3$AlC$_2$. This strengthened Ti$_3$AlC$_2$ through solid solution hardening, without deteriorating the oxidation resistance.

In this study, dense, monolithic Ti$_3$Al$_{0.7}$Si$_{0.3}$C$_2$ compounds were oxidized at 900°C and 1000°C in air for 100 h. Ti$_3$Al$_{0.7}$Si$_{0.3}$C$_2$ oxidized into rutile-TiO$_2$, α-Al$_2$O$_3$, and amorphous SiO$_2$, accompanied with the evaporation of carbon. The oxide scales consisted primarily of an outer TiO$_2$-rich layer, an intermediate Al$_2$O$_3$-rich layer, and an inner (TiO$_2$+Al$_2$O$_3$+SiO$_2$)-mixed layer. These triple-layer oxide scales formed were not oxidation resistant because of the evaporation of carbon, and the formation of TiO$_2$ and voids. Thick but adherent oxide scales formed during high-temperature oxidation.

This work is the outcome of a Manpower Development Program for Energy & Resources supported by the Ministry of Knowledge and Economy (MKE), Korea.
As the PbO content in the glass increases, structural changes take place in the network. However, the ligand environment of VO$_2^+$ (VP) seem to remain the same in the compositional region from $x=0.3$ to $x \leq 0.5$). This suggests that VO$_5$ polyhedra remain in the same position in the glass network.

The electronic absorption spectrum of VO$_5^2^+$ (VP) could be explained successfully in terms of the energy level diagram developed for VO$_5$ polyhedron treated as distorted trigonal bipyramid.

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Microwave-assisted Preparation of Metal-doped Titania Nanotubes
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Titania nanotube (TNT) has attracting many researches due to its various applications in dye-sensitized solar cell, sensor, photocatalysis, and others with the advantages of being nontoxic, stable in wide pH range, and transparent to visible light. TNT has been synthesized mainly with hydrothermal, templating, and anodic oxidative approaches. The present study uses a modified sol-gel method to synthesize transition metal-doped titania nanoparticles which served as a raw material for the preparation of metal-doped TNT by the use of a temperature-controlled microwave process.

The volumetric heating process with the microwave at 150 degrees centigrade could save more than 90-95% of heating time, as compared with the conventional autoclave process. The TNTs obtained in this study have a length of a few micrometers with a diameter of 8-10 nm. The TNTs are open-ended. The TNTs occurred in anatase form as shown by the results from both x-ray diffraction (XRD) patterns and x-ray absorption spectroscopy (XAS). The metal doping process significantly increases the N$_2$-based surface area for the TNT with a typical surface area of $>$300 m$^2$ g$^{-1}$ that is about twice the surface area of TNT obtained in a previous study with microwave heating. Over-doping the metals would lead to a decrease in the TNT surface areas. Red-shifting phenomena were observed with the metal-doped TNT, as compared with the pure TNT and titania nanopowders. Major fractions of the metallic ions were chemically reduced as indicated by the x-ray absorption near-edge spectroscopy (XANES).

Sol-Gel Derived Crystalline AZO Nano-Powders Prepared by Supercritical CO2 Drying
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Polycrystalline sol-gel derived nano-sized aluminum-doped zinc oxide (AZO) powders were successfully synthesized by supercritical carbon dioxide drying of the AZO precursor solutions without calcination. It is also shown that the extent of drying of the precursors plays a decisive role in whether or not crystalline AZO powders can be produced. In addition, it was demonstrated that the properties of the supercritically dried sol-gel derived crystalline AZO nano-powders are superior to those of AZO powders prepared by traditional sol-gel process with calcination procedure.

Effect of Si, Sc, Cr Doping in the Microstructural, Optical and Discharge Characteristics of MgO Thin Films
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While MgO thin films have many attractive qualities for use in plasma Display Panels, there is a potential for vast improvements by optimizing the microstructural characteristics of the films. The physical and surface properties of the MgO protective layer influence the discharge characteristics, and enhance the lifetime and stability of the panel. Silicon and multiple (Silicon, Scandium, Chromium) doped MgO protective layers have demonstrated higher electron emission, which decreases the firing voltage of the panel. Multiple doped MgO protective layers have much lower firing voltage as compared to pure and only Si doped MgO layers. In this article we describe the preparation of the pure (undoped), silicon doped and multiple doping of silicon, scandium, chromium (with varying concentration of each) thin films using conventional reactive electron beam technique.
and their microstructural properties as studied by x-ray diffraction, scanning electron microscopy and atomic force microscopy.

For doping of Si and Si, Sc, Cr in the MgO thin films, a 30,300,500 ppm amount of Si and Si, Sc, Cr was added to the MgO pellet prior to deposition. The microstructural studies of the pure, Si and Si, Sc, Cr-doped (in varying concentrations) MgO films demonstrate the influence of Si, Sc, Cr doping on the film orientation and surface morphology. The Transmission studies showed 90% transmittance in the visible range for all the films. The importance of an Optimum amount of Si, Sc, Cr doping is seen in our study, where the initial improvement in discharge characteristics with doping is followed by deterioration on further increase in the Si and multiple doping. The microstructural attributes of the MgO films are correlated to the observed changes in discharge characteristics in the context of varying amount Si Sc, and Cr doping.

**A01699-04972**

**Influence of Hydrogen in Magnetron Sputtering Deposition of MgO Thin Films for Plasma Display Panels**

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Large secondary electron emission (SEE) coefficient, low sputtering yield and large band gap have made MgO an important material for dielectric protective layer in plasma display panels (PDPs). In this article, we report the influence of hydrogen addition on the microstructure and secondary electron emission characteristics of MgO films deposited by pulsed DC reactive magnetron sputtering technique. During deposition flow rate of hydrogen was varied by keeping Ar to O₂ flow rate constant. Deposited MgO films were characterized by X-ray diffraction (XRD), atomic force microscopy (AFM), UV- VIS spectrophotometer, spectroscopic ellipsometry (SE). Secondary electron emission property was studied by secondary electron emission tool (SEE tool). From experimental results, it was found that hydrogen addition during sputtered deposition of MgO thin film improved its microstructure and created trapped level inside it which resulted in its improved secondary electron emission properties and stable firing voltage. At 5 sccm hydrogen flow rate we achieved the lowest and stable firing voltage and increased secondary electron emission coefficient (γₑ) for hydrogen flow rate.

**A01742-03030**

**Water-Induced Degradation in Fluorine-Doped Tin Oxide Thin Films**

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Transparent conducting oxides are widely used as electrodes in various optical devices and there have been extensive investigations with regard to the reliability. For indium-doped tin oxide (ITO) and aluminium-doped zinc oxide (AZO), a failure mechanism known as water-induced degradation has been reported, in which the oxides are reduced to metals by hydrogen from the electrolysis of water. This failure mechanism is important for transparent conducting oxides as water is a basic environmental factor and electricity is always present for electrodes. In this paper water-induced degradation in fluorine-doped tin oxide (FTO) will be studied and a comparison over water-induced degradation among several transparent conducting oxides will be conducted.

**A01743-03395**

**Synthesis and Characterization of Zinc Oxide (ZnO) Nanomaterials through the Horizontal Vapor Phase Growth Method**

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Zinc Oxide nanomaterials were synthesized on glass substrate through the non-catalytic horizontal vapor phase growth method. Nanobelts with average width of 100 nm were grown when the growth temperature was set at 1200°C and 1000°C. Nanowires with mean diameter of 2 micrometers in length. Increasing the growth time corresponded to the growth of nanowires with average length of 10 micrometers. EDX analysis revealed that varying the growth temperature and growth time affects the amount of oxygen vacancy of the synthesized nanomaterials. XRD measurements showed that the synthesized nanostructures have a hexagonal wurtzite structure with growth preference in the (001) and (101) directions. Thermostat-temperature photoluminescence spectra showed an intense ultraviolet emission (UV) at 3.26 eV with weak emissions in the visible light regime. The
strong UV emission can be attributed to the recombination of free excitons through an exciton-exciton collision process. Meanwhile, the deep level emissions are caused by oxygen vacancies in ZnO and the emission results in the recombination of the photogenerated hole with an electron occupying the oxygen vacancy. It is proposed that growth mechanism involved in the formation of ZnO nanostructures is the vapor-solid process because no catalyst was used.

A01759-03087

Thin Film of Flexible Porous Metal-Organic-Frameworks by Dip-coating Method

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Porous metal-organic frameworks (MOF) combine a high and regular porosity and the presence of organic groups inside network. The easy tuning of the size, shape and composition offers a unique environment for the host-guest chemistry. Moreover, our group has recently reported a new class of flexible hybrid solids which modulate their pore size upon adsorption of organic molecules. This reversible “breathing” effect varies between 50 and 235 % of volume cell unit, depending on the structure and the length of the linker. These characteristics allow the potential application of these bulk solids in very important fields as gas storage, drug release, separation or catalysis.

Making thin films of MOFs would of a high interest due to the enormous prospects in nanotechnology for such films but examples are still quite scarce. To date, only a few studies have been reported dealing with the nucleation of MOF particles on substrate such as the work reported by Fischer et al. on the selective nucleation and growth of MOF-5 on a modified Au substrate. We proposed for the first time a simple dip-coating method for the preparation of optical quality thin film of a porous flexible MOF.

The porous hybrid solid MIL-89 (MIL: Material Institut Lavoisier) is built up from trimers of iron(III) octahedra linked to muconate dianions to create a 3D framework with a 1D pore channel system along the c axis and interconnected cages. The structure of MIL-89 is highly flexible and can swell, according to the nature of the adsorbed molecules, up to 150% in volume with a maximum pore size ≈11 Å.

Homogeneous thin films of MIL-89 have been obtained by deposition of colloids using the dip-coating method. The evolution of particle size and the study of the crystallisation MIL-89 phase have been investigated. Finally, the flexibility of the resulting film was studied by environmental ellipsometric porosimetry and indicated a reversible increase in thickness upon adsorption of water.

A01772-03105

Colossal Electroresistance and Current-induced Multilevel Resistivity Switching in Nd0.5Ca0.5MnO3 and La0.5NiMnO3

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We investigated electroresistance in two insulators of different electronic and magnetic ground states: charge-orbital ordered antiferromagnetic Nd0.5Ca0.5MnO3 (NCMO) and charge disordered ferromagnetic LaNi0.5Mn0.5O3 (LNMO). A systematic study of dc and pulsed current–voltage (I-V) characteristics of varying pulse width or period along with simultaneous temperature measurements suggest that hysteresis and negative differential resistance (NDR) observed at high current densities during dc current sweep in these compounds vanish systematically with increasing period of the pulsed current, thus underlining the importance of Joule heating in the NDR phenomenon.

In the regime of negligible Joule heating with low currents, it is shown that we can induce bi-level and multi-level resistivity (ρ) switching by a sequence of pulses with controlled pulse width or period even at fixed current amplitude. It is found that ρ increases abruptly (∼37 % for NCMO at 100 K and ∼17 % for LNMO at 300 K) upon increase of pulse period from 50 ms to 100 ms for a fixed pulse width (25 ms) and current (I = 2 mA). Similar resistive switching effects of different magnitudes were also found with variations in the pulse width for a fixed period and our results suggest that these materials can be exploited for non-volatile memory based on resistivity.

A01796-03540

Hydrophobic ZnO Nanostructured Thin Films on Glass Substrate by Simple Successive Ionic Layer Absorption and Reaction (SILAR) Method

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Over the past few decades, the rapid developments in application of nanostructured materials demands precise control over their size and shape. As an important wide band gap materials, Zinc oxide (ZnO) is an II–VI semiconductor, is particularly important material for its both scientific as well as industrial applications. In the recent years, water repellent behavior is a very important property governed by both the chemical composition and the geometric features of the surfaces. Morphology and size controlled synthesis of the ZnO nanostructures was achieved by optimizing the
synthesis parameters such as temperature, reaction time and the surface roughness of the substrate. In the present work, ZnO nanostructured thin films were grown on glass substrates by a simple successive ionic layer absorption and reaction method (SILAR) process at relatively low temperature for self cleaning application. SEM image clearly shows the surface morphology with cluster of spindle nanostructured with length various around 350nm. Structural analysis reviles that the grown ZnO NRs exhibit (002) reflection with higher intensity, indicating the c-axis orientation of the ZnO nanostructure film. Photoluminescence (PL) spectra of ZnO nanostructures film exhibit a UV emission around 385nm and visible emission in the range around 420 – 500nm. Good water repellent behavior were observed for ZnO nanostructured film without any surface modification.

Inkjet Printing Approach to Fabricate Non-sintered Dielectric Film with High Packing Density for 3D Package Integration Technology

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We have successfully demonstrated the inkjet printing method to create Al₂O₃ films without a high temperature sintering process. The packing density of the Inkjet-printed Al₂O₃ films is more than 80% which is very high compared to the value obtained from the same films synthesized by other conventional methods such as film casting, etc. The voids inside the films which are less than 20% of the entire film volume are filled with the polymer resin thru the infiltration process. This resin infiltration is also implemented by the inkjet printing process right after the film fabrication process. The printed films of highly packed Al₂O₃ lead to reasonably good material properties. The microstructures of the printed Al₂O₃ films are investigated by Scanning Electron Microscope (SEM) to understand the degree of packing density in the printed films. Dielectric loss of the printed film is also measured. It shows a reasonably low value since the high packing density of the printed Al₂O₃ films.

Thermal Conductivity Measurement of Inkjet-printed Non-sintered Al₂O₃ and AlN Thin Films by Differential 3ω Method

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The thermal conductivity of Al₂O₃ and AlN thin film synthesized using an inkjet printing has been studied by the differential 3ω technique. Al₂O₃ and AlN films are printed on Cu substrate without a successive sintering process. High packing densities of those films are obtained by optimizing the various printing parameters. The packing density of the inkjet-printed films is more then 80% in the case of Al₂O₃ printed layer. The voids in the films (~ less than 20% of the entire printed volume) are filled with polymer resin. In order to measure the thermal conductivity of those non-sintered printed layers, we choose a differential 3ω technique which has been well known to measure thermal conductivity for multi-layer thin films. Since there is polymer resin in the printed layers, we intentionally deposit a protection layer right on top of the printed Al₂O₃ and AlN layers before preparing a heater structure on the sample.

Effect of Surface-layer on Giant Dielectric Properties of Li₀.₀₅Ti₀.₀₅Ni₀.₉₀O Ceramic

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We studied the effect of surface-layer on the giant dielectric properties of Li₀.₀₅Ti₀.₀₅Ni₀.₉₀O (LTNO) ceramic prepared by a polymer pyrolysis route. The phase composition and microstructure were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive x-ray spectrometer (EDS), respectively. The dielectric and electrical properties of the prepared LTNO sample were investigated as functions of frequency (10⁵-10⁶ Hz) and temperature (-50-150°C). Microstructure and phase composition analyses revealed that the microstructure of the LTNO ceramic exhibited a core/shell structure. Our results proved that the surface-layer had a great influence on the dielectric properties of the LTNO ceramic. Two thermally activated dielectric relaxations
were detected in the dielectric spectra of the as-sintered sample. The activation energy ($E_a$) for these relaxations was found to be 0.294 and 0.378 eV for the high- and low-frequency relaxations, respectively. Interestingly, when the surface-layer of the as-sample was removed (abbreviated as polished-sample) from the both sides of the pellet as-sample, the low-frequency relaxation disappeared and the activation energy of the remaining high-frequency relaxation remained constant ($E_a \sim 0.288$ eV). By using complex impedance and ESD analyses, it was suggested that both the as-sintered and polished samples were electrically inhomogeneous, consisting of semiconducting grains (i.e., semiconductive Li-doped NiO particles) and Ti-rich insulating boundaries and surface-layers. Moreover, the surface-layer effect might also be attributed to the difference in oxygen vacancies between the inner part and surface-layer, which cause a change in electrical conductivity between them. Therefore, the low-frequency relaxation with higher activation energy value was strongly indicated to the effect of insulating surface-layers. The origin of the observed giant dielectric constant in our LTNO samples should be ascribed based on the Maxwell-Wagner polarization (i.e., interfacial polarization) at the insulating layers in the bulk ceramic and at the surface-layers.

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Fabrication and Some Properties of Textured Ceramics by Colloidal Processing in a Strong Magnetic Field

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The controlled development of texture is one of the ways for effectively improving properties of ceramics. Recently, high magnetic fields with a field strength up to 14 T is readily available without the use of liquid helium due to the development of superconducting technology. These new magnets have been used in studies of many fields, such as crystal alignment, levitation, separation, etc. We have demonstrated the new processing of textured ceramics with a feeble magnetic susceptibility by colloidal processing in a high magnetic field and subsequent heating. The principle of the process is that a crystal with an anisotropic magnetic susceptibility will rotate to an angle minimizing the system energy when placed in a magnetic field. To obtain the oriented materials with feeble magnetic susceptibilities, the following conditions are necessary: (1) the particle should be single crystal and well dispersed, (2) crystal structure should be non-cubic to yield an anisotropic magnetic susceptibility, (3) magnetic energy should be larger than thermal motion energy, (4) the viscosity of the suspension should be low enough to rotate the particles with a low energy, and (5) grain growth is necessary to obtain a highly oriented structure especially when spherical particles are used.

As colloidal processing, slip casting and electrophoretic deposition (EPD) have been conducted successfully. Slip casting is a powerful method to prepare dense and fine grained bulk ceramics and the EPD is useful for preparing laminated ceramics. The slip casting in a high magnetic field confers several advantages and it is possible for this type of processing to be applied to non-cubic ceramics, such as alpha-alumina, titania, zinc oxide, tin oxide, hydroxyapatite, aluminium nitride, silicon carbide, silicon nitride, etc. Also textured ceramics with complicated structure can be fabricated by reaction sintering, such as beta-alumina, SiC-mullite-alumina nanocomposite, beta-Si3N4, etc. However, when we use whisker or plate-like particles, special attention is necessary owing to the effect of gravity energy that is the highest energy in the colloidal dispersion system.

We have also demonstrated that EPD in a high magnetic field is an excellent method to fabricate crystalline textured ceramic thick bodies. Here, the direction of the electric field relative to the magnetic field can be altered to control the dominant crystal faces. Crystalline-textured controlled laminated composites can be fabricated using EPD by varying the angle between the vectors of electric field and magnetic field. This method can be also applied to prepare crystalline-oriented or specific crystal face thin films for functional applications, such as dielectric ceramics, thermoelectric devises.

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In the ceramic/metal joining and coating, the most difficult problem is how to overcome poor adhesion of ceramic layers by their cracking and/or peeling arising from their intrinsic brittleness. On the basis of accumulated results and discussion, we propose a novel concept and technology of formation “Growing Integration Layer” [GIL] between ceramics and metallic materials to improve the adhesion performance. Those GIL(s) can be prepared from a component of the metallic materials by chemical and/or electrochemical reactions in a solution at low temperature of RT-200°C. They have particular features: 1) Widely diffused interface(s), 2) Continuously graded layers grown from the bulk(substrate), 3) Low temperature process, etc. BaTiO₃ or SrTiO₃/TiOₓ GIL films on Ti plates formed by hydrothermal-electrochemical method showed good adhesion. CaTiO₃/Al₂O₃/Ti₂Al GIL films on TiAl exhibited excellent adhesion and anti-oxidation performances: they could be sustained for 10 times cyclic oxidation test at 900 C in air for 5 hrs. The GIL strategy is effective for many metallic alloys and bulk metallic glassed because they generally contain active components(s).

On a Ti-base Bulk Metallic Glass, we could succeed to prepare bioactive titanate nano-mesh layer by hydrothermal-electrochemical techniques at 90-120°C. Similarly, bioactive oxide layers could be prepared on different Bulk Metallic Glasses.

Thermal Conductivity of Bi-Sr-Ca-Cu-O Superconductors

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Using the latest approach of electron-phonon line widths for various scattering mechanisms in the relaxation time approach, the thermal conductivity of Bi-Sr-Ca-Cu-O superconductors has been analyzed successfully. The concept of electronic thermal conductivity and lattice thermal conductivity has been ruled out in the present formulation.

Synthesis of MoSi₂ Based Composite Powder by Mechanochemical Reduction of MoO₃

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In this study, a MoSi₂ based composite powder was produced by mechanochemical synthesis. MoO₃ powder with various amounts of silicon and carbon were mixed together. Mixtures were subjected to severe mechanical activation using a high energy planetary ball mill. The milling operation on a suitable mixture of reactants resulted in carbosilicothermic reduction of MoO₃ and simultaneous formation of an ultra fine MoSi₂ / MoC composite powder. The synthesized product was characterized by scanning electron microscope and X-ray diffraction. XRD analysis indicated that the composite powder could be obtained after 27 hours of milling. Calculation of the mean crystallite sizes of the product phases indicated that they could be in nano scale by prolonged milling.

Giving a New Life to an Old Material ZnO

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Atomically defined heterostructures composed of ZnO and (MgZn)O were grown by pulsed laser deposition (PLD) and plasma-assisted molecular beam epitaxy (MBE). PLD films had been used for making p-ZnO/n-ZnO light emitting diodes (LEDs), but it was difficult to extend the research for making p-(MgZn)O due to impurities in the target. MBE enabled us to make highly pure (MgZn)
O and nitrogen doped p-(MgZn)O on +c ZnO substrate. Ultraviolet electroluminescence could be clearly observed for LEDs having p-(MgZn)O and ZnO active layers. This technology also drastically improved the mobility of two-dimensional electron gas accumulated at the ZnO/(MgZn) O interfaces. The highest value of the mobility exceeds 20,000cm2/Vs, giving threefold improvement from that grown by PLD. Zero-resistance in Shubnikov-de Haas oscillation and clear plateau in quantized Hall resistance were observed in MBE grown heterostructures.

We also discover that a π-conjugated conducting polymer blend, poly(3,4-ethylene dioxy thiophene) poly(styrene sulfonate) (PEDOT:PSS), can be used as an ideal electrode for making Schottky junctions with n-ZnO. Since this polymer is transparent to ultraviolet, the junctions can be used as ultraviolet photo-detectors. The Schottky junction is also used for controlling the 2DEG density with a field effect transistor geometry.

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Spectroscopic Identification Valence and Conduction Band Electronic Structure and Intrinsic Defects in Complex Oxides

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Advanced spectroscopic techniques such as O K edge spectroscopy, and soft X-ray photoelectron spectroscopy studies of HfO2 and TiO2 carry over directly to complex oxides, as do the generalizations for symmetry adapted molecular orbital and defect state electronic structure. New to these studies are (i) virtual bound state spectroscopy (VBSS) for revealing empty transition metal and defect state resonances above the vacuum continuum threshold, as well as (ii) pre-edge O K spectroscopy (PEKS) that distinguishes between mono- and di-vacancy defects. The information gained from these studies is particularly significant for identification of the electronic structure required for functionality enhancements, e.g., multiferroic behavior in complex oxides with two or more cations in which the local site symmetries of these ions are different, as in cubic and hexagonal perovskites. This paper identifies for the first time important enabling degrees of freedom in composition that either involve (i) the same TM atom species, but in different ion states, as in (La,Sr)MnO3, where Mn is a 3+ ion with d0 occupancy in the LaMnO3 layers, and Mn is a 4+ ion with d0 occupancy, in the SrMnO3 layers, or (ii) alloy additives with different valence states that promote structure changes and Jahn-Teller distortions that enable specific functionalities, e.g., super-exchange ferromagnetism.

The discussion addresses cubic and hexagonal perovskite complex oxides that form a basis set for complex oxides in general. The first and simplest class are cubic d0 complex oxides, e.g., SrTiO3, and d2, n>0 alloys in which local site symmetries can be modified by substitution of for Ti-atoms with TM atoms having more than 2d electrons in their neutral atomic states, e.g., Mn and Ni. The conduction and valence electronic structures at the respective band edges of titanates and manganites are qualitatively similar to those of their respective elemental oxides, and defect and alloy atom electronic states are readily detected in VBSS and PEKS spectra that reveal complementary d\textsuperscript{0} electronic structures.

The O K edge spectroscopic characterization of d\textsuperscript{0} persovskite complex oxide electronic structure is incomplete and requires (i) extensions into the VBSS and PEKS regimes, augmented with (ii) high-resolution SXPS to distinguish between occupied and empty TM electronic states, and mono- and di-vacancy defects. These defects have for the first time been correctly characterized in terms of d\textsuperscript{0} electronic structures. Previous attempts to calculate defect state properties with modified DFT methods are inherently flawed, and erroneously redistribute the electronic charge in vacancy defect sites over extended rather than localized TM electronic states. Examples of d\textsuperscript{0} complex oxides include LaMnO3 as (La,Sr)MnO3, as model systems and engineered d\textsuperscript{0} oxides formed by alloy atom introduction into d\textsuperscript{0} complex oxide hosts, including titanates and scandates.

A new approach for depositing transition metal elemental oxides, HfO2 and TiO2, directly onto to Ge substrates without an intervening Ge-O-N interfacial transition region represents a pathway for introducing complex oxides into Si-based CMOS ULSI.

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Dimensional Constraints and Percolation Theory: Pathways to Physical Mechanisms for Reducing Intrinsic Bonding Defects in Advanced High-κ Gate Stack Dielectrics

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Extension of O K edge X-ray absorption spectroscopy from the conduction band states regime at X-ray energies between 528 and 545 eV, into the pre-edge regime, <528 eV, and vacuum continuum, >545 eV provides a pathway to study final states for (i) intra-d state and f-state transitions in the pre-edge regime, and (ii) anti-bonding virtual bound states in the vacuum continuum. Incorporation of symmetry adapted linear combinations of atomic states into molecular orbital states provides a theoretical basis for identifying the electronic structure of occupied transition
metal d-states, and well intrinsic mono- and di-vacancy defects in TiO$_2$ and ZrO$_2$ and HfO$_2$, respectively.

These advances in O K edge XAS are used to complement empirical approaches for defect reduction in thin film high-k gate dielectrics. This approach combines spectroscopic studies with physical mechanisms for macroscopic strain reduction where (i) percolation controlled nano-scale strain-reducing chemical bonding phase separations applies to non-crystalline films, and (ii) nano-scale dimensional or diphasic control of grain-size in nano-cry stalline films, each substantially reduces intrinsic bonding defects: monodiphase control of grain-size in nano-cry stalline films, to non-crystalline films, and (ii) nano-scale dimensional or strain-reducing chemical bonding phase separations applies reduction where (i) percolation controlled nano-scale studies with physical mechanisms for macroscopic strain gate dielectrics. This approach combines spectroscopic empirical approaches for defect reduction in thin film high-k dielectrics, no electron trapping is detected after X-ray and γ-ray stressing at levels used for rad-tolerant microelectronic devices.

The nano-cry stalline Hf Si oxynitride alloy in (i) is a ternary extension of the 50% SiO$_2$-50% S$_3$N$_4$ composition used by major chip producers since about 2000 as a pathway to achieving EOTs to about 1 nm, and thereby achieving aggressive down-scaling of lateral dimensions as well. In the ternary alloy, Hf is introduced into ~1/6 of the four-fold coordinated network sites. This is at the percolation threshold for macroscopic strain relief. The Hf-atoms are trapped in a compliant tetrahedral bonding arrangement as evidenced by a crystal field splitting of ~ 2.2 eV, as compared with 3.5 - 4 eV for seven- and eight-fold coordinated Hf in monoclinic- and tetragonal-HfO$_2$ grains. This low-strain non-crystalline film has a low density intrinsic bonding defects, <10$^{11}$ cm$^{-2}$. Unlike nano-crystalline Hf-based high-k dielectrics, no electron trapping is detected after X-ray and γ-ray stressing at levels used for rad-tolerant microelectronic devices.

Spinels-ferrites have long been the subject of extensive investigations by virtue of their extraordinary magnetic and magnetostrictive properties. Recently, they have brought about intensive scientific investigation because the combinations with piezoelectric materials, such as composite films and multilayers, turned out to be candidates for room-temperature multiferroic structures.

Magnetoelectric (ME) coupling effects in these types of multiferroic structures are caused by the interfacial strain. In other words, the strain generated by the piezoelectric phase under an applied electric field is coupled to the magnetostrictive phase mechanically, and this consequently changes the magnetic moment. However, the ME coupling effect arising from an alternatively stacked layered structure is known to be significantly suppressed by the clamping effect exerted by a non-piezoelectric single-crystalline substrate.

To overcome this limitation, we have adopted a piezoelectric single-crystalline substrate, 0.72PMN-0.28PT (PMN-PT), which is known to exhibit giant electrostrictive strains. Epitaxial NiFe$_2$O$_4$ (NFO) films were then grown by pulsed laser deposition on (001) planes of these substrates. XANES and EXAFS experiments under dc electric fields were performed to investigate the structural modulation of the NFO layer. This study clarifies the ME coupling arising from the piezoelectric-magnetostrictive effect in atomic scales. In addition to this, we observed the enhancement of the remnant magnetization ($M_r$) under an applied dc electric field, indicating that a strain at the NFO/PMN-PT interface effectively enhanced magnetization. The first-principles DFT calculations further support the observed enhancement of $M_r$ under an epitaxially constrained state. We suggest this type of bi-layer structure can significantly enhance the degree of the ME coupling by an electric-field control of $M_r$. 

**Symposium D - Functional Ceramic Materials, Oxide Thin Films and Heterostructures**

**Electric-Field-Induced Modulation of Magnetization in Epitaxial NiFe$_2$O$_4$ Films Grown on PMN-PT**

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PdO-modified Zinc Stannate Thick Film Resistors as H₂-gas Sensors

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Nano-zinc stannate ceramic powders was synthesized by the coprecipitation method assisted ultrasonic. The material was confirmed by XRD, TEM and EDS. PdO-modified ZnSnO_3 thick films were obtained by incorporating palladium chloride as an additive and prepared by indirect heating procedures. Sintering converted palladium chloride into palladium oxide. Surface morphology of gas sensing layer was characterized by SEM. It was shown that PdO-modified ZnSnO_3 thick film exhibited more sensitive than pure ZnSnO_3 to H₂ and high sensitivity as well as good selectivity was found to be at 280°C. The effect of amount of PdO in ZnSnO_3 on the gas response, response time and recovery time of the sensor were also studied. The origin of the gas response was attributed to the surface catalytic reaction of hydrogen gas with O^− (ads). This work is supported by supported by Nature Science Foundation of ChongQing (No.8430), China.

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The Structure and Microwave Properties of Dielectric Tunable (Ba,Sr)TiO₃-based Composite Ceramics

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The dielectric tunable (Ba,Sr)TiO₃-based (BST) composite ceramics are fabricated via the conventional solid-state reaction method. The phase structure, microstructures, dielectric tunability, and microwave properties of composite ceramics are investigated. The XRD and backscattered electron image analysis shows that the two-phase structures of BST perovskite and AB₂O₄ spinel structure were formed. The temperature and frequency dependence of the dielectric permittivity indicate a crossover from ferroelectric with the diffuse phase transition to relaxor behavior. The dielectric permittivity exhibits relaxor behavior with the frequency-dependent T_m satisfying Vogel-Fulcher formula. The dielectric tunability and microwave properties of the (Ba,Sr)TiO₃-based composite ceramics were optimized compared with pure (Ba,Sr)TiO₃-based material. The dielectric constant of composite ceramics can be tailored from high dielectric constant to low dielectric constant and the tunability is still maintained at relatively high level. The high tunability is due to the small cooperative displacements of Ti⁴⁺ ions in the close packed oxygen octahedral of BST system, which may be ascribed to the decrease of the oxygen vacancy concentration. The Q value is gradually increasing with Mn content, potentially due to the microstructural evolution of BST lattice structure characterized by Raman spectra. The composite ceramics with high Q value at L-band can be useful for potential tunable microwave device applications in wireless communication system and phase shift devices.

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Room-Temperature Ferroelectricity and Giant Magnetoelectric Coupling in Mn-doped InP

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Multiferroics combine two or more of the properties of ferromagnetism (or antiferromagnetism), ferroelectricity, and ferroelasticity. The quest for multiferroic materials is of great technological and fundamental importance. One of the most important issues in multiferroism is to find a new ferroelectromagnet that possesses a strong magnetoelectric (ME) coupling at room temperature.

In this work, we have designed and fabricated a [001]-oriented Mn-doped InP (InP:Mn) thin film which simultaneously exhibits canonical ferroelectric and ferromagnetic hysteresis loops up to and above room temperature. More importantly, a strong ME coupling that is two orders of magnitude higher than the best previously reported was observed at 3 K. The magnitude of the magnetically induced polarization (∆P) is larger than 6 μC/cm² at 3 K with a periodic reproducibility in ∆P under a varying magnetic field.

To understand the behavior at the atomic scale, we performed first-principles DFT calculations. The DFT study indicates that the direction of the spontaneous polarization is parallel to [111] of zinc-blende structure and the multiferroism arises from the atomic displacement around a Mn-vacancy complex. The DFT calculations further suggest that the polarization reversal is accompanied with some changes in the direction of spin moment as a consequence of the ME coupling. Analysis of the polarization response on the basis of the Landau theory indicates that the degree of the intrinsic ME coupling remains strong even at the magnetic transition temperature, ~380 K.
Enhanced Magnetization and Trigonal Distortion of $t_{2g}$ Orbital in Epitaxially Constrained BiFeO$_3$ Thin Films with Rhombohedral Symmetry

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Multiferroics are an interesting group of materials that show ferroelectric, ferroelastic, and (anti)ferromagnetic ordering simultaneously. The coupling of these order parameters and the enhancement of the ferroic properties caused by the epitaxial constraints have stimulated the possibility of new multifunctional devices.

An epitaxially constrained BiFeO$_3$ (BFO) thin film is known to exhibit a remarkably enhanced ferroelectric polarization and saturation magnetization, though there are still some controversial arguments about the enhancement by the misfit strain. In order to clarify the origin of the enhanced magnetization, we have fabricated both the [111]-oriented epitaxial BFO thin film with R3c symmetry (with the thickness range between 30 and 200 nm). The degree of the in-plane epitaxial strain was assessed by examining XRD patterns and reciprocal space mapping (RSM). XPS, NEXAFS, and XANES studies indicate that the present BFO films are exclusively characterized by trivalent Fe ions with the absence of any divalent Fe ion.

The saturation magnetization ($M_s$) of these epitaxial films increased substantially with decreasing film thickness and did correlate well with the calculated epitaxial misfit strain. On the basis of polarized x-ray absorption spectroscopy analysis, we will discuss the splitting of $t_{2g}$ orbital into $a_{1g}$ and $e_{g}$ orbitals arising from a trigonal distortion as a possible origin of the observed enhanced magnetization under a highly constrained state.

Low-temperature Growth and Switching Characteristics of Epitaxial BiFeO$_3$ Thin Films on Pt/MgO(001)

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BiFeO$_3$ (BFO), the most promising material for room-temperature applications of multiferroics, has stimulated the possibility of a new ferroelectric device because of its pronounced ferroelectric polarization as well as the coupling of ferroic order parameters at room temperature. However, the well-known thin-film capacitor structure based on BFO is currently fabricated at an elevated temperature using a conducting SrRuO$_3$ (SRO) buffer layer.

Here we report that we are able to lower down the growth temperature to 500°C by replacing a SRO-buffered SrTiO$_3$ (SRO/STO) substrate with a (001)-oriented MgO substrate coated with an epitaxial Pt layer. The in-plane XRD analysis and the reciprocal space mapping revealed that both Pt and BFO layers were grown epitaxially with pseudo-tetragonal symmetry. Analysis of the switching behavior further showed that the films were electrically fatigue-free and had good charge-retention characteristics compared to typical capacitors based on SRO/STO.

Powder-In-Closed-Tube (PICT) Fabrication of Bi$_2$Sr$_2$CaCu$_2$O$_8$/Al Superconducting Tape

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Bi$_2$Sr$_2$CaCu$_2$O$_8$/Al superconducting tape was successfully fabricated using the powder-in-closed-tube (PICT) method. PICT method involves packing of Bi-2212 powder into an aluminum tube, pressing, and sintering. The Bi-2212 powder was packed into an aluminum tube, which is used as sheathing material, by closing both ends of the tube. The tube filled with Bi-2212 powder was then applied with force by uni-axial pressing. For the final heat treatment, the samples were separated into two batches; first batch were annealed for 300 minutes, (750°C for 30 minutes and 600°C for 270 minutes) and the second batch were annealed for 600 minutes (750°C for 30 minutes and 600°C for 570 minutes), both at a rate of 30°C min$^{-1}$. X-ray diffraction verified the existence of Bi-2212 in
the tape. The graph of the magnetic susceptibility versus temperature showed that the critical temperature ($T_c$) of the samples is approximately 79K. Scanning electron microscope (SEM) images showed that the sample sintered for 600 minutes attained more spatial homogeneity than the sample annealed for 300 minutes. The hardness test results also agree with the SEM images which showed that the tape sintered for 600 minutes is more compact, thus harder than that of the tape sintered for 300 minutes.

Control of Y$_2$BaCuO$_5$ Particle Formation in Bulk, Single Grain Y-Ba-Cu-O High Temperature Superconductor

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The trapped field of undoped, bulk, single grain Y-Ba-Cu-O (YBCO) fabricated by top seeded melt growth (TSMG) has been measured in order to investigate the field generating potential of this material. Bulk samples were prepared from precursor powders that incorporated Y$_2$O$_3$ of particle size 20 to 50 nm, rather than Y$_2$BaCuO$_5$ (Y-211), which is used more commonly, in order to introduce nano Y-211 inclusions in the superconducting YBa$_2$Cu$_3$O$_y$ (Y-123) matrix. Relatively small bulk samples of diameter 20 mm processed from this precursor are observed to trap a peak magnetic flux density of nearly 0.8 T at 77 K. This is the first time that such a trapped field of this magnitude has been observed in undoped YBCO (i.e., in the absence of chemical additions). The increase in trapped field is accompanied by an associated enhancement in $J_c$ by up to a factor of five over applied magnetic field up to 4 T at 77 K compared to standard single grain YBCO fabricated by precursor powders that contain the Y-211 phase.

Resistive Switching Memory Devices Composed of Binary Transition Metal Oxides Using Sol-Gel Chemistry

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We describe a novel and versatile approach for preparing resistive switching memory devices based on binary transition metal oxides (TMO). Titanium isopropoxide (TTIP) was spin-deposited onto platinum (Pt)-coated silicon substrates using sol-gel chemistry. The sol-gel derived layer was converted into TiO$_2$ film after thermal annealing and then the top electrode was coated onto the TiO$_2$ films to complete device fabrication. When an external bias was applied to the devices, switching phenomena independent of the voltage polarity (i.e., unipolar switching) were observed at low operating voltages (about 0.6 $V_{\text{RESET}}$ and 1.4 $V_{\text{SET}}$). In addition, it was confirmed that the electrical properties (i.e., retention time, cycling test and switching speed) of sol-gel derived devices were comparable to those of vacuum deposited devices. Furthermore, our approach can be extended into a variety of binary transition metal oxides such as niobium oxides. The reported approach offers new opportunities to prepare the binary TMO-based resistive switching memory devices allowing facile solution processing.

How Do Gas, Temperature and Oxigen Pressure Change the Conductivity of Metal Oxide Semiconductor (MOS) Thin Film? : A Theoritical Study with Point Defect Theory

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A study of the mechanism of oxidating and reducting gas response on the metal oxide semiconductor (MOS) thin film surface has been done. To formulate the effect of reducting and oxidating gas adsorption to MOS’s charge carrier concentration, we use the quasi-chemical equations as the analysis tool representing the mechanism of reaction between the point defect of MOS’s surface and the gas on thermodynamic equilibrium. The choice of the point defect system that is the natural state of the surface and bulk of the metal crystalline semiconductor is to relate description between the effect of the gas response mechanism and MOS’s electrical parameter. At a point purpose, the Flockhardt’s point defect model of MOS’s surface is used as a basis understanding of surface reaction considered within the point defect theoretical frame. Furthermore,
we introduce the quasi-chemical equation of the reaction between the point defects of the metal oxide surface and oxidating gas (O$_2$ and CO$_2$), and also the reducting gas (C$_2$H$_5$OH, NH$_3$ and CO). The formulation has been done in the thermodynamical equilibrium between the surface and bulk by considering gas sensor operating temperatur of the MOS thin film. From the developing of theoretical model, we find that a number of charge carriers (hole-electron) of the MOS depend on oxygen pressure, gas concentration and temperatur.

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The Effects of Surface Reaction, Point Defect Diffusion and Gas Diffusion on the Sensitivity of Metal Oxide Semiconductor (MOS) Thin Film Gas Sensor: A Theoretical Study with Point Defect Theory

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Theoretical study of the dependence of gas diffusion in metal oxide semiconductor (MOS) thin film on the MOS’s gas sensor sensitivity has been done. Point defect theory in Flockhardt’s MOS surface structure model is used as theoretical framework to view the dependence of the absorption of reducting gas (C$_2$H$_5$OH, NH$_3$ and CO) and oxidating gas (O$_2$ and CO$_2$) on the concentration of charge carrier. The theory of point defect diffusion relating to the role of polaron and small polaron as charge carrier is also used to formulate the mobility of charge carrier and the conductivity of MOS’s gas absorption. The effects of gas transport phenomena in material to MOS’s conductivity and sensitivity are investigated theoretically by the assumption that the moving gas in a MOS thin film obey Knudsen diffusion. Considering the operational temperature of MOS thin film gas sensor, The formulation is designed to a thermodinamically equilibrium state between the MOS’s surface and interior. From the developing of theoretical model, we find that the sensitivity of MOS in gas absorption process depend on the oxygen gas pressure, gas concentration, temperature, thin film thickness, the porous radius and gas molecular weight. The results of our study have agreement with experimental results.

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Design of Interconnections of Novel Complex Oxide Nanoparticles

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To enhance physical properties, it is known well that polarization rotation mechanism is very important for dielectric and piezoelectric materials. Up to date, enhancement of dielectric properties was reported for barium titanate (BaTiO$_3$, BT) nanoparticles with size of around 100 nm, and this enhancement can be explained using this polarization rotation mechanism. To investigate the mechanism, mesoscopic particle structure of BT nanoparticles were investigated using synchrotron radiation X-ray diffraction (XRD) method and Rietveld analysis. For this objective, BT nanoparticles with various particle sizes from 10 to 150 nm were prepared by the 2-step thermal decomposition method of barium titanyl nanooxalate with a size of 30 nm. Various characterizations revealed that these particles were impurity-free, defect-free, dense BT particles. The powder dielectric measurement clarified that dielectric constant at room temperature was 30,000 for BT nanoparticles with sizes from 40 to 63 nm. Using these particles, a relationship between dielectric property and mesoscopic particle structure was investigated. Moreover, to apply the polarization rotation mechanism for property enhancement, a new material design using different complex oxide nanocubes was proposed. In this study, a novel complex oxide accumulation of BT and strontium titanate (SrTiO$_3$, ST) nanocubes was prepared. For this objective, a concept of molecular recognition was applied in this study. As a result, using DNA base pair such as adenine and thymine, 3-dimentional artificial superlattice accumulations were prepared without any external fields, and checkerboard-like structure composed of BT and ST nanocubes was successfully prepared with a size of around 100 nm. However, this size is too small to measure dielectric property. Thus, some external fields such as electric and magnetic-fields were applied during self organization of BT and ST nanocubes, and thus, their sizes became over 10 mm. Their dielectric property will be reported at the presentation.
In this study, Li(Ni$_{1/3}$Co$_{1/3}$Mn$_{1/3}$)$_2$O$_4$ together with new material compositions of Li(Ni$_{1/3}$Co$_{1/3}$Mn$_{1/3}$-xM$_x$)$_2$O$_4$ (M=Al, Fe, Mg, Zn; x=0.11, 0.22, 0.33) and Li(Ni$_{1/3}$Co$_{1/3}$Mn$_{1/3}$-yM$_y$)$_2$O$_4$ (x=0.11, 0.22, 0.33) were synthesized in the form of fine powders by the Pechini method and subsequently were calcined to obtain the final ceramic oxide powder. The phase, particle size, and morphology analyses were carried out on calcined powders with X-ray diffractometry, particle size analysis, and SEM, respectively. The d.c. (four probe method) electrical characterization was performed on the pellets sintered at 1000°C for 4 h.

The phase analysis revealed the formation of solid solutions of appropriate α-NaFeO$_2$ layered structure in the materials but with the presence of a secondary phase in trace amounts in some compositions. The resultant refinement patterns of the Le-Bail fitting to the X-ray diffraction data of the materials are in good agreement between the observed and calculated patterns. Most of the ceramic oxide materials prepared in this study show having appropriate particle size for LIB positive electrode. In general, the SEM show sponge-like agglomerates with well-defined, compact, submicron size primary particles.

2016 stainless steel coin cells, assembled in an argon-filled glove box were used for electro-chemical testing of the materials in cathodes. The electrolyte used was 1.2M LiPF$_6$ in 3:7 (weight ratio) ethylene carbonate:ethyl methyl carbonate. The electrochemical experiments were run in the glove box using EG&G Princeton Applied Research Model 263 and Solartron 1287 potentiostats controlled by the CorrWare software. The cells were tested using galvanostatic charge/discharge mode. Used potential window is 3.0 and 4.5 V. For Li(Ni$_{1/3}$Co$_{1/3}$Mn$_{1/3}$)$_2$O$_4$, much better electrochemical performance was observed for the materials calcined at 1000°C, with specific capacity close to 180 mAhg$^{-1}$. Conversely, the material calcined at 800°C shows only a capacity of 140 mAhg$^{-1}$. As a whole some of Li(Ni$_{1/3}$Mn$_{1/3}$Co$_{1/3}$-xM$_x$)$_2$O$_4$, M=Fe, Al, Mg materials show a higher first cycle charge capacity than LiCoO$_2$.

All together, this study shows potentiality of Li(Ni$_{1/3}$Co$_{1/3}$Mn$_{1/3}$)$_2$O and Li(Ni$_{1/3}$Mn$_{1/3}$Co$_{1/3}$-xM$_x$)$_2$O, M=Fe, Al and Mg oxide ceramics prepared by Pechini method, for LIB positive electrode application.
A02028-03495

Synthesis and Gas Sensing Properties of Zinc Oxide Nanostructures Obtained By a Novel Microwave Based Technique

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We describe a novel route for the synthesis of nanostructured zinc oxide powder using a modified kitchen microwave. A SiC-based composite showing a strong absorption of microwave was used as a microwave heater. The composition of the suscepter was optimized to reach rapidly the highest temperature possible. Tests showed that high temperatures exceeding 1700°C can be reached in less than hundred second exposure to microwaves. The SiC-based composite was used as a heater to prepare ZnO nanopowders by evaporation and oxidation of metallic zinc in the ambient atmosphere of the microwave oven. The SEM micrographs showed the presence of complex nanostructures including needle-like, tetrapods, sheets and spherical ZnO particles. The dimensions of the tetrapods vary from 40 to 80 nm diameter and about 1-1.5 µm length.

The gas sensing properties of the nanopowders were tested at different temperatures and for different hydrogen compositions of the atmosphere (500-1500 ppm). The results showed a fast response of ZnO based sensor. The resistance recovers its initial value after H2 elimination. It was observed that the response time was shorter than the recovery time. The sensing mechanism and the behavior of the device are discussed in terms of desorption and adsorption of charged species at the surface affecting the depletion layer and the creation/annihilation of charged oxygen vacancies.

A02041-03508

Polaronic Relaxation in Colossal Dielectric Constant Perovskite Ceramics CaCu3Ti4O12

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The dielectric properties of CaCu3Ti4O12 (CCTO) single phase ceramics were investigated in the frequency range from 1 Hz to 10 MHz. A mean-field model was used to derive the dielectric behavior of polaronic relaxation. The relaxation properties are found to be perfectly described by this polaronic model, indicating that the dielectric relaxation is intimately related to the hopping motion caused by localized charge carriers. Further internal friction study indicates that the polarons are associated with oxygen vacancies.

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A02036-04585

Barium Strontium Titanate Ferroelectric Tunable Photonic and Phononic Crystals

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Theoretical simulations and experimental investigations on the development of electro-optically tunable photonic crystals and thermally tunable phononic crystals based on ferroelectric Ba0.7Sr0.3TiO3 (with Curie temperature at ~35°C) are presented. One-dimensional photonic crystal consists of five periods, with each period consisting of a 90 nm thick Ba0.7Sr0.3TiO3 layer and a 10 nm thick MgO layer was fabricated using pulsed laser deposition. A photonic bandgap with a centre wavelength at ~464 nm has been observed in the transmission measurement which is consistent with simulation using the plane wave expansion (PWE) method and the transfer matrix method. A 2 nm shift towards the longer wavelength is observed when a dc voltage of 240 V (E~12 MV/m) has been applied across the coplanar electrodes on the film surface. The result suggests that the electric field induced change in the refractive index of Ba0.7Sr0.3TiO3 is ~0.5%.

The shear and transverse wave velocities of Ba0.7Sr0.3TiO3 ceramics as a function of temperature have been determined using the ultrasonic immersion technique. A large variation in the wave velocities has been observed across the Curie temperature of Ba0.7Sr0.3TiO3. A phononic crystal composed of Ba0.7Sr0.3TiO3 square rods (width = 200 µm) in an epoxy matrix (width 65 µm) has been fabricated using the dice-and-fill method. The temperature dependence of the bandgaps of the phononic crystal has been measured by the reflection spectra obtained using the ultrasonic pulse-echo technique. Thermal tuning of the phononic bandgap has been observed and the results are in good agreement with the phononic bandstructure calculation by the PWE method.

Acknowledgement:
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**Structural and Optical Analysis of Self Sustainable Spindle Shaped ZnO Nanostructured Thin Films**

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Chemical bath deposition (CBD) is an inexpensive and low temperature method often perceived to impart some advantages to fabricate complex nanostructured thin films. Highly oriented 1-D Zinc oxide (ZnO) nanostructures derived by CBD technique have been widely investigated for their electrical, photoelectronic, catalytic, and photochemical properties. The nucleation and grain growth of 1-D ZnO in the low temperature CBD bath were catalyzed with the aid of metal ion pre-activation so called “seed layer” deposition. But, the presence of separate seed layer, is undesirable for charge transport in optoelectronic devices. Therefore, a modified CBD technique is essential for preparing highly oriented ZnO nanostructured films without seed layer. Accordingly, in the present work, highly oriented spindle shaped ZnO nanostructured thin films were grown on glass substrate without seed layer using triethanolamine (TEA) as a complexing agent in aqueous medium. Tunable spindle shaped ZnO nanostructures were achieved by varying concentration of TEA (0.1, 0.2 and 0.3M) in the chemical bath at 70°C in aqueous medium. The film deposited at 400 mTorr had the highest (002) peak with the largest estimated grain size. Outside this window, the crystallinity and c-orientation of grains is lost. The microstructure of the films was investigated by Atomic Force microscopy (AFM). Optical transparency of the films was about 85%. The transparency of the films decreased with substrate heating. The films produced were highly resistive, which might provide new alternatives for the synthesis of ZnO thin films aimed for SAW devices.

**SrZrO$_3$-based Thin Films For Resistive Switching Memory Application**

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Several emerging nonvolatile memories (NVMs) such as ferroelectric memory, magnetoresistive rams and ovonic universal memory are being developed for possible applications. Resistive random access memory (RRAM) is another interesting competitor in the class of NVMs. The RRAM is based on a large change in electrical resistance when the memory film is exposed to voltage or current pulses. The ideal RRAM should have the superior properties of reversible switching, long retention time, multilevel switching, simple structure, low operating voltage and current, and small size. Metal oxides and organic materials were found to have resistive memory properties. This presentation reviews the ongoing research and development activities on future resistive NVMs technologies incorporating metal oxides memory materials and describes the possible basic mechanisms for their bistable resistance switching behaviors. The current results of SrZrO$_3$-based thin films resistive memory research in our Laboratory are reported. The effect of processing and dopant on the switching properties of SrZrO$_3$-based thin films and consequently the devices is discussed.
A02103-03621

Electrical Transport of Ce doped La-Ca-Mn-O System

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We have studied the low temperature (below transition temperature $T_P$) resistivity behavior of Ce doped La$_{1-x}$Ca$_x$MnO$_3$. Polycrystalline samples of compositions La$_{0.95}$Ca$_{0.05}$MnO$_3$ (sample1), La$_{0.9}$Ca$_{0.1}$MnO$_3$ (sample2) and La$_{0.85}$Ce$_{0.15}$MnO$_3$ (sample3) were prepared by solid state route method. From the XRD studies, it is revealed that all samples exhibit the single phase and all the peaks indexed according to orthorhombic structure (Space group Pbnm) having variant lattice parameters. The temperature dependence of the resistance was measured between 4 K to room temperature. It is found that all samples show a systematic variation in metal to insulator transition at room temperature $T_P$. The systematic variation in metal to insulator transition at room temperature $T_P$ of Ce doped La-Ca-Mn-O system is attributed to collapse of the minority spin band. The deviation from this quadratic temperature below 55K is attributed to collapse of the minority spin band. The data showed $T^2$ dependence from 55 to 190 K consistent with the electron - electron scattering. In the temperature range above 190 K, the two-magnon cross-correlation response, materials whose electricity is induced by spin ordering are of vital interest. In this presentation, we would like to discuss two typical examples of magnetically-induced electric polarization in well-known ferrites with distinct crystal structures; One is Y-type hexagonal Ba$_2$(Mg,Zn)$_2$Fe$_{12}$O$_{22}$ and the other is orthogonal RFeO$_3$ with perovskite-type structure.

A02132-03672

Magnetic Control of Electric Polarization in Fe-oxides

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5. Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Miyagi, Japan

Magnetic materials which possess electric polarization as well have recently been investigated extensively from the viewpoints of fundamental interest and possible application for devices. Particularly, to obtain gigantic cross-correlation response, materials whose electricity is induced by spin ordering are of vital interest. In this presentation, we would like to discuss two typical examples of magnetically-induced electric polarization in well-known ferrites with distinct crystal structures; One is Y-type hexagonal Ba$_2$(Mg,Zn)$_2$Fe$_{12}$O$_{22}$ and the other is orthogonal RFeO$_3$ with perovskite-type structure.

A02132-03672

Dilutely doped Anatase TiO$_2$: New Science and Applications

Satishchandra OGALE
Physical and Materials Chemistry Division, National Chemical Laboratory, Maharashtra, India

Dilutely doped titania have been at the focus of scientific attention for quite some time due to their unique transport and optical properties as well as realization of room temperature ferromagnetism therein when doped with transition element ions. In this talk I will present and discuss some of our recent research results on thin films of niobium and cobalt doped anatase TiO$_2$. In the case of Nb doped TiO$_2$ films grown under a specific window of oxygen pressure we provide clear evidence of cation vacancy induced magnetic effects by using the electrical characterization, x-ray photoelectron spectroscopy (XPS) and x-ray absorption spectroscopy (XAS) measurements. These results are adequately supported by first principle calculations. In the case of low temperature grown cobalt doped anatase titania, we show significant enhancement of the so called resistive switching effect. We attribute this enhancement to the management of oxygen vacancies in the film due to the necessity of charge balance by the non-isovalent nature of the dopant. Finally some results on dilutely doped nanophase titania will also be presented and discussed.

2 K. A. Bogle, M. N. Bachhav, M. S. Deo and S. B. Ogale, Advanced Functional Materials (Submitted)

Work supported by BRNS (DAE) and DST, Govt. of India.

A02126-03658

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field. Zn doping reduces the polarization rapidly, while changing the magnetic anisotropy. The observed behavior of polarization is discussed in terms of the spin-current model.

At the ground state of $R\text{FeO}_3$ both of the Fe and $R$ spins show antiferromagnetic structure with weak ferromagnetic moment of Fe. When the ordered $R$ moment has the same component as the Fe spins, for example, $A$, or $G$, component, then the ferroelectric polarization show up along c axis due to the exchange striction between Fe spins and $R$ moments. In these compounds, magnetic manipulation of electric polarization is also demonstrated.

Tunable Two-Dimensional Electron Gases at Oxide Interfaces

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Two-dimensional electron gases (2-DEGs) based on conventional semiconductors such as Si or GaAs have played a pivotal role in fundamental science and technology and lead to intriguing new devices, e.g., the high electron mobility transistor. Recent work has shown that 2-DEGs can also exist at oxide interfaces. These electron gases typically result from reconstruction of the complex electronic structure of the oxides, so that the electronic behavior of the interfaces may differ from that of the bulk. In the presentation I will give an overview of our studies of the unusual electronic properties of the special interface between the two insulators LaAlO$_3$ and SrTiO$_3$ and report on the latest results of our experiments where the electronic properties of the interface can be tuned by external electric fields - a key function for such oxide interfaces to be used in nanoscale electronic devices.

Mechanical Properties of Graded TiC$_x$N$_y$ Films Deposited by HCD Technique

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Properties of mono- and graded hard coating of TiCN, deposited on AISI-M2 tool steel produced by a low temperature Hollow cathode discharge (HCD) ion coating technique have been examined. OM, SEM, roughness testing and hardness testing were used to study the film composition, structure and mechanical properties. Experiments indicated that the increment of carbon element will increase the deposition rate, while the impact on deposition rate by adding Argon gas is not that predicative. Under the low temperature, the graded films appear have an over-stoichiometric composition. The appearance of a dimple structure affects the characteristics of the thin film. Moreover, on the examined condition of GC1, the influence of dimple structure on the film’s roughness can reach up to 3.8%. In this research, graded film is better than monolayer film in terms of higher hardness value and better adherence.

Electric-field Control of Magnetic Domain Structures in Ferroelectric/Ferromagnetic Heterostructures

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Multiferroics are multifunctional materials in which ferroelectricity, ferromagnetism, and ferroelacticity coexist. Due to the strong couplings among spin, charge, orbit, and dipole, some novel physical properties and functions have been found in multiferroic materials. For example, one can control magnetic moment by applying an electric field, and control electric polarization by applying a magnetic field, respectively, due to the magnetoelectrical coupling between the coexisting ferroelectricity and ferromagnetism. Multiferroic materials are of important potentials especially for novel memory with a high density, magnetic/electric/press sensors with an extremely high sensitivity, and also for the renascent spintronic devices. So far, there still exist some critical obstacles or challenges in multiferroic materials with a single phase. (1) very few types of single-phased multiferroic materials exist in nature, and high pressure is needed usually to synthesize single-phased multiferroic materials; (2) the magnetic transition temperature is lower than room temperature usually; (3) the magnetoelectrical coupling is weak and only exist at low temperature (below magnetic and ferroelectric transition
temperature), which is a severe drawback for realizing the cross-control of physical properties. Comparing to the single-phased materials, multiferroic heterostructures or superlattices composed of ferromagnetic and ferroelectric layers with high phase transition temperatures and a large magnetization/polarization, respectively, are more promising for both fundamental researches and future applications.

In this talk, we present the electric-field control of magnetic domain structures in Fe/PMN-PT heterostructures. An Fe film of 100 nm was deposited on PMN-PT substrates with 15 nm Cu capping by e-beam evaporation. The magnetic domain was observed by means of magnetic force microscopy (MFM) with an applied electric field in situ between Fe film and PMN-PT substrates. The magnetic domain of Fe films on PMN-PT shows a striped structure without applied electric field, however, the configuration changes gradually with increasing the applied electric field, and disappeared finally when the electric field was 56V/cm. The electric-field control process of magnetic domain structures in Fe/PMN-PT heterostructures is reversible after removing the applied electric field. More results and discussion will be given in this talk.

Detection of Low Concentration CO in Air Using ZnO:Al/Au Thin Films Deposited By Co-sputtering
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Al-doped ZnO (ZnO:Al) thin films were deposited on self-assembled Au nanodots fabricated on SiO2/Si (100) substrates by rf co-sputtering from separated metallic Zn and Al targets for CO gas sensing application. Morphological analysis of the films obtained by SEM and AFM revealed a very high surface roughness compared to the case without Au dots. The surface composition of ZnO:Al/Au thin films analyzed by XPS showed the surface structure like ZnO/Au composites with the Au/Zn atomic ratio on the surface = 0.165. The ZnO:Al/Au thin films were sensitive to CO even at a low concentration of 5 ppm and exhibited a high sensitivity to CO in dry air in a wide range of working temperature from 150 to 400 °C with a maximum sensitivity of 640% to 20 ppm CO gas at a temperature of 250 °C. Moreover, the ZnO:Al/Au thin film sensors showed fast response and recovery to CO gas in comparison with conventional Al-doped ZnO films. These enhancements are attributed to the double role of Au dots; Au dots played the role of a buffer layer for the abnormal grain growth of ZnO:Al thin films, resulting in high surface roughness and the role of catalyst through the spillover effect during gas detecting process for the ZnO:Al/Au thin films. These results suggest that the ZnO:Al/Au thin films are suitable for the detection of low levels of CO in air: <9 ppm - limit value for the protection of human health - according to European and US standards for the maximum daily 8-hour mean of exposure to CO.

Characterization of TiO2 Coating Prepared by a Modified Radio Frequency Sputtering Deposition Method
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TiO2 thin films were deposited on glass surface by a radio frequency sputtering (RF Sputtering) deposition method. After coating, they were annealed for 2 hrs from 350°C to 500°C. XRD, TG, AFM, RS (Raman Spectroscopy) and SEM were applied to study and characterize the samples. The effects of deposition parameters on crystal structure, thickness, surface characterization, photo catalytic activity and hydrophilic properties of the films were evaluated.

The effects of relative oxygen and argon partial pressure, cathode current, bias voltage external magnetic filed, coating time, vacuum pressure and post annealing temperature were studied and evaluated.

The composition and phase growth from amorphous phase were also analyzed, which were Anatize and Rutile phases.
The Effects of Annealing Temperature and Barium Oxide on the ZnO Varistor Degradation

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One of the main problems in the route of ceramic semiconductor varistor development is the degradation and increasing of the leakage current. In this research, the effect of Barium Oxide and post-annealing on leakage current and electrical properties were studied. Due to the large ion size of Ba\(^2+\) (~0.134 nm), it has low solubility in the ZnO structure. The amount of BaO was varied from 0.25 mol% up to 1 mol%. The amount of other additives (\(\text{SnO}_2\), \(\text{Co}_3\text{O}_4\), \(\text{MnO}\), \(\text{Cr}_2\text{O}_3\), \(\text{NiO}\) and \(\text{B}_2\text{O}_3\)) was kept constant.

The samples were sintered at 1250°C for 1 hr and annealing was conducted 2 hr at 600°C. The electrical properties were measured and the microstructure and phase analysis were conducted by SEM and XRD.

The grain size was about 12.3 mm to 12.6mm. The relative density was lowered with BaO content (from 98.6% down to 94.9%). The amount of leakage current was lowered from 7(mA) down to 4.7 (mA). The non-linear index was increased from 48 to 56 (at 0.15 mol% of BaO) and then was lowered down to 49 (at 1 mol% of BaO). The amount of absorbed energy was changed from 115 J/cm\(^3\) (in the sample without BaO) to 195 J/cm\(^3\) (in the sample with 0.5 mol% of BaO) and down to 145 J/cm\(^3\) (in the sample with 1 mol% of BaO).

The results are discussed with respect to BaO amounts and formed phases in the system and microstructure features.

Vibration Damping with Control on Friction due to Oxide Thin Film Layer to Eliminate Noise in a Brake Friction Composite

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R&D, Daimler AG, Stuttgart, Germany

Novel brake friction composite design with heterostructures has been designed for Mercedez Benz model for braking to eliminate noise while keeping the good virtues of friction and wear. Fundamental mechanisms of control of the advanced matrix with oxide film layer controlling the friction characteristics was achieved in this exercise with good control on composite material wear. AS A DESIGN INPUT to minimize thermal fatigue damage the difference between the thermal expansion coefficients of the matrix and the fiber materials were minimized while designing the system. The issue was addressed with oxide layers as a flexible fiber coating helped ease the thermal stresses at the interfaces which would otherwise affect the friction by an incremental value and would have otherwise caused noise.

Oxide thin film layer and its role in friction control has been validated for vibration with an increased random motion of molecules resulting in deformation. These vibrations have dampenned out but bodies got heated up to as high as 450degree C. Toughness as a measure of modulus relating to noise and increase in deformation strength was achieved in this exercise. An advanced software was designed and scheduled for testing vibration and noise resulting in accurate measurements.

Highly Oriented PZT Films Grown On Si with Improved Ferroelectric and Piezoelectric Properties

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2. Faculty of Engineering Physics & Nanotechnology, Vietnam National University, Viet Nam
3. Faculty of Chemistry, Vietnam National University, Viet Nam

Thin films perovskite-type oxide Pb(Zr,Ti)O\(_3\) (PZT) have been grown on Si(001) substrate by pulsed laser deposition. To minimize the effect of the native amorphous SiO\(_2\) layer on the Si surface, a thin buffer layer of YSZ has been pre-deposited in Ar then O\(_2\) environments prior to the deposition. SrRuO\(_3\) and La(Sr,Mn)O\(_3\) were chosen as the next buffer layers with different combinations for subsequent PZT deposition. The obtained films are highly oriented with smooth surface. By replacing the buffer layer of SrRuO\(_3\) by La(Sr,Mn)O\(_3\) and using a suitable substrate temperature, the oriented direction of PZT films can be tuned from (110) to (001) direction. The films exhibit high leakage current in the range of 10\(^{-6}\) A/cm\(^2\) and well-defined butterfly shape of capacitance-voltage dependence. The polarisation-electrical field measurements show hysteresis loops with high remanent polarisation around 25 \(\mu\)C/cm\(^2\).

To characterize piezoelectric properties, four-point bending type samples have been prepared utilizing lithography technique. High effective transverse piezoelectric coefficients \(e_{31}\) of above 10 C/m\(^2\) were determined by using a double beam laser interferometer. The obtained characteristics of these PZT films are important for piezoelectric thin film fabrication and design for practical applications as silicon-based devices in micro-electromechanical systems (MEMS) technology.
Preparation and Photocatalytic Properties of the Mesoporous Fe-doping Nanometer TiO$_2$

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In order to extend the optical absorption of TiO$_2$ to the visible spectral region and improve the photocatalytic property of the TiO$_2$ photocatalyst, various transition metal ions have been added into the titania oxide as doping agent. In the present study, Fe-doped mesoporous TiO$_2$ photocatalyst was prepared by hydrolysis precipitation method with Ti(OBu)$_4$ as precursor, HAC as complexation reagent, and Fe(NO$_3$)$_3$·9H$_2$O as doping agent at room temperature. The composition and microstructure of the prepared mesoporous Fe-TiO$_2$ were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM) and N$_2$ adsorption-desorption isotherm. The photocatalytic properties of mesoporous TiO$_2$ in the degradation of methyl orange solution were determined. The effects of different molar ratio of doped agent, heat treatment temperature and concentrations of TiO$_2$ on the photocatalytic properties of mesoporous Fe-doping TiO$_2$ were also studied investigated. The optimum molar ratio of Fe/Ti and heat treatment temperature were obtained. The results showed that with doping of Fe, anatase-TiO$_2$ became more stable and the phase transformation from anatase to rutile was blocked. The particle sizes of Fe-TiO$_2$ were in the range of 10~20 nm. The mesoporous titania with Fe/Ti molar ratio of 0.05 shows a narrow distributing pore diameter of 9.7 nm, large special surface area of 103.407 m$^2$/g and pore volume of 0.061 cm$^3$/g. The optimal heat treatment temperature of Fe-TiO$_2$ powder was 500°C. Under these conditions photocatalytic decomposition rate of methyl orange solution reached the highest, up to 99% under 3 h irradiation with ultraviolet light.

Nonlinear Dynamics of Domain Wall Propagation in Epitaxial Ferroelectric Thin Films

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Ferroelectric (FE) domains have been studied for past decades because of scientific importance in microscopic aspects such as multi-domain formation, stability, and pattern at equilibrium as well as technological applicability in multi-functional devices such as FE random access memories, actuators, and sensors. Quite recently, lots of piezoresponse force microscope (PFM) studies have provided us new insights on the properties of FE domains, including inhomogeneous nucleation process and the fractal nature of their rough surfaces. Note that most works on FE domains have been focused on their static properties. In spite of its scientific and technological importance, we have limited understandings on how the FE domain wall propagates in terms of time. We suggest to prospect FE domain wall from the view of nonlinear responses, which follow the predictions of the statistical physics on surface growth.

We investigated the ferroelectric domain wall propagation in epitaxial Pb(Zr,Ti)O$_3$ thin film over a wide temperature range (3-300 K). To widen the accessible region of $T$ and $E$, we used switching current measurement results, combined with direct velocity data from PFM images. Using this newly developed technique, we measured the domain wall velocity under various electric fields and found that the velocity data is strongly nonlinear with electric fields, especially at low temperature. We found that, as one of surface growth problems, our domain wall velocity data from ferroelectric epitaxial film could be classified into the creep, depinning, and flow regimes due to competition between local pinning sites and elasticity. The measured values of velocity and dynamical exponents indicate that the ferroelectric domain walls in the epitaxial films are fractal and pinned by a disorder-induced local field. This work provides us new insights on how domain walls propagate inside epitaxial FE thin films.
**Oxide Nanoelectronics On Demand**

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Electronic confinement at nanoscale dimensions remains a central means of science and technology. We demonstrate nanoscale lateral confinement of a quasi-two-dimensional electron gas at a LaAlO3-SrTiO3 interface. Control of this confinement using an atomic force microscope lithography technique enabled us to create tunnel junctions and field-effect transistors with characteristic dimensions as small as 2 nanometers. These electronic devices can be modified or erased without the need for complex lithographic procedures. Our on-demand nanoelectronics fabrication platform has the potential for widespread technological application.

**Effect of Calcium Substitution on Dielectric Behavior of BaTi0.85Sn0.15O3**

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Effect of Calcium substitution at Ba site in the system Ba1-xCa2xTi0.85Sn0.15O3 has been investigated. Compositions in the range 0.14≤x≤0.20 were prepared by solid state ceramic method. Solid solution was found to be formed in the compositions with x=0.14 and 0.16 among the compositions investigated. These samples have cubic structure. Scanning Electron microscope shows presence of grains in the range.

These samples exhibit diffuse phase transition at low temperature. Diffuseness of the peak increases with increase in x. Variation in temperature of maxima, Tm in dielectric constant, ε′ vs T plots with x indicates that for x=0.14 Ca2+ exclusively occupies Ba site while in x=0.16, a small amount (0.015 atomic percent occupies Ti site).

**Effect of Different Ge Concentrations on Phase Change Materials**

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Phase Change Random Access Memory (PCRAM) is one of next-generation non-volatile memory with the most potential due to its many good memory characteristics such as long data retention; high endurance and fast write performance. PCRAM is based on phase change from high resistance amorphous state to low resistance crystalline state and vice versa in nano-timescale. The GeSbTe (GST) system is a commonly used ternary system with many widely used phase change materials such as Ge2Sb2Te5 which has fast and repeatable switching performance. However, the actual phase change mechanism and the effect of each element on the phase change characteristics
remain unclear. There is a search for the ideal phase change material with high switching speed and high stability. There were previous works that suggests that Ge in GST plays a crucial part in the nano-second phase change effect through an umbrella flip of its bonds during phase change. In this work, different Ge concentrations are mixed in different phase change materials such as Ge$_2$Sb$_2$Te$_5$ and Sb$_2$Te$_3$ and the effect of Ge on important phase change characteristics such as crystallization and melting point would be of great importance in the future search of the ideal phase change material.

According to the polarization-dependent $O$ K-edge XAS study, the piezoelectricity of GFO is reasoned to be closely related to a small off-center distortion along the crystallographic $b$-axis while the magnetic easy axis is oriented parallel to the $c$-axis with a non-negligible contribution of the orbital magnetic moment to the total magnetic moment. Thus, it is highly important to prepare a thin-film capacitor with its growth direction parallel to the $b$-axis for systematic experimental investigations of its piezoelectric/ferroelectric properties as well as its direction-dependent magnetoelectric (ME) coupling characteristics.

Considering the above described backgrounds, we have fabricated Ga$_{2-x}$Fe$_x$O$_3$ (GFO with $x=1$–1.2) thin films on the (111) plane of yttrium-stabilized zirconia (YSZ) substrate which is coated with an epitaxially grown Pt layer by using pulsed laser deposition (PLD). XRD analysis revealed that our GFO films were preferentially grown along the [010] direction (i.e., along the $b$-axis), which is suitable for ferroelectric and ME coupling characterizations. In the presentation, we will discuss the onset of the ferroelectricity and the magnetodielectric responses (i.e., the change in the relative dielectric permittivity under a bias magnetic field) in details.
heterojunction solar cell. These promising results will be presented and discussed.

A02320-03974

Ferroelectric to Paraelectric Transitions in the System $\text{Ba}_{1-x}\text{La}_x\text{Ti}_{1-x}\text{Cr}_x\text{O}_3$

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Effect of simultaneous substitution of La and Cr at Ba and Ti site respectively in equimolar fractions in the system $\text{Ba}_{1-x}\text{La}_x\text{Ti}_{1-x}\text{Cr}_x\text{O}_3$ has been studied in the single phase solid solution compositions which are prepared by solid state diffusion controlled thermochemical reaction. Formation of single phase has been confirmed by powder X-ray diffraction. Microstructure has been studied using Scanning Electron Microscope. Measurement of dielectric properties at a few selected frequencies shows that Curie temperature $T_c$ decreases with increase in $x$ and nature of ferroelectric to paraelectric transformation also changes.

A02320-04832

Magneto-electrical Studies in LSMO-BTO Bulk and Nano Composites

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In recent years, the multiferroics have drawn the attention of scientists world wide. In view of this, an effort has been made to synthesize bulk and nano composite of the system $\text{La}_x\text{Sr}_y\text{MnO}_3$ with $x=0.10$. In this process, a composite sample in the bulk form by solid state route and thin film of thickness 80nm on STO and LAO substrates by dc magnetron sputtering method have been synthesized. For this a typical composition $\text{La}_x\text{Sr}_y\text{MnO}_3$ (which is magnetic) has been prepared by chemical route and $\text{BaTiO}_3$ (which is ferroelectric) by solid state route. Single phase formation of these samples has been confirmed separately by using x-ray diffraction studies. This typical composite is expected to exhibit multifunctional properties.

Resistivity measurement on the bulk sample over the temperature range 77 K to 373 K exhibits a peak around 198 K which appears due to metal-insulator transition ($T_{\text{MI}}$) around the peak temperature. The sample of composite thin films has been found to have highest intense peak of LSMO and BTO which confirmed the formation of LSMO-BTO thin film composite. Resistivity measurements of this film on LAO and STO substrate have shown $T_{\text{MI}}$ at 203K and 198K respectively. It has been observed that composite on LAO substrate has shown lower resistivity as compared to the thin film on STO substrate. However, the sample with thin film on LAO substrate has exhibited higher magnetoresistance (MR) in the magnetic field of 0.3T. In view of this, when the sample with LAO substrate was prepared in the atmosphere of mixture of gases Ar/O$_2$ with ratio 80/20 and 60/40. The magnetoresistance has been found to improve when heat treated in the atmosphere of Ar/O$_2$ mixture with ratio 80/20.

A02335-04012

Enhanced Efficiency of Dye-Sensitized Solar Cells Utilizing ZnO Thin Film with Different Light-scattering Layers as Photoelectrodes

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Dye-sensitized solar cells (DSSCs) have found a wide practical application as a cost-effect alternative to p-n junction solar cells. However, the improvement of solar-to-electric energy conversion efficiency is an urging problem for commercial application. An effective strategy to the enhancement of efficiency is to build a light scattering layer with both enhanced photo-capture efficiency and the optical adsorption in photoelectrode films in DSSCs.

In this work, a novel double-layered ZnO film as photoelectrode was prepared and used in DSSCs that allowed the significant improvement of solar cell performance ranging from the short circuit current to open circuit voltage and energy conversion efficiency ($\eta$). The bilayer-structural ZnO film is composed of an overlayer of 200-300 nm-sized plate-like ZnO and an underlayer of ZnO monodisperse aggregates assembled by ~20 nm-sized ZnO particles. The $\eta$ values of DSSCs based on submicrometer-sized plate-like ZnO and ZnOmonodisperse aggregates were 0.81% and 2.34%, respectively. But, after submicrometer-sized plate-like ZnO particles were introduced as an overlayer coating on the underlayer covered with uniform ZnO monodisperse aggregates, $\eta$ enhanced from 2.34% to 3.44%, corresponding to a 47% increment. The improved performance is explained by the enhanced double light-scattering layers and effective light absorption.
This work points out the importance of light-scattering layers in DSSCs as a key to improve light-to-electric conversion efficiency, and may provide useful insight for different light-scattering applied in semiconductor thin films as photovoltaics and sustainable materials.

A02361-04040

Growth Control of ZnO Nanorod Density by Sol-gel Method

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Preparation of ZnO nanorods and their growth control over density by the sol-gel processing conditions were studied as a function of seed layer thickness as well as annealing temperatures. The seed layer films were deposited both on transparent conducting oxide (FTO) and glass substrates at different thicknesses from 50 – 100 nm by spin coating method were for the synthesis of ZnO nanorods. Equimolar concentrations of the zinc nitrate hydrate and hexamethylenetetramine (HMT) used as the precursor solutions were taken in a 100 ml beaker and the substrates were placed at the bottom of the beaker which is covered with cap and heated to 70 to 95°C, and maintained for different growth times. Finally, the substrates were removed from the aqueous solution, rinsed with double distilled water, and allowed to dry in air at room temperature. The experiment was repeated for various precursor concentrations and growth times. Experiments carried out at reaction temperatures below 65°C indicated no growth of ZnO nanorods, but formed opaque nature of films, which may be attributed to a thermal barrier for the growth of nanorods. ZnO nanorods synthesized at 85°C in the present work exhibit a good crystalline structure with a hexagonal plane. It is observed that the density of nanorod growth increases as the precursor concentration increases as well as at 70 nm seed layer thicknesses as well as annealed at 200°C. The diameters of ZnO nanorods are found to increase from 150 - 400 nm as the concentration increases. Further studies on the growth mechanisms at different substrates and with processing conditions are underway.

A02400-04144

ZnO Based Transparent Conductive Multilayer Films for Dye Sensitized Solar Cell Applications

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Multilayer coatings consist of three alternative layers ZnO/Ag/ZnO (named as ZAZ) have been applied to dye sensitized solar cells (DSSC). We have used the simultaneous RF and dc sputtering method for deposition of ZnO and Ag layers.

The electrical and optical properties of the transparent conductive multilayers were studied using four-point probe and UV-Visible spectrophotometer. Mesoporous TiO₂ electrodes for DSSC were coated on multilayer ZAZ by spin coating and low temperature sintering method. A ZnO covered TiO₂ (denoted as ZnO/TiO₂) film was also prepared by incorporating small quantity of ZnO in a TiO₂ matrix by chemical vapor deposition method. The properties of the film and DSSC are compared with well known transparent metal oxides ITO and FTO. These DSSC on the ZAZ coating yielded an overall cell efficiency of 4.91 % at one sun light intensity. The dye sensitization process with the low cost mercurochrome is sensitive in this case of ZnO based multilayer.

A02408-04125

Effect of Volume Fraction and Molding Temperature on the Electric Properties of PZT/PVC Composites

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In recent years, combining a ferroelectric ceramic and a polymer matrix to form a flexible composite with coupling properties in between that of two individual phases has been pursued for sensors in medical applications and hydrophones. For hydrophone applications, a useful piezoelectric material should have high piezoelectric voltage coefficient (g₃₃), piezoelectric strain coefficient (d₃₃) and high electromechanical coupling coefficient (Kₚ), low density and acoustic impedance (Z). The PZT/nanocrystalline PVC composites are prepared by hot-pressing technique. The microstructure of all composites was investigated by XRD and SEM. The effect of volume fraction and molding temperature on dielectric, piezoelectric properties and P-E hysteresis of PZT/PVC composites was investigated. The optimal electric properties have been obtained in 0.5PZT/0.5PVC composite at molding temperature being 150°C, showing, eᵣ=45.66, tanδ=0.0286, d₃₃=18.5 pC/N, g₃₃=45.78 mV·m·N⁻¹, Kₚ=0.226, d₃₃·g₃₃=0.85×10⁻¹⁵ Pa⁻¹, Z=11.25×10⁶ kg·s⁻¹·m⁻², and could be used for hydrophone materials in the further.
Photoemission Study on the Degree of Ta doping on Anatase (001) and Rutile (001) TiO₂ Single-crystal and their Structure Stability

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The mechanism of whether the Ta really substituting the Ti atom or not in Ta-doped TiO₂ and their stability in anatase and rutile phase, was investigated systematically using X-ray Photoelectron spectroscopy (XPS) and Ultraviolet Photoemission (UPS). By doing partial atomic substitution (i.e. in this study 5% Ta) to Ti, we found that Ta linewidth in rutile phase is more narrower compared to that of anatase, indicating that the Ta is more localized in rutile phase rather than in anatase phase. From UPS we found that the valence band of the Ta-doped TiO₂ (both for anatase and rutile) near the Fermi level is shifted to the lower binding energy, indicating the n-type doping of the system. Furthermore, systematic sputtering using Ar⁺ on the surface of these two different phase of TiO₂ provides the evidence that it is more easy to create the defect state, i.e. Ti³⁺ state, in anatase phase rather than in rutile phase. These results indicated that rutile phase is more stable structure compare to that of anatase.

Large-Scale Growth of Single-Crystalline TiO₂ Nanowires and Their Visible-Light Photocatalytic Activity

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Titanium dioxide (TiO₂) has been extensively studied as a promising material for photocatalysis, and gas sensor applications. High-efficient photocatalytic reactivity of TiO₂ can be very promising for electrodes of solar cells, the elimination of pollutants, and the photogeneration of hydrogen from water. However, photocatalytic efficiency of TiO₂ under visible light is very low because of its large band gap of 3.0-3.2 eV. Many approaches have been made to improve the photocatalytic activity of TiO₂ under visible light irradiation, such as transition metal doping and non-metallic doping. Furthermore, one-dimensional (1-D) TiO₂ nanostructures, such as nanowires and nanotubes, can yield high photocatalytic activity because of their high specific surface area. In this presentation, we report some results of TiO₂ nanowire growth by oxidation of Ti films with ethanol as oxygen source and their visible-light photocatalytic characteristics.

TiO₂ nanowires were grown on as-sputtered Ti films by a direct oxidation method. Ti films were initially deposited onto Si (100) substrate by sputtering system. The Ti films were subsequently loaded into a quartz-tube furnace. The ethanol was used as oxygen source, and bubbled at 10 °C with the gas flow rate at 150 cm³/min. The ethanol vapor was introduced into the furnace for 1-90 min. The chamber pressure and growth temperature were varied in the range of 4-100 Torr and 600-850 °C, respectively.
The uniform and straight TiO$_2$ nanowires were formed with a high density. The length and diameter of the nanowires was ~7-9 µm and ~40-50 nm, respectively. X-ray diffractometer and high-resolution transmission electron microscopy studies revealed that the nanowires had a single-crystalline structures and grew along the [110] axis. The TiO$_2$ nanowires showed high-efficient visible-light photocatalytic activity, which were evaluated by the decomposition of methylene blue under visible light excitation. We will further discuss the growth mechanism and the photocatalytic characteristics of TiO$_2$ nanowires under visible and ultraviolet light.

**Preparation and Properties of Lead-free 0.94(K$_{0.5}$Na$_{0.5}$)NbO$_3$-0.06LiNbO$_3$ Piezoelectric Ceramics with Excess Alkali Elements**

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Since the discovery of PbZrO$_3$-PbTiO$_3$ (PZT) ceramic in the 1950s, lead-based piezoelectric materials have been widely used in sensors, actuators, transducers, and many other electronic devices. The increasing applications of these devices generate a more and more aggravated environmental concern because these traditional lead-based ceramics typically contain more than 60% lead by weight. During the last several years, intensive efforts have been made to develop lead-free piezoelectric materials to replace the lead-based compositions. One of the most promising candidates as lead-free piezoelectric materials is potassium sodium niobate system. However, the potassium and sodium precursor chemicals are highly volatile during high temperature sintering, which brings about the difficulty in the composition control for the resulting ceramic. Therefore it is crucial to develop appropriate processing methods and reduce the sintering temperature to prevent evaporation of alkali elements during heat treatments.

In this work, the preparation method for 0.94(K$_{0.5}$Na$_{0.5}$)NbO$_3$-0.06LiNbO$_3$ (KNN-LN) piezoelectric ceramics by conventional solid state reaction has been refined. Different amounts of excess K$_2$CO$_3$, Na$_2$CO$_3$, Li$_2$CO$_3$ have been introduced and investigated systematically. The perovskite phase of KNN-LN ceramics have successfully been obtained with sintering temperature as low as 1000°C. The excess alkali elements have promoted the ceramic densification process through liquid phase sintering mechanism. However, it has been observed that over excess alkali carbonates added into the system could lead to an undesired Li-rich second phase of Li$_3$NbO$_2$.

There is also a decrease in the density when the amount of excess carbonates increases, which has resulted in higher dielectric losses. The optimal excess carbonates added into the KNN-LN system that produces high relative density at 1000°C has been determined. The electrical properties of the obtained samples, including loss tangent, dielectric constant, remnant polarization, and piezoelectric constant (d$_{33}$) are to be reported.

**Detailed Studies of Multiferroic Properties in LaFeO$_3$-PbTiO$_3$ Solid-solutions**

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Multiferroic materials are those that possess more than one ferroic properties that are coupled. However, in literature this term loosely refers to materials that exhibit both ferroelectric and magnetic orders. Coexistence of ferroelectric and magnetic orders in a material in itself poses a challenging problem. Magnetoelectric multiferroic materials are promising candidates for the applications such as spintronics, information storage, magnetic field sensors and actuators. Usually solid solutions have too week magnetoelectric coupling to be measured at room temperature. Invariably, transition metal d-electrons that are essential for the formation of magnetic moment disturb the lattice distortion required for ferroelectric behavior, making them poor insulators. Broad transitions in dielectric spectra are observed in many of these multiferroic candidates; in literature these broad transitions are either reported as relaxor ferroelectrics with frustrated ferroelectricity or are explained on the basis of the Maxwell–Wagner capacitor model. An important issue faced by the researchers in the field is thus establishing the origin of broad transitions usually observed in dielectric spectra.

In this report, we introduce a new multiferroic candidate (Pb$_{1-x}$La$_x$) (Ti$_{1-x}$Fe$_x$)$_3$O$_7$; $x$=0.1, 0.2, 0.3 with finite magnetoelectric coupling at room temperature. The detailed dielectric studies exhibit a unique coexistence of a pure ferroelectric transition and a frequency dependent broad transition. The maxium of this frequency dependent broad transition are observed to shift towards lower temperatures with increasing LF content (x). Temperature dependent P-E loops clearly distinguish a pure ferroelectric hysteresis and lossy loops (corresponding to these broad transition related dielectric anamoly). The lossy hysteresis loops, which were taken in the temperature range of broad transitions, show frequency dependent behavior. This is unusual for good ferroelectric and verifies that these broad transitions have no ferroelectric origin. Dielectric data in the frequency range of broad transitions were fitted to the Vogel–Fulcher law. Fitting parameters for x=0.2 sample thus obtained, are $E_a$ =0.6906 eV, $\omega$=4.18x10$^{11}$ Hz, and...
The FIR transmission spectra show four main phonon vibration energies exhibit splitting and shifting as Co doping increases in the sample. A detailed analysis of the FIR and Raman measurements is presented.

Magnetization (M) vs. Temperature (T) profile (ZFC and FC) clearly indicates that these broad transitions in dielectric spectrum may have magnetic origin. Also low and high temperature magnetic measurements were done.

The magnetoelectric coupling measurement methods are very carefully chosen to nullify any type of the artifacts due to Maxwell Wagner capacitor and magnetoresistance. The enhancement of the magnetization with electrical poling has been demonstrated and clearly showed the magnetoelectric behavior in LF-PT solid solutions.

The rare-earth manganites RMnO$_3$ compounds belong to the class of multiferroic materials are hot topic in recent solid-state research. In this paper we report the optical properties of GdMn$_{1-x}$Co$_{x}$O$_3$ (x= 0-0.8). Far-infrared (FIR) and Raman spectroscopy were carried out in the energy range of 150 – 700 cm$^{-1}$ at room temperature. The samples were prepared by solid-state reaction. The samples phases were studied by powder X-ray diffraction and the structures were refined using the Rietveld refinement program.

The FIR transmission spectra show four main phonon vibration modes around at 190, 250, 400 and 580 cm$^{-1}$ that corresponding to external, torsional, bending and stretching respectively. It is shown that the FIR active vibration energies exhibit splitting and shifting as Co doping increase. The Raman spectra were obtained with excitation wavelength of 632.8 nm. The most intense spectral feature in HoMnO$_3$ sample is the highest frequency line at 610 cm$^{-1}$. This high frequency line is shifted as Co doping increases in the sample. A detailed analysis of the FIR and Raman measurements is presented.

Domain Imaging Mechanisms in Ultrathin Ferroelectric Structures

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Ferroelectric nanostructures have scaled both vertically and laterally down to the nanometer range. The amount of available displacement charges in nanoislands of only a few dozen nanometers lateral extension is insufficient for detection by probe stations in a (modified) Sawyer-Tower configuration. Piezoresponse force microscopy has therefore become an indispensable tool for the investigation of ferroelectric nanostructures, with qualitative imaging capabilities of the piezoelectric tensor and polarization inversion down to a few nanometer resolution. The increasing demand of quantitative data is unlikely to be satisfied by this method due to various limitations that will be discussed. As the thickness of ferroelectric films decreases down to a few nanometers, piezoresponse force microscopy even suffers from a complete loss of domain contrast. This observation will be quantitatively discussed in terms of polarization reversal and electrostriction measurements at the second harmonic.

This presentation will discuss the next generation of scanning probe techniques for ultrathin ferroelectric structures including scenarios where the reduced vertical dimension itself opens the way to novel detection schemes.

Effect of Nitride Flow Rate of TiCrVZrTa High-entropy Alloys on Structure and Characterization

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Multi-element high-entropy alloy is considered to be applicable to protective coating. The TiCrVZrTa metallic and nitride film was prepared under various argon/nitrogen flow rate by dc magnetron sputtering. The structure, morphology, and mechanical property were investigated in detail. Under pure argon atmosphere, the TiVCrZrTa metallic films are amorphous structure with crack. When the Ar+N$_2$ mixed working gases were added in the deposition process, from the result, the TiVCrZrTa nitride film exhibits a typical FCC structure at lower nitrogen flow rate. The films are columnar structure with pyramid surface morphology. As the nitrogen flow rate increases, the FCC becomes amorphous structure, implying that introducing excess amount of nitrogen will inhibit the grain growth and decrease the crystalline of the film. The
original pyramid surface morphology becomes round and columnar structure becomes hard to discriminate. The surface roughness of nitride films tend to become smooth. Moreover, it is found that introducing higher amount of nitrogen can cause superior mechanical property.

Ceramic-polymer Composite Thick Films by Aerosol Deposition for the Application of Integrated Substrates

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In conventional ceramic process, high temperature sintering above 1000°C is essential to achieve the densification of ceramic body, which makes it difficult to integrate the electronic components due to high shrinkage and heterogeneous bonding. Low temperature co-fired ceramic (LTCC) has been suggested as an alternative to overcome such drawbacks, but it still requires the sintering temperature above 700°C. In the point of view above mentioned, aerosol deposition method (ADM) used in this experiments might be a promising technique to fabricate a ceramic based substrate for 3-dimensional integration modules. It does not require the high temperature sintering process because it is possible to grow a highly packed ceramic thick film at room temperature. In this presentation, fabrication process and unique properties of thick film substrates grown by ADM for the application of high frequency application will be shown. To satisfy the requirements of low permittivity, low dielectric loss, and high thermal conductivity, some kinds of candidates of ceramic materials were reviewed and their electrical properties were compared. In addition, the effects of small content of polymers, which were incorporated in the films to reduce the internal stress and increase the plasticity, will be discussed through the characterization of their microstructures.

Effect of Deposition Temperature on Alumina Films Obtained by Atmospheric Pressure Chemical Vapor Deposition

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This work has been studied on a quartz substrate by low deposition temperature by using atmospheric pressure chemical vapor deposition (AP-CVD). These films were obtained by using aluminum trichloride precursor gas with choosing different substrate temperature and amounts of argon carrier gas mixed with oxygen. Film deposited at low temperature with a high Ar flow rate showed high surface roughness and cracked surfaces. The kinetic study was showed on the film growth rate limiting step, molecular size and gas phase reaction constants. The molecular species has increases with increasing deposition temperature of 4.7 Å for 350°C and 6.53 Å for 400°C respectively. The film had a higher compositional Al/O ratio at higher temperature. The thickness and microstructure film of the alumina deposited was observed using scanning electron microscopy and atomic force microscopy. Optical properties, i.e. refractive index and optical transmittance, were also measure.

Modification of Physico-chemical Properties of Sm3+ Doped Ceria by Addition of Hydrogen Peroxide

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Rare earth doped nano ceria has lot of applications in different fields like fuel cells, oxygen sensors, polishing agents etc. So it has become challenging to synthesis soft agglomerated and mono-dispersed rare-earth doped ceria. Effect H2O2 on synthesis and powder properties of nanocrystalline Sm3+ doped sample has been explored by treating the mixture of Ce(NO3)3 and Sm(NO3)3 (4:1) with NaOH in presence and absence of H2O2. The as-prepared samples were calcined at 600°C for 1h. The calcined products were characterized by x-ray diffraction (XRD). Crystallite size were calculated from the X-ray peak broadening the and it was found to be 11 and 17nm for the samples prepared in presence of H2O2 (named as SDC) and in absence of H2O2 (named as SDCN). DLS technique suggests that SDC nano powders are soft agglomerated whereas the SDCN are comparatively hard in nature. The obtained surface area for SDC were found to be higher than that of SDCN. High resolution transmission electron microscopy were employed to investigate the particle size and morphologies of the samples. The histograms of particle size distribution showed that particle sizes of SDC samples
were lower than that of SDCN samples. The reason behind the obtained better powder properties of SDC over SDCN is due to presence of peroxo (-O-O-) linkage which has been unequivocally proved by Raman spectroscopy. This peroxo (-O-O-) linkage formed during synthesis of doped ceria in presence of H2O2. The mechanism of formation of lower particle size can also be understood from Raman spectroscopy. The samples prepared in absence of H2O2 form hydroxide precipitate by reaction of metal ions and OH ions. During condensation, the precipitate eliminates H2O and form -M-O-M- (M= metal ion) network. Besides -M-O-M- bonds, the samples prepared in presence of H2O2 form peroxo linkage (-M-O-O-M-) also. Subsequently, upon calcination this O-O linkages break down which ultimately leads to the lower particle size and higher surface area containing samples.

Moreover, temperature programmed reduction studies revealed that the doping in conjunction with the use of H2O2 leads to enhanced reduction properties over multiple cycles. Interestingly, SDC also showed the drop in conversion temperature of CO to CO2 about 100°C proving it to be a better catalyst.

**Synthesis and Photoluminescence Properties of (Gd1-x,0.06≤y≤0.15) phosphors**

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Green-emitting (Gd1-x,0.06≤y≤0.15) phosphors were successfully synthesized by ultrasonic spray pyrolysis. As-synthesized and annealed (Gd1-x,0.06≤y≤0.15) phosphors showed high-quality powder characteristics, i.e., fine size as well as smooth, regular, and spherical morphology. The excitation and emission spectra were obtained at 540 and 147nm, respectively, and the excitation spectrum was calibrated with sodium salicylate which had constant quantum efficiency which constant quantum efficiency in the VUV region. The characteristic emission peaks, which were caused by the transition from 5D2 to 7Fj (j=3-6) of Tb3+, were detected at 489, 543, 585, and 621nm, respectively, indicative of green light emission. The emission intensity of (Gd1-x,0.06≤y≤0.15)PO4 increased with Tb content. (Gd1-x,0.06≤y≤0.15)PO4 was the optimal composition showing the highest emission intensity. We demonstrate that the partial incorporation of Zn for Gd is desirable for improving photoluminescence properties.

**Thermoelectric Properties of Ca3Co4-xFe2O9 (0≤x≤0.15) for Power Generation**

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We successfully prepared nano-sized Ca3Co4-xFe2O9 (0≤x≤0.15) powders by solution combustion process. Plate-like grains were observed in as-sintered Ca3Co4-xFe2O9 ceramics. The as-sintered Ca3Co4-xFe2O9 samples had a monoclinic symmetry. The samples for the measurements of thermoelectric properties were cut out of the sintered bodies in the form of rectangular bars of 2×2×15 mm. For thermopower measurements, a temperature difference in the sample was generated by passing cool Ar gas over one end of the specimen placed inside a quartz protection tube. The sign of the Seebeck coefficient was positive over the measured temperature range, indicating that the major conductivity carriers were holes. The electrical conductivity and Seebeck coefficient of the Fe-added Ca3Co4-xFe2O9 were much higher than those of Fe-free Ca3Co4O9. In this study, we discussed the thermoelectric properties of the Ca3Co4-xFe2O9 samples, depending on Fe content.

**Electronic Reconstruction at Oxide Interfaces**

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The polar nature of LaAlO3 is crucial in understanding the conductivity at SrTiO3-LaAlO3 interfaces. For every unit cell of LaAlO3, electric potential is built up, albeit partly screened by counteracting ionic displacements within the charged layers. After a critical number of LaAlO3 unit cells, electronic reconstruction is though to take place, in which charge is transferred from the LaAlO3 surface to the SrTiO3-LaAlO3 interface.

Apart from the critical role of induced oxygen vacancies in the SrTiO3, also reconstruction at the LaAlO3 surface can be expected to have a large influence on the interface electronic reconstruction. In order to study a system that is less sensitive to oxygen vacancies and surface contamination effects, it is beneficial to cap the LaAlO3 surface with a nonpolar material such as SrTiO3. We have studied coupled complementary interfaces by performing transport experiments at various oxygen deposition pressures and variable LaAlO3 and SrTiO3 capping layer thicknesses.
These experimental findings now lead to a detailed microscopic understanding of the ionic, electronic and orbital reconstruction at the two interfaces, including the nature of the charge carriers.

Dielectric Property Studies of Heterogeneous PZT(52/48)/DEG Composites

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Ferroelectric lead zirconate titanate ceramics Pb(ZrₓTi₁−ₓ)O₃ (PZT) are largely used in sensing and actuating applications due to their remarkable piezoelectric and dielectric properties. They have been the subject of extensive research in both experimental and theoretical physics. Above the Curie temperature (Tc), at which a reversible ferroelectric (polar) to paraelectric (non polar) phase transition occurs, perovskite-type PZT is cubic while below Tc it becomes tetragonal for Ti-rich compositions or rhombohedral for Zr-rich compositions. Generally, the composition range x= 0.50–0.54, corresponds with the morphotropic phase boundary (MPB) separating the tetragonal from rhombohedral region. The science of embedded capacitor is a sophisticated technology with the congregation of both performance and functionality requirements for future electronic devices. One of the major hindrances for implementing this technology is the lack of dielectric materials with promising dielectric properties. Polymer based composite is considered as a solution to the problem mentioned hitherto. Polymer/ceramic composites can be used in forming capacitors because they combine the process ability of polymers and high dielectric constant of ceramics. One of the promising embedded capacitor materials is a polymer/ceramic composite which is a ceramic particle-filled polymer. It is a material utilizing both high dielectric constant of ceramic powders and good process ability of polymers. Particularly epoxy/ceramic composites have been investigated and studied due to their compatibility with printed writing boards (PWB).

In the present work, we have synthesized morphotropic phase boundary compound PbZr₅₃Tio₄₈O₃ (PZT 52/48) by sol-gel method. The phase formation and morphology of the PZT (52/48) powders were studied by X-ray diffraction and scanning electron microscope. 2 wt % of PZT (52/48) powder is dispersed in diethylene glycol (DEG) using ultrasonic vibrator and magnetic stirrer. Dielectric property of the composite was studied by an Impedance Analyzer (Agilent 4294A, USA). We observed very high dielectric constant (εₘₐₓ = 13593 at 40 Hz with bias volt = 0 V) for the heterogeneous composites which decreased to the value of 7114 with bias voltage of 20 V. This heterogeneous PZT(52/48)/DEG composite can easily find application as a passive component in an electronic systems.

Enhanced Dielectric Properties of Low Temperature Sintered Ferroelectric PMN-PT (65:35) Ceramics

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Solid solutions of PbMg₁/₃Nb₂/₃O₃ (PMN) and PbTiO₃ (PT) ceramics are one of the most extensively studied materials for their magnetoelectric properties. The optimal concentration 0.65PMN – 0.35PT has a morphotropic phase transition between the rhombohedral and tetragonal ferroelectric phases. The dielectric and piezoelectric constants for these PMN-PT compositions (0.3<ₓ<0.35) are significantly high. Composites of PMN-PT with magnetostrictive materials like Terfenol-D have paved a way for a new generation of device applications. PMN-PT solid solutions are usually synthesized using high temperature (~1200°C) solid state reaction route. However, this route always leads to a high amount of Pb loss and formation of pyrochlore phase at high temperature during sintering process. We report here a relatively newer method of synthesizing this material using a columbite precursor method at lower temperatures. Furthermore, a sintering aid (flux) in the form of LiF, was also used to further lower the sintering temperature to ~ 875°C (~81% of theoretical density and highly resistive, ~ 10¹² Ω cm). The phase purity of the samples was verified using XRD and the morphology using SEM. Dielectric studies of the sample showed very high dielectric constant (~10⁷) and low dielectric loss (0.9) at low frequency. From ferroelectric studies, the value of saturation polarization, remnant polarization and coercive field are found to be 16.7 μCm⁻², 12.2 μCm⁻² and 11.4 kVcm⁻¹ respectively. These results will be discussed in detail.
Textured Growth of Terbium Iron Garnet Thin Films as a High Coercivity Media

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Materials exhibiting coupling between electric and magnetic properties, for example induction of polarization by magnetic field or vice versa, attracted a great attention due to their promising applications in magnetic field sensors, and electric–write magnetic read memory devices. Majority of these materials in single phase, exhibit such a fascinating effect only in the presence of significantly high driving field. However, recently we have reported ~0.4% change in dielectric constant only at 10kOe of applied magnetic field in (110) oriented single phase epitaxial TbIG thin films deposited on gadolinium gallium garnet substrate (GGG). The change in dielectric constant were about an order of magnitude smaller than the single crystal and may be explained due to compressive strain and clamping by the substrate and change in magnetic easy axis from [1-11] to [110]. The in-plane and out of the film plane coercivity of (110) oriented epitaxial TbIG thin films on GGG substrate were nearly 500Oe and 200Oe respectively. Interestingly, in the present study, a textured growth of TbIG thin films on Pt/Si(100) substrate exhibit about one order of magnitude enhancement in the in-plane and out of the film plane coercivity values. These TbIG thin films were deposited in pure oxygen atmosphere (0.16 mbar) at room temperature by pulsed laser deposition technique. Films were subsequently subjected to the annealing at 900 °C in air. The detailed study of results and discussion of the TbIG thin films will be presented.

Transmitting Conducting Oxide Layers for Thin Film Silicon Solar Cells

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Optical confinement plays an important role in case of thin film solar cells due to its role in facilitating a near complete absorption of above band gap light in photoactive layers which are not thick enough to sufficiently absorb light in a single pass. Recent advances in either varying the band gap of the materials or using multi-junction structures have extended the absorption strength of the solar cells. Moreover, use of nano-dots to adapt the absorption characteristics and up-conversion or down-conversion of light are the techniques are also being followed to address this absorption issue from two directions. However, the deterioration of electronic quality of the new materials, has so far limited such techniques to succeed and light trapping process is still a sought after method to enhance current in solar cells. We will discuss here mainly the use of metal oxide layers such as transmitting conducting oxide (TCO) in thin film silicon solar cells where they are used at front and back regions.

For amorphous silicon (a-Si) solar cells, SnO₂:F has been the most favored TCO for the high temperature resistant substrates such as glass or metal foils. Doped (B, Al, Ga) ZnO layers have recently received much attention due their high stability against atomic hydrogen and its broad range of processing temperatures, that allows to fabricate solar cells even on plastics. SnO₂:F TCOs with naturally grown pyramidal surface textures by APCVD are commercially available and U-type TCOs from Asahi Co. have delivered state of the art efficiencies for a-Si type of cells. Textured etched sputter deposited ZnO on the other hand has shown superior results for nanocrystalline silicon (nc-Si) type of cells. However, CVD methods, such as LPCVD and ETP have shown the possibilities of as depositing textured ZnO. Three concepts have recently come into picture, which define the required surface texture of the TCOs. (1) The feature size of the textured surface has to be designed appropriately for the type of i-layer in the cell for optimal scattering. A high roughness TCO is used for nc-Si cell whereas the a-Si cell performance is best for small roughness (such as Asahi U-type TCO). A combination of these types of surfaces is now considered to be appropriate for tandem cells that use a-Si and nc-Si as component cells. W-textured SnO₂:F (HU type) developed by Asahi Co. is one such example. (2) Rough surfaces cause defective regions in the silicon i-layers that lead to shunting paths. Adapting/modifying a high scattering rough surface to achieve high open circuit voltages is an effective way to obtain high efficiencies. (3) The scattering has to be predominantly at the metal oxide/silicon surface. Use of thick ZnO in combination with white reflectors, instead of metals, will be a possible trend to avoid surface plasmon absorption loss. The paper will discuss all these issues for high efficiency thin film silicon solar cells.
Oxide Film Assisted Dopant Diffusion in Silicon Carbide

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Oxide film is commonly used as an insulator, and therefore not usually used as a source of dopant in semiconductors. In this paper, we will describe a general process in which an oxide film can be used to introduce dopants in semiconductors, with special emphasis on the application of this technique in the doping of silicon carbide (SiC) for metallization purposes.

SiC is recognized as a wide band gap semiconductor material highly suitable for high temperature and high power electronics. Due to the low impurity diffusion rate in silicon carbide, thermal diffusion is often done at high temperatures. For 6H- and 4H-SiC, the diffusion temperature is in the range of 1700-2300°C. Dopant activation following ion implantation in hexagonal SiC polytypes also requires a high activation temperature in excess of 1500°C. These processing temperatures are not suitable for 3C-SiC/Si because they exceed the melting point of silicon which is about 1414°C. In any case, the high activation temperature or high diffusion temperature required to activate or introduce dopants in SiC necessitates the use of precautionary measures such as silicon overpressure to prevent decomposition of the surface. A low-temperature diffusion or dopant activation technique that can reduce surface deterioration without the need for elaborate surface protection technique is highly preferable.

In this paper, we will describe a process in which an oxide film is used to actively assist in dopant diffusion in silicon carbide. This process involves depositing a thin film consisting of an oxide of the impurity on silicon carbide, followed by annealing in an oxidizing ambient. The impurity atoms then diffuse from the thin film into the near-surface region of silicon carbide with a diffusion rate higher than that without oxide assistance. This enables the doping of silicon carbide to be done more efficiently at lower temperatures.

The suitability and applicability of this method in other semiconductors such as Si and the III-nitrides will be discussed.

Characterization of a Refractory Crucible for the Drawing of Silicon Ingots for Photovoltaic Applications

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Our work aims is to develop a future industry photovoltaic polycrystalline technology in Algeria, for rational and economical use of local materials. This technology uses silicon crystal aggregate in high-purity 99.9999% Si for the drawing of polycrystalline silicon ingots. Each ingots drawing requires the use of a refractory crucible. This work is mainly devoted to characterize these crucibles and possibly develop. To do this, waste crucibles used had already been the subject of this characterization by various analytical techniques namely: Analysis by X-ray diffraction, X-ray fluorescence, dilatometer, a scanning electron microscope, density, porosity and specific density. The results obtained showed that the crucible consists of cristobalite which is a variety of silica that forms at high temperature with a high content of SiO2 which is equal to 99,322% and traces of other oxides as impurities. The chemical composition SiO2 makes it compatible with silicon Si (minimum contribution of impurities on silicon melted). Its melting point 1713 °C which is higher than the other varieties of silica and its variations and dimensional stability at high temperatures it confers resistance to temperatures of silicon processing. The low porosity makes it able to contain the molten silicon.

Interplay of Nanoscale Phases in Some Ferroelectric Oxides

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The ever growing micro-to-nano electronic industries called for investigation of materials system in many aspects, not limiting to the scaling down technologies of electronic devices but also the understanding of nanoscale phases or domains or other entities by which the system behaves. In the past decades, our group has been engaged in a range of studies focusing on the interplay of structure and properties in dielectrics and ferroelectrics. Our works encompassed ferroelectric, anti-ferroelectric as well as relaxor materials in many bulk and thin-film complex oxides. Further, we have pursued investigation into the nontraditional behaviors including nonlinear, temporal and coupled interactions. This paper highlights our recent research and presents a systematic view of the interplay among dipoles in multi-phase systems.
**A02584-04415**

**Structure and Photoactivity of ZnO Derived from Microwave Assisted Chemical Routes with Different Precursors**

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Photoactivity under UV irradiation for the decomposition of acid red 27 in water was studied on ZnO powders derived from wet chemical route by microwave assisted with different precursors, comparing with P25 titania.

Rate constant dye decomposition kazo increased with decreasing crystalline size of nano powders. It tended to increase at 10-20 nano meters range, mainly due to the QE of fine powders. The dependences of kazo on full width at half maximum intensity (FWHM) were common for three samples, ZnCl2, ZnSO4, Zn(Ac) with similar preparation conditions. Acetate based powders is giving high photo activity due to high crystallinity and high adsorptivity particularly for Acid red 120°C by chmical vapor deposition at low-pressure of 50 Torr. The mono-silane and acetylen gases were used as a source material of SiC. Before β-FeSi2 film deposition, the 3C-SiC-buffered Si(100) and Si(111) substrates were treated in a solution of HCl : H2O : H3O (1:1:10) and then etched in buffered HF (HF : NH4F = 1:6). The β-FeSi2 films (150 nm thick) were deposited by co-sputtering of iron and silicon in an argon atmosphere. A 2-inch Fe-Si alloy disk with silicon tips was used as a target. The silicon/iron atomic ratio of the films was controlled by changing the areas of the tips. The deposition temperature and deposition rate were 750°C and 1.5 nm/min, respectively. The chamber pressure during deposition was kept constant at 3 × 10⁻³ Torr.

It was ascertained by x-ray diffraction measurement that the (100)-oriented β-FeSi2 films were epitaxially grown on both of 3C-SiC on Si(100) and Si(111) substrates. The full-width at half maximum of the rocking curve of the β-FeSi2 800 diffraction peaks was 1.6° and 0.5° on 3C-SiC-buffered Si(100) and Si(111), respectively, which were the same level as that of epitaxial β-FeSi2 films on Si substrates. The epitaxial (100) β-FeSi2 film on 3C-SiC-buffered Si(100) was constructed with double domains, while the (100) epitaxial film on 3C-SiC-buffered Si(111) substrates contained a three-domain structure. The epitaxial relationship between (100) β-FeSi2 and (100) 3C-SiC was identified as Type B, which differed from that between (100) β-FeSi2 and (100) Si (Type A).

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**A02628-04510**

**Epitaxial Growth of β-FeSi2 Thin Film on SiC**

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Recently β-FeSi2 has been attracting attention as a material for Si-based light emitters and detectors operating at wavelengths used for optical fiber communication. This is because the β-FeSi2 has a band gap of approximately 0.78 eV, and a very large optical absorption coefficient of over 10⁹ cm⁻¹ at 1 eV. While there have been numerous reports on preparation of β-FeSi2 thin film over the last decade, Si single crystal has been used as a substrate. We think that the hetero-epitaxial growth of β-FeSi2 on the other semiconducting materials can be applied to the optical device and to clarify its physical properties. In this article, we report on the hetero-epitaxial growth of the silicide semiconductor thin films on the silicon-carbide (SiC) semiconductors, (100) and (111) plane of 3C-SiC, by using rf-magnetron sputtering. The 3C-SiC has a band gap of 2.23eV and a cubic-phase crystal structure with a space group of F43m. The 3C-SiC layer has been epitaxially grown on (100) and (111) plane of Si substrates.

Substrates were 3C-SiC films of approximately 1 μm thickness on Si(100) or Si(111), which were prepared at 105 cm⁻¹ at 1 eV. While there have been numerous reports on preparation of β-FeSi2 thin film over the last decade, Si single crystal has been used as a substrate. We think that the hetero-epitaxial growth of β-FeSi2 on the other semiconducting materials can be applied to the optical device and to clarify its physical properties. In this article, we report on the hetero-epitaxial growth of the silicide semiconductor thin films on the silicon-carbide (SiC) semiconductors, (100) and (111) plane of 3C-SiC, by using rf-magnetron sputtering. The 3C-SiC has a band gap of 2.23eV and a cubic-phase crystal structure with a space group of F43m. The 3C-SiC layer has been epitaxially grown on (100) and (111) plane of Si substrates.

**A02633-04515**

**Tailoring Glass Surfaces via Internal Cationic Diffusion**

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In this paper, we report a new way to create functional surfaces on the transition metal containing oxide glasses. In terms of chemistry and structure, the created surfaces are classified into two groups. One group is the nano-crystalline surface layer enriched in earth alkaline oxides, and the other is the nano-amorphous surface layer enriched in silica. The nano-crystalline surface is generated by subjecting the glass to an oxidation process in atmospheric air at the glass transition temperature for a certain duration. During this process, a fraction of the transition metal ions will be transformed from low to high valence state, and this causes electron holes to migrate from the glass surface towards the glass interior. To maintain the charge neutrality, the network modifying ions must diffuse from...
the interior towards the surface, i.e., the so-called outward diffusion occurs. At the surface, the modifying cations react with oxygen ions to form the corresponding oxides, the formation of which is driven by the negative Gibbs free energy of the reaction. It is found that the magnesium ions are the most mobile, and hence, the formed periclase (MgO) is the predominant nano-crystalline phase in the surface layer. The origin of this phenomenon is discussed in the present work. As a mirror effect, the nano-amorphous surface layer enriched in silica is generated by subjecting the glass to a reduction process. The direction of the cationic diffusion during reduction is just the opposite of that of the outward diffusion, i.e., the so-called inward diffusion occurs. However, whether the inward diffusion happens strongly depends on the partial pressure of the reducing gas (e.g. hydrogen). The fascinating part of the new approach is that both the direction and the extent of the diffusion are controllable, and hence, the thickness of both types of layers can be tailored. The formation of the nano-layers can considerably contribute to improving physical and chemical functions of glassy materials. We clarify the kinetic and thermodynamic mechanisms of both outward and inward diffusion. We also describe the perspectives of our new approach, in particular, with respect to the potential applications of the approach in creating functional surfaces on non-oxide glasses, and even on crystalline materials.

References:

Electromagnons, Ferroelectric Soft Modes and Magnetoelectric Coupling in Multiferroics

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In our contribution we give an overview of dielectric, magnetic, magnetocapacitive, magnetostrictive and lattice dynamics properties of cubic EuTiO3 and hexagonal YMnO3. Obtained results will be generalized on other magnetoelectric multiferroics.

The perovskite EuTiO3 is not typical multiferroics. It exhibits and antiferromagnetic structure below \( T_N = 5.3 \) K, but the ferroelectricity does not take place because the quantum fluctuations prevent the creation of long range ferroelectric order. Its permittivity increases on cooling similarly to classical quantum paraelectrics SrTiO3, saturates below 30 K and sharply drops down at \( T_c \), giving the evidence about the strong magnetoelectric coupling. Moreover, below \( T_c \) permittivity exhibits a huge dependence on the magnetic field (magnetocapacitive effect). Our infrared reflectivity studies confirm, that the temperature and magnetic dependence of permittivity can be explained by temperature and magnetic field shifts of the lowest energy polar phonon (soft mode). We propose a model based on spin correlations and magnetization (both dependent on temperature and external magnetic field) which explains the phonon and permittivity changes with temperature and magnetic field. We also show, that the same model can be successfully used for explanation of anisotropic magnetostriction, which we measured in the same material along and parallel to the external magnetic field.

We measured not only bulk, but also strained EuTiO3 thin films. Infrared spectra of 22 nm thick -0.9% compressively strained EuTiO3 film deposited by reactive molecular-beam epitaxy on a (100) LSAT substrate exhibit stiffening of all phonons. Moreover the soft mode splits below 150 K providing evidence for a structural phase transition near this temperature. Unfortunately, from our IR spectra we cannot distinguish whether the phase transition is ferroelectric or only antiferrodistortive.

Hexagonal YMnO3 exhibits ferroelectric phase transition near 1280 K and antiferromagnetic phase transition below 80 K. Our infrared reflectivity spectra taken up to 950 K are in agreement with an improper ferroelectric type of the phase transition (tripling of the unit cell), because we observed only small phonon softening and negligible increase of permittivity on heating to 950 K.

In our THz spectra of YMnO3 one magnetic resonance excitation appears in the antiferromagnetic phase. It is impossible to determine from our nonpolarized THz spectra, whether the magnon contributes only to magnetic susceptibility, how it should be by classical antiferromagnetic resonance, or whether it is activated also in dielectric permittivity due to its coupling with phonons. In the later case, such magnon is called electromagnon and it was observed recently in THz spectra of orthorhombic RMnO3 (R=Tb, Gd, Y-Mn) magnetoelectric multiferroics, where the ferroelectric order is induced by spiral magnetic order due to Dzyaloshinskii-Moria interaction. Nevertheless, the coupling of magnetic order with the phonons we proved by observation of phonon hardening near \( T_N \), which is responsible for pronounced decrease of static permittivity near \( T_N \). Electromagnon should have specific infrared selection rules. Therefore new polarized THz studies performed on hexagonal YMnO3 single crystal are in progress.

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Multiferroics, which display simultaneous magnetic, electric, and ferroelastic ordering, have drawn increasing interest in recent years due to their multi-functionality for a variety of device applications.

BiFeO$_3$ (BFO) is the most studied material exhibiting multiferroism at room temperature. We observed Raman scattering from magnons (also known as electromagnons, meaning they possess electric dipole moment due to magnetoelectric coupling) in BiFeO$_3$ thin films grown on (111) SrTiO$_3$ substrates at cryogenic temperatures; the temperature dependence of the magnon at 18.2 cm$^{-1}$ approximates an $S^2/5$ Brillouin function up to the temperature (280 K) at which the magnon becomes overdamped. The diverging cross-section and the frequency-shift at 140k and 200 K imply a spin-reorientation transition as in orthoferrites.

The high temperature phase transition in BFO thin films was studied by Raman scattering in the temperature range from 300 – 1173K. The low frequency intense sharp modes appearing at 136, 172 and 212 cm$^{-1}$ showed anomalies around the magnetic phase transition $T_N$ (673K) that could be interpreted as an experimental evidence of perturbation of antiferromagnetic ordering at the magnetic phase transition (Spin phonon coupling). We also observed an orthorhombic order-disorder $\beta$ phase between 820 and 925 °C, and established the existence of a cubic $\gamma$ phase above 925 °C before reaching the decomposition temperature. The transition to the cubic phase causes an abrupt collapse of the band gap toward zero (insulator-metal transition) at the orthorhombic-cubic transition.

We also discovered a new room-temperature, single-phase magnetoelectric multiferroic, PbZr$_{0.52}$Ti$_{0.48}$Fe$_{0.13}$W$_{0.07}$TiO$_3$, with polarization loss (<1%), and resistivity (typically 108 ohm.cm) equal to or superior to BFO, with enormously greater magnetoelectric effect: switching not from +Pr to -Pr with applied H, but from Pr to zero with applied H of less than a Tesla. We switched 70 $\mu$C/cm$^2$ at H=0.5T at 293K, which is more than 1000x greater than the nC/cm$^2$ values in rare-earth manganites at cryogenic temperatures. This switching occurs not because of a conventional magnetically induced phase transition, but because of dynamic effects: Increasing H lengths the relaxation time by x500 from <200 ns to >100 ms, and it couples strongly the polarization relaxation and spin relaxations. The diverging polarization relaxation time accurately fits a modified Vogel-Fulcher Equation in which the freezing temperature, $T_v$, is replaced by a freezing field, $H_v$, that is 0.91 Tesla, with activation energy $E_a = 0.26$ eV and attempt frequency $T_0 = 40$ MHz.

ZnO nanorods are synthesized by hydrothermal method on ITO glass. First, we clean the indium tin oxide (ITO) glass using acetone, ethanol, and deionized water sequentially. In ZnO nanorods synthesis process, Zinc acetate dehydrate solution is used as the seeding layer and spin-coated on the ITO glass substrate for the subsequent growth of nanorods. Then, it is baked at 350°C for 20 minutes in ambient. The hydrothermal reaction solution for ZnOnanorod fabrication is prepared by mixing 25 mM zinc nitrate hexahydrate (Zn(NO$_3$)$_2$·6H$_2$O) and 25 mM hexamethylenetramine (C$_6$H$_4$(NH$_2$)$_2$) in 200 mL of deionized water in a glass beaker. During the growth, the glass beaker is placed in an electric-heated thermostatic water bath and maintained at 92°C for 15 minutes. Subsequently, the sample is removed from solution and washed with ethanol and deionized water repeatedly and baked for 30 minutes in ambient. Then two kind of devices are prepared as ITO/ZnO/ZnO nanorod/Alq$_3$/Al and ITO/ZnO/ZnO nanorod/MEH-PPV/Al. The p-type polymer MEH-PPV is dissolved in chloroform with a concentration of 5 mg/mL and then spin-coated on as-grown ZnO nanorods layer. Alq$_3$ and electrode Al are deposited by the thermal evaporation method in a high vacuum condition of 2×10$^{-4}$ Pa.

Under DC bias, the band-edge ultraviolet (UV) electroluminescence (EL) at 380 nm and the defect related emission of ZnO nanorods at 640 nm and 747 nm are detected in both devices. In addition, the exciton emission of MEH-PPV at 580 nm and 510nm of Alq$_3$ is observed respectively in two kinds of devices. Compared two devices, it is found that the ultraviolet electroluminescence is stronger in ITO/ZnO/ZnO nanorod/MEH-PPV/Al than in ITO/ZnO/ZnO nanorod/Alq$_3$/Al. Along with the increasing of the applied voltage, the UV EL becomes predominant and is much stronger than other emissions. The mechanism of the ZnO band edge emission and background emission are discussed.

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Magnetoelectric Effects in Composite Thin Films of Ferroelectric and Magnetic Oxides

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Nanostructured thin films of ferroelectric (e.g., BaTiO$_3$, PZT, and BiFeO$_3$) and magnetic oxides (e.g., cobalt-ferrites and nickel-ferrites) prepared by physical deposition techniques (e.g., pulsed laser deposition) and chemical solution processing (e.g., sol-gel spin-coating method) have recently become new routes to multiferroic magnetoelectric (ME) composites. In comparison to bulk ME ceramic composites, the nanostructured thin films provide more degrees of freedom, such as lattice strain or interlayer interaction, for modifying the ME behavior. These films also offers a way to investigate the physical mechanism of the ME effect in nanoscale. The coupling interaction between two oxides in the ME nanostructures could be still due to an elastic interaction as was the case in bulk composites. However, the mechanical constraints arising from the film-on-substrate structure and the bonding between the two nanostructured phases could significantly affect the ME coupling interactions. Two kinds of nanostructured ME thin films, i.e., 1-3 type nanostructures (vertical heterostructures) consisting of magnetic oxide nanopillars vertically embedded into a ferroelectric film, and 2-2 type heterostructures (horizontal nanostructures) consisting of alternating layers of a ferroelectric perovskite and magnetic oxide, have been studied. Such ME nanostructures present simultaneously ferroelectric and ferromagnetic responses at room temperature. In particular, obvious ME coupling effect has been observed in the nanostructured oxide films. In this work, we discuss the nanostructured ME oxide films both in experimental and theoretical.

Synthesis of Corundum Nanocrystals

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The preparation methods of nanocrystalline corundum were developed. All of them are based on the decomposition of aluminium containing compounds.

1. Aluminium lactate was decomposed via autoclaving for an hour in steam at 400°C with formation of fine crystalline boehmite (AlOOH). Its crystals have size 50-200 nm. When heated at 1200°C this fine crystalline boehmite transformed to alpha-alumina powder without strong aggregates. The initial habitus of the boehmite particles preserved.

2. After the decomposition of aluminium hydroxy-acetate under the same conditions fibrous boehmite formed. The fibers have thickness 300-500 nm and length up to 10 microns. The fibrous structure of the boehmite powder preserved after calcination and formation of alpha-alumina at 1200°C in air and even after heating in vacuum at 1500°C. Such crystals, for instance, may be used for ceramic filters manufacture.

3. Terephthalic acid-aluminium hydroxide formed crystals with supramolecular structure. The synthesized structure decomposed on calcination at 700°C with formation of the gamma alumina, which at 1250°C transformed into alpha-alumina nanocrystals without strong agglomeration and preserving the initial isometric grain habitus.

4. The aluminium hydroxide (hydrargillite) formed fine boehmite crystals when hydrothermally treated at 200°C.
in acidic media. This boehmite had grain size ~300 nm and at 1250°C formed alpha-alumina powder without sintering and change of the initial grain habitus.

5. Fine metallic aluminium powder was dissolved in water ammonia solution and stabilized with organic additives. When treated in autoclave this system gave aluminium hydroxide fibrous crystals with size ~50 nm.

**Structural and Electrical Properties of BiFeO$_3$ Thin Films Prepared on SRO Buffer Layers of Varied Thickness**

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SrRuO$_3$ (SRO) buffer layers of different thicknesses are prepared by using different sputtering time. Cross-sectional analysis using scanning electron microscope, confirms the continuous SRO buffer layer deposition. Surface morphology analysis reveals that the SRO grain sizes are varied by using different sputtering time. A close comparison between the BiFeO$_3$ (BFO) films RF sputtered on SRO buffer layers of different thicknesses revealed that the crystallization, surface morphology and electrical properties of the BFO structure are all strongly affected by the bottom SRO buffer layer deposition. Thick (large-grained) SRO buffer layer is able to assist the growth of BFO film with better crystallinity and lower leakage current, while fatigue and dielectric measurements reveals that the BFO films deposited on thin (small-grained) SRO buffer layer contained more structural defects. Investigation shows that SRO buffer layer of varied grain-sizes are crucial in determining the electrical performances of the resultant BFO films deposited upon them.

**Effect of Heat Treatment on Structural and Optical Properties of Sol-gel Spin Coated Nanocrystalline TiO$_2$ Thin Films**

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TiO$_2$ thin films were prepared from a titanium isopropoxide precursor by sol–gel spin coating method followed by three different annealing temperatures. The chemical constituents present in the deposited TiO$_2$ films have been identified using energy dispersive analysis of x-rays. Optical properties of the films were characterized by UV-Vis spectroscopy and Photo Luminance spectra. Crystalline structures by X-ray diffraction and Raman spectra. The morphology of the film surfaces as obtained by SEM techniques could be also correlated with the results of our AFM studies. The as deposited films have been found to be amorphous in nature. The crystalline quality has been observed to improve with annealing temperature. The annealed TiO$_2$ films have been found to exhibit anatase phase with grain size ranging from 10 nm to 25 nm. The crystallite size can also conformed from TEM images.

**Fatigue Behavior of Heterolayered Lead Zirconate Titanate Thin Films**

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Fatigue behavior of heterolayered Pb(Zr$_{0.53}$Ti$_{0.47}$)O$_3$ films with alternating layers of Pb(Zr$_{0.48}$Ti$_{0.52}$)O$_3$ and Pb(Zr$_{0.72}$Ti$_{0.28}$)O$_3$ are studied as a function of thickness, temperature, and frequency. At the early stage of polarization fatigue, a wake-up phenomenon is observed which is particularly apparent as the number of alternating layers decreases. There is a frequency dependence of the polarization fatigue, whereby the heterolayered film fatigues earlier at lower frequency. The wake-up phenomenon was reduced with increasing temperature, causing the heterolayered Pb(Zr$_{0.5}$Ti$_{0.5}$)O$_3$ films to fatigue at a relatively lower number of switching cycles. The fatigue behavior of the heterolayered Pb(Zr$_{0.5}$Ti$_{0.5}$)O$_3$ films is accompanied by an increase of leakage current and a stronger domain wall pinning in the fatigued films. From these studies, it is concluded that during the polarization fatigue, injected electrons cause formation of space charges which impedes the domain switching.
Pulsed Laser Deposition (PLD) of the Gd$_2$O$_2$S:Tb$^{3+}$ Thin Films

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Over the last decade, luminescent properties of inorganic phosphors have been extensively investigated to make flat panel displays such as field emission displays (FEDs), plasma display panels (PDPs), and thin film electroluminescent devices (TFEL). This process has always been accompanied by improvements in the phosphors used. It is highly desirable to develop novel low-voltage phosphors with high efficiency and chemical stability under electron beam bombardment in a high-vacuum system for the next generation of field emission displays [1] and [2].

Gd$_2$O$_2$S:Tb$^{3+}$ luminescent thin films were grown on Si (10 0) substrates using pulsed laser deposition (PLD) in O$_2$ gas ambient, vacuum and at substrate temperature of 600$^\circ$C. The influence of the substrate temperature on the structural and morphological properties (surface chemical composition) of the films was investigated using X-ray diffraction (XRD), atomic force microscopy (AFM), and scanning electron microscopy (SEM) and Energy dispersive spectrometry (EDS). Photoluminescence (PL) measurement of the Gd$_2$O$_2$S:Tb$^{3+}$ luminescent thin films show green emission at the 545 nm (5D$^4$ - 7F$^5$ transition) wavelength. Surface characterization of thin films devices from Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) will be investigated in our next study.

References:
A Novel and Facile Synthesis of Hybrid Silica Nanocapsules and its Application in Fluorescence Imaging

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In the preparation of nanostructured materials, it is desirable to adopt bottom – up approach to synthesize new materials with controlled size, shape, and property. By utilizing artificial or natural template for directing the precipitation of inorganic precursors, we can create interesting novel nanostructured inorganic materials. On the basis of these concepts, thus, we successfully synthesized hybrid silica nanocapsules via a facile and benign approach of templated condensation at the core-shell interfaces of non-ionic PEO based block copolymer micelles. They are truly nanosized, which are ~15 nm and highly stable in aqueous environment, with part of the PEO block of the micelles extending out from the surface of the silica into the solution. The hybrid silica nanocapsules, which are non-cytotoxic and can be internalized by brain macrophages (BV-2) cells, are shown to be an effective fluorescence imaging marker by encapsulating fluorescent conjugated polymers into the core of the nanocapsules.

Positron Annihilation Studies on Gamma-Irradiated Barium Zirconate Ceramic

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Positron lifetime measurements, in general, can be used to study cationic and neutral vacancies in oxides. To ascertain the existence of such vacancies if any, at room temperature, in gamma irradiated barium zirconate ceramic (BaZrO3) the positron lifetime measurements have been carried out using BaF2 based positron lifetime spectrometer with a time resolution of 240 ps. The positrons on entering a solid will rapidly lose their kinetic energy via dissipative processes like electronic excitation and ionization, electron-hole pair creation and phonon interactions. As the positrons are able to survive for a few hundred picoseconds in the solid, their diffusion through the solid will be decisive in their trapping at the specific sites of eventual annihilation.

The samples were synthesized by the conventional ceramic method and found to be phase pure with good crystallinity. The γ - irradiation of the ceramic samples was carried out using ‘Gamma Chamber 900’. The dose delivered was 10 kGy with a dose rate of 2.17 kGy/hour. Positron lifetime measurements were carried out in reference and γ-irradiated BaZrO3 at room temperature. Measurements in the reference sample show the existence of only one lifetime component, implying that all the positrons annihilate in the defect free bulk of the reference sample. Lifetime in reference sample = 180 ± 2 ps. On the other hand, it is seen that even in the γ- irradiated sample, there exist only one lifetime component implying that the positrons annihilate at a single site with a lifetime of 179 ± 2 ps. This value of lifetime is almost same as that of the reference sample indicating that either cationic or neutral vacancies are not formed in BaZrO3 due to gamma irradiation, or, if they have been formed, they are mobile at room temperature leading to the recovery of these defects. In other words, either they are not formed or they do not survive at room temperature. If, by any chance, there exist only negative ion vacancies i.e. oxygen vacancies in BaZrO3 following gamma irradiation, positron could not detect these defects directly. This is due to the reasoning that the effective charge of oxygen vacancies is positive and positron being positively charged particle cannot detect these defects.

Labile Ferroelastic Nanodomains in Bilayered Ferroelectric Thin Films

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The ability to engineer and control domain behavior in ferroic materials systems such as ferroelectrics is of immense practical as well as fundamental interest. In the special case of thin film ferroelectrics that have a large ferroelastic self-strain associated with their phase transformation, a key aspect is the interaction of this self...
strain with the boundary conditions of the film. In this presentation we show, by depositing a strongly tetragonal ferroelectric thin film on a soft rhombohedral bottom layer, that these elastic interactions result in a ferroelastic domain structure in the tetragonal film that is susceptible to external perturbation. High-resolution piezoresponse force microscopy images demonstrate gross movement (nm scale) of the ferroelastic domains under local bias. This movement creates enhanced electromechanical response, which make the system attractive for novel sensor and nanoelectronic applications.

The observed results are supported by a linear elastic theoretical model that investigates ferroelastic domain fractions in a heteroepitaxial bilayer consisting of (001) tetragonal PbZr1-xTiO2 and (001) rhombohedral PbZr1-xTiO2 on a thick (001) passive as a function of the lattice misfit strain between layers and the substrate, by considering the self-strain in each layer and the indirect elastic interaction between the layers. In particular, we provide a numerical analysis of the relative domain fractions in the tetragonal layer of a (001) PbZr1-xTiO2/(001) PbZr1-xTiO2 and (001) PbZr1-xTiO2/(001) PbZr1-xTiO2 bilayer structure as a function of the tetragonal layer thickness on (001) LaAlO3, (001) SrTiO3 and (001) MgO. It is found that the self-strain coupling between the tetragonal and rhombohedral layers leads to an excess elastic energy in the tetragonal layer, resulting in a 2 to 3 times increase in the ferroelastic domain volume fraction of the tetragonal layer compared to single-layer films of similar thickness. These results show alternate ways of engineering ferroelastic domain structures in ferroelectric thin films.

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Hierarchically Nanostructured ZnO for Dye-Sensitized Solar Cells
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Nanostructured ZnO aggregates are synthesized via a hydrolysis route using zinc acetate dehydrate and diethylene glycol (DEG) as the precursors under carefully controlled conditions. They are then studied as the photoanodes in dye-sensitized solar cells, where the doctor-blade technique is employed to assemble a crack-free layer thickness of ~8µm from the ZnO nanoaggregates together with ethyl cellulose (EC) and terpineol. An energy conversion efficiency of 3.68% (under 100mW cm−2) is achieved by using 11wt % ZnO sintered at 400°C. In this paper, the various processing parameters and operation mechanisms involved in the synthesis of ZnO nanoaggregates and solar cell assembly will be presented and discussed.

Templated Mesoporous Titania for Dye-Sensitized Solar Cell Applications
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Mesoporous titania films were successfully prepared via an amphiphilic triblock copolymer templated sol-gel route following the evaporation-induced self-assembly mechanism. Layer-by-layer deposition was applied to thicken the films up to micrometer scale while the mesopore organization was retained. High degree of crystallization was achieved with the crystallite size of ~10 nm regardless of the film thickness, due to the spatial confinement of mesopores. The multilayered titania films as photoanodes were integrated into the dye-sensitized solar cells, demonstrating a complex interplay of factors determining the cell performance. An enhanced efficiency of 6 – 7% was achieved for the mesoporous titania films at the thickness of 5 – 6 µm, dependent on the mesostructural morphology, film thickness, pore accessibility, optical transparency and presence of micro-cracks.

Co-sensitization of Porous TiO2 by PbS Quantum Dots and Ruthenium Based Organic Dye N719
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PbS quantum dots (QD) have been successfully prepared by dipping the TiO2 paste into the solutions of Pb(NO3)2 and Na2S. Various quantum sizes were obtained by adjusting the dipping time and number of cycles. The PbS quantum dots were characterized by UV-visible absorption & TEM. Performance of the solar cells constructed from PbS-sensitized TiO2, N719-sensitized TiO2, and PbS & N719-cosensitized TiO2 has been studied. PbS QDs are shown to elevate the open circuit voltage (Voc) and subsequently increase the efficiency from 5.9%, given by N719-sensitized TiO2, to 6.4%, given by PbS & N719-cosensitized TiO2. The operating mechanisms involved in the co-sensitization are presented and discussed.

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Modulated Charged Defects and Their Effects on Electrical Behaviors of BiFeO$_3$-based Thin Films

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The charged defects structure in Bi$_{1-x}$La$_x$FeO$_3$ (BLF) and La-10% and Mg-2% codoped BiFeO$_3$ (BLFM) thin films, as well as their relation to electrical behaviors are investigated. Through temperature-dependent conductivity and XPS analysis, it is demonstrated that La doping suppresses but Mg doping increases the concentration of both oxygen vacancies (OVs) and Fe$^{2+}$ ions. Correspondingly, the leakage mechanism evolves from grain boundary and space charge limited conduction of BLF ($x$=0.2 and 0.1) to Poole-Frenkel emission of BLM and BiFeO$_3$ with the variation of dominant defects from OVs to Fe$^{2+}$ ions. The formation of defect complexes between the acceptors and OVs is suggested to interpret the highest activation energy of BLM film, while the dielectric relaxation is resulted from the migration of OVs. Furthermore, compared with BiFeO$_3$, BLF ($x$=0.2) film reveals a raperd increase in C-V curve and smaller dielectric loss, deviating from the Rayleigh law. It is proposed that due to the depressed charged defects in BLF ($x$=0.2) film, an enlarged distance between two nearest pinning centers is resulted, allowing the reversible bending movement of pinned 180-degree domain walls, which contributes to the dielectric permittivity nonlinearity without inducing loss.

Effects of Anodization Parameters on Titania Nanotube Arrays and the Performance of Dye-sensitized Solar Cells

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Dye sensitized solar cells (DSSCs) have attracted considerable attention as potential, cost-effective alternatives to silicon-based photovoltaic (PV) devices. The efficiency of DSSCs with a liquid electrolyte can reach as high as 11%. One of the factors limiting the performance of the DSSCs is the electron collection efficiency through the mesoporous titania layer. Titania nanotube arrays (TNAs) aligned perpendicular to electron collection electrodes could enhance electron transport and reduce recombination with redox electrolytes, leading to the higher charge collection efficiency. As a consequence, titania nanotube arrays replacing the conventional nanoparticle titania thin films is expected to improve the PV performance of DSSCs. Highly ordered, closely packed, and vertically oriented titania nanotube arrays with lengths exceeding 10µm were fabricated by anodizing titanium in an electrolyte composed of ammonium fluoride, ethylene glycol and deionized water. The microstructural morphology of the TNAs were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD) and were adjusted by modifying the anodization voltage and time over a wide range. After anodization the nanotubes walls are amorphous, but they can be transformed into anatase of ca. 50 nm in crystallite size by annealing in air at 450°C for three hours. Anodization voltage and time greatly influence the photovoltaic performance of dye sensitized solar cells based on the TNAs by adjusting the available surface area. The resulting efficiency enhancement is dominated by the variation of $J_{sc}$. A promising efficiency of 4.16% ($J_{sc}$ 7.68mA/cm$^2$, $V_{oc}$ 0.803 and FF 67.4%) under AM 1.5 100mW/cm$^2$ illumination was achieved.

Thickness Dependent Properties of Epitaxial BiFeO$_3$ Multiferroic Thin Films

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Epitaxial BiFeO$_3$ multiferroic thin films of different thicknesses were grown by radio frequency sputtering on SrTiO$_3$ single crystal substrate with a SrRuO$_3$ buffer layer. The ceramic target used for sputtering had a 10% bismuth excess to compensate the likely loss in association with evaporation at high temperature. Crystal structure of the epitaxial films were characterized by using high-resolution X-ray diffraction (XRD), showing the epitaxial growth. Ferroelectric and dielectric properties were investigated by using the Radiant workstation (Radiant Technologies) and an impedance analyzer (Solartron Gain Phase Analyzer). High remnant polarization with low leakage current was obtained in our epitaxial thin films. The effects of strain and stress arising from the lattice mismatch and thermal mismatch will be presented and discussed.
peaks corresponding to the commensurate/incommensurate modulation of oxygen octahedra, polar clusters of A-site ion ordering and B-site ion ordering, respectively, were observed in the composition with x=0.25. With decreasing the radius difference between A1- and A2-ions (increasing x), the dielectric relaxations especially the one originating from the polar clusters of A-site ion ordering tended to increase significantly and overlap the diffuse ferroelectric peak, which was completely overlapped for x≥0.75. This process just reflected the increased disordering degree of both A- and B-site ions, and the analysis of ultrasonic attenuation strongly supported the above conclusions on dielectric relaxations and their structural origins. The ultrasonic attenuation peak around 100K corresponded to the freezing process of the dielectric relaxations, and the fluctuation with composition of the ultrasonic attenuation peaks between 150 and 260K suggested the possible structure variation.

Dielectric Properties Bi$_{1.15}$Nd$_{0.85}$Ti$_{3}$O$_{12}$ Ceramic with a Core-Shell Structure
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Bi$_{1.15}$Nd$_{0.85}$Ti$_{3}$O$_{12}$ Ceramics were successfully prepared with a core-shell structure of the grains. The Bi$_{1.15}$Nd$_{0.85}$Ti$_{3}$O$_{12}$ was calcined by the traditional approach at 800°C and then the particles were submerged by excessive melt sodium chloride at 850°C for a certain time. After that, the Cl- and relative soluble cations were washed off by deionized water. The surface-modified particles were pressed into a pellet and sintered at 1100°C. The dielectric loss tangent δ of the modified ceramics researched down to ~0.002, which is about 8-10 times lower than the reported data. At the same time, the dielectric constant was not affected.

Epitaxial Functional Oxide Films: A View From the World of Ferroelectrics
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Epitaxial oxide films display an amazingly broad spectrum of cooperative phenomena that allow functional coupling to external stimuli. However, the complexity of these typically multicomponent systems has made understanding their structure-property relationships a grand challenge in materials science. Using the example of metalorganic chemical vapor deposition of epitaxial PbTiO$_3$, we will illustrate how in-situ synchrotron x-ray scattering and fluorescence can be utilized to address this challenge for the study of the synthesis and physics of functional complex oxides incorporated into various representative heterostructures. This includes deposition onto novel substrate materials such as DyScO$_3$, which offer an alternative to SrTiO$_3$ for control of epitaxial strain. In addition, the polar nature of substrates such as DyScO$_3$ impose a different electrical boundary condition than would arise at a non-polar substrate surface such as SrTiO$_3$ (001). We observe that both substrate surface charge as well as charge linked to the gas environment above a film can impose a strong bias on the polarization of the film. Indeed, changing the partial pressure of oxygen in the gas above ultrathin PbTiO$_3$ films on SrRuO$_3$ can induce inversion in the sign of the polarization, i.e. chemically-induced switching occurs. Most interestingly, we observe that under certain conditions nucleation is suppressed and switching can occur continuously, i.e. by uniform decrease and inversion of the polarization amplitude rather than by formation and motion of domain walls, indicating that the intrinsic coercive field is reached. Since switching at the intrinsic coercive field has never been achieved in oxide ferroelectrics using applied electric field, our results suggest that the barrier to nucleation is large during chemical switching. We will review our experimental results on the effect of interface charge compensation on the ferroelectric phase transition, how interface charge is related to the gas-phase chemistry above a film under different processing conditions, and explore links to surface reconstructions that have been found for this system.

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Engineering Exotic Phenomena at Ferroelectric Oxide Interfaces: A First-principles Perspective

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Thanks to recent advances in epitaxial oxide growth, ferroelectric oxide heterostructures and superlattices can nowadays be realized with atomic-level precision. This is bringing the field of ferroelectrics to an entirely new level and offers tremendous new possibilities for creating artificial multifunctional materials and devices [1]. First-principles calculations constitute a powerful tool to clarify the physics of oxide heterostructures at the atomic level and to guide the experimentalists toward the design of artificial nanostructures with enhanced or, eventually, totally new properties. After a brief introduction, I will focus on two concrete examples. In a first part, I will illustrate how it is possible to couple ferroelectric and antiferrodistortive structural instabilities at the inter-layer interfaces of PbTiO$_3$/SrTiO$_3$ superlattices in order to induce an improper ferroelectric behavior and obtain unusual dielectric properties of direct interest for technological applications [2]. In a second part, I will report recent first-principles results concerning ferroelectric tunnel junctions and deduce some general rules for the design of optimized structures.

Work done in collaboration with Eric Bousquet, Patrick Hermet, Daniel Bilc, Frederico Novaes, J. Iniguez, P. Ordejon Matthew Dawber, Céline Lichtensteiger and Jean-Marc Triscone and supported by the FAME NoE, the MaCoMuFi and OxIDes Strep projects and the VolkswagenStiftung.


Optimization of Thickness of Magnetron Sputtered MgO Thin Films Thickness as Protective Layer for Plasma Display Panels

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Low sputtering yield and large band gap have made Magnesium oxide (MgO) an important material for dielectric protective layer in plasma display panels (PDPs). In this work, we report the influence of thickness of MgO layer on the secondary electron emission and firing voltage. A major improvement in PDP performance depends on the thickness of Magnesium oxide film as the protective layer due to its high secondary electron emission and anti-sputtering properties. In this work, we deposited MgO thin films varying the deposition time using pulsed dc magnetron sputtering technique. Thin film characteristics such as crystal orientation, surface morphology, optical properties (transmittance & reflectance) and secondary electron emission coefficient were systematically investigated by X-ray diffraction (XRD), Atomic Force Microscope (AFM), UV-VIS spectrophotometer and Secondary Electron Emission Tool (SEE-Tool) respectively. It was observed that surface roughness increased with thickness of MgO thin films. We found that secondary electron emission (SEE) coefficient is dependent on the MgO thickness. Mgo thin films with thickness ~900 nm have lower SEE coefficient than films having thickness ~400 nm. The reasons behind lower SEE coefficient of thicker MgO thin films is studied. If the MgO layer is too thin (~100 nm), a few secondary electrons
generated within the MgO film and results in lower SEE coefficient. Optimized thickness (400 nm-600nm) of MgO thin films will be useful for as protective layers for future plasma display panels technology.

**Effect of Deposition Parameters on Structural and Surface Characterization of Magnetron Sputtered MgO Thin Films**

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Low sputtering yield and large band gap have made Magnesium oxide (MgO) an important material for dielectric protective layer in plasma display panels (PDPs). In this work, we report the influence of deposition parameters on structural and surface characterization of pulsed dc sputtered MgO thin films. We deposited MgO thin films varying the different parameters using pulsed dc magnetron sputtering technique. Thin film characteristics such as crystal orientation, surface morphology, optical properties (transmittance & reflectance) and secondary electron emission coefficient were systematically investigated by X-ray diffraction (XRD), Atomic Force Microscope (AFM), UV-VIS spectrophotometer and Secondary Electron Emission Tool (SEE-Tool) respectively. It was observed that surface roughness increased with thickness of MgO thin films. We found that secondary electron emission (SEE) coefficient is dependent on the MgO thickness. MgO thin films with thickness ~400 nm have lower SEE coefficient than films having thickness ~ 300 nm. The reasons behind lower SEE coefficient of thicker MgO thin films is studied. If the MgO layer is too thin (~100 nm), a few secondary electrons are generated within the MgO film and results in lower SEE coefficient. Optimized thickness (400 nm-600nm) of MgO thin films will be useful for as protective layers for future plasma display panels technology.

**Characterization of Si doped MgO Thin Films for Plasma Display Panels**

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Plasma display panels (PDPs) have emerged as the successful technology for large area flat panel display with a wide viewing angle. A major improvement in PDP performance depends on the doping of Magnesium oxide (MgO) film as the protective layer due to its high secondary electron emission and anti-sputtering properties. In this work, we deposited Si (30 ppm) doped MgO thin films at different substrate temperature and different oxygen partial pressure by using electron-beam evaporation method. Thin film characteristics such as crystal orientation, surface morphology, optical properties (transmittance & reflectance) and secondary electron emission coefficient were investigated by X-ray diffraction (XRD), Atomic Force Microscope (AFM), UV-VIS spectrophotometer and Secondary Electron Emission Tool (SEE-Tool) respectively. It was found that the crystal orientation, crystal size and surface morphology are dependent on substrate temperature as well as the oxygen partial pressure. From the results, it was observed that substrate temperature and oxygen partial pressure is a crucial parameter for the crystal orientation. All the films were found more than 90% transparent in the visible range.

**Novel Ferroelectric Capacitor for Non-volatile Memory Storage and Biomedical Tactile Sensor Applications**

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We report on novel ferroelectric thin film compositions for use in non-volatile memory storage and biomedical tactile sensor applications. The Lead Zirconate Titanate (PZT) composition was modified by Lanthanum (La³⁺) (PLZT) and Vanadium (V⁵⁺) (PZTV, PLZTV) doping. Hybrid films with PZTV and PLZTV as top layers are also made using seed layers of differing compositions. La³⁺ doping decreased the coercive field, polarization...
and leakage current, while increasing the relative permittivity. V⁵⁺ doping, while having similar effects, results in an enhanced polarization, with comparable dielectric loss characteristics. Complex doping of both La³⁺ and V⁵⁺ in PLZTV, while reducing the polarization relative to PZTV, significantly decreases the coercive field. Hybrid films have a greater uniformity of grain formation than non-hybrid films, thus decreasing the coercive field, leakage current and polarization fatigue while increasing the relative permittivity. Analysis using X-Ray Diffraction (XRD) verified the retention of the PZT perovskite structure in the novel films. PLZT/PZTV has been identified as an optimal ferroelectric material due to its desirable ferroelectric, piezoelectric, and pyroelectric properties. Complex doping of both La³⁺ and V⁵⁺ results in an enhanced polarization, with comparable dielectric loss characteristics. Complex doping of both La³⁺ and V⁵⁺ in PLZTV, while reducing the polarization relative to PZTV, significantly decreases the coercive field.

The processing of the ferroelectric layer is of central importance to BioMEMS as this layer provides the piezoelectric properties and also the processing step must be compatible with the fabrication sequence. Sol-gel method is well suited for BioMEMS application because films with reproducible compositions and properties can be prepared with desirable thickness at acceptable processing temperatures and low cost. The scope of this presentation will cover ferroelectric materials research, device design and processing technologies that is suitable for biomedical applications.

Multiferroic BiFeO₃ has attracted great interest in potential applications to spintronic devices due to its room temperature magnetoelectric coupling. BiFeO₃ is also considered as a candidate for lead-free nonvolatile memories because of its high remanent polarization and piezoelectric response. The characterization and control of domain walls are critical issues for the fabrication of the epitaxial BiFeO₃ thin films desirable for devices. In this paper we will report the effect of interfacial structure and strain on the structures and properties of domain walls in epitaxial BiFeO₃ thin films fabricated by sputtering on SrTiO₃, Si, and TbScO₃ single crystal substrates. The domain wall structures of BiFeO₃ films with different boundary conditions were characterized by high-resolution transmission electron microscopy (HRTEM). The electronic properties of different domain walls in epitaxial BiFeO₃ thin films were determined by electron energy-loss spectroscopy (EELS) using a spherical aberration corrected cold field emission gun TEM. TEM imaging reveals that the domain configuration and domain wall structure strongly depend on strain, interfacial structure, and crystallographic symmetry of substrates. The TEM results correlate highly with the electrical and ferroelectric properties measured from the same films, indicating the mechanism of domain wall contributions to leakage and multiferroic properties of BiFeO₃ materials.

Multilayered Ferroelectric Micro Sensors for Biomedical Applications
S. E. VALA V AN
New Materials Technology Development Centre, School of Applied Science, Republic Polytechnic, Singapore

Ferroelectric functional thin films fit naturally into the burgeoning field of microelectro-mechanical systems (MEMS), since their superior ferroelectric, piezoelectric and pyroelectric properties make them ideal materials for micro-sensors and actuators. Lead zirconate titanate and its derivatives, such as PbTiO₃, Pb(La,Zr,Ti)O₃ and Pb(Zr,Ti,Sn)O₃, etc., are widely used because of their large electromechanical coupling coefficients, temperature stability and high resistance to depolarization from mechanical stress and high driving voltages. The recent research on piezoelectric sensors and actuators is moving toward miniaturization to achieve better resolution, precise mechanical displacements and high power densities. These requirements are needed in applications such as biomedical, manufacturing, information processing and automotive industries. In the biomedical sector, several key piezoelectric MEMS are being introduced for fabricating devices such as lab-on-a-chip, micropumps, drug delivery, tactile, imaging and molecular recognition sensors.

The processing of the ferroelectric layer is of central importance to BioMEMS as this layer provides the piezoelectric properties and also the processing step must be compatible with the fabrication sequence. Sol-gel method is well suited for BioMEMS application because films with reproducible compositions and properties can be prepared with desirable thickness at acceptable processing
An Investigation of Structural, Magnetic and Dielectric Properties of \( R_2 \text{NiMnO}_6 \) (\( R = \text{rare earth, Y} \))

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We have investigated the structure, magnetic and dielectric properties of the double perovskite oxides \( R_2 \text{NiMnO}_6 \) where Ni and Mn atoms are ordered at 2c and 2d sites, respectively. All the phases are ferromagnetic insulators exhibiting relatively low dielectric loss and dielectric constants in the range 15–25. The ferromagnetic ordering temperature of the \( R_2 \text{NiMnO}_6 \) series seems to correlate better with the radius of \( R^{3+} \) ions than with the average Ni–O–Mn angle (\( \omega \)) in the double perovskite structure. These results are consistent with all samples having \( \text{Mn}^{3+} \) and \( \text{Ni}^{2+} \) with minimal anti-site disorder.

Fabrication and Mechanism of Large Area and Uniform Silver Dendritic Micro-structures

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The development of novel nanostructured materials with controlled shapes and ordered arrangement has stimulated great research for seeking novel properties and tailorable functions. In particular, the synthesis of metal nanostructures with controllable shapes received much attention in the modern materials chemistry. Beyond their aesthetic appeal, these new structures are compelling for multiple fundamental and practical technological reasons. First, their synthesis has motivated experimental progress in understanding the intrinsic shape-dependent properties of metal nanocrystals. Second, some of these structures feature optical and electrical properties that make them desirable for emerging applications including biolabels, photovoltaic behavior, chemical sensing, and surface enhanced Raman scattering, among others. Third, some of these structures enable elucidation of the particle growth mechanism, which in turn may make possible the prediction and systematic manipulation of the final nanocrystal structure. Finally, these new materials provide new template systems for the further generation of different structures. In this paper, we extend the colloidal crystal template technique to the electrochemical synthesis of silver dendritic array. In a typical process, the colloidal polystyrene spheres and electrodeposited ZnO were used as the primary and secondary template, respectively. Then, the periodic silver dendritic structures were prepared on the ZnO template by electrodeposition. The experimental results show that most of these dendritic cells have a two-level structure with a whole size near 700nm and present an order hexagonal periodicity. We also studied the main effective parameters, such as the distance between two electrodes and the concentration of surfactant, and growth mechanism of this silver dendritic structural array.

Synthesis of ZnO Nanorods and Highly Textured Films by Hydrothermal Route

Tong LI; H. M. FANG; J. M. XUE; J. DING

Department of Materials Science and Engineering, National University of Singapore, Singapore

Due to numerous potential applications of high quality ZnO thin or thick films and the great advantages of hydrothermal synthesis in both cost and environmental impact, a significant effort recently has been made for growth of highly textured ZnO films via hydrothermal method. However, considering the requirement of hydrothermal growth on small lattice mismatch between ZnO films and the substrates, most research now is confined to employ spinel or GaN-buffered sapphire as the substrates for ZnO films synthesis. In this study, we report the hydrothermal growth of highly textured ZnO film on an as-grown ZnO thin film fabricated by pulsed laser deposition (PLD), which could actualize the growth of highly textured ZnO films on various substrates such as quartz, silicon, glass, etc.

In this study, highly textured ZnO films were successfully synthesized by hydrothermal route at 80°C. The highly textured ZnO thin film with a thickness of 20nm grown on quartz by PLD was employed as the substrate. A dense and continuous ZnO film (ca. 2μm) was further grown on the substrate in an aqueous solution consisting of \( \text{Zn(NO}_3\text{)}_2\cdot6\text{H}_2\text{O} \), ammonium nitrate at the PH value of approximately 10.5 for 24h. Scanning electron microscopy, X-ray diffraction, PL and Hall effect were used to investigate the structural, optical, and electrical properties of the ZnO films. X-ray diffraction analysis showed that the thin film was highly textured and exhibited wurtzite structure and c-axis orientation. The rocking curve of this hydrothermal ZnO thin film with a halfwidth of 2.7° was compared with the seed layer deposited by PLD (3.1°). This study lays the foundation for growing high-quality ZnO films with large area on different substrates, which could be a promising technique for various applications.
Solid State Catalysis Of Epitaxial Oxide Thin Films

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Catalysis is increasingly playing an important role in the development of various nano-materials, chemicals, fuel cells, food and energy processing. It provides an alternative and faster route to lowering the activation energy for chemical reactions, which may be prohibited or slowed due to intrinsic kinetic barriers. Although most catalysis processes are restricted to surfaces, catalysis inside a solid influencing its bulk and thin film properties is yet to be recognized and exploited. Here, we demonstrate the growth of epitaxial SiO\textsubscript{2} and HfO\textsubscript{2} thin layers with the help of solid state catalysis. Under conventional growth conditions, in the former case epitaxial growth is pretty unlikely and in the latter case very high growth temperatures are required. High quality epitaxial SiO\textsubscript{2} thin layers were achieved through solid state reactions with the help of the alkaline catalysts Cs or Rb. HfO\textsubscript{2} epitaxial films were grown by pulsed laser deposition at far reduced temperatures using Co as a catalyst. The mechanism of the catalysis process will be discussed.

Femto-Second Laser Excitation Studies Of Oxide Thin Films and Heterostructures

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Oxide thin-film heterostructures offer unique opportunities for combining materials with various functionalities, providing a versatile pathway to create novel oxide electronic devices. In particular, studies have recently shown high-mobility two-dimensional conducting planes emerge at the interface between insulating oxide heterostructures of LaAlO\textsubscript{3} and SrTiO\textsubscript{3}. Subsequently, various other physical properties such as magnetism and superconductivity were also observed at the interfaces of these heterostructures. However, apart from the exploratory work, the in-depth investigation of the unusual properties of this electronic system has only just begun.

The real-time dynamical studies of charge carriers out of equilibrium can give detailed microscopic information about electronic correlation at these interfaces, which can be read out with time-resolved optical spectroscopy on a sub-picosecond time scale. Here we will discuss our current experiments using femtosecond laser pump and probe technique to investigate the real-time dynamics of the electrons and holes at the LaAlO\textsubscript{3}/SrTiO\textsubscript{3} interfaces.

Ta Doped TiO\textsubscript{2}: Substitutionality and Bandgap Variation

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We have investigated the substitutionality, bandgap and the lattice constant evolution of anatase TiO\textsubscript{2} films deposited by Pulsed laser deposition as a function of Ta doping. It is shown that there is complete Ta substitution in the Ti lattice for almost all the concentrations using Rutherford backscattering / ion channeling measurements. There is a strong oxygen pressure and temperature dependence of the substitutional fraction. The bandgap of the doped TiO\textsubscript{2}, measured by UV-Vis spectroscopy, shows a blue shift. This appears to be more consistent with the formation of an alloy TaxTi1-xO\textsubscript{2} with a bandgap given by a linear combination of the bandgaps of the end members than the band filling effects (Moss-Burstein shift). The origin of this blue shift will be presented and discussed along with the experimental results.
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