SYMPOSIUM D

D: Materials and Devices for Smart Systems

November 30 - December 5, 2003

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* Invited paper

TUTORIAL

FT D: Smart Materials-Classifications, Devices, and Composites
Sunday, November 30, 2003
10:30 AM - 5:00 PM
Room 202 (Hynes)

The tutorial will cover the "fundamentals" (designs and fabrication methods) of smart materials, devices, and composites, and their engineering applications for young students, as well as engineers in the fields of materials science, and electrical, mechanical, and civil engineering, etc. The lectures consist of three ninety-minute presentations by specialists in: a) material science of sensor/actuator materials; b) features and classification of smart materials and devices; and c) smart composite systems and their engineering applications. The intention of this tutorial is to give attendees the new concept of smart/intelligent material, fundamental design, fabrication processes and features of each smart/intelligent material, devices, and smart composites, and their potential applications for the industries, including the ease/difficulty/cost of setting each up, and future directions.

Instructors:
Manfred Wuttig, University of Maryland
Victor Giurgiutiu, University of South Carolina
Hiroshi Asanuma, Chiba University

SESSION D1: Ferroelectric and Piezoelectric Materials and Devices
Chairs: Dragan Damjanovic and Qiming Zhang
Monday Morning, December 1, 2003
Room 202 (Hynes)

8:30 AM *D1.1
Recent Progress In Transducers Actuators And Sensors For Smart Systems. L. Eric Cross, Materials Research Institute, Penn State University, Universtiy Park, Pennsylvania.

All High strain electro-active dielectric actuators are basically electrostrictors, yet the manner in which the phenomenon is manifest is startlingly different between several of the systems of current interest. In soft polymeric elastomers, very large electrostrictive strains (over 200%) are a direct consequence of the Maxwell Stress exerted by the attractive force between unlike charges on the opposite electrode surfaces. Current low permittivity silicone and acrylic elastomers have many attractive potential applications, but do need uncomfortably high electric fields (over 100 Volts/micron) for full performance. Developments to lower the needed fields and improve force levels will be discussed. For high energy electron irradiated or chemically modified Polyvinylidene difluoride: Trifluoroethylene P(VDF:TrFE) Copolymer, the relaxor like modification of the ferroelectric form permits field induced electrostrictive strains up to 7% and high coupling piezoelectric properties under bias field, again however, E fields required are uncomfortably high and efforts to modify these systems will be considered.In polymer sensors an exciting development from Tampere Finland is the polymer foam space charge electrets which have d33 comparable to ceramic PZT, but ultra-low permittivity resulting in spectacular dhgh figures of merit for shallow water hydrophones. In the stiffer inorganic polycrystal ceramics it is essential to engineer phase instability to achieve high strain and polability, and the focus is on systems with morphotropic phase boundaries. Lead zirconate titanate (PZT) is still the 'work horse' of polycrystals, but the recently discovered monoclinic phase at the MPB composition is stimulating new thinking on the origins of the useful properties. New MPBs in the Bi3+B3+O3:PbTiO3 solid solutions include polabe polycrystals with wider temperature range and exciting new possibilities in both bulk and thin film forms. For BiFeO3 based systems, better control of conductivity is opening new possibilities for the most difficult transduction, namely that between electric and magnetic properties i.e. high magnetoelectric coefficients. The quite outstanding piezoelectric properties in the single crystal lead zinc niobate:lead titanate and lead magnesium niobate: lead titanate MPB compositions close to the MPB are now fully authenticated, but the complex of phase stabilities, dielectric and piezoelectric properties and switching responses still call for more detailed studies and a more complete explanation.

9:00 AM *D1.2
Enhanced Piezoelectric Property of Piezoelectric Single Crystals by Domain Engineering. Satoshi Wada and Takaaki Tsurumi; Department of Metallurgy & Ceramics Science, Tokyo Institute of Technology, Tokyo, Tokyo, Japan.

Engineered domain configuration is one of the domain engineering techniques and makes piezoelectricity of ferroelectric single crystals extremely enhanced. In this study, this engineered domain configuration was induced into barium titanate single crystals as a typical lead-free piezoelectric single crystals, and the piezoelectric property was investigated as a function of (1) crystal structure, (2) crystallographic orientation, (3) domain size. As a result, in the orthorhombic barium titanate single crystals, when electric field (E-field) was applied along [001] direction, the maximum piezoelectric property was observed and its piezoelectric properties were larger than those of PZT ceramics. Moreover, domain density of tetragonal barium titanate single crystals was controlled by temperature and E-field, and their piezoelectric properties were measured. The barium titanate crystals with high domain density exhibited the much higher piezoelectric properties than those of barium titanate crystals with low domain density. The above results revealed that what kind of engineered domain configurations is the best for the piezoelectric application.

9:30 AM D1.3
Polarizable Charge Equilibration Force Field and Application to Phase Transformation in Ferroelectrics. Qinsong Zhang, Tahir Cagin, Alejandro Strachan and William A Goddard; California Institute of Technology, Pasadena, California.

We present the Polarizable Charge Equilibration force field to include self-consistent atomic polarization and charge transfer in molecular dynamics of materials. The charge on each atom is partitioned into a Gaussian-shaped core with fixed (positive) charge (4 for Si) and a Gaussian-shaped shell with variable charge. The shell charges can flow from atom to atom based on the QEq (Charge equilibration) scheme of Rappe and Goddard. The restoring force between a core and its shell is given by the electrostatic interaction between the two charge distributions. The long range London dispersion and short range Pauli repulsion effects are described by two body potentials (Morse or Lennard-Jones) that act between all atoms (no exclusions). Using the above model, we determined all the parameters in the force field for BaTiO3 directly from Quantum Mechanics calculations (DFT with GGA). We find that the model leads to correct temperature sequence for the transitions between the four phases of BaTiO3. With the model it is practical to consider simulations of large unit cells to study domain boundaries, surface reconstruction, the role of defects and the effects of temperature, pressure, and applied electric fields and stress.

9:45 AM D1.4
Mixed Ordering in Pb-based complex perovskites.
K. Z. Baba-kishi, C. W. Tai, X. Meng, H. L. W. Chan and C. L. Choy; Department of Applied Physics, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong.

High-resolution transmission electron microscopy (HRTEM) studies of the ceramics $(x)Pb(In_{1/2}Nb_{1/2})O_3$:$(1-x)Pb(Mg_{1/3}Nb_{2/3})O_3$, with x=0.1, 0.3 and 0.6 show ordered nano-regions along the [001] direction. The nano-regions are separated from each other but randomly distributed. The contrast exhibited by each region is distinct from the neighbouring region, caused by the clustered ordering of a specific cation concentration. The state of mixed order creates a superstructure whose intensity tends to vary depending on the distribution of the nano-ordered regions. The ideal 1:1 ordering of a B-site cation pair along the [110] direction creates a long-range order with a superstructure of its own. However, mixed ordering occurs along [001] direction, can be highly random in nature and might also be associated with localized short-range order. The presence of the superstructure reflections and diffuse streaking along [001] is dependent on the degree of mixed ordering. HRTEM studies of the three compounds show various degrees and forms of mixed ordering. Simulations carried out illustrate the ways in which these regions are ordered primarily by preferential clustering of one of the three cations in any one of the nano-regions. Six clusters of Mg-Nb, In-Nb and In-Mg of which three clusters are 1:1 ordered and the remaining three clusters are disordered, are incorporated into the simulations. Simulations include models of the diffraction patterns in which a simple perovskite structure ABO_3 with space group Pm3m is extended into three distinct ordered unit cells, containing the B-site cations In, Mg and Nb. A variety of different conditions in which ordered and disordered clusters were combined to simulate the HRTEM images will be described in detail. The origin of the superstructure reflections and diffuse streaking and the influence of mixed ordering on the dielectric properties will be discussed.

10:30 AM *D1.5
Highly Efficient Piezoelectric Actuators for Active Vibration Control. Enrico Colla, Ganesh Suyal, Sandrine Gentil and Nava Setter; Ceramics Laboratory, EPFL Swiss Federal Institute of Technology, Lausanne, Vaud, Switzerland.

Small actuators providing large displacements and reasonably high forces are required for vibration control in a wide range of applications. This need is rarely satisfied by currently available compact actuators. We report on such a family of low cost / high performance disk bender actuators produced by combining efficient design and fabrication methods. The properties of these actuators are intermediate between those of standard bimorphs, used for very large displacements but providing rather small forces, and those of low voltage stack multilayers, which provide quite large forces but are generally heavier, larger and very expensive for equivalent displacements. The absence of any external mechanical amplification mechanism makes the actuators particularly efficient and ideal for active vibration damping applications within buildings or for noise control by emission of controlled sound in antiphase. In order to lower the driving voltages, multilayer disk benders can also be fabricated with the same technique. The actuator displacement and force properties are not influenced by the number of layers. Patterning of the internal and external electrodes for the application of appropriate voltage configurations enables the development of actuators with highly specialised functionalities. The fabrication method enables the downsizing of these piezodevices to satisfy MEMS requirements too.

11:00 AM D1.6
Novel High Power Piezoelectrics for Transformers and Actuators. Shashank Priya[1] and Kenji Uchino[2]; [1]Penn State University, University Park, Pennsylvania; [2]Electrical Engineering, Penn State University, University PArk, Pennsylvania.

High Power piezoelectric materials are required for numerous devices such as piezoelectric transformers, high frequency actuators, ultrasonic mototrs and current sensors. All these applications demand a material that exhibits a combination of hard and soft properties, i.e., high piezoelectric, dielectric and electromechanical constant along with low dielectric and electromechanical losses. Further the properties should be nonhysteretic with field and temperature. This study illustrates the development of new family of PZT-PZN-PNN based ceramic materials whose property can be tailored to meet these requirements. The results will be shown which conclusively proof that this material can generate high electrical power as compared to other available choices.

11:15 AM *D1.7
50 years of Piezoelectric Transformers. Trends in the Technology. Alfredo Vazquez Carazo, Face International Corp., Norfolk, Virginia.

The initial idea of a piezoelectric transformer (PT) corresponds to C.A. Rosen, K. Fish, and H.C. Rothenberg and is gathered in the U.S. Patent 2830274, applied in 1954. Fifty years later, this technology has become one of the most promising alternatives to replace the magnetic transformers in a wide range of applications. PTs convert electrical energy into electrical energy by making use of acoustic energy. These devices are typically manufactured using piezoelectric ceramic materials that vibrate in resonance. With an appropriate design it is possible to step-up and step-down voltage between the input and output of the transformer without the use of wires or magnetic materials. The technology did not reach commercial success until early the 90s. During this period, several companies, mainly in Japan, decided to introduce PTs for applications requiring small size, high step-up voltages, and low electromagnetic interference (EMI) signature. This type of PTs where developed based on optimizations of the initial Rosen concept. That's why they are typically referred as Rosen-type PTs. Today's, PTs are used for backlighting LCD displays in notebook computers, PDAs, and other handheld devices. The PT yearly sales estimate was about 20 millions in 2000 and industry sources report that production of piezoelectric transformers in Japan is growing steadily at a rate of 10% annually. The reliability achieved in this application and the advances in the related technologies (materials, driving circuitry, housing and manufacturing) have currently motivated an enormous interest and confidence in expanding this technology to other applications fields. Currently, the industry is moving in two directions: the low-cost product market and the value-added product market. Prices of PTs have been declining in recent years, and this trend is expected to continue this year that may make this technology a serious candidate to replace the magnetic transformers in cost-sensitive applications. As for the value-added product market, leading makers are reportedly focusing on more value-added products. Two key points are miniaturization and higher output power. Piezoelectric transformers for power applications require lower output impedances, high power capabilities and high efficiency under step-down conditions. Among the different PT design proposed as alternative to the classical Rosen configuration, Transoner laminated radial PT has been demonstrated as the most promising technology for achieving high power level. Higher powers than 100W, with power densities in the range of 30-40 W/cm2 has been demonstrated. Micro-PTs are currently being developed with sizes of less than 5mm diameter and 1mm thickness allowing up to 0.5W power transfer and up to 50 times gain. Smaller sizes could be in the future integrated to power MEMs systems. This paper summarizes the state of the art on the PT technology and introduces the current trends of this industry.

11:45 AM D1.8
Phase field simulations of polarization switching under an applied electric or stress field. Jie Wang[1], S.Q. Shi[2], L.Q. Chen[3] and Tong-Yi Zhang[1]; [1]Department of Mechanical Engineering, Hong Kong University of Science and Technology, Hong Kong, Hong Kong; [2]Department of Mechanical Engineering, Hong Kong Polytechnic University, Hong Kong, Hong Kong; [3]Department of Materials Science and Engineering, The Pennsylvania State University, University Park, Pennsylvania.

The polarization switching in a ferroelectric subjected to an electric field or a stress field is simulated using a phase field model based on the time-dependent Ginzburg-Landau equation, in which both the long-range electric and the long-range elastic interactions are taken into account. Domain switching takes place when the electric field or the stress field exceeds a threshold value. The temporal evolution of polarization switching shows that the switching is a process of nucleation new domains following by the disappearance of old domains and growth of new domains, which is accomplished through domain wall motion. An electrical load can induce both 1800 and 900 polarization switching, while a mechanical load can produce only 900 polarization switching. The macroscopic polarization or strain is obtained by averaging polarizations and strains over the simulated size at the final stable domain configuration under each given electric field or stress load, correspondingly. The simulation results successfully reveal the hysteresis loop of the macroscopic polarization versus the applied electric field, the butterfly curve of the macroscopic strain versus the applied electric field, and the macroscopic strain response to an applied compressive stress.

SESSION D2: Shape Memory Alloys
Chairs: Kanryu Inoue and Eckhard Quandt
Monday Afternoon, December 1, 2003
Room 202 (Hynes)

1:30 PM *D2.1
Application of Active Materials to Microactuation. Kaushik Bhattacharya, Mechanical Engineering and Materials Science, California Institute of Technology, Pasadena, California.

The development of effective microactuators remains an important challenge in MEMS. Active materials – shape-memory alloys, ferromagnetic shape-memory alloys and ferroelectric materials – offer promising opportunities. However, many practical difficulties have limited the optimal utilization of such materials. This talk will describe a strategy for microactuation using active materials which makes explicit and optimal use of the underlying microstructure; it shows that large displacement and force can be generated from small volumes by the manipulation of the underlying microstructure or domain patterns. The talk will describe the theoretical considerations underlying the strategy and current experimental efforts based on ferromagnetic shape-memory alloys and ferroelectric perovskites.

2:00 PM *D2.2
Elastic and Magnetic Properties of Ni2MnGa and Fe7Pd3 alloys. Jun Cui[1], Qingzhen Huang[2] and Manfred Wuttig[1]; [1]Material Science and Engineering, University of Maryland, College Park, Maryland; [2]NIST Center for Neutron Research (856), National Institute of Standards and Technology, Caithersburg, Maryland.

NiMnGa and FePd are two prototypical ferromagnetic shape memory alloys that differ in details of the transformation mechanism and in the state of chemical order in the high temperature state. This talk will be divided into two parts, and address these two alloys respectively. The first part of the talk will focus on the elastic properties of NiMnGa and show that, in addition to the well known softening of (C11-C12)/2, the elastic constant C11 also softens as the Curie temperature is traversed. The elasticity of polycrystalline NiMnGa films reflects the softening of (C11-C12)/2 in the austenitic state as well as magnetoelastic characteristic of the ferromagnetic phase. The second part of the talk will focus on the structural and magnetic transitions in Fe7Pd3 alloy. Evidence shows that after cooling from the Curie temperature (490C), there are three sudden changes of saturation magnetization near 300C, 10C, and -60C respectively. Structural transitions are responsible for these three changes, and are studied using high-resolution neutron powder diffractometer and Rietveld method. In addition, a study of effects of heat treatments on chemical order in Fe7Pd3 alloy will be presented. This study was aimed to improve the magnetocrystalline anisotropy which is essential for large field induced strain.

2:30 PM D2.3

Combinatorial Thin Film Synthesis of NiMnAl Ferromagnetic Shape Memory Alloys Using MBE Technique. Ralf Hassdorf[1], Juergen Feydt[1], Sigurd Thienhaus[1], Rene Borowski[1], Markus Boese[2], Thomas Walther[1] and Michael Moske[1]; [1]Research center caesar, Bonn, Germany; [2]Dept of Chemistry, Bonn University, Bonn, Germany.

Ferromagnetic shape memory alloys have attracted much attention as potential high-performance magnetically controlled actuator materials due to the fact that they show large field-induced strains accompanied by the rearrangement of twin variants in the martensitic state. So far, numerous candidate systems have been investigated in this respect including binary alloys like FePd or FePt as well as the Heusler-type alloys Ni_2MnGa, Ni_2MnAl, or very recently CoNiAl and CoNiGa. In bulk form, these materials come up with high magnetostriction values comparable or even superior to the value of the giant-magnetostrictive Terfenol-D. The real challenge by now is to fabricate thin films of these materials while overcoming metallurgical constraints and to provide them with the same order of functionality as in the bulk counterparts. In this context, we present a study demonstrating the advantage of combinatorial thin film synthesis by using molecular beam epitaxy. Here, NiMnAl alloy composition spreads were grown onto four-inch wafer substrates. Compositional variations ranging from 10 to 15 at.% relative to each constituent enable a direct comparison of the chemical-structural relationship with respect to martensitic transformation and to magnetic ordering as well as an efficient identification of the emerging phase stability regions. In our study, we set the primary focus on the structural aspects of the transformation behavior confirmed by X-ray microdiffraction in combination with a specially designed heating stage. Notably, high-resolution TEM imaging of the respective composition areas reveals patterns of a long periodic stacking order indicating crystallographic reorientation in the martensitic state. Stress relief upon transformation as observed by thermal processing in a stress apparatus using a capacitance bending beam technique ranges from 50 to 200 MPa depending on the composition. Vibrating-sample magnetometry so far suggests magnetic ordering to occur well below ambient temperature. The compositional and microstructural aspects of the phase stabilities will be discussed.

2:45 PM D2.4

New smart composites composed of ferromagnetic shape memory alloy particles and polyer matrix for sensors and actuators. Hideki Hosoda[1], Shinsuke Takeuchi[1], Tomonari Inamura[1], Kenji Wakashima[1] and Shuichi Miyazaki[2]; [1]Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan; [2]Institute of Materials Science, University of Tsukuba, Tsukuba, Ibaraki, Japan.

Since most ferromagnetic shape memory alloys (FSMAs) are brittle, the enhancement of ductility is strongly required. In order to improve workability and ductility of FSMAs, a new smart composite composed of FSMA particles (FSMAPs) and polyer is proposed. The FSMAPs are easily fabricated by mehcanical crush, and isotropic mechanical and shape memory characteristics are obtained while anisotropic properties appear for the composites containing shape memory alloy wires or plates. The isotropic properties are advantageous for shaping and forming. In this paper, the design concept, mechanical and shape memory properties of FSMAPs/polyer smart composites are described. Besides, the ability of the composites for sensors and actuators is pointed out.

3:30 PM *D2.5

Combinatorial investigation of ferromagnetic shape memory alloys. Ichiro Takeuchi, [1]Dept of Materials Science and Engineering, University of Maryland, College Park, Maryland; [2]Small Smart Systems Center, Department of Materials Science and Engineering and Center for Superconductivity Research, University of Maryland, College Park, Maryland.

We have developed a thin film composition spread technique for rapidly mapping the ternary phase diagrams of metallic alloy systems in search of ferromagnetic shape memory alloys. A UHV three-gun magnetron co-sputtering system is used to deposit composition spreads whose natural compositional distribution contains large fractions of ternary phase diagrams. The spreads are created on 3-inch Si wafers, and the typical thickness of the film ranges from 300 nm to 1 micron. Spread films are deposited at room temperature, and they are annealed at 500-600 °C in vacuum following the deposition. Wavelength dispersive spectroscopy is used to map the exact composition distribution of every wafer. Room-temperature scanning SQUID microscopy is used to obtain the magnetic field distribution of the spread patterned into 2 mm x 2 mm square grids. Obtained distribution is converted to quantitative remanent magnetization mapping using a numerical algorithm. This allows us to identify composition regions that are ferromagnetic at room temperature. In order to detect composition regions which are reversible martensites, we use wafers where arrays of cantilevers are micromachined prior to the spread deposition. By monitoring the thermally induced actuation of all the cantilevers on the spread wafer simultaneously by visual inspection, we can detect regions which undergo martensitic transitions. Scanning x-ray diffractomety of spread wafers is also used to identify regions which display structural phase transitions. By combining the information extracted from both types wafers, we can construct functional ternary phase diagrams. To date, we have looked at a variety of ternary systems. We have found new composition regions which are both ferromagnetic and reversible martensites in the Ga deficient region of the Ni-Mn-Ga system and the Al deficient region of the Ni-Mn-Al systems. A clear relationship between the martesitic transition temperature and the Curie temperature has been observed for a wide range of compositions. This work was performed in collaboration with O. O. Famodu, M. A. Aronova, K.-S. Chang, J. Hattrcik-Simpers, S. E. Lofland, F. C. Wellstood, L. Knauss, and M. Wuttig.

4:00 PM *D2.6

Design of Ferromagnetic Shape Memory Alloy Composites and Their Actuators. Minoru Taya, Mechanical Engineering, University of Washington, Seattle, Washington.

Recently, strong attentions are paid to ferromagnetic shape memory alloys (FSMAs) as a key actuator material, owing to its fast actuation speed, yet providing large force and stroke. FSMAs are driven by applied magnetic field and/or magnetic field gradient, which cause the deformation due to the martensitic phase transformation, and hence can provide very fast actuation speed with reasonably large strain and stress capability. Among various FSMAs, the polycrystalline FePd alloy is practically promissing to be applied as an actuator material, owing to the good mechanical properties, such as shape memory effects, superelasticity and high ductility, and so on. Since the uniform (constant) magnetic field alone is found to be disadvantageous as this would provide a modest force, we proposed so-called "hybrid mechanism" for actuator applications. The key step in the mechanism is the stress-induced martensite phase transformation produced by applied magnetic field gradient, thus enhancing the displacement, as the stiffness of Fe-Pd is reduced from stiff to soft during the austenite to martensite transformation. The price of Pd is very expensive, thus, we are searching for alternative FSMA, and come up with FSMA composites which are composed of ferromagnetic material and shape memory alloy (SMA). This talk will discuss the hybrid mechanism of FSMA and FSMA composites and present recent results of three-dimensional (#D) phase transformation diagram based on thermodynamics. This #D diagram will be used effectively by design engineers who want to design fast responsive FSMA based actuators. This talk introduces also several designs of FSMA and FSMA composite based actuators.

4:30 PM D2.7

Reversible Martensitic Transformation in A Ferromagnetic CoNiAl Alloy. H. Ersin Karaca[1], Ibrahim Karaman[1] and Hans J. Maier[2]; [1]Department of Mechanical Engineering, Texas A&M University, College Station, Texas; [2]Lehrstuhl fur Werkstoffkunde, University of Paderborn, Paderborn, Germany.

In recent years ferromagnetic shape memory alloys have attracted increasing interest because of the ability to obtain one order of magnitude higher recoverable magnetic field induced strain (MFIS) than the other active materials. The main requirements for large magnetic field induced strain are: low twin boundary energy, high strength of matrix, high magnetocrystalline anisotropy energy and saturation magnetization. A recently discovered ferromagnetic shape memory CoNiAl alloy has promising shape memory characteristics for conventional and magnetic shape memory applications. In this study we have demonstrated that these alloys have low pseudoelastic stress hysteresis, high strength for dislocation slip, large pseudoelastic and two way shape memory strain, large pseudoelastic temperature window, and low stress for martensite reorientation. These findings satisfy the thermomechanical requirements to obtain MFIS. Additionally, high melting point, low density, good corrosion and oxidation resistance may result in the replacement of conventional SMAs with CoNiAl in most applications. This work is supported by Army Research Office, Contract No. DAAD 19-02-1-0261.

4:45 PM D2.8

Nonlinear Stress-Based Control of a Rotary SMA-Actuated Manipulator. Mohammad Elahinia, Michael Seigler and Mehdi Ahmadian; Mechanical Engineering, Virginia Tech, Blacksburg, Virginia.

In this paper a nonlinear stress-based controller is designed to position a single-degree-of-freedom shape memory alloy (SMA) actuated manipulator. A three part model was constructed based on the dynamics/kinematics of the arm, the thermomechanical behavior of SMA's, and an assumed heat transfer model consisting of electrical heating and natural convection. Both sliding mode control and inverse dynamics control are used to calculate a desired stress, based on the position error. The desired stress is compared with the actual stress which is computed using an Extended Kalman Filter. The stress error is then used for control via a proportional-integral controller. Numerical simulations are performed to investigate tracking performance as well as other issues such as robustness. The results demonstrate that the variable structure controller designs are highly accurate in tracking both stationary and variable input signals.

SESSION D3: Electroactive Polymer
Chairs: Ji Su and Qiming Zhang
Tuesday Morning, December 2, 2003
Room 202 (Hynes)

8:30 AM *D3.1
Ferroelectrets: Polymer-foam space-charge electrets with exciting fundamental and applications-relevant properties. Reimund Gerhard-Multhaupt and Michael Wegener; Department of Physics, University of Potsdam, Potsdam, Brandenburg, Germany.

Recently, the term "ferroelectrets" has been suggested for the new class of highly insulating and suitably foamed polymers with internal space charge that have become a major focus of electret research [1]. The first step in their preparation is foaming and void optimization by means of suitable drawing processes, solvent evaporation, foaming agents, gas expansion, etc. Because of relatively large microvoids, the polymer foams are mechanically quite soft with rather low elastic moduli and acoustic impedances. After charging or poling, they contain stable charge layers on the internal void surfaces. These charge layers form a "giant dipole" whose dipole moment is easily varied by mechanical or electrical stresses. The resulting electrical or mechanical response of the foamed and charged polymer represents a direct or inverse quasi-piezoelectric effect, respectively. Because of the very high deformability of the polymer microvoids, the observed electromechanical effects can be very large, often dwarfing the corresponding values of polar piezoelectric polymers and sometimes exceeding the coefficients of commercial piezoelectric materials. In addition to these effects, the new materials exhibit some pyroelectricity which is often quite small and thus makes them rather insensitive to temperature changes. From a general perspective, the novel foamed and charged polymers are composites of gas bubbles in a polymer matrix. As in other composite materials, the properties of the material are critically dependent on the size and shape distribution of the voids and on the mechanical and electrical behavior of the interface, i.e. the internal void surfaces. The development of cellular polypropylene (PP) as an electret-transducer material since the mid-eighties is briefly described, and the most relevant experimental techniques for polymer-foam ferroelectrets are introduced. Porous fluoropolymer electrets and their investigation as single- or multiple-layer piezoelectrics are also considered in view of their better thermal and temporal stabilities. Typical results are discussed in view of the potential of polymer ferroelectrets for device applications which include flat microphones and loudspeakers, active noise-cancelling panels, flat and curved ultrasonic transducers without or with patterning of the active area, electromechanical sensors for vibration detection (e.g. machinery, musical devices, security systems) and for dynamic biomedical measurements, etc. [1] Reimund Gerhard-Multhaupt, "Less can be more - Holes in polymers lead to a new paradigm of piezoelectric materials for electret transducers," IEEE Trans. Diel. Electr. Insul., Vol. 9, No. 5, pp. 850-859 (October 2002). Several contributions by researchers from Austria, China, Finland, Germany and Poland in the Proceedings of the 11th International Symposium on Electrets, IEEE Catalog No. 02CH37383, IEEE Service Center, Piscataway, NJ 2002.

9:00 AM *D3.2
Dielectric Elastomer Artificial Muscle Devices for Actuation, Sensing, Generation and Smart Structures. Roy Kornbluh, Ron Pelrine, Qibing Pei, Marcus Rosenthal, Harsha Prahlad, Scott Stanford, Neville Bonwit, Richard Heydt, John Heim and Seajin Oh; SRI International, Menlo Park, California.

Electroactive polymers (EAPs) offer the potential to overcome limitations of traditional smart material and transducer technologies. A promising class of EAP is dielectric elastomers. Dielectric elastomer transducers are rubbery insulating polymer materials with compliant electrodes that have a large electromechanical response to an applied electric field. The strains and elastic energy of dielectric elastomer actuators are larger than those observed in any other field-activated materials. Because of their unique characteristics, dielectric elastomer transducers are under development for a wide-range of devices including multifunctional muscle-like actuators for biomimetic robots, microactuators for MEMS, conformal loudspeakers, haptic displays, shoe-mounted generators for harvesting the energy of walking, replacements for electromagnetic and pneumatic actuators for industrial applications, and shape control of space structures. Dielectric elastomers have shown promise in each of these applications. The success or failure in a given application will depend on the achievable performance and lifetime as well as the electrical driving requirements.

9:30 AM D3.3
Nematic Elastomer Actuators. Banahalli R Ratna[1], Amritha Srinivasan[2], Young Jin Kim[2] and Jawad Naciri[1]; [1]Center for Bio/Molecular Science and Engineering, Naval Research Laboratory, Washington, District of Columbia; [2]Geo-Centers Inc, Washington, District of Columbia.

Anisotropic freestanding films and fibers of nematic elastomers from laterally attached side-chain polymers show promise as linear actuators with large strain and retractive force. The orientational order of the liquid crystal side groups imposes a conformational anisotropy in the polymer backbone. When a large change in the order parameter occurs, as at the nematic-isotropic phase transition, there is a concomitant loss of order in the backbone which results in a contraction of the film/fiber in the direction of the director orientation. In this presentation we show that induced and strain blocked stress of these elastomers can be easily tuned by varying the nature and composition of the crosslinking material. The effect of multidentate crosslinkers on the alignment and hence the mechanical properties of the film will be discussed. We will also present results on the actuation induced by a reorientation of the liquid crystal mesogens by an applied field.

9:45 AM D3.4
A Multiscale Computational Model of Soft Elasticity and Director Reorientation in Nematic Gels. Antonio DeSimone, SISSA, International School for Advanced Studies, Trieste, Italy.

Nematic gels combine large deformations and entropic elasticity of elastomers with tunable optical properties peculiar to nematic liquid crystals. In addition, they exhibit soft elasticity (in analogy with the superelasticity of shape memory alloys) as a consequence of a symmetry-breaking phase transformation from an isotropic phase (in which the nematic mesogens are randomly oriented) to a spontaneously deformed uniaxial phase (in which the nematic mesogens are aligned). The phase transformation can be triggered by several mechanisms (temeprature changes, irradiation by UV light) and the material has been proposed as a suitable candidate for applications such as artificial muscles. The mechanical response of nematic gels is highly nonlinear. Modelling superelasticity requires the resolution of complex domain patterns which arise in connection with loading-induced phase changes. This is most effectively done through a coarse-grained hyperelastic model, in which fine-scale spatial oscillations are carefully accounted for in the definition of an effective energy density, and then averaged out from the kinematics. Algorithmically, this amounts to taking a suitable convex envelope of the microscopic free-energy density of the system. Finite element simulations based on the effective energy density are dramatically more effective than those based on the macroscopic free-energy. We will report on numerical simulations of stretching experiments on thin sheets of nematic elastomers. The use of the coarse-grained model enables us to simultaneously resolve the macroscopic mechanical response (i.e., deformed shape, stress-strain curves) and the underlying microscopic mechanisms (evolution of domain structures, local reorientation of the nematic director). Comparison of numerical results with measured stress-strain diagrams and with X-ray scattering experiments confirms the robustness of our approach.

10:30 AM *D3.5
Molecular Engineering and Morphological Control of Electrostrictive Graft Elastomers. Ji Su, Advanced Materials and Processing, NASA Langley Research Center, Hampton, Virginia.

Electrostrictive graft elastomers developed at NASA Langley Research Center demonstrate promising electromechanical properties for lightweight actuation devices. The desired electromechanical properties of the elastomers can be tailored and optimized by molecular engineering (chemically) and morphological control (physically). This presentation will review recent results on 1) the effects of chemical composition and molecular morphology on electromechanical properties; 2) the effects of process parameters on the material morphology; and 3) a computational study on mechanisms of electrostriction in the material. In addition to the material development, progress in design and fabrication of electroactive polymer (EAP)-based electromechanical devices will also be presented. These include fabrication of polymer-based micro-electro-mechanical systems (MEMS), hybrid electromechanical actuators, and multi-layered EAP actuators as well as other technical issues related to the development of EAP-based devices such as electrodes and configuration design.

11:00 AM D3.6
High-Dielectric-Constant All-Organic/Polymeric Composite Actuator Materials. Cheng Huang[1], Ji Su[2] and Qiming Zhang[1]; [1]Materials Research Institute and Electrical Engineering Department, The Pennsylvania State University, University Park, Pennsylvania; [2]Advanced Materials and Processing Branch, NASA Langley Research Center, Hampton, Virginia.

Among various electroactive polymer (EAP) actuator materials developed recently, the class of EAPs whose responses are stimulated by external electrical fields (often known as the field type EAPs) is especially attractive due to their high strain level and elastic energy density. However, for most field type EAPs, dielectric constant is low, generally less than 10. Consequently, these polymers usually require high electric fields (>100 MV/m) to generate high elastic energy density which limits their applications. In this talk, we will investigate some avenues to significantly raise the dielectric constant and electromechanical response in field type polymeric materials. By exploiting an all-organic composite approach in which high-dielectric-constant organic particulates were blended with a polymer matrix, a polymeric-like material can reach a dielectric constant higher than 400, which results in a significant reduction of the applied field to generate high strain with high elastic energy density. An all-polymer high-dielectric (dielectric constant K>1,000 at 1 kHz) percolative composite material was fabricated by the combination of conductive polyaniline particles ($K>10^5$) within a fluoroterpolymer matrix (K>50). These high-K polymer hybrid materials also exhibit high electromechanical responses under low applied fields. In addition, a three-component all-organic composite was designed and prepared to improve the dielectric constant and the electromechanical response, as well as the stability of the composites, in which a high-dielectric-constant organic dielectric phase and an organic conductive phase were embedded into the soft dielectric elastomer matrix.

11:15 AM D3.7
Simulations of High-Strain Electrostrictive Chlorinated Terpolymers. George J. Kavarnos[1] and Thomas Ramotowski[2]; [1]Department of Chemistry, University of Rhode Island, Kingston, Rhode Island; [2]Naval Undersea Warfare Center, Newport, Rhode Island.

Chlorinated poly(vinylidene fluoride/trifluoroethylene) terpolymers are remarkable examples of high strain electrostrictive materials. These polymers are synthesized by copolymerizing vinylidene fluoride and trifluoroethylene with small levels of a third chlorinated monomer. The electromechanical responses of these materials are believed to originate from the chlorine atom, which, by its presence in the polymer chains and by virtue of its large van der Waals radius, destroys the long-range crystalline polar macro-domains and transforms the polymer from a normal to a high-strain relaxor ferroelectric. To exploit the strain properties of the terpolymer, it is desirable to understand the structural implications resulting from the presence of the chlorinated monomer. To this end, computations have been performed on model superlattices of terpolymers using quantum-mechanical based force fields. The focus has been on determining the energetics and kinetics of crystallization of the various polymorphs that have been identified by x-ray diffraction and fourier transform infrared spectroscopy. The chlorinated monomer is shown to act as a defect that can be incorporated into the lamellar structures of annealed terpolymer without a high cost in energy. The degree of incorporation of the chlorinated monomer into the crystal lattice is controlled by annealing conditions and ultimately determines the ferroelectric behavior of the terpolymers. (This work was supported by the Office of Naval Research).

11:30 AM D3.8
Synthesis and Characterization of Cross-Linked Electroactive Polymer Actuator. Nathan Vandesteeg[1], Timothy Swager[2] and Ian Hunter[3]; [1]Materials Science and Engineering, MIT, Cambridge, Massachusetts; [2]Chemistry, MIT, Cambridge, Massachusetts; [3]Mechanical Engineering, MIT, Cambridge, Massachusetts.

A novel cross-linked conducting polymer has been synthesized for use as an electroactive actuator. The polymer combines a conjugated backbone of phenylene and thiophene rings with a polyurethane-based elastomeric linker. Conducting polymers of a nature similar to the backbone, such as polypyrrole and polyaniline, have been shown to actuate via intercalation of ions into and out of the polymer upon electrochemical stimulation. For this material, ionic motion is enhanced by the cross-links, which are joined to the backbone with several segments of ethylene oxide, a known ionic conductor. In addition, the cross-links provide mechanical stability and enable synthesis of freestanding films for active and passive testing. This paper will discuss the synthesis of the novel monomer precursor to the cross-linked conducting polymer. It will then focus on polymerization and cross-linking techniques, followed by active and passive characterization of the material.

11:45 AM D3.9
Large Strain Polypyrrole Actuators. Patrick A. Anquetil[1], John Madden[2], Derek Rinderknecht[1], Nathan Vandesteeg[1] and Ian Hunter[1]; [1]Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; [2]Electrical and Computer Engineering, The University of British Columbia, Vancouver, British Columbia, Canada.

A typical limitation of polypyrrole based conducting polymer actuators is the low achievable active strains (2% recoverable, 7% max) that they exhibit when activated in a common propylene carbonate / tetraethylammonium hexafluorophosphate electrolyte. Mamlian skeletal muscle, on the other hand, exhibits large recoverable strains on the order of 20%. Such large strains are desirable for applications in life-like robotics, artificial prostheses or medical devices. We report herein the measurement of recoverable strains in excess of 16% (21% max) for polypyrrole activated in the 1-butyl-3-methyl imidazolium tetrafluoroborate liquid salt electrolyte. This breakthrough in conducting polymer actuator technology will impact many engineering fields, where a lightweight, large displacement actuator is needed. Benefits and trade offs of utilizing ionic liquid electrolytes for higher performance polypyrrole actuation are discussed.

SESSION D4: Ferroelectric, Piezoelectric, and Dielectric Materials and Devices
Chairs: Nava Setter and Satoshi Wada
Tuesday Afternoon, December 2, 2003
Room 202 (Hynes)

1:30 PM D4.1
Phase Transition-piezoelectric Anisotropy Relations in Perovskite Ferroelectrics: BaTiO3 and PbTiO3. Marko Budimir, Dragan Damjanovic and Nava Setter; Ceramics Laboratory, EPFL, Lausanne, Switzerland.

Enhanced piezoelectric response along a nonpolar direction in complex relaxor ferroelectrics, such as Pb(Zn1/3,Nb2/3)O3-PbTiO3 and Pb(Mg1/3,Nb2/3)O3-PbTiO3, has been in the center of the ferroelectric research in the past several years. This phenomenon is not restricted to relaxor ferroelectrics solid solutions and has been reported in simple perovskites, for example in KNbO3 and BaTiO3. Although the piezoelectric effect is by far largest in relaxor ferroelectrics, the simple compositions can serve as convenient model materials. They exhibit the same sequence of crystal phases (cubic-tetragonal-orthorhombic/monoclinic-rhombohedral) as the complex solid solutions, the crystal phases are well defined, and they do not exhibit mesoscopic structure often present in complex materials. In this paper, the anisotropy of the longitudinal piezoelectric coefficient d33 is investigated as a function of temperature in BaTiO3 and PbTiO3 using Landau-Ginsburg-Devonshire approach. We show that presence of phase transitions in BaTiO3 leads to large d33 along nonpolar directions. The reason for this is that in the vicinity of a phase transition in which polarization vector changes its direction (tetragonal-orthorhombic/monoclinic, orthorhombic/monoclinic-rhombohedral), the shear piezoelectric coefficient(s) becomes very high resulting in an enhanced d33 along a nonpolar axis. The effect is particularly pronounced in the orthorhombic/monoclinic phase where piezoelectric anisotropy is influenced by the presence of both the high temperature tetragonal and the low temperature rhombohedral phase. In PbTiO3, which exhibits only tetragonal ferroelectric phase, the shear piezoelectric effect is weak and d33 has its maximum along the polar axis at all temperatures. The obtained results are general and can be extended to other ferroelectric compositions, including complex relaxor ferroelectrics, where a phase transition can be induced by temperature, composition (morphotropic phase boundary) or electric field.

1:45 PM D4.2
Modified $BiFeO_3$-$PbTiO_3$ Morphotropic Phase Boundary Piezoelectric Ceramics for Smart Systems. Jinrong Cheng[1,2] and L.Eric Cross[1]; [1]Materials Research Institute, Penn State University, State College, Pennsylvania; [2]School of Materials Science and Engineering, Shanghai University, Shanghai, China.

$BiFeO_3$-$PbTiO_3$ (BF-PT) crystalline solutions have been modified by La^{3+}, Ga^{3+} and Ba^{2+} substituent. We have found that the modified

BF-PT was a family of morphotropic phase boundary (MPB) piezoelectric solid solution with excellent piezoelectric properties. Two major problems of earlier BF-PT, high conductivity and switching field, have been solved in our modified systems. La increased the insulation resistivity up to 10^{13} Ω.cm at room temperatures and made BF-PT poled into a completely piezoelectric state. Ga provided more polarizability with La substituent. With increasing Ba content, the ferroelectrics (FE) to relaxor ferroelectrics (RFE) transformation was observed for the La and Ga- co-modified BF-PT. Therefore, it is flexible to tailor the performance of BF-PT by different concentrations of the substituent. In the vicinity of MPB, the Curie temperature (T_c) of above 400^{o}C can be achieved in La-10 at% modified BF-PT, whereas a piezoelectric d_{33} constant of 295 pC/N was measured for the one with La-20 at%. The modified BF-PT showed comparable performances to conventional $Pb(Zr,Ti)O_3$ (PZT) ceramics, but in significantly lead reduced forms. In addition, the dielectric strength of above 100 kV/cm and high coercive field made the modified BF-PT a potential application for high power electromechanical components.

2:00 PM *D4.3
Effect of piezoelectric vibration on electrical properties of YSZ film prepared by MOCVD. Hiroshi Masumoto and Takashi Goto; Institute for Materials Research, Tohoku University, Sendai, Japan.

Mechanical vibration produced by a piezoelectric actuator was applied to an ionic conductor film to increase the electrical conductivity at low temperature. Yttria-stabilized zirconia (YSZ) ionic conductor films were prepared on MgO substrates by metalorganic chemical vapor deposition (MOCVD) using $Zr(dpm)_4$ and $Y(dpm)_3$ as sauce materials. Cubic-YSZ films containing 8mol%Y_2O_3 with a significant <100> preferred orientation were obtained at 973 K. The films in 1.4 μm thick consisted of fine grains of about 400 nm in size and had a columnar structure. The YSZ films showed ionic conduction because the complex impedance spectra had clear spikes at a low frequency region. The activation energy of electrical conductivity was about 0.99 eV that was almost in agreement to that of YSZ films prepared by other methods. The YSZ film was placed on a multilayer piezoelectric actuator composed of seven sheets of PZT (lead zirconate titanate) films. The effect of piezoelectric vibration on electric properties of the YSZ film was investigated. The resistivity of the YSZ film decreased with increasing the amplitude of piezoelectric vibration. The electrical conductivity of the YSZ film under the vibration at 115 kHz and 40 V was about 2×10^{-6} $\Omega^{-1}m^{-1}$ at 90 °C. This value was more than 10 times greater than that of YSZ film without vibration. The cyclic expansion of YSZ lattice might accelerate the jumping of oxide ions between vacancy cites.

2:30 PM D4.4
ZnO Based SAW Delay Line Sensor: Fabrication and Characteristics. Farid Hassani[1], Shahrokh Ahmadi[1], Can Korman[1], Mona Zaghloul[1], Shiva Hullavarad[2], R.D. Vispute[2] and T. Venkatesan[2]; [1]Electrical and Computer Engineering, The George Washignton University, Washington, DC, District of Columbia; [2]Center for Superconductivity, Dept. of Physics, University of Maryland at College Park, College Park, Maryland.

ZnO, a well-known piezoelectric material, is used to develop micro-scale Surface Acoustic Wave (SAW) delay line sensor. In this work, SAW delay line sensors are fabricated employing ZnO films that are deposited by Pulse Laser Deposition (PLD) and RF sputtering techniques. Films are characterized prior to device fabrication by X-ray diffraction (XRD) for film crystalline quality, UV-visible transmission spectroscopy for optical characteristics, and Atomic Force Microscopy (AFM) for surface morphology. Interdigitated SAW electrodes producing surface acoustic waves in the hundreds of MHz are developed by photolithography and metalization techniques. SAW delay line sensor device testing, measurement and characteristics on both PLD and RF sputtered ZnO films are presented and compared.

2:45 PM D4.5
Fabrication of lead zirconate titanate thick film disks for micro transducer devices. Takashi Iijima, Sachiko Ito and Hirofumi Matsuda; Smart Structure Research Center, AIST, Tsukuba, Japan.

A combination of the preparation techniques for the piezoelectric lead zirconate titanate (PZT) films and the micro machining of Si is considered to be an effective way to fabricate microelectromechanical systems (MEMS), such as piezoelectric micro-transducer devices for the electrical and medical fields. To achieve these devices 5- to 100-μm-thick PZT films are required. Thin film preparation processes using chemical solutions, like sol-gel, have the advantage of a low firing temperature and dense microstructure despite the low film deposition rate compared with the screen printing process. Therefore, a chemical solution deposition (CSD) process is considered to be an attractive way to ensure compatibility with the Si micro-machining process and to fabricate MEMS devices. In this study, disk shape PZT thick film devices were successfully fabricated. Crack free 5-to10-μm-thick $Pb_{1.1}(Zr_{0.53}Ti_{0.47})O_3$ films deposited onto $Pt/Ti/SiO_2/Si$ substrate were prepared using a CSD process. A 0.5 M PZT precursor solution was prepared from trihydrated lead acetate, titanium i-propoxide, zirconium n-propoxide, and 2-methoxyethanol as the solvent. The process of spin coating and pyrolysis at 500°C was repeated five times, and then the precursor films were fired at 700°C for 5 min. This sequence was repeated 30 times. Pt top electrode and PZT layer were etched by reactive ion etching (RIE) process with Ar/CHF_3 mixture gas, and 100 to 1000-μm-diameter PZT micro disks were fabricated. The relative dielectric constant and dissipation factor were $\epsilon r=$ 1130 and $tan\delta=$ 0.02, respectively. The ferroelectric properties and piezoelectric displacement properties of the fabricated disks were measured dynamically with atomic force microscope (AFM). The 100-μm-diameter PZT micro disks showed well saturated $P-E$ hysteresis curves and butterfly shape displacement curves with a bipolar applied field of 5Hz. The remnant polarization and coercive field were $Pr=$ 14 $\mu C/cm^2$ and $Ec=$ 25 kV/cm, respectively. The piezoelectric constant calculated from unipolar driven displacement with AFM was about 220 pm/V. This means that the ferroelectric and piezoelectric properties of the micro disks were comparable with that of the bulk PZT ceramics. Investigation of the resonance frequency for the PZT micro disk are undertaken now.

3:30 PM D4.6
Exploration of magnetoelectric thin films using superlattice composition spreads. Kao-Shuo Chang, Materials science & engineering, U. of Maryland, College Park, Maryland.

Magnetoelectric materials are attractive due to the co-existence of charge polarization and magnetization. The magnetoelectric effect can be used to induce change in magnetization by applying electric field and induce electric polarization from applied magnetic field. One way to obtain magnetoelectric materials is by combining piezoelectric and piezomagnetic materials. Such materials have previously been pursued in bulk composites. We are exploring fabrication of artificial magnetoelectric thin films by creating superlattice structures where piezoelectric materials and piezomagnetic materials are modulated in periods of multiple unitcells. In particular, we fabricate superlattice composition spreads where one end of the spreads is a pure piezoelectric material and the other end is a pure piezomagnetic material. This technique allows us to 1) study coupling of the two properties at nanometer level and 2) systematically investigate mixing and changing of the two physical properties as a function of average composition which continuously varies across the spread. In order to fabricate a superlattice spread, a series of alternating gradient thickness deposition controlled at atomic layer level is performed for two targets with end compositions using combinatorial pulsed laser deposition. We have thus far fabricated BaTiO3 - CoFe2O4 and PbTiO3 - CoFe2O4 spreads. The lattice parameters of these materials are such that they can be grown together in a pseudo hetero-epitaxial manner. Microwave microscopy and scanning SQUID microscopy are used to determine the respective ferroic property across the spreads. We found that the composition region in the middle of the spread can often exhibit reasonable dielectric constants and magnetization simultaneously. Thus, this technique allows us to select the composition region and the layering structure where magnetoelectric effects can be potentially observed in individual samples.

3:45 PM D4.7
Precursor Suspension Processing of 65PMN-35PT Ceramics and Thick Films. Wei-Heng Shih, Hongyu Lou and Wan Y Shih; Materials Science & Engineering, Drexel University, Philadelphia, Pennsylvania.

We report a novel precursor suspension method where 65PMN-35PT ceramics and thick films can be processed at lower temperatures. The precursor suspension is made of PMN powders, Ti-isopropoxide, and Pb-acetate in an ethylene glycol solution. The sintering temperature for the ceramics occurred at 1000°C and good dielectric and piezoelectric properties were obtained. The formation temperature for the perovskite phase in thick films occurred at 800°C. However, the sintering temperature remains higher than 800°C and the perovskite phase cannot be sustained at such temperatures. The PMN powders used in the PMN-PT thick film were produced using $Mg(OH)_2$-coated Nb_2O_5 powders. The size of the core Nb_2O_5 particles was found to affect the coating behavior of $Mg(OH)_2$ and the sintering behavior of PMN-PT. To improve the sintering of PMN-PT thick films, studies involving the control of the solids loading and the dispersion of the particle suspensions will be presented.

4:00 PM D4.8
Synthesis and Characterization of Lead Strontium Titanate Thin Films by a Sol-Gel Method. Wei-Heng Shih, Y S Chiu, Scott McNamara, Hongyu Lou and Wan Y Shih; Materials Science & Engineering, Drexel University, Philadelphia, Pennsylvania.

It was shown that the addition of strontium in the precursor of lead titanate in ethylene glycol improves the rheological properties of the precursor solution and helps the sol-gel deposition of thin films. Perovskite-phase lead strontium titanate (PST) powders and PST films on Ti substrate were obtained after heat treatment at 450°C. Sr doping decreases the tetragonality of PT powders and thin films. Dielectric properties of PST are better than that of lead titanate (PT). Deposition of PST films on Si substrate was also studied. Heat treatment of each layer at 450°C followed by a final 650°C annealing resulted in perovskite PST films with good dielectric and ferroelectric properties. Results of PST thin films of different thickness as well as films with different strontium concentrations will be presented.

4:15 PM D4.9
PZT Thin Film on Silicon Wafer by a Sol-Gel Method for Sensor Applications. Wei-Heng Shih, Zuyan Shen and Wan Y Shih; Materials Science & Engineering, Drexel University, Philadelphia, Pennsylvania.

Piezoelectric unimorph cantilevers have been shown to be excellent sensors for liquid viscosity and density determination, mass detection, in-situ detection of cells, proteins, and specific antigen-antibody binding. In order to fabricate miniaturized microcantilever sensor, we investigate the synthesis of PZT thin films on silicon wafers. Lead acetate, titanium isopropoxide, and zirconium propoxide precursors with ethylene glycol as solvent were used to deposit PZT thin films in a clean room condition. Good ferroelectric PZT thin films were obtained with a dielectric constant of 800 and the saturated polarization of 94 $\mu C/cm^2$. By adjusting the precursor concentration, spinning speed and time, micron-thick films can be obtained without excessive repeated depositions.

4:30 PM D4.10
Synthesis and characterization of Ca_{1-X} $Sr_xCu_3Ti_4O_{12}$ thin films for dielectric applications. R. Guzman, Maharaj Singh Tomar and R.E. Melgarejo; Department of Physics, University of Puerto Rico, Mayaguez, USA, Puerto Rico.

There is a great deal of interest in $CaCu_3Ti_4O_{12}$ system for dielectric properties in microwave applications. In the present work we have studied Ca_{1-x} $Sr_xCu_3Ti_4O_{12}$ system for different compositions i.e. for x = 0.0, 0.05, 0.1, 0.3, 0.4, 0.5, 0.7, 0.9, 1.0. The material is synthesized by chemical solution route and thin films were deposited by spin coating. Both powder and thin films were investigated by X-rays, Raman spectroscopy and dielectric spectroscopy. The result indicates stochiometric films for all compositions and high dielectric values have been achieved.

4:45 PM D4.11
Nanoscale investigation of polar structure of (1-x)$PbMg_{1/3}Nb_{2/3}O_3$-x$PbTiO_3$ single crystals. Vladimir Shvartsman[1], Maciej Wojtas[2], Sergey Vakhrushev[3] and Andrei Kholkine[1]; [1]Ceramic and Glass Engineering, University of Aveiro, Aveiro, Portugal; [2]Faculty of Chemistry, University of Wroclaw, Wroclaw, Poland; [3]A. F. Ioffe Physico-Technical Institute, St. Petersburg, Russian Federation.

Relaxor ferroelectrics based on $PbMg_{1/3}Nb_{2/3}O_3$ (PMN) attract significant interest due to their excellent dielectric and piezoelectric properties. The peculiar properties of relaxors are attributed to the formation of nano-sized polar regions with a short-range order, while macroscopic ferroelectric order does not develop in the absence of sufficient external electric field. In solid solution between relaxors and "classical" ferroelectrics the gradual transformation from relaxor to typical ferroelectric behavior is observed. Even though these compositions were intensively investigated in the past, little attention has been paid to the studies of domain structure and its evolution under variation of external conditions. The common optical methods have limited resolution and are appropriate only for the observation of "normal" domains having the sizes of the order of microns. Recently, the Piezoresponse Force Microscopy (PFM) has been successfully applied for the investigation of ferroelectric materials at the nanoscale. The advantages of the PFM are extremely high spatial resolution (down to few nanometers) and high sensitivity to local polarization, which make this method well suitable to study the relaxors. We report here our results of the investigation of local piezoelectric properties of (1-x)$PbMg_{1/3}Nb_{2/3}O_3$-x$PbTiO_3$ (PMN-PT) single crystal by PFM. Piezoelectric contrast has been observed in 0.9PMN-0.1PT single crystals indicating spatial distribution of polarization and related to the existence of polar clusters. The observed domain structure is found to drastically change with increasing Ti content. In particular, in the composition with x=0.2 the domain structure already combines both relaxor and ferroelectric features. Finally, in 0.65PMN-0.35 PT only micron-size ferroelectric domains have been observed. The evolution of the polar structures under temperature and external electric field was also investigated. The nature of the observed phenomena is discussed based on the current understanding of the relaxor state in ferroelectrics and possible influence of PFM instrumentation.

SESSION D5: Poster Session: Electroactive Polymer 2
Chair: Cheng Huang
Tuesday Evening, December 2, 2003
8:00 PM
Exhibition Hall D (Hynes)

D5.1
Abstract Withdrawn

D5.2
High-Dielectric-Constant Fluoroterpolymer-Based Electrostrictive Polar Elastomers. Cheng Huang, Sabrina Chowdhury and Qiming Zhang; Materials Research Institute and Electrical Engineering Department, The Pennsylvania State University, University Park, Pennsylvania.

Field-type electroactive polymers (EAPs) are promising for high-performance actuators and artificial muscles due to their high electromechanical conversion efficiency. There are two main classes of field-type EAPs, one is P(VDF-TrFE) based relaxor ferroelectric electrostrictive polymers developed by Penn State, which has high modulus (~1GPa), but relatively low strain (~5%), and the other is soft dielectric elastomers based on Maxwell stress effect developed by SRI, which has high strain (~100%), but low modulus (~1MPa). When field-type EAPs are applied in biomedical field, higher performance such as high strain, modulus and elastic energy density is required, especially for the development of artificial heart. The key issue is that these polymers usually require high electric fields (>100 MV/m) to generate high elastic energy density which limits their applications. For Maxwell stress (electrostatic force) based electromechanical materials (MSEAP), both the field induced strain and electromechanical coupling factor are proportional to the ratio of the dielectric constant, K to modulus, Y of the electroactive materials. Hence, a high strain and high coupling factor under low electric fields require either a high dielectric constant or a high ratio of K/Y. By optimalizing the ratio of K/Y, higher strain can be realized under lower electric field. In this paper, with the combination of the electromechanical properties of hard electrostrictive ferroelectric polymers and soft dielectric elastomers, three methods will be utilized and demonstrated based on polymer molecular engineering: 1) Improved electromechanical performance from high-dielectric-constant fluoroterpolymer polar elastomers by electron irradiation or chemical cross linking; 2) Polymer blends or semi-interpenetrating polymer networks (sIPN) based on high-dielectric-constant fluoroterpolymers and soft dielectric elastomers; 3) Synthesis and characterization of hard ferroelectric/ soft dielectric block and graft copolymer elastomers: new dielectric-ferroelectric polymers with large induced strains under low electric field. In these block elastomers, high-dielectric-constant telechelic fluoroterpolymer hard segments constaining reactive terminal groups in opposite chain ends was first synthesized, which can be extended to the long chains by polycondensation with telechelic dielectric soft segments.

D5.3
Nanostructured Actuators: Conducting Polymers Integrated into Polynorbornene-Derived Scaffolds. Hyun A Kang and Timothy M. Swager; Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Conducting polymer-based electroactive polymer (EAP) actuators have found numerous applications. Well-defined nanostructures are expected to amplify the electrochemical response of EAPs, facilitating greater dimensional changes. Toward this end, efforts are directed to gain control over the microstructures of such materials. Three different norbornene derivatives having electroactive pendant groups were copolymerized with a 7-oxanorbornene derivative via ring-opening metathesis polymerization. In addition to rendering mechanical strength and flexibility, the poly(7-oxanorbornene) backbone is expected to facilitate ion transport across the bulk material. The cyclic voltammograms of the electropolymerized block copolymers displayed reversible redox waves over a broad range of potentials. Processing of EAPs via electrospinning facilitates fabrication of devices having enhanced switching speed of actuation. The morphologies of these block copolymers studied by various microscopic techniques will be also discussed.

D5.4
Structural and Electromechanical Study of Poly(vinylidene fluoride-trifluoroethylene) Defected by Chlorinated Termonomers. Rob Klein[1], James Runt[1,2] and Qiming Zhang[1]; [1]Materials Research Institute, Pennsylvania State University, University Park, Pennsylvania; [2]Materials Science and Engineering, Pennsylvania State University, University Park, Pennsylvania.

The PVDF-TrFE copolymer has found wide application in electromechanical devices, due to its ferroelectric properties. Recently, high-energy electron-irradiated PVDF-TrFE copolymer has been shown to produce large electrostrictive strain. Results indicate that the terpolymer made by adding chlorofluoroethylene (with varying arrangements of the fluorines) to the VDF-TrFE chain can improve upon the irradiated copolymer, with an increase in strain response and reliability and a decrease in processing cost. Through better understanding of the relation between termonomer choice, morphology, and crystallization effects and the electromechanical properties, the strain and energy density of PVDF-TrFE-based terpolymer can be further optimized. The conformation and morphology of random terpolymers formed primarily from poly(vinylidene fluoride-trifluoroethylene) with small amounts of chlorofluoroethylene or chlorotrifluoroethylene were studied by X-ray diffraction, infrared spectroscopy, differential scanning calorimetry, and polarization loops. Results indicate that quenching from a high crystallization temperature forms a more polar phase, whereas crystallizing at a lower crystallization temperature produces a more non-polar phase. Essentially, the termonomer disrupts the VDF-TrFE polarization coherence enough that the crystallized phase has conformations with varying degree of polarity, with preference dictated by the thermal history. The effect of the termonomer appears to be due to inclusion. The width of the (001) X-ray peak indicates very thin coherence regions, less than 25 angstroms, although the lamellar thickness is greater than 150 angstroms. If established by a properly chosen crystallization temperature, the crystalline regions can be polarized with applied electric field, or relaxed into a non-polar state heating above the Curie temperature, which is about 35 degC. The crystallization arrangement also determines the strain response and electromechanical coefficients, which are crucial for electromechanical energy conversion.

D5.5

Device Design and Mechanical Analysis of Conducting Polymer Actuators. Bryan D. Schmid[1], Peter Madden[1], John Madden[2] and Ian Hunter[1]; [1]Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; [2]Electrical and Computer Engineering, University of British Columbia, Vancouver, British Columbia, Canada.

Conducting polymers, specifically polypyrrole, have tremendous potential as actuators. Various device designs using polypyrrole as an actuator were theoretically investigated and built for potential advancements in current technology and practice. Two key limitations to using conducting polymers in practical devices include the need for actuation out of a liquid electrolyte solution and the small strains produced by the polymer. Development of a polypyrrole trimorph design and various encapsulation techniques were tested as potential solutions to previous strain and actuation environment limitations. The trimorph design is composed of two mechanoactive polypyrrole electrodes separated by a gel electrolyte and ion-permeable layer. Several encapsulation techniques tested expand the environmental conditions under which actuation is possible. The trimorph design and encapsulation techniques are proven, viable solutions for actuator design. Trimorph samples tested in this paper consisted of two ~25 μm thick polypyrrole sheets, 65 μm thick nylon mesh, and a BF_4 based electrolyte gel. Final trimorph sample dimensions were 145 mm by 30 mm with an average thickness of 280 μm. The trimorph strips demonstrated successful actuation out of a liquid electrolyte, produced forces of 0.2 N and strain curvatures up to 133 m^{-1}, while demonstrating a greater than 3 month shelf life.

D5.6

Analysis and Modeling of Electro-Mechanical Coupling In An Electro-Active Polymer-Based Actuator. Thomas A. Bowers[1], Neville Hogan[1], Patrick Anquetil[1], Rachel Zimet[2] and Ian Hunter[1]; [1]Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; [2]Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

A nonlinear constitutive model is formulated for electro-active polymers (EAP) to describe the energetic coupling between electrical and mechanical domains. The polymer is modeled as a multi-port energy storage element with inputs from the electrical and mechanical domain. Using energy conservation methods, the general relationships between stress, strain, voltage, and charge are determined. The solution to the axial loading boundary condition is developed fully and compared to a linear model published by Madden and a nonlinear electrochemical model published by Mazzoldi et al. Additionally, experimental results from a conducting polymer actuator composed of polypyrrole are used to validate the electro-mechanical coupling model. It is found that the correlation between the model and experimental data is very good for strains up to 4% and potentials up to 1 Volt; these are within the typical operating range of polypyrrole. The model is sufficiently simply to allow real-time control while also exceeding Madden's linear model in its ability to accurately predict polymer behavior in normal operating ranges.

D5.7

Synthesis and Characterization of Sulfated Poly-β-Hydroxyether Doped Polypyrrole Actuators. Rachel Zimet[1], Patrick Anquetil[2], Hsio-hua Yu[3], Timothy Swager[3] and Ian Hunter[2]; [1]Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; [2]Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; [3]Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Conducting, electro-active polymers are very promising materials due to their potential applications in biorobotics, especially muscle-like actuators. New molecules exhibiting molecular conformational changes are being developed, but films synthesized from these new molecules are often lacking in durability and mechanical robustness compared to Polypyrrole. A polyanionic dopant can be added to help improve the mechanical properties of the film. However, the new molecular actuator systems are not well understood and the effects of the polyanionic dopant on actuator properties unknown. For this reason, Polypyrrole (a well-studied electro-active polymer) was deposited in the presence of 0 to 2%wt sulphated poly(β-hydroxyether) (SPHE) to form a robust, electro-active polymer composite. The polymeric anion presented herein has a high molecular weight (>1000 g/mol) and a flexible polymer chain. It replaces the salt typically used to dope the conducting polymer during electropolymerization of the polymer film. The resulting film's electrical, passive mechanical and active mechanical properties were investigated, and it was determined that they varied by an order of magnitude depending on the concentration of SPHE present in the deposition solution. Results have shown that it is possible to tailor the electrical conductivity between 20 S/m and 3 S/m and elasic modulus between 0.04 GPa and 0.2 GPa through co-deposition of Polypyrrole and SPHE. In addition, the active strain can be varied by much as 90% for a given set of electrochemical conditions.

D5.8

High Strain P(VDF-TrFE) based Terpolymer for BioMEMs and Microfluidic Devices. Feng Xia[1], Rob Klein[1], Francois Bauer[2], Srinivas Tadigadapa[1] and Q. M. Zhang[1]; [1]The Penn State University, University Park, Pennsylvania; [2]Institut Franco-Allemand de Recherches de Saint-Louis, Saint-Louis, France.

BioMEMs and microfluidic devices have gained a lot of attention in recent years due to their emerging applications in biochemical analysis, medical diagnosis, chemical analysis and synthesis, drug discovery and drug delivery, biosensing and biomimetic systems. The materials requirements for bioMEMs are biocompatible, chemically modifiable, easy to fabricate, economic, compliable and smart. Among various materials, the electro polymeric materials can best meet these requirements. Recently, we developed a group of P(VDF-TrFE) based terpolymers which have very high strain level and high energy density. The longitudinal and transverse strain of these materials can reach about 7% and 4.5%, and the elastic energy density is around 1.1 J/cm3, which are very attractive for the development of polymeric pump, valve and other microfluidic components for all polymer-based bioMEMs and microfluidic integrated system. This paper reports on the recent efforts on developing these microfluidic components using the electroactive terpolymers for bioMEMs, including the multilayer fabrications, surface modification from hydrophobic to hydrophilic by using oxygen plasma etching, and polymer-based valve and pump.

D5.9

Effects of UV and Gamma Irradiation on the Ferroelectric Behavior of Poly(vinylidene Fluoride Trifluorethylene) Copolymers. Luiz Oliveira de Faria[1], Cezar Welter[2] and Roberto Luiz Moreira[2]; [1]Materials and Nuclear Fuel, Centro de Desenvolvimento da Tecnologia Nuclear, Belo Horizonte, Minas Gerais (MG), Brazil; [2]Physics, Universidade Federal de Minas Gerais, Belo Horizonte, Minas Gerais, Brazil.

P(VDF-TrFE) copolymers with 50% of trifluorethylene were irradiated with gamma and UV rays in order to investigate the effect of photodegradation in their ferroelectric properties. Dielectric and calorimetric investigations have confirmed that gamma irradiation provokes the appearance of relaxor ferroelectric features in the copolymer. These induced relaxor properties are thought to be linked to the loose of stability of the ferroelectric domains, provoked by interruption of the long-range dipolar interaction. FTIR spectrometry revealed the presence of NH molecules which may take part of gamma induced cross-linking bonds, probably affecting the ferroelectric domains stability. On the other hand, the UV irradiation does not induce ferroelectric relaxor features on the copolymer. However, it was found that the dielectric loss changes from an Arrhenius to a Vogel-Fulcher-like behavior during the ferroelectric to paraelectric phase transition, with a decrease in the activation energy when compared to the gamma irradiated one. These results could have direct impact in the electromechanical response of the copolymer.

D5.10

Development of Totally Synthetic Glucose-Responsive Polymer Gel for Use as a Novel Type of Insulin Delivery Device. Akira Matsumoto and Kazunori Kataoka; Graduate School of Engineering, The university of Tokyo, Tokyo, Japan.

Over the past several decades, stimuli-responsive polymer gels have attracted a great deal of research interest. A series of stimuli that includes heat, pH, electric fields and light have been demonstrated to induce abrupt changes in the physical properties of polymer gels. This knowledge led to development of various types of stimuli-responsive, hence "self-regulated" systems so-called "intelligent" materials. Among wide range of their applied fields, applications in the medical field such as "drug delivery systems" have been a constant research topic. We attempt to develop a totally synthetic, glucose-responsive polymer gel system, aiming for use in a self-regulated insulin delivery system to treat diabetes. As a synthetically available sensor moiety for the blood sugar level, we paid particular attention to the unique ability of phenylborate derivatives to form a reversible, covalent bonding with glucose. We have previously reported that a gel composed of N-isopropylacrylamide and 3-acrylamidophenylboronic acid exhibits a reversible volume change synchronized with a change in the glucose concentration, through which the sufficiently controlled and pulse-shaped release of insulin was achievable at 28 C in a pH 9.0. This work is intended to provide a synthetic methodology to optimize the operational conditions of the system to be physiological. The approach involves the use of a newly synthesized phenylborate derivative possessing an appreciably low pKa as glucose sensing moiety, as well as the adoption of a different type of main chain structure that exhibits critical solution behavior in the range close to physiological temperature. Evaluations of glucose dependent changes in the swelling degree of the resultant copolymer gels for varied pH and temperatures revealed a markedly improved sensitivity near the physiological conditions. Discussion will be presented in terms of the effect of the modulated copolymer gel structure on the glucose responsive behavior.

SESSION D6: Poster Session: Ferroelectric, Piezo, and Dielectric Materials and Devices
Chair: Feng Xia
Tuesday Evening, December 2, 2003
8:00 PM
Exhibition Hall D (Hynes)

D6.1

Study of structural ordering in $(0.3)Pb(In_{1/2}Nb_{1/2})O_3$:$(0.7)Pb(Mg_{1/3}Nb_{2/3})O_3$ ceramics by transmission electron microscopy. C. W. Tai and K. Z. Baba-kishi; Department of Applied Physics, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong.

Transmission electron microscopy (TEM) studies of the ceramics $(0.3)Pb(In_{1/2}Nb_{1/2})O_3$:$(0.7)Pb(Mg_{1/3}Nb_{2/3})O_3$, abbreviated 0.3PIN:0.7PMN have revealed long-range structural order (LRO), short-range order (SRO) and mixed-order (MO). The ceramics, synthesized using mixed-oxide route, have important properties exhibiting negligible slim P-E loop and non-hysteresis electromechanical behaviour at room temperature. TEM observations of the LRO domains and their antiphase boundaries imaged along <110> and <112> directions show non-uniform size distribution ranging from 20 to 150nm across. Domains in the proximity of grain boundaries within a grain are several times larger than the domains at the centers of the grains. High-resolution TEM images taken along [001] show clusters of lattice fringes with different contrast. Simulations indicate that the combinations of the three different cations, Mg^{2+}, In^{3+} and Nb^{5+}, create a state of mixed-ordering arrangement in the B-sites. The clusters are therefore chemically district regions. In addition to the 1:1 long-range order, these clusters form the mixed structural ordering resulting in nano-scale ordered domains. Mixed ordering is a highly random process and takes place by the combinations of the various cations present, thus creating a superstructure. Diffuse scattering and streaking were also observed in selected-area diffraction patterns along various directions particularly along high-order zone axes. The streaks form specific mosaic patterns, indicative of short-range ordering, which could partially originate from A-site disorder. In 0.3PIN:0.7PMN, three different combinations of the B-site cations, Mg-Nb, In-Nb and Mg-In, exist simultaneously. The electrical charges of the Mg^{2+}:Nb^{5+} and In^{3+}:Nb^{5+} pairs balance in the ratio 1:2 and 1:1, respectively. The charges of Mg^{2+}-In^{3+} pair cannot simply balance in a perovskite structure, although the overall electrical charge of the solid solution is neutral. Such localized order and disorder result in different sizes of domains with slight misorientations. They give rise to the continuous diffuse streaking between the fundamental Bragg reflections instead of a set of distinct reflections generated by long-range periodic structure. In addition to the LRO, which can also be found in certain other Pb-based complex perovskite materials,a new mixed state of structural orderings, SRO and cluster order in different scales were observed. This mixed ordering contributes to chemical inhomogeneity, which has implications on the property. Consequently, the behavior of the ferroelectric relaxor is enhanced, especially affecting the temperature dependence of the dielectric properties.

D6.2

Nanoscale ferroelectric properties of PZN-PT single crystals studied by scanning force microscopy. Igor Bdikin, Vladimir Shvartsman and Andrei Kholkin; Department of Ceramics and Glass Engineering, CICECO, University of Aveiro, Aveiro, Portugal.

The nature of the excellent electromechanical properties of ferroelectric single crystals of solid solutions $(1\text{-}x)(Zn_{1/3}Nb_{2/3})O_3\text{-}xPbTiO_3$ is intensively investigated during last several years. These crystals are expected to replace traditional piezoelectric materials such $Pb(Zr,Ti)O_3$ in actuators, transducers and smart systems. Recently, the Scanning Force Microscopy has been modified to study the piezoelectric properties at the nanoscale. This new technique, called Piezoresponse Force Microscopy (PFM), was applied for the visualization of domain structure by means of the piezoelectric effect and for the measurements of local piezoelectric coefficients by applying voltages and measuring the corresponding response. In this work, high-resolution PFM piezoelectric studies have been performed on $(1\text{-}x)Pb(Zn_{1/3}Nb_{2/3})O_3\text{-}xPbTiO_3$ (PZN-PT) single crystals of the composition close to the morphotropic (rhombohedral-tetragonal) phase boundary (x=0.045-0.08). Irregular domain patterns with the typical sizes 20-100 nm have been observed on the (001)-oriented surfaces of unpoled samples. On the contrary, (111)-oriented surfaces exhibited normal micron-size regular domains with the domain boundaries directed along allowed crystallographic planes. The existence of nanodomains in the (001)-oriented crystals was attributed to the relaxor nature of PZN-PT where small polarization clusters may form under zero-field-cooling (ZFC) conditions. The average size of these polarization clusters is $\sim$ 10 nm at room temperature as estimated earlier from neutron scattering.[1] It is hypothesized that the presence of the highly polarizable Ti^{4+} ions in PZN-PT promotes interaction between clusters, which thus can reorient and merge into the micron-size domains upon cooling. These nanodomains can be, in principle, considered as the nuclei of the opposite polarization state that ease the switching process for this particular crystal cut. Local piezoelectric hysteresis has been also performed by PFM on the nanometer scale. Similar switching behavior of (111)- and (001)-oriented PZN-PT crystals suggests that their superior piezoelectric properties are related to the domain wall motion and is not an intrinsic property of the material. Nanoscale data are complemented with conventional dielectric, ferroelectric and piezoelectric measurements. 1. D. La-Orauttapong, J. Toulouse, Z.-G. Ye, W. Chen, R. Erwin, and J. L. Roberts, Phys. Rev. B 67, 134110 (2003).

D6.3

Off-center Cu^{2+} Ions in Ferroelectric Lead Germanate. Michael P. Trubitsyn, Victor M. Duda and Yurii D. Krokhmal'; solid state physics dept., Dniepropetrovsk national university, Dniepropetrovsk, Ukraine.

Near ferroelectric phase transitions off-center impurities may play an especially important role. In the abstract we report the results of electron paramagnetic resonance (EPR) measurements, performed on lead germanate $Pb_5Ge_3O_{11}$ (PGO) crystals doped with copper ions. The anisotropy of the Cu^{2+} EPR spectra shows that paramagnetic ions occupy three structurally equivalent positions of C_1 symmetry conjugated by C_3 axis. The spectra have been described by spin-hamiltonian with nearly axial g and hyperfine A tensors: g_Z=2.410, g_X=2.066, g_Y=2.069; A_Z=10.88 mT, $|A_X| \approx |A_Y| \leq 0.5$ mT (T=293 K). The temperature evolution of Cu^{2+} spectra have been studied in the interval from 200 to 450 K. On heating the drastic line width broadening occurs and above 320 K three low symmetry (C_1) Cu^{2+} spectra transform into one broadened line, demonstrating C_3 local symmetry. The data obtained give evidence that Cu^{2+} substitute for Pb^{2+} host ions in positions of C_3 point symmetry and occupy three off-center positions shifted from trigonal lattice point in (001) plane. Thermally activated hopping of Cu^{2+} ions between off-center positions motionally averages the low symmetry triclinic spectra. Since the off-center localisation has to induce the electric dipole moments, to confirm this assumption we have investigated the dielectric response of PGO:Cu^{2+} and revealed the dielectric losses peaks typical for thermally activated relaxation processes. EPR and dielectric data testify for Arrenius like temperature behaviour of the Cu^{2+} hopping rate and permit to estimate its parameters ($\tau_0{}^{-1} \sim 10^{12}$

Hz, W=0.24 eV). The static and dynamic properties of Cu^{2+} centers have been attributed to pseudo Jahn-Teller effect. Influence of the orientable Cu^{2+} centers on the PGO properties near ferroelectric phase transition is discussed on the basis of semi-phenomenological approach.

D6.4

Temperature Dependence of Piezoelectricity in $Pb(B_{1/3}Nb_{2/3})O_3$ based single crystals (B=Zn, Mg).

Park jong sung[1], Park Hyu-min[2], Yi Jae-Yun[1], Lee Jung-Kun[3] and Hong Kug-Sun[1]; [1]School of Materials Science and Engineering, College of Engineering, Seoul National University, seoul, South Korea; [2]Korea Standards Research Institute, Daejeon, South Korea; [3]Materials Science and Technology Divison, Los Alamos National Laboratory, Los Alamos, NM87545, New Mexico.

Dependence of piezoelectricity on temperature in $Pb(B_{1/3}Nb_{2/3})O_3$ (B=Mg, Zn) based single crystals will be presented. Though ultra-high piezoelectric properties are observed in (001) oriented rhombohedral $Pb(B_{1/3}Nb_{2/3})O_3$ - $PbTiO_3$ (B=Mg, Zn) crystals (PBN - PT), there are a few limitations to prevent the application of piezoelectric relaxor based single crystals. Most of these problems are related with the temperature stability of piezoelectricity, which decreases the reliable operation of piezoelectric materials. In this study, the temperautre effect on piezoelectricity is evaluated precisely and the modified material to compensate these shortcomings is investigated. The temperature dependence of piezoelectric properties was found to be closely related with the phase transition behavior of <001> oriented crystals. With decreasing the stability of E-field engineered state, the piezoelectric properties of crystals deteriorated. Also, the addition of materials with high Tc to PBN based crystals lead to stabilize the engineered domain configuration and the piezoelectricity against the temperature.

D6.5

Some Properties of Incommensurably Disordered Rb_2ZnCl_4Crystals. Michael P. Trubitsyn, solid state physics dept., Dniepropetrovsk national university, Dniepropetrovsk, Ukraine.

Applying of the radiospectroscopic techniques has allowed to achieve the remarkable progress in studying of the incommensurably modulated phases. Just the local nature predetermines the successes of the magnetic resonance methods, since the spatial averaging of structural distortions in many respects causes the inefficiency of the macroscopic experimental methods. In particular, the valuable information on static and dynamic properties of incommensurate systems has been obtained via EPR investigations of Mn^{2+} probe in Rb_2ZnCl_4(RZC) crystals. It has been shown that Mn^{2+} centers, substituting for Zn^{2+} ions, are localised in chloride tetrahedral complexes whose rotations result in modulation of the structure. The EPR data give evidence that Mn^{2+} is a suitable probe, correctly reflecting the properties of the crystal bulk. In the temperature interval of paraelectric-incommensurate transition point T_i=304.4 K the EPR spectra of Mn^{2+} ions have been studied for various orientations of the external magnetic field. The singularity spectra, typical for modulated phases, have been observed on cooling below T_i. The computer simulations of the singular line shape permit to estimate the critical exponents of the order parameter β=0.35 and correlation length ν=0.64. It has been shown, that just below T_i the local line width of singular peaks is influenced by amplitude fluctuations of the modulation wave whereas the contribution of phase fluctuations dominates in the middle, flat part of the singular spectrum. The values of the critical exponents β and ν confirm the non-classical character of the RZC critical properties corresponding to the universality class of 3d XY Heizenberg systems.

D6.6

Structural and Dielectric Properties of CaCu3Ti4O12 Thin Films. Vinay Gupta[1], Rasmi R Das[2], Pijush Bhattacharya[2], Yuri I Yuzyuk[2], William Perez[2] and Ram S. Katiyar[2]; [1]Department of Physics, University of Delhi, Delhi, India; [2]Dept. of Physics, University of Puerto Rico, San Juan, Puerto Rico.

Recently, there is a constant surge for the enhancement of dielectric permitivity of oxide thin films for various functional devices. CaCu3Ti4O12 (CCT) was found to be an interesting material that exhibiting bulk dielectric constant over 104. In this study we have used pulsed-laser-deposition technique to grow CCT thin films on Pt/TiO2/SiO2/Si and LaAlO3 substrates. During the thin film deposition, the substrate temperature was varied in the range of 300-750oC with a constant oxygen pressure of 200 mTorr. As-grown films are post-annealed at 750oC in order to improve the crystalline quality of the films. X-ray diffraction showed polycrystalline nature of the films. A systematic variation of the grain size with substrate temperature was studied using atomic force microscopy. Micro-Raman spectroscopy was used to study the vibrational modes of the CCT thin films in comparison with the bulk ceramics. The dielectric properties of the films were studied in metal-insulator-metal configuration. Films grown at higher substrate temperature exhibited highest value of dielectric permittivity (~2200). The temperature dependent dielectric properties were carried out to understand the exact mechanism of the ionic conductivity and the origin of high dielectric constant in CCT thin films. Detailed results on structural and dielectric properties will be discussed in close correlation with the processing conditions

D6.7

Structural and dielectric properties of $Ca_{1-x}Mg_xCu_2Ti_4O_{12}$. Luis A. Bermudez and Maharaj Singh Tomar; Department of Physics, University of Puerto Rico, Mayaguez, USA, Puerto Rico.

$Ca_{1-x}Mg_xCu_2Ti_4O_{12}$ is seems to be a novel dielectric ceramic material for microwave applications due to its high dielectric constant value. This material has been synthesized by chemical route for different compositions and thin films have been deposited by spin coating. X-ray diffraction and Raman spectroscopy were used for detailed characterization of this material for both powder and thin films. X-ray diffraction shows highly stochiometric films for different compositions. Dielectric measurement indicates high value of dielectric constant, results will be presented.

D6.8

Piezoelectric Transformers for Space Applications.
Alfredo Vazquez Carazo, Face International Corp., Norfolk, Virginia.

Despite piezoelectric transformers (PTs) being invented in the late 50s, this technology did not reach commercial success until early the 90s. During this period, several companies, mainly in Japan, decided to introduce PT technology for applications requiring small size and low electromagnetic interference (EMI) signature. Since then, PTs have been used as step-up transformers for the CCFL inverter used for backlighting the liquid crystal display of notebook computers and personal digital assistants (PDA). In these applications, PTs provide i) high voltage gain ratios, ii) high power density - typically about 10W/cm3 -, iii) high output impedance, iv) high efficiency, and v) low EMI. Currently, PT applications for CCFL are limited to 5-8W. In the last decade, the interest in PTs has moved toward a second group of applications beyond CCFL backlighting use. Companies in U.S., Japan, and Europe are now investigating the use of PTs for power applications, including battery chargers, linear and compact fluorescent ballasts, DC/DC converter, power supplies and others. In these applications, compared to the CCFL, the requirements include i) step-down transformers, ii) high power transformers, iii) high efficiency power conversion, iv) low output impedance, v) input to output isolation and v) low content of EMI. New topologies of PTs have been proposed to address higher levels of power conversion than those available with the classical Rosen type PTs (typically use for 5-8W with power densities of about 5-10W/cm3). This is the case for the laminated piezoelectric transformers, Transoner®, developed, patented and commercialized by Face Electronics. In these transformers power densities of over 40W/cm3 have already been reported for step-down applications. A third group of applications has been recently proposed by Face Electronics and granted under two ongoing SBIR projects for use in the new generation of communication satellite systems as well as space research. Among others, these applications include i) high voltage power supplies for driving Traveling Wave Tubes amplifiers used for satellite communication, and ii) high voltage igniters for controlling the ignition process of Pulsed Plasma Thrusters used for positioning the new generation of small satellites. These applications are characterized by a combination of high power and high voltage requirements (making them doubly complex), the need for high reliability and, in some cases, the ability to withstand extreme environmental conditions. This paper introduces the ongoing research on space applications for PTs.

D6.9

Effects of Electrode Configurations on Internal Stress Distribution of Multilayer Actuators. Dong-Kyun Lee[1], Ji-Won Choi[1], Deuk-Young Han[2], Hyun-Jai Kim[1] and Seok-Jin Yoon[1]; [1]Thin Film Materials Research Center, Korea Institute of Science and Technology, Seoul, South Korea; [2]Department of Electrical Engineering, Konkuk University, Seoul, South Korea.

The internal stress distribution in multilayer actuator was analyzed by Finite Element Method (FEM) program. Around the edge of conventional inter-digital electrodes, the non-uniform electric field generated the stress concentration, which caused the ceramic to crack. To decrease the stress concentration, various internal electrode configurations were presented such as plate-through type, slit-insert type, and float-electrode type. The simulated results indicate that the float-electrode type can decrease the stress concentration of inter-digital type in approximately 1/3.

D6.10
PZT-Driven Micromagnetic Optical Devices. Hiroyuki Takagi, Masahiko Mizoguchi, Jaehyuk Park, Kazuhiro Nishimura, Hironaga Uchida and Mitsuteru Inoue; Toyohashi University of Technology, Toyohashi, Aichi, Japan.

A spatial light modulator (SLM) is real-time programmable device capable of modifying amplitude, phase and polarization of an optical wave front by an electrical control signal. So far, various types of reusable SLMs with two-dimensional pixel arrays have been intensively developed. A magneto-optic spatial light modulator (MOSLM) has the advantages of high switching speed, robustness, nonvolatility, and radioactive resistance. However, because the conventional MOSLM is driven by a large current, large power consumption is required. To improve the disadvantage, we developed two PZT-driven MOSLMs by different fabrication processes for a magnetic garnet film, which were driven by electric field and external magnetic field. In our first PZT-driven MOSLM, a PZT film was fabricated by using a sol-gel method [1]. Although a driving current of the PZT-driven MOSLM was less than 1/10 time as compared with that of the conventional MOSLM, a magneto-optic layer (Bi:YIG) was damaged by a high temperature process for crystallization of the PZT film. In second device, an aerosol deposition method (ADM), which produces a film by using accelerated material particles with impact energy [2], was applied to a PZT film on the YIG layer. Moreover, in order to give effective stress by the PZT film to the YIG layer, we simulated the structure of the PZT-driven MOSLM by using a finite element method. The developed MOSLM was driven by a voltage of 5 V between X and Y drivelines, and an external bias field of 68 Oe. As the result, the selected pixels in the PZT-driven MOSLM were independently switched. [1] J. H. Park, H. Takagi, J. Park, and J. K. Cho, K. Nishimura, H. Uchida and M. Inoue, J. Appl. Phys. 93, 8525-8527 (2003). [2] J. Akedo and M. Lebedev, J. Crystal Growth 235, 415-420 (2002).

SESSION D7: Shape Memory Alloys-2
Chairs: Kaushsik Bhattacharya and Manfred Wuttig
Wednesday Morning, December 3, 2003
Room 202 (Hynes)

8:30 AM *D7.1
Fabrication and Characterization of TiNi/Al Smart Composites. Gyu Chang Lee[1], Jun Hee Lee[2] and Young Chul Park[3]; [1]Materials & Processes Research Center, Research Institute of Industrial Science & Technology(RIST), Pohang, Kyungbuk, South Korea; [2]Department of Metallurgical Engineering, Dong-A University, Pusan, South Korea; [3]Department of Mechanical Engineering, Dong-A University, Pusan, South Korea.

Al alloy matrix composite with TiNi shape memory fiber has been fabricated by hot pressing and ingot casting to investigate the effect of processing variables on microstructures and mechanical properties. The main focus is on the reduction ratio of rolling on mechanical properties and diffusion layer formation by heat treatment at the interface between the TiNi fiber and Al matrix. Through SEM micrographs and EDS analysis, good interface bonding has been observed. The stress-strain behavior of the composites was evaluated at temperatures between 363K and room temperature as a function of prestrain. The results showed that yield stress at 363K was higher than that of the room temperature. Furthermore, yield stress of this composite increases with increasing the amount of prestrain, and it also depends on the volume fraction of fiber and heat treatment. The smartness of the composite is given due to the shape memory effect of the TiNi fiber which generates compressive residual stress in the matrix material when heated after being pre-strained. Microstructural observation has revealed that interfacial reactions occur between the matrix and the fiber, creating two intermetallic layers. Finite element analysis was used to predict the mechanical properties of TiNi/Al composite. The FEA results represented the experimental results very well.

9:00 AM D7.2
Fabrication and characterization of freestanding NiMnGa thin films. Holger Rumpf[1], Achim Gilles[1], Manfred Wuttig[2] and Eckhard Quandt[1]; [1]Smart Materials, Caesar, Bonn, Germany; [2]Department of Materials Science, University of Maryland, College Park, Maryland.

Ni50Mn30Ga20 thin films were deposited by DC magnetron sputtering. Free-standing thin films of 3 - 10 micrometers in thickness were fabricated by depositing on thin glass plates and subsequent removing of the substrate. Thermal annealing led to polycrystalline films revealing shape memory properties as demonstrated by cantilever deflection technique and differential scanning calorimetry. Transformation temperatures and reaction enthalpies of the martensitic transformations were strongly influenced by the applied annealing temperatures. Vibrating sample magnetometer measurements revealed a further dependency of the ferromagnetic hysteresis on the applied thermal annealing. Magnetostriction of Ni50Mn30Ga20 films sputtered on Mo cantilevers revealed only a small effect of about 300 ppm limited due to the polycrystalline structure of the film. Financial support provided by the Office of Naval Research (N00014-02-1-0231) is gratefully acknowledged.

9:15 AM D7.3
Theory and Modeling of Gradient Shape Memory Alloy Actuators. Alexander Roytburd[1], Victor Roytburd[3] and Julia Slutsker[2]; [1]University of Maryland, College Park, Maryland; [2]NIST, Gaithersburg, Maryland; [3]Rennseleaer Polytechnic Institute, Albany, New York.

A new concept of bending actuation in which the transformation in a SMA active layer proceeds by the movement of phase interfaces or intermediate two-phase zone is presented. The interface movement is determined by gradient of temperature or composition across the thickness of an active layer. The bending of the film is a result of self-strain in a transformed part of the film. Calculation of the elastic energy of internal stresses show that there is an equilibrium position of interface, which can be shifted by a thermodynamic driving force. Thus, it is possible to obtain reversible movement of interface by changing temperature. Combining SMA film with top or/and bottom passive layers of different elastic properties, thickness and misfit it is possible to optimize the actuating deformation. The kinetics of interface movement is quantitatively described by solving a Stefan-type problem with an equilibrium temperature at the interface between phases dependent on interface position. The computational technique has been developed to control dynamics of the actuator through the variation of temperature at the top and the bottom of the active layer. The effect of compositional gradient on dynamic of actuation is discussed. The work is supported by AFOSR Grant#F4962001101110.

9:30 AM D7.4
Fabrication and Characterization of Fe-Pd Shape Memory Thin Films. Yuki Sugimura and Joost J. Vlassak; DEAS, Harvard University, Cambridge, Massachusetts.

Thermally activated shape memory materials such as Ni-Ti alloys are widely used as functional materials in sensors and actuators. However, heat transfer issues in these materials may limit their application in devices that require rapid response. Magnetically driven shape memory effect in select ferromagnetic alloys offers potential for faster response time as well as contactless actuation, expanding the range of application of these materials. Ferromagnetic shape memory alloys such as the Fe-Pd system have been studied in detail in bulk form by a number of researchers. While thin films are more suitable for miniature devices research activities in Fe-Pd thin films have begun only recently. The thermoelastic austenite-to-martensite transformation takes place over a composition range of approximately 28 to 32 atomic percent Pd in bulk Fe-Pd alloys. In recently published papers, Fe-Pd thin films were deposited from alloy targets of specific composition, making it difficult to vary the Pd content in a systematic manner. In this paper we present the results from fabrication and characterization of Fe-Pd thin films deposited using Fe and Pd elemental targets. Films are produced by dc magnetron sputtering in an UHV chamber equipped with three independent confocal sputter guns. The effect of process parameters and post-deposition thermal treatments on film composition, microstructure and stress are examined by Rutherford backscattering, x-ray diffraction, electron backscatter diffraction and substrate curvature technique.

9:45 AM D7.5
Phase Transformation in Ni-rich Ti-Ni and Ti-rich Ti-Pd Shape Memory Alloys. Minoru Nishida, Materaials Science and Engineering, Kumamoto University, Kumamoto, Japan.

Near equiatomic Ti-Ni and Ti-Pd alloys undergo thermoelastic martensitic transformation from B2 to B19' and B19 structures upon cooling, respectively. The former is technologically importaant materials with their superior shape memory and superelastic properties. The latter is expected to be high temperature shape memory materials, since the transformation temperatures are around 800K. In order to complete the further improvement of both the alloys, various heat treatment have been proposed so far. In the present study, the phase separation processes in Ni-rich Ti-Ni and Ti-rich Ti-Pd alloys are briefly summarized. Subsequently, the mechanism of multistage martensitic transformation in both the alloys are discussed on the basis of thermal analyses and TEM observations. We demonstrate that the multistage transformation in the Ni-rich Ti-Ni alloy is considered to be a kind of artifact, which strongly depends on heat treatment atomosphere. On the other hand, that in Ti-rich Ti-Pd alloy is closely related to in-situ precipitation of Ti2Pd during the transformatuion cycles.

10:30 AM D7.6
Thermo-Mechanical Characteristics of Ti-Ni-Cu Shape Memory Alloy Fabricated by Pulse-Current Pressure Sintering Method. Hideki Kyogoku[1], Takeshi Kadomura[1], Shinichiro Komatsu[1], Fusahito Yoshida[2] and Toshio Sakuma[3]; [1]Mechanical Engineering, Kinki University, Higashihiroshima, Hiroshima, Japan; [2]Mechanical Engineering, Hiroshima University, Higashihiroshima, Hiroshima, Japan; [3]Central Research Institute of Electric Power Industry, Komae, Tokyo, Japan.

In this research, we attempted to fabricate the Ti-Ni-Cu shape memory alloys by elemental powders by means of a pulse-current pressure sintering method that can produce high-density sintered compacts in a very short sintering time. We investigated the fabrication conditions of Ti-Ni-Cu alloys and the influence of Cu content in the alloys on the tensile and thermo-mechanical characteristics by experiments. The relative density of the as-sintered compacts was around 97% at any Cu content. The microstructure, tensile properties and thermo-mechanical characteristics of the as-sintered compacts were improved greatly by performing a solid-solution treatment and a shape memory treatment. The yielding behavior due to the stress-induced martensite in stress-strain curves changed with Cu content, and it became the lowest at some 9at% in Cu content. The tensile strength and elongation of the alloy with Cu content around 9at% were more than 400 MPa and 6%, respectively. The alloys of higher Cu contents showed a superelastic-like behavior, and the shape memory characteristics of the alloys were confirmed to be superior. The recovery stress of the alloys was almost the same level of that of the wrought materials. The cyclic deformation behavior of the alloys became stable after 15 cycles.

10:45 AM D7.7
Crack Tip Fields and Fracture Behavior in a NiTi Shape Memory Alloy Sheet. Wei Tong, Nian Zhang, Changjin Xie and Hong Tao; Yale University, New Haven, Connecticut.

Shape memory alloys such as NiTi are often used in MEMS and other applications in the form of thin strips. Fatigue failure is one of important design considerations in many NiTi shape memory alloy products. We present an experimental investigation on the crack tip deformation fields and fatigue crack growth kinetics in a NiTi thin sheet of 0.25mm thick. The material is 54.8wt% Ni and 45.2wt% Ti with only a trace amount of O, H, and C. The external mechanical and thermal loads are controlled to induce various levels and size of crack tip deformation fields in each load cycle. Experimental results on the possible correlation on the crack tip field characteristics and the fatigue crack growth kinetics will be presented for the NiTi sheet. A micromechanical analysis of the crack tip field will also be given to identify some possible mechanisms on the fatigue damage in NiTi shape memory alloys.

11:00 AM D7.8
Phase Stability of Single Crystalline Co-Ni-Ga Shape Memory Alloy. D. L. Schlagel[1], T. A. Lograsso[1,2] and A. O. Pecharsky[2]; [1]Metal & Ceramic Science Program, Ames Laboratory, Ames, Iowa; [2]Materials Science and Engineering, Iowa State University, Ames, Iowa.

Single crystals of Co48Ni22Ga30 have been synthesized using the modified Bridgman method. The ability to solidify and retain single phase B2 austenite was found to depend not only on the starting composition and growth rate, but also the ability to maintain sufficiently high cooling rates to avoid the precipitation of a Co-rich FCC phase during post-solidification cooling. DSC measurements on the single crystal found the Ms, Mf, As, and Af to be 35.7, -1.8, 34.1 and 72.2°C, respectively. On subsequent heating the B2 phase was found to partially decompose into the Co-rich phase at temperatures exceeding 350°C. The decomposition of the single phase B2 phase was tracked by DSC, high temperature powder diffraction, and microstructural observation. Restoration of the crystal to single phase B2 austenite required annealing of the crystal at temperatures above 1125°C.

11:15 AM D7.9
High Transition Temperature Shape Memory Alloys for Micro-actuator Systems. Elizabeth Baldwin and Afsaneh Rabiei; Department of Mechanical and Aerospace Engineering, North Carolina State University, Raleigh, North Carolina.

A new generation of thin film shape memory alloy (SMA) for MEMS micro-actuator has been developed, in which film structure and chemistry are optimized, for enhanced higher transition temperature, higher strain recovery rate as well as reduced actuation time by improving the heat transfer rates. Thin film TiPdNi was produced using Ion Beam Assisted Deposition (IBAD) technique followed by post processing heat treatment. Shape memory properties of thin film TiPdNi and their mechanical properties showed a great improvement over conventional NiTi thin films processed using sputtering technique. This is partly due to their higher transition temperature and their reduced thickness that affects the actuation time by improving the heat transfer rates. Furthermore, the service-life of the new thin film SMAs increased by improving their micro structural properties. The desire to introduce this innovative technology to the field of SMA micro-actuators is based on two primary advantages of IBAD process over existing technology used to apply thin film SMAs. First, the chemical composition and grain size of the applied coating can be precisely controlled over a wide range of values. Second, the SMA can be deposited as thin films ≤ 2 μm thick with smaller grain size, much denser than films applied using sputter deposition technology. These coatings provided a higher interfacial strength and better fracture resistance than the plasma spray coatings. The effects of various processing parameters, and post processing heat treatment, on mechanical, and microstructural properties as well as their SMA properties were studied.

11:30 AM D7.10
Crystallography of Ni-Ti SMA in Austenite and Martensite by X-ray Diffraction Study. Mitsuo Notomi and Hajime Kayano; Mechanical Engineering, Meiji University, Kawasaki, Kanagawa, Japan.

The crystal structures of the austenite and martensite in Ni-Ti shape memory alloy (SMA) are studied by X-ray diffraction. First, the program that estimates the crystallographic data of an alloy and analyzes its X-ray diffraction pattern is developed on the basis of the theory of the intensity of a particular diffraction peak. The patterns by this program are verified by the comparison with those of single crystals of Si, Ni and Ti. Next, we measure the diffraction pattern of Ni-Ti not using powder sample but also wire one by X-ray diffractometer because a wire of Ni-Ti SMA is widely used for application as an actuator and so on. Moreover, the crystal structure should be damaged if powder sample is made of the wire by milling and the curvature of wire surface has the same effect as the aggregate of powder for X-ray scattering. The diffraction pattern of Ni-Ti SMA is measured at the elevated temperature and its crystal structure is austenite phase. This pattern corresponds with the analyzed one from B2 structure using the program. Next, at room temperature the diffraction pattern of Ni-Ti SMA that is martensite is measured. This pattern is compared with the B19' crystal structures presented by K. Otsuka et al., R. F. Hehmann et al., G. M. Michal et al. and Y. Kudoh and et al. Although the x-ray pattern almost corresponds with that by Kudoh, it remains the peak of B2 structure even thought the temperature is below the martensite transformation and the wire shows the shape memory effect. In order to consider the difference of B19' structures by each researcher, the relationship between the unit cell volume and the atomic ratio of Ni and Ti is obtained. The unit cell volume is proportional to the atomic weight percentage of Ti.

11:45 AM D7.11
Characterization of Nitinol for Smart Structural Systems Using Aces Methodology. Shivananda Pai Mizar and Ryszard J Pryputniewicz; Department of Mechanical Engineering/CHSLT-NEST, Worcester Polytechnic Institute, Worcester, Massachusetts.

SMAs are engineering alloys that recover extensive amounts of deformation when subjected to a temperature change. The recovery of deformation is a result of a phase transformation from martensite to austenite. For example, for NiTiNOL, a very popular SMA, this transformation occurs at 60°C. Actuation is a type of shape memory process that utilizes deformation recovery that is used in smart structural systems. These systems are designed to detect and control structural deformations due to external time varying loads encountered during functional operation. In this paper, the actuating properties of NiTiNOL are characterized using an analytical, computational, and experimental solution (ACES) methodology. Analytical models are utilized to calculate the deformation under static and dynamic loads. Finite element method (FEM) is used for computational investigations. Optoelectronic holography (OEH) is the experimental method utilized to obtain temperature dependent behavior of NiTiNOL. As a result of the phase transformation, NiTiNOL undergoes a stiffness change indicated by a change in the modulus of elasticity from 33 GPa to 73 GPa. Deformations of NiTiNOL samples subjected to static loads at different temperatures were measured indicating recovery of 85% due to the phase transformation. In addition, amplitudes of vibration were measured as a function of excitation frequency and temperature to determine the effect of phase transformation. The results show effectiveness of NiTiNOL in development of smart structural systems.

SESSION D8: Electroactive Polymer-3
Chairs: Roy. D. Kornbluh and Mohsen Shahinpoor
Wednesday Afternoon, December 3, 2003
Room 202 (Hynes)

1:30 PM *D8.1
Ionic Polymer-Metal Composites as Smart Materials under Subzero Temperature Conditions. Kwang J. Kim and Jason Paquette; Mechanical Engineeering Department and Nevada Ventures Nanoscience Program, Univ. of Nevada, Reno, Nevada.

This paper presents a description of Ionic Polymer-Metal Composites (IPMCs) as an alternative solution for cold operation actuators. This is because of their capability for actuation with relatively low voltages, durability and capability of operating within the subzero regime ($T < O$ C). The building block material of IPMCs experiences phase changes within the base polymeric material that results in an alteration of the performance of the material in terms of actuator performance. An experimental apparatus is constructed in order to have a controlled temperature environment in which to analyze the material. The results are presented and interpretated to show that there is definte promise for these low temperature polymeric actuators to operate in practical applications. Also, an effective process technique, nanocompositing, was attempted to obtain the optimized properties of the base polymeric materials.

2:00 PM D8.2
Hybrid Actuation in Coupled Ionic/Conducting Polymer Devices. Matthew Bennett and Don Leo; Mechanical Engineering, Virginia Tech, Blacksburg, Virginia.

Ionic polymer membrane actuators represent a relatively new and exciting entry into the field of smart materials. Several key limitations of these transducers have prevented them from experiencing widespread use, however. For example, the bandwidth of these devices is limited at very low frequencies by characteristic relaxation and at high frequencies by the low elastic modulus of the polymer. In this paper, an overview of the initial results of work with hybrid ionic/conducting polymer actuators is presented. These hybrid actuators are devices that combine the electromechanical coupling of ionic polymer actuators and conducting polymer actuators into one coupled device. Initial results show that these hybrid devices have the potential to offer marked advantages over traditional ionic polymer membrane transducers, including increased stress and strain generation and higher actuation bandwidth. Details of the preparation of these devices and performance metrics are presented and comparisons to baseline materials are made.

2:15 PM D8.3
Temperature-Responsive Polymers with Tailored Onset of Response. Evangelos Manias and Mindaugas Rackaitis; Materials Science & Engineering, Penn State University, University Park, Pennsylvania.

Stimuli responsive materials are central in applications involving chemical sensing and/or stimuli-driven actuation. A systematic series of temperature-responsive polymers were synthesized and studied, and the onset of their T-response was tailored by design of their monomer. Their T-response was studied both for their water solutions, and when they were end-tethered on a surface. Within the monomer, a balance of hydrophilic (ethylene oxide, EO) and hydrophobic (ethylene) segments provides the possibility to fine-tune the lower critical solution temperature (LCST) point. Solubility studies and phase diagrams were done for the solutions, whereas water contact angle, ellipsometry, and atomic force microscopy were carried out for the end-grafted polymers, as a function of grafting density. A universal dependence of the temperature of response on the monomer composition is found, and the onset of the response can be tailored by design in the range of 5-70°C in water. Model microfluidic devices employing these polymers as T-responsive gates were also realized.

2:30 PM D8.4
All-Electrical Soft-Tissue Stiffness Measurement Using Piezoelectric Cantilevers. Wei-Heng Shih, Anna Markidou, Stephen T. Szewczyk and Wan Y. Shih; Materials Science & Engineering, Drexel University, Philadelphia, Pennsylvania.

Piezoelectric unimorph cantilevers were explored for all-electrical soft-tissue elastic moduli measurements. The elastic moduli of soft tissues were obtained by simple electrical means using a dual electrode design: a driving electrode for force generation and a separate sensing electrode for deformation measurement. Accurate Young's modulus measurements were demonstrated with both indentation and compression tests on model gelatin samples as well as blood vessel plaques. Direct shear modulus measurements were also demonstrated using cantilevers with a L-shaped tip. Potential clinical applications for such a method were studied, mainly, the possibility of a stand-alone instrument for non-invasive detection or diagnosis of diseased tissue. The advantages of piezoelectric unimorph soft-tissue stiffness sensors include simple all-electrical measurements, accurate elastic moduli measurements at very small strains (<1%), local stiffness measurements that offer potential for tissue imaging. Further miniaturization will allow simple electrical soft-tissue stiffness imaging on the micrometer- and nanometer-scale.

2:45 PM D8.5
Exploration of an Electroactive Polymer as an Actuator for Microrobotics. Christie Bielmeier and Wayne Walter; Department of Mechanical Engineering, Rochester Institute of Technology, Rochester, New York.

The development of lightweight low power consumption actuators is critical to the development of micro-robotics. The Electroactive Polymer (EAP), Nafion N-117, meets these requirements. In the actuation of an EAP, the current does not remain constant over time. A circuit model for current draw versus time to best predict the current behavior has been developed for Nafion N-117. While the material mimics a parallel plate capacitor, it has been found that capacitance plays no role in achieving steady state current levels. This development is critical to understanding and developing the material as a viable actuator. This paper discusses the necessary developments required for the EAP actuator to be utilized in microrobotic devices. Specifically, the development of a lightweight reliable electrode for EAP material actuation, the current draw versus time model and equations, and microrobot applications are discussed.

3:30 PM D8.6
Switchable Window Based on Electrochromic(EC) Polymers. Chunye Xu, Lu Liu, Susan E. Legenski, Dai Ning and Minoru Taya; University of Washington, Seattle, Washington.

Switching window technology is a continuously growing field due to its current and potential applications for architectural, vehicular and aircraft windows, buildings, sunroofs, eyeglasses, and numerous type of displays. Whether its purpose is energy conservation, functional use or purely aesthetic, switchable windows have a promising future. Electrochromic polymers are the basis for switchable window technology. The polymers change color under different electric potentials. When a small electric potential is applied, the electrochromic polymers change state and the window changes between transparent and opaque. Even though electrochromic polymers have been around since the early 1990s, our latest work is an advance because they invented new electrochromic polymers and a new system of laminating these components together to make a window. A large contrast ratio and rapid switching EC polymer device which consists of a laminated two-layer structure between two electrodes was prepared. The new design consists of an ITO glass electrode, a cathodic EC polymer film, a solid electrolyte and a counterelectrode that replaces the anodic EC polymer and ITO electrode. Several EC polymers, such as, Poly[3-methyl-3-propyl-3,4-dihydro-2H-thieno(3,4-b)(1,4)dioxepine] (PProDOT-MePro) were synthesized as cathodic EC polymers. Carbon-based counterelectrode was prepared for comparison with Au-based counterelectrode. Screen printing was utilized for carbon-based counterelectrode. Lithography and sputtering were used for Au patterned glass counterelectrode. Several kinds of polymer gel electrolyte were prepared for solid-state applications. Color change of high contrast ratio of transmittanceof the window is rapidly obtained when even less than 2V is applied. The repeatability of color changeable EC polymer windows was estimated by the method of electrochemistry and spectrophotometry.

3:45 PM D8.7
Self-assembly and applications of magnetorheological suspensions in microfluidic devices. Ramin Haghgooie and Patrick Doyle; Chemical Engineering, MIT, Cambridge, Massachusetts.

Magnetorheological (MR) fluids are suspensions of colloids which acquire dipole moments under application of a magnetic field. Traditionally, these colloids have been used in macroscopic applications, such as controllable dampers, where the ability to tune their bulk rheological properties with a magnetic field has been exploited. We have recently shown that the microstrucutures formed by the colloids in thin gaps can be used to efficiently separate DNA in microfluidic devices (Doyle et. al. 2002). Here we investigate the self-assembly of MR fluids in microchannels using Brownian Dynamics simulations and compare our results to microscopy experiments. We study geometries in which the characteristic length scale of the colloidal microstructure is comparable to the channel dimensions. In this regime, we find that the crystal structure deviates from the usual hexagonal lattice commonly found for the 2-D self-assembly of particles interacting through long-range repulsive interactions. Further fundamental understanding of self-assembly in confined geometries will allow for greater control of the porosity of colloidal matrices used for biomolecule separations in microfluidic devices.

4:00 PM D8.8
***In − situ* characterisation of photoactuating and photorheological polymers.** C Sabey[1], M Moniruzzaman[1], G

Fernando[1], R Badcock[1], J D Talbot[1], D Winter[1], J Akhavan[2], E Kronfli[2] and A Bandara[3]; [1]Centre for Photo-Analysis and Photo-Manipulation, Cranfield University, Swindon, Wiltshire, United Kingdom; [2]Department of Environmental and Ordnance Systems, Cranfield University, Swindon, Wiltshire, United Kingdom; [3]School of Chemical and pharmaceutical Sciences, Kingston University, Kingston-upon-Thames, Surrey, United Kingdom.

This paper reports on the design and validation of three $in-situ$ measurement techniques for characterisation of photorheological and photoactuation phenomena. Results from these $in-situ$ techniques are presented and used to investigate isomerisation and recovery effects in azobenzene and spyropyran polymer systems with, and without, flexible spacer groups. A new $in-situ$ technique for the measurement of photoviscosity effects in polymer solutions has been developed using a cone and plate rheometer. The new technique permits simultaneous irradiation of the photoresponsive polymer solutions and continuous measurement of its viscosity in a controlled environment. An additional benefit of the technique is the greatly reduced sample volume compared to traditional capillary viscometers. Techniques for the $in-situ$ measurement of the photoactuation effect in polymer systems are discussed, including image analysis and interferometric methods. The effect of environmental factors such as temperature are considered and steps taken to minimise them. The resulting measurement techniques provide an accurate means of measuring the effect of irradiation on the photoactuating polymers in solution and as solids. A range of polymers with photoresponsive azobenzene and spiropyran groups in the polymer side chains have been synthesised and evaluated using these three techniques. The photoviscosity and photoactuation effects under irradiation have been measured in a variety of solvents. The effects of incorporating a flexible spacer group in the side chain of the azobenzene polymers is examined and its effect on the photorheology mechanism is discussed. The reproducibility of the isomerisation and recovery cycling of the spiropyran based polymer systems under alternate irradiation at different wavelengths is examined and discussed.

4:15 PM D8.9
Preparation and Properties of the Novel Nanocrystal and Polymer Composite Films for Electro-Optic Applications. Quan Ren[1,3], Dong Xu[2], Yuk Tak Chow[3], Andy Hau-Ping Chan[3] and Pak Lim Chu[3]; [1]Department of Optics, Shandong University, Jinan, Shandong, China; [2]State Key Laboratory of Crystal Materials, Shandong University, Jinan, Shandong, China; [3]Optoelectronics Research Centre, City University of Hong Kong, Hong Kong, China.

The novel nanocrystals and polymer composite thin films for the applications of electro-optical devices were prepared. The transparent polymer with a high glass transmission temperature, polyetherketone, has been selected as the host. $PbTiO_3$ has been selected as the electro-optic basic units (guest), due to its electro-optic properties and high melting point. The $PbTiO_3$ nano-crystals have been synthesized successfully by the chemical solution decomposition method. The size of the $PbTiO_3$ nanocrystals was estimated to be 30-40 nm under a transmission electron microscopy. The weight ratio of $PbTiO_3$ nanoocrystals to polymer polyetherketone was 15 %. The composite thin films of $PbTiO_3$ / polyetherketone were prepared by spin-coating technique. The films are then electrically poled to induce asymmetry in the material by heating the films to a temperature close to their glass transition and applying an electric field. The refractive indices of the un-poled composite $PbTiO_3$ / polyetherketone film are found to be 1.55165 for 633 nm and 1.65352 for 414 nm by using the Metricon prism coupling system. In the poled composite $PbTiO_3$ / polyetherketone film sample, the TE- and TM-indices differences are found to be 0.02945 for 633 nm and 0.03915 for 414 nm, showing very good optical anisotropy properties. Dielectric constant ϵ of the film is determined to be 7.32 at room temperature at 100 KHz using an Impedance Analyzer. The electro-optic coefficient, γ_{33}, of poled $PbTiO_3$ / polyetherketone composite film is measured to be 18.34 pm/V at 633 nm under room temperature by the simple reflection technique. The factor $F_2 = n^7\ \gamma^2\ /\epsilon$, figures of merit, is estimated to be 1546. In addition, a relaxation process was observed in the time range of 28 days. The relaxation time constant was calculated to be 5836 min.

SESSION D9: Poster Session: Carbon Nanotube, Sensor, and Magnetic Based Materals
Chair: Rob Klein
Wednesday Evening, December 3, 2003
8:00 PM
Exhibition Hall D (Hynes)

D9.1
Novel actuating system based on a composite of single walled carbon nanotubes and an ionomeric polymer. Igor Levitsky[1], Peter Kanelos[2] and William Euler[2]; [1]Emitech, Inc., Fall River, Massachusetts; [2]Department of Chemistry, University of Rhode Island, Kingston, Rhode Island.

We report the fabrication and characterization of a novel composite material based on purified single walled carbon nanotubes (SWNTs) and the ionomeric polymer Nafion. SWNTs were airbrushed from a chloroform suspension onto both sides of a Nafion membrane (180μm) and the electromechanical properties of the composite material were explored. Cantilevers of the composite material were cut on all sides in order to create autonomy between the two layers of carbon nanotubes. The outer two layers of carbon nanotubes acted as electrodes in order to pass electrical current through the system, and the mechanical response (displacement, excitation and relaxation behavior) was monitored. Under this design the mechanical response could be characterized, with respect to the electrical signal, as a function of: voltage, waveform (AC vs. DC), and frequency (AC). Data was also compiled to gauge the effect of size and thickness of each individual layer of the system. Reference samples (graphite/Nafion and sputtered gold/Nafion) did not exhibit mechanical actuation under the same conditions. FTIR, UV-Vis, and cyclic voltammetry (CV) were applied to study the physical chemical and electrochemical properties of the interface between the polymer and the SWNTs. An analytical model for current decay was considered, and is in agreement with the experimental data. The possible mechanisms of actuation in this novel composite are discussed. The results show this configuration to be a promising new electromechanical actuator for potential use in MEMS and NEMS technological applications.

D9.2
Sterilizing Properties of Carbon Nanotube Composites. Roger Jagdish Narayan and Bryan Bell; Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia.

Hydrogen-free carbon nanotube composites have been created that possess unusual free radical generation properties on photosensitization. These films have the ability to nonspecifically kill bacterial and human cells, thereby sterilizing a surface. These nanocomposites have been formed by pulsed laser ablation of carbon and simultaneous bombardment of nitrogen ions generated by Kaufman ion source. A pulsed excimer laser (l=248 nm, ts=25 ns) was used to ablate the high purity graphite target with energy density in the range 2-3 J cm-̂2, which gives an average power density of around 108̂ W/cm2̂. To produce atomic and nitrogen ions from a very stable N2 molecule, the ultra high purity grade nitrogen was used as an input to the Kaufman ion source which makes an angle of 45 degrees with the substrate. The beam energy of N+̂ and N2̂+ used for these experiments was ~500 eV and two beam currents (10 and 20 mAngstrom) were used in the defocused geometry. The silicon substrates were kept at 600 degrees C while ablating the carbon target. Bonding characteristics were determined using x-ray photoelectron spectroscopy, microstructure was determined using high resolution transmission electron microscopy (HRTEM), and short range atomic order was studied using radial distribution function analysis of the electron diffraction patterns. The C is core-level peak was detected by XPS to shift from 284.6 eV BE for graphite to 284.3 eV for carbon nanocomposites, indicating a weaker C-C bonding caused by the curvature of graphene sheets. The C1s peak can be deconvoluted into three main components, corresponding to surface carbon, C=N and C-N, respectively, in the order of increasing binding energy. The typical N content is thus estimated to be in the range 15-20 at.%. TEM studies allow us to conclude that the material consists presumably of the sp2̂-bonded ribbons wrapped around the normal to the surface. These ribbons remain parallel within +/-15 degrees to the substrate surface normal and the interlayer order extends to approximately 15-30 Angstrom. These features were seen irrespective of the specimen tilt in the microscope suggesting the curved nature of these planes. The textured nature of these films was also seen in the electron diffraction patterns obtained in the selected area diffraction mode of the cross-section samples. In vitro testing revealed significant antimicrobial activity against Staphylococcus aureus. Possible applications include the functional surfaces of dialysis equipment, scalpels, and other sterile equipment.

D9.3
In Situ Raman Spectroelectrochemistry Study of Single-Wall Carbon Nanotube Mat.+. Sanju Gupta[1], M. Hughes[2], A. H. Windle[2] and J. Robertson[1]; [1]Engineering, University of Cambridge, Cambridgeshire, United Kingdom; [2]Metallurgy and Materials Science, Univeristy of Cambridge, Cambridgeshire, United Kingdom.

Electrochemically induced strain in carbon nanotubes is of interest for actuator applications, the potential of which as "artificial muscles" is realized recently [1]. This is because of the outstanding physical (electrical and mechanical) properties of single-wall nanotubes and therefore it is not surprising that their electro-mechanical properties are unusual. One of these many key applications of carbon nanotubes is based on the electrochemical double-layer (ECDL)-separates charges on an electrode from the ionic charges in solution. Briefly, when a sheet of single-wall carbon nanotubes is dipped into an electrolyte, it expands or contracts if a voltage is applied between them and a counter electrode. Based on prediction, actuation is due primarily to changes in orbital occupation and band structure with a secondary contribution arising from double-layer electrostatic effects. However, the relative contribution and interaction between each of these mechanisms is unclear. We aim to elucidate actuation behavior using Raman spectroscopy (RS) and built a linear actuator from SWNT mat. The measurements were performed in various oxygen-free aqueous electrolytes (LiCl, NaCl, KCl, KBr, K2SO4, and CaCl2) in order to clarify the role of counter-ion, if any. The variation of carbon bonding in the nanotubes with applied potential was studied by in-situ Raman spectroscopy, because it can detect changes in C-C bond length (dC-C), through two of the most prominent bands [radial breathing mode; RBM at ∼190 cm-1, and G band at ∼1590 cm-1]. In addition, the intensities of both the modes vary with the occupation of the bonding and anti-bonding states due to charge transfer. We found a strong, electrolyte dependent variation in intensity of both modes with applied voltage and follow a quasi-parabolic behavior. While the shift in frequency of RBM does hardly change within the experimental uncertainities, the variation in the highest frequency G-band (ranging 1593 - 1602 cm-1) with applied potential displayed somewhat dependence on the electrolyte used. The estimated in-plane strain deduced indirectly from Raman measurements is of the order of ∼0.25% [2] and the charge transfer (fc) is ∼0.004, which is significantly lower if compared with several GIC acceptor compounds. This is to say that though this is in agreement with the proposed mechanism [1], but the degree of variation (i.e. coupled electro-chemo-mechanical response of SWNT mat) does depend upon the type of counter-ion used. The cyclic voltammetry (CV) and ac impedance behavior of the nanotubes is described briefly. These findings of electron transfer processes in SWNT sheets besides actuators opens a gamut of practical applications include hydrogen storage/fuel cells, batteries, super/ultra capacitors, and micro-electrodes for neuro-physiology. [1] R. Baughman et. al. Science, 284, 1340 (1999). [2] S. Gupta et. al. Diamond and Related Materials (2003) (Submitted).

D9.4

Production of High Quality Single-Walled Carbon Nanotubes in A Nano Agglomerates Fluidized Bed Reactor.
Qixiang Wang, Guoqing Ning, Fei Wei and Guohua Luo; Department of Chemical Engineering, Tsinghua University, Beijing, China.

Single-walled carbon nanotubes (SWNTs) have attracted much attention due to their unique structural, mechanical, chemical, and electrical properties. Catalytic chemical vapor deposition (CCVD) through decomposition of hydrocarbons is one of the most challenging synthesis methods to produce SWNTs in large scale. In this paper, high quality SWNTs prepared in a fluidized bed reactor (FBR) was reported. The reactor is a vertical quartz tube with the inner diameter of about 50 mm. Several catalysts particles used for the production of SWNTs were prepared with Fe and/or Co metal nanoparticles carried on the surface of MgO, Al2O3, SiO2, ZSM-5 etc. About 100g catalysts powders were loaded on the gas distribution grid. The reaction temperature was controlled at not less than 1123 K. The carrier gas of argon was used to maintain turbulent fluidization of the catalysts powders in FBR. The hydrogen gas was used to reduce the catalysts and to synergize the growth of SWNTs with methane. The products were spherical agglomerates of SWNTs bundles tangling with the catalysts powders. By optimizing the process parameters of CCVD, the products of SWNTs with the diameter about 0.8 - 1.5 nm were prepared, without any carbon by-products, such as multiwalled carbon nanotubes (MWNTs) or amorphous carbon. The quality and quantity of SWNTs products were characterized by the methods of high resolution transmission electron microscopy (HRTEM), scanning electron microscopy (SEM), Raman spectra, thermal gravimetric analyzer (TGA), X-ray diffractometer (XRD), etc. Our method gives a higher yield of SWNTs and would be a commercially viable (effective and continuous production at a low cost) process. The authors gratefully acknowledge the support of the National Natural Science Foundation of China (No.20236020).

D9.5

Hybrid Organic-Inorganic Polymeric Networks For Biosensor Applications. Nuttawee Niamsiri[1], Daniel F. Schmidt[2], Deepak Shah[2], Terri A. Wilson[3], Emmanuel P. Giannelis[2] and Carl A. Batt[1]; [1]Food Science, Cornell University, Ithaca, New York; [2]Material Science & Engineering, Cornell University, Ithaca, New York; [3]Chemical & Biomolecular Engineering, Cornell University, Ithaca, New York.

Our research focus is on the synthesis of innovatively engineered biodegradable polymeric materials for the development of low-cost, disposable biosensors that can be used for medical diagnostics, for the rapid detection of hazardous biological and chemical contaminants in the environment and food processing industry. Polyhydroxyalkanoates (PHAs) are biopolyesters naturally synthesized by numerous bacteria as carbon and energy storage compounds, and are suitable candidates for the production of novel biodegradable plastics. Since they possess material properties similar to various synthetic thermoplastics and elastomers, they have been extensively studied as environmentally friendly substitutes for conventional petroleum-based polymers such as polypropylene. However, due to the fact that unmodified PHAs possess limited stability towards many standard polymer-processing techniques, the aim of our research group is to develop novel hybrid polymer networks based on PHAs that further expand their applications. To date, we have successfully produced a variety of chemically distinct PHA using specially designed bioreactors. In this project, PHA-PDMS (Polydimethylsiloxanes) hybrids were formed through a crosslinking reaction between functional group present in the PDMS and complementary groups present in the PHA. The resulting material has properties quite similar to standard PDMS rubber, but unlike such PDMS, this material is potentially biodegradable. Additionally, this material represents the first of its kind reported to date. Currently, our primary research goal is to investigate the feasibility of using these novel PHA-PDMS hybrid elastomers for constructing microfluidic networks on chip-based biosensor devices. In addition, our lab is presently characterizing the biodegradability of the crosslinked PHA-PDMS materials.

D9.6

Degradation processes in new type resistive oxygen sensors.
Thomas Schneider[1], Wolfgang Menesklou[1], Harumi Yokokawa[2] and Ellen Ivers-Tiffee[1]; [1]Universitaet Karlsruhe (TH), Institute of Materials for Electrical and Electronic Engineering, Karlsruhe, Germany; [2]AIST Tsukuba, Energy Electronics Institute, Tsukuba, Japan.

New types of oxygen sensors with increased sensitivity are required to maintain operation of combustion engines within the desired A/F ratios under lean burn conditions and the future challenges cylinder-selective regulation, life-cycle emission control and on-board diagnostics. For these purposes, resistive oxygen sensors are attractive given their increased sensitivity, their short response times as well as their simple structure. Acceptor doped perovskite type metal oxides of the composition $Sr(Ti,MT)O_{3-\delta}$ (MT: transition metal Fe, Cr, ...) are commonly used for the sensitive layer. Contrary to donor doped sensor materials, $Sr(Ti,Fe)O_3$ shows a long term stable and temperature independent characteristic. In the presented planar exhaust gas sensor yttria doped zirconia (YSZ) is used as substrate with an Al_2O_3 protective layer doped with lanthanides as intermediate layer. To obtain distinct signal values an interdigital contacting for a high EMC performance is realized on top. The chemical stability of the intermediate and sensitive materials as well as their chemical compatibility at the interfaces have a significant impact on the performance and the long term behavior of the exhaust gas sensors. Degradation processes due to changes in composition, interdiffusion and the formation of secondary phases at the interfaces between the layers decrease the performance of the device. Depending on the material composition and the operating conditions, different types of degradation processes can be observed: Interdiffusion of cations at the interfaces between intermediate and sensitive layer affects the electrical properties of the exhaust gas sensor, while formation of insulating secondary phases at the interfaces between substrate, intermediate and sensitive layer interferes with the chemical reactions. Chemical Potential Diagrams based on the thermodynamic properties of the present compositions provide decisive information about the stability of the applied materials and the formation of parasitic secondary phases resulting in a severe degradation of the sensitive layer. By the use of Chemical Potential Diagrams a prediction of the stability of advanced materials and a preselection of applicable compositions becomes feasible without costly long term stability tests.

D9.7

Synthesis and VOC gas sensing properties of Polypyrrole/MoO3 Nanohybrids. Kouta Hosono, Ichiro Matsubara, Norimitsu Murayama, Woosuck Shin and Noriya Izu; National Institute of Advanced Industrial Science & Technology, Nagoya, Japan.

Gas sensors based on organic-inorganic hybrid materials are demonstrated. The synthesis of organic-inorganic hybrid materials aims at combining the physical and chemical properties of inorganic and organic components. The interesting point of such materials is that with the large number of chemical and structural modifications available. For designing organic-inorganic hybrid sensors, the organic and inorganic components take part in molecular recognition and transduction of chemical signals to measurable resistance changes, respectively. In this paper, we will present the preparation of organically hybridized inorganic materials and their VOC gas sensing properties. We have synthesized polypyrrole intercalated MoO3 hybrid materials, (PPy)xMoO3. A semiconducting-like transport is observed for the (PPy)xMoO3 pressed pellets, and the resistivity value is 9.6 Ωcm at room temperature. The (PPy)xMoO3 pressed pellets show a distinct response to VOCs by increasing in their electrical resistivity, which could be induced by the incorporation of VOC molecules into the interlayers of (PPy)xMoO3. (PPy)xMoO3 exhibits higher sensitivities to polar analytes such as formaldehyde and acetaldehyde, whereas it showed almost no response to toluene and benzene.

D9.8

Effect of sample preparation procedure on the magnetotransport properties of polycrystalline bulk Sr_2FeMoO_6double perovskite. N. Rama[1,2], T Komalavalli[1,2], K Chandrasekaran[1] and M.S. Ramachandra Rao[1,2]; [1]Materials Science Research Centre, Indian Institute of Technology, Madras, Chennai, India; [2]Department of Physics, Indian Institute of Technology, Madras, Chennai, India.

After the discovery of room temperature magnetoresistance (MR) in Sr_2FeMoO_6 by Kobayashi et. al. in 1996 [1], there is a renewed interest in these systems. However, after intense research it was found that MR (which results due to intergranular tunneling) in these systems is highly dependent on preparative route and conditions. We have therefore studied the effect on the MR by using two different sets of compounds in the solid state route. We have used strontium nitrate as the source of Sr in one case (sample 1) and strontium carbonate (sample 2) in the other case. Fe_2O_3 and MoO_3 are the sources of Fe and Mo respectively in both cases. We have characterized these two compounds by X-ray diffraction and the superlattice reflection peak (111) which denotes the ordering of the Fe and Mo sublattice is seen only in the case of sample 1 and not in sample 2. The absence of this peak indicates high degree of disorder in the lattice of sample 2 which is detrimental to potential applications like spintronics. It is seen from the resistivity and MR plots that sample 1 shows lower resistance at room temperature and higher low field MR than that of sample 2. These results will be explained in detail with respect to the difference in the acidity of the anion involved. [1]. K.I. Kobayashi et al. Nature **395** (1998) 677

D9.9

A Trial on Amplification of Magneto-Surface-Acoustic-Waves In Highly Magnetostrictive Metal Films By Means of Electron Bunching. Takeshi Kawahata, Naohisa Obata, Kazuhiro Nishimura, Hironaga Uchida and Mitsuteru Inoue; Toyohashi University of Technology, Toyohashi, Aichi, Japan.

An amorphous $Fe_{80}B_{20}$ thin film, which is highly magnetostrictive metal, shows a soft magnetization property after annealing. We applied the magnetostrictive thin film to propagation path for magneto-surface-acoustic-wave (MSAW) [1]. In our fabricated MSAW device, a phase velocity of surface acoustic wave was controlled widely by an external magnetic field because of ΔE effect of the $Fe_{80}B_{20}$ thin film. However, since the $Fe_{80}B_{20}$ film was a conductive alloy, an eddy current loss in the thin film increased by increasing a driving frequency for a MSAW, and the eddy current loss attenuated a surface acoustic wave. Therefore, the $Fe_{80}B_{20}$ film was not applicable to a MSAW device in the frequency range above several 100 kHz. In this report, we investigated amplification of MSAW by an electron bunching that was used in a traveling-wave tube in order to compensate the attenuation. We used a hybrid structure of $Fe_{80}B_{20}$/InSb thin films for amplification of a MSAW, because a speed of an electron in an InSb thin film is accelerated more than that of the SAW with a low voltage. The InSb thin film was deposited on a 128 degree Y-cut, X-propagation $LiNbO_3$ substrate by an ion beam sputter system. After annealing at 475 degree C for 90 minutes in vacuum, the (111)-oriented InSb thin film with an electron mobility of 860 cm^2/Vs was obtained. The amorphous $Fe_{80}B_{20}$ thin film with a coercive force of 1.5 Oe and a magnetostriction of 26 ppm was fabricated by a magnetron sputter system. An interdigital transducer (IDT) with a strip width of 8 mm was fabricated by photolithography. We observed a SAW of 125 MHz in our developed device with a hybrid structure of $Fe_{80}B_{20}$/InSb thin films. [1]N. Yokokawa, S. Tanaka, M. Inoue and T. Fujii, Jap. J. Appl. Phys., Suppl. 30-1, 182-184 (1991)

SESSION D10: Poster Session: Photonic, Optic and other Novel Materials
Chair: DaeYong Jeong
Wednesday Evening, December 3, 2003
8:00 PM
Exhibition Hall D (Hynes)

D10.1

Negative refraction by a prism and imaging by a flat slab of microwave photonic crystal. Patanjali V Parimi[1], Wentao Lu[1], John Derov[2], Plarenta Vodo[1], Srinivas Sridhar[1] and Beverly Turchinetz[2]; [1]Department of Physics and Electronic Materials Research Institute, Northeastern University, Boston, Massachusetts; [2]AFRL/SNHA, Hanscom, Massachusetts.

A conventional optical lens cannot focus light onto an area smaller than a square wavelength and the image formed by such lens is limited by the aperture size and fabrication quality due to curved surfaces. However, a superlens characterized by negative refraction can change the way optics works. Recent theoretical studies indicate that negative refraction is possible in photonic crystals (PC) in various regimes of the band structure. In this report we demonstrate negative refraction of microwaves in a metallic PC prism and imaging by a flat slab of a dielectric PC. Two dimensional refraction and imaging measurements are carried out in a parallel plate waveguide. Negative refraction is observed in different frequency regimes between 7 and 12 GHz. Using a flat lens of the dielectric PC, for a subwavelength point source at 2.25 cm from the surface of the PC, we have observed an image of similar size on the far side at 2.75 cm. The image formed by the flat lens due to negative refraction is a real 3D image. The experimental results are in excellent agreement with band structure calculations and simulations of wave refraction. The photonic crystal materials display low attenuation and are easily fabricated. The present results can be scaled to optical frequencies and pave way for several interesting applications. Work supported by the National Science Foundation and the Air Force Research Laboratories, Hanscom, AFB.

D10.2

Evaluation of the Optical Characteristics of Blue Phosphors for Plasma Color Display. Zhenhua Wang[1], Jieping Jian[1] and Longmei Zhao[2]; [1]Shanghai Yuelong New Materials Co. Ltd., Shanghai, Shanghai, China; [2]Shanghai Office of Rare Earth Materials Developing & Application, Shanghai, Shanghai, China.

Quality of plasma color display depends much on the optical characteristics of the phosphors. Chromaticity forms an important part of the fundamental parameters of color display systems. FCC and EBU have successively formulated chromaticity standards for phosphors. It is therefore seen that chromaticity provide an important indicator of the quality of the optical properties of a phosphor. On the other hand, brightness of a display screen is obviously dictated by the brightness of the phosphor used, so phosphor brightness must also be an important characterization of its light emission properties. However, the brightness and chromaticity coordinates of a blue phosphor are interrelated. It is often difficult to evaluate the quality of blue phosphors by its brightness and chromaticity. Listed in table 1, are the sintering temperature vs. optical properties of the phosphors obtained therefrom. The y value should preferably be around 0.06 according to EBU chromaticity standards. Thus the color purity is also satisfactory. However, it is seen in table 1, all samples with good color purity gave their brightness relatively low. This makes it difficult to evaluate their optical properties. In fact for all color display systems, including plasma color display, electro-optical parameters must be tuned to obtain white balance at specified brightness satisfying the requirements on its tristimulus values X,Y,Z. Thus in order to judge whether a blue phosphor is up to standards, considerations should be given not only to brightness, but also, and more importantly, to their tristimulus values. While its Y stimulus determines its brightness, stimulus Z is mainly contributed by its blue phosphor. Blue phosphors in fact contribute little to its brightness. It is therefore appropriate to choose the Z stimulus rather than its brightness as the main indicator of the blue phosphor quality. As shown in table 1, with increasing sintering temperature, its Z stimulus values increases accordingly, giving better and better optical properties. The fluorescent spectrum of such samples indicates that with blue phosphors, Z stimulus values vary linearly with its emission intensities. This leads to the same conclusion that it is appropriate to take its Z stimulus rather than brightness as an indicator of its quality. Another set of experimental data on the thermostability of blue phosphors also points to fact that brightness is not a good indicator of its quality and should be replaced by its Z stimulus.

D10.3

Characteristics of Dopant Activation by Sequential Lateral Solidification (SLS). Yong-Hae Kim, Choong-Yong Sohn, Choong-Heui Chung, Young-Wook Ko and Jin Ho Lee; Information Display Team, Electronics and Telecommunications Research Institute, Daejeon, South Korea.

Low temperature poly-Si (LTPS) TFTs have been widely studied for active matrix displays with integrated circuits, such as AMLCD and AMOLEDs. Among the methods of dopant activation in LTPS process, excimer laser annealing is the most efficient on and is a candidate for dopant activation on plastic substrate. In this paper, we study the characteristics of dopant activation on poly-Si films crystallized by sequential lateral solidification (SLS). The ion implantation is used to introduce phosphorous/boron ions to poly-Si film with 2E15 ion/cm^2 for n+/p+ type doping respectively. The dopant activation on excimer laser energy is analyzed with sheet resistance, SEM, raman spectroscopy and SIMS. The sheet resistance is compared with that of rapid thermal annealing (RTA). Sheet resistance of n+/p+ doped poly-Si film decreases exponentially as the excimer laser energy increases. The minimum sheet resistance of n+ doped poly-Si film is 150 ohm/sheet which is near to that of RTA and occurs at the beginning energy of nucleation. The minimum sheet resistance of p+ doped poly-Si film is 180 ohm/sheet which is a half to that of RTA and occurs above the nucleation energy. Raman signal of n+ doped poly-Si film shows single peak at all laser energy around $515cm^{-1}$ and it's intensity shows maximum at the beginning energy of nucleation. However, raman signal of p+ doped poly-Si film shows single peak at low laser energy and double peak at high laser energy around $510\sim520cm^{-1}$. SIMS profile on the laser energy is analyzed.

D10.4
Vanadium Dioxide Thin Films for Thermo-Optical Switching. Lijun Jiang and William N. Carr; New Jersey Institute of Technology, Newark, New Jersey.

Vanadium dioxide (VO_2) thin film undergoes a semiconductor-to-metal transition at about 68°C. The phase transition is accompanied with a drastic change in its optical transmittance and reflectance, which makes it a good candidate material for optical switching devices. Previous works have been focused on the study of the film properties. In this paper, we present the integration of VO_2 films into MEMS structures by the method of vanadium metal lift-off followed by thermal oxidation. The active VO_2 film was made by e-beam evaporation of pure vanadium target followed by thermal oxidation in oxygen ambient. Synthesized VO_2 film displays a phase transition at 65-68°C. It shows sharp switching for the infrared light at the transition. Results for VO_2 on a highly reflective metal layer is strongly dependent on the thickness of VO_2 film. The spectroscopic ellipsometer and stylus measurements reveal that the film thickness increases about 2.2 times after oxidation. The optical switching has a hysteresis of about 15°C between the heating and cooling branches. The scanning electron microscope (SEM) result shows a film microstructure with 50-100nm columnar grains. The measurements of the optical switching at different points across the film prove good uniformity of the oxidation. The effects of the micromachining process steps on its optical switching after VO_2 deposition will be presented. The study shows that VO_2 film with sharp thermo-optical switching can be successfully integrated into MEMS processing using lift-off followed by thermal oxidation.

D10.5
Optical Constants of Vanadium Dioxide Films and Design of a Solar Energy Control Window. Masato Tazawa, Hideo Asada, Gang Xu and Ping Jin; ISEM, AIST, Nagoya, Japan.

Vanadium dioxide (VO2) shows an abrupt change of the optical properties in the wavelength region longer than about 800 nm at the transition temperature of 68 C. At the temperature VO2 undergoes a phase transition from the low temperature semiconductive phase to the high temperature metallic phase. Doping tungsten reduces the transition temperature even to the freezing point. In this report, first we determined the optical constants of VO2 films in both metallic and semiconductive phases by ellipsometry from 380 to 1700 nm in wavelength. The Lorentz model with two oscillators provided the ellipsometric parameters very close to the measured values of the low temperature phase and the Lorentz model combined with the Drude model for the high temperature metallic phase. Second, using the optical constants, we designed a solar energy control window using a stacked film consisted of titanium oxide (TiO2), vanadium oxide (VO2), and indium tin oxide (ITO) for the upper, middle, and lower layer, respectively, on glass substrate. The TiO2 layer was used as an anti-reflection coating in the visible region and the ITO layer was used as a reflector in the wavelength region longer than about 2500 nm. The designed stacked film shows high transmission in the visible region and high reflection in the far-infrared region. In the near infrared region between 800 and 2500 nm, the reflectance changes according to the temperature because of the phase transition of the VO2 layer. Namely, this window reflects at high temperature and transmits at low temperature the near infrared part of the solar radiation.

D10.6
Star-like Aromatic Conjugated Polymers and Dendrimers for OLEDs. Irina A. Khotina[1] and Ludmila M Bronstein[2,1]; [1]Polymer synthesis, A.N.Nesmeyanov institute of the organoelement compounds, Moscow, Russian Federation; [2]Chemistry Department, Indiana University, Bloomington, Indiana.

The solution of the problem allowing synthesis of effective blue emitters can be found in synthesis of polymers with very branched fragments containing no other substituents than benzene rings. Polyphenylene synthesis based on cyclocondensation of acetylacetylaromatic compounds by trimerization cyclocondensation in the presence of acidic catalysts gives branched polymers with exclusively 1,3,5-substituted triarylbenzenes. The polymer chain growth occurs due to the interaction of three acetyl groups with new benzene ring formation. This reaction has a drawback - a polymer contains many defect fragments due to side reactions, which can quench the luminescence. The major side reactions are an incomplete cyclization, i.e. dimerization instead of cyclization with formation of b-methylhalcone groups, formation of linear and cyclic vinylene-containing products and also pyrylium salts. The main approach for the synthesis of luminescent branched polyphenylenes was the creation of defect-free structures. A branched defect-free oligophenylene (with 13 phenyl ring) was synthesized using cyclocondensation of [1-(4-acetylphenyl)-3,5-diphenyl]-benzene. This molecule presents a second generation of phenylated dendrimer with 1,3,5-triphenylbenzene (TPB) as a dendrimer center. Here all byproducts were eliminated by purification after synthesis and the final product showed intense photoluminescence in solution. The PL QY is 72%. We believe that higher generations of such dendrimers might result in enhancement of photoluminescence properties. However, as always with dendrimers of a higher generation, the synthesis is usually laborious and time-consuming. Another possibility is to lengthen the arms of the dendrimer center (obtained by cyclocondensation of acetylaromatic compounds) using some polymerization technique (for example, Ni0-catalyzed dehalohenation) providing defect-free structure. This approach should result in the branched polymers containing star-like fragments. New polymers were prepared starting from 1,3,5-tri(p-bromophenyl)benzene synthesized by cyclocondensation of p-bromoacetophenone. To prevent 3D crosslinking in Ni0-catalyzed reaction with bromides, we needed to convert tribromides to dibromides, i.e. to substitute one bromo-group (out of three) in tribromide for phenyl ring using Suzuki- reaction, i.e., interaction of tribromide with phenylboronic acid. The product prepared was used in Ni0-catalyzed polymerization (polymer 1) together with 1,3,5-tri(p-bromophenyl)benzene, as was used for direct polymer synthesis (polymer 2), but in this case, the equimolar amount of bromobenzene was added to keep the reaction mixture from gelation due to the decreasing of overall functionality of the system. This polymer 2 is characterized a remarkably high PL QY (96%) and also showed bright fluorescence in a solid state under UV irradiation at 366 nm, so these polymers can be considered as promising materials for OLED applications.

D10.7
Improvement of secondary electron emission property of MgO protective layer of an alternating current plasma display panel by addition of some oxides. Ari Ide[1], shigeto Murakami[2] and Yoshikazu Tanaka[3]; [1]International Innovation Center, Kyoto University, Kyoto, Japan; [2]Graduate School of Engineering, Kyoto-University, Kyoto, Japan; [3]Sanwa Kenma Ltd., Uzi, Japan.

In order to improve secondary electron emission coefficient of a protective layer in a plasma display panel, TiO2, SnO2, Al2O3, and ZnO are added to MgO films. We applied electron beam evaporation method for preparing the films. The proportion of doped-oxide was varied 0.5, 1, 2 mole-percent with each oxide. Rutherford backscattering spectroscopy (RBS), X-Ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) were performed to measure the chemical state and the composition of the prepared films. Secondary electron emission coefficient of these films was also measured. The doped-oxides vary their secondary electron emission particularly. The result means that adding oxide to MgO films has great influence on their secondary electron emission coefficient.

D10.8
Ion beam processing of MgO thin films with large secondary electron emissivity. Ari Ide[1], Hiroshi Nomura[2] and Yuji Tsukuda[3]; [1]International Innovation Center, Kyoto University, Kyoto, Japan; [2]Graduate School of Engineering, Kyoto University, Kyoto, Japan; [3]Sanwa Kenma Ltd., Uji, Japan.

Thin film of MgO is widely used as a protecting layer of plasma display panel (PDP). We prepared the MgO thin films using ion beam-assisted deposition (IBAD) technique with the aim of controlling the crystal orientation, density and composition of the films. Oxygen ion beam was utilized to irradiate the growing films. The ion beam irradiation was performed by electron cyclotron resonance (ECR) type ion source. The flux of evaporated MgO was produced using an electron gun. Energy and current density of the ion beam as well as deposition rate were taken as the parameters to control the deposition. Crystallinity, density and composition of the films were measured using X-ray diffraction (XRD) and Rutherford backscattering spectroscopy (RBS). We measured also the secondary electron emission coefficients of the films with a newly developed apparatus. Experimental results suggest that the ion beam irradiation during film growth strongly influences the crystal properties and the composition of the films to have the best orientation for high efficiency secondary electron emission.

D10.9
Effect of Postdeposition Annealing Temperature and Atomsphere on the Properties of ZnO Thin Films.
Omima M, Hamad[1], Gabriel Braunstein[1], Harshad Patil[2] and Neelkanth Dhere[2]; [1]Physics, UCF, Orlando, Florida; [2]FSEC, UCF, Cocoa, Florida.

The effect of post-deposition heat-treatment, in different gas atmospheres, upon the electrical and optical properties of ZnO thin films, grown by rf sputtering deposition, have been investigated. Samples of undoped, and Al doped, ZnO have been heat treated in air, oxygen, nitrogen, and forming gas (7% H_2 + 93% N_2), at temperatures ranging from 300°C to 900°C. Hall effect, electrical conductivity, and optical measurements revealed significant changes as a function of the annealing temperature and atmosphere. Films annealed at high temperatures show increases in sheet resistance and optical transmittance. The sheet resistance of the as-prepared ZnO is 2.1 x 10^2 Ω / , and it increases to values as high as 3.1 x 10^5 Ω/, after annealing at 900°C in oxygen. The average transmittance increases (in the VIS-NIR range) from 70%, for the as prepared ZnO films to > 80% for the film annealed at 500°C. The carrier concentration, Hall mobility, optical band gap, and index of refraction also show significant changes upon annealing. These changes are discussed in terms of the crystalline structure of the films.

D10.10
Smart Heterostructures Based on Solid Solution ZnCdHgTe.
Galina Khlyap[1] and Peter Sydorchuk[2]; [1]General Physics, State Pedagogical University, Drogobych, Ukraine; [2]General Physics, State Pedagogical University, Drogobych, Ukraine.

Smart narrow-gap semiconductor materials belonging to the A2B6 compounds are seemed to be of particular interest for design of optoelectronics devices used in automobile and vehicle navigation. The heterojunctions based on epitaxial layers ZnCdHgTe and wide-gap monocrystalline substrates CdTe (ZnTe) are of essential importance owing to the symplicity and reliability of the growth technology (modified liquid-phase epitaxy or pulse laser evaporation technique). Different methods of hetero-components doping give a wide range of possibilities to change the main parameters of the energy band diagram which appears as a principal characteristic of the active element. Furthemore, the studies of the charfe carriers characteristics (in particular, mobility, space distribution, lifetime, etc.) provide a sound information for simulation of active element as a whole. The investigation of the experimental data obtained from the symplest electric-field measurements apeears as a reasonable base for computing modeling of the graded-gap heterostructures. The abstract reports results of experiment and numerical simulation for active elements based on graded-gap heterostructures CdTe(ZnTe)/ZnCdHgTe. The main parameters of the energy band diagram are calculated and the numerical algorithm for the device simulation is also presented.

D10.11
Pulsed Electron beam Deposition - A Simple and Versatile Thin Film Deposition Technique for Developing Materials for Smart Systems.
K. S. Harshavardhan, M Strikovski and J Kim; Thin Films, Neocera, Inc, Beltsville, Maryland.

Pulsed Electron beam Deposition is a relatively new thin film deposition technique. A high current (1500 A), high voltage (15 kV) electron beam of about 100 ns pulse width, created in a low pressure Paschen discharge regime is used as the primary energy source to ablate a given target material resulting in the formation of a thin film on a substrate. The high power density at the target facilitated by the pulsed nature of the beam permits deposition of complex materials such as $YBa_2Cu_3O_7$, Ba-$SrTiO_3$ etc., with stoichiometric composition control. The Pulsed Electron Deposition, in several aspects, is similar to the well-established Pulsed Laser Deposition technique but differs from the fundamental viewpoint of beam-target interaction. The additional advantage of Pulsed Electron Deposition is realized in its ability to deposit optically transparent materials (with respect to the laser wavelength) such as SiO_2 and Al_2O_3 and some special polymers. The technique is scalable and cost effective and hence could become the preferred method in high throughput manufacturing environment where pulsed techniques are sought for accomplishing specific functionality (composition control, high density, deposition at lower temperatures etc) in the deposited films. This presentation will briefly introduce the basics of this deposition technology to the materials scientist and will present experimental data obtained on several technologically important smart materials systems such as high temperature superconducting films (YBCO, GdBCO), para-electric Ba-$SrTiO_3$, hard, Hydrogen-free Diamond like Carbon and PTFE films.

SESSION D11: Active MaterialS Systems and MEMS
Chairs: Yasubumi Furuya and Jiangyu Li
Thursday Morning, December 4, 2003
Room 202 (Hynes)

8:30 AM *D11.1
Selection of Materials and Sensors for Health Monitoring of Composite Structures.
S. Mark Spearing[1] and Seth Stovak Kessler[2,1]; [1]Aeronautics and Astronautics, MIT, Cambridge, Massachusetts; [2]Metis Design Corporation, Cambridge, Massachusetts.

Embedded structural health monitoring systems are envisioned to be an important component of future transportation systems. The principal challenge in designing an SHM system is the choice of sensors, and a sensor layout which can detect unambiguously the relevant structural damage. This paper focuses on the relationship between sensors, the materials of which they are made, and their ability to detect structural damage. Sensor selection maps have been produced which plot the capabilities of the full range of available sensor types vs. the key performance metrics (power consumption, resolution, range, sensor size, coverage). This exercise is used to define a system architecture utilizing piezoceramic sensors, operating in several modes (acoustic emission detection, strain detection, Lamb wave generation/sensing and global modal response). Experimental and analytical results are presented for in-situ damage detection in composite materials using piezoceramic sensors. Test results and corresponding analysis is presented for coupons and built up structures, including sandwich panels, stiffened panels and cylinders containing representative damage (delaminations, impact damage, off-axis ply cracking and penetrations). Modal analysis methods are shown to be effective for detecting the presence of some forms of damage in composite structures. but provide little information regarding the size, location or severity of the damage By comparison Lamb wave techniques are shown to be sensitive to all types of damage, and present the possibility of estimating damage location and size. The results of these analyses are discussed and conclusions for the architecture of future SHM systems are presented, in addition recommendations for the development of improved sensor configurations and materials are made.

9:00 AM *D11.2
Biomimetic Approach of the Composite Materials: Towards the Active Health Monitoring Concept.
Piere-Francois Gobin[1], M. Salvia[2], N. Godin[1], J. Courbon[1], P. Guy[1] and Y. Jayet; [1]Material Science, INSA, Villeurbanne, France; [2]IFOS, Ecole Centrale de LYON, Ecully.

The classical NDE techniques of periodical maintenance are just now evolving towards the continuous health monitoring of materials and structures. Taking account of the relation of this approach with the biomimetic notion of smart materials, it seems useful to extend this passive concept to a more active one specially in the case of composite materials. Effectively, if sensors and actuators are embedded in a composite structure before curing in order to monitor and improve the processing parameters, they are able, remaining in the structure, to assume the health monitoring and the optimization of the usage in the next stages of the life. Moreover, in slightly damaged systems, it becomes reasonable to use the results collected by the sensors for one on line tentative prediction of the residual life. Finally in the last stage of the life, when the material is deeply damaged, it is sometimes possible to slow down, and more rarely, to heal the damage.

9:30 AM *D11.3
Smart Materials and Nondestructive Evaluation.
Manabu Enoki[1] and Teruo Kishi[2]; [1]Department of Materials Enigineering, The University of Tokyo, Tokyo, Japan; [2]National Institute for Materials Science, Tsukuba, Japan.

The smart materials and structural systems are based on the concept to incorporate sensors, actuators, etc. in the materials themselves and to integrate (fuse) materials with the structures, thereby imparting such functions as to (1) self-evaluate the occurrence and deterioration of strains and damages, (2) self-control vibrations and noises, (3) self-change their own shape depending on conditions, and (4) self-control the propagation of damages and repair them as the need arises. We report the recent research and development project in Japan on the smart materials and structural systems, which was a project under the NEDO's (New Energy and Industrial Technology Development Organization) system to conduct research and development on industrial and scientific technologies in conjunction with colleges and universities. This report shows examples of application to composite materials such as, (1) development of health monitoring technology, (2) development of smart manufacturing technology, (3) development of active/adaptive structure technology, (4) development of materials and elements for actuator, and (5) airplane body demonstrator for demonstration test. Also recent

development of nondestructive evaluation technique such as laser based AE (acoustic emission) to detect microfracture process in smart materials will be reported.

10:30 AM D11.4
Tailoring Strains Through Microstructural Design.
Luc J. Vandeperre and William J. Clegg; Department of Materials Science and Metallurgy, University of Cambridge, Cambridge, United Kingdom.

When developing structures, which respond with a strain upon application of a stimulus such as temperature or an electric field, the fixed relation between the response of a given material and the magnitude of the stimulus puts severe constraints on the design. This problem is commonly dealt with either by design of the shape of the actuator or sensor, or by selecting a material with the appropriate coefficients in response to the field. However, when actuating or sensing functions need to be incorporated within other structures, shapes must be as simple as possible, and the environment where the structure is to be used can put limits on materials selection. Such limitations can be overcome by incorporating design into the microstructure of materials. By combining two materials into a structure, which allows levering of the strains, one can obtain a much wider range of coefficients than predicted by the rule of mixtures. For example, using aluminium and Invar, with respective coefficients of thermal expansion of 24×10^{-6} K^{-1} and 1.5×10^{-6} K^{-1}, a coefficient of thermal expansion as low as -360×10^{-6} K^{-1} was obtained. It will be shown how simple variations of the microstructure obtained by combining simple triangular elements can yield a wide variety of properties: isotropic and anisotropic coefficients, linear and non-linear responses, and materials with graded properties. Initial production of sheet-like materials was performed by a combination of photolithography and soldering. Predictions of both the coefficient of thermal expansion as well as mechanical properties will be compared with experiments on a model system of copper and Invar.

10:45 AM D11.5
Development of a wireless MEMS inertial system for health monitoring of structures. Ronald Kok, Cosme Furlong and Ryszard J Pryputniewicz; Mechanical Engineering Dept./CHSLT, Worcester Polytechnic Institute, Worcester, Massachusetts.

In this paper, advances in development of instrumentation and methodologies for wireless health monitoring of structures using MEMS inertial sensors are presented. Development of new materials and structures has increased the demand for accurate modeling of their mechanical behavior. Such modeling includes determination of dynamic properties of structures, such as their natural frequencies, mode shapes, and damping factors. Dynamic properties can be determined using analytical and computational methodologies, but are only limited to simple structures since they are insufficient to study complex structures. In order to study complex real-world structures, experimental modal analysis is applied. Rapid development of microelectromechanical systems (MEMS) have led to progressive designs of high-resolution, lightweight, and low-actuation power inertial sensors. These MEMS sensors provide an optimum alternative to traditional sensors, which have larger masses and may modify the dynamic characteristics of the structures being investigated. Studies presented in this paper include utilization of multiple MEMS inertial sensors to determine dynamic properties of structures. To ensure that the sensors are attached to appropriate locations for optimal results, both, Fourier and Wavelet analyses are utilized in order to perform time and frequency studies of the responses of specific test structures. To verify the results obtained with MEMS sensors, noninvasive methodologies, such as laser optoelectronic holography (OEH), are utilized to determine the natural frequencies, mode shapes, as well as to investigate the effects that attachment of MEMS sensors may have on the dynamic characteristics of the test structures. In addition, the test structures are also modeled with analytical and computational methods for correlation with the experimental results. Considering that multiple MEMS sensors are utilized, wiring and integration of those sensors might become complicated and monitoring of structures across long distances becomes limited. Therefore, a miniaturized wireless system that utilizes commercially available RF components is being developed and its current functionality is demonstrated.

11:00 AM D11.6
A New Techinque for Measuring Mechanical Response of Thin Released Films for MEMS. Rongjing Zhang, Doron Shilo, Guruswami Ravichandran and Kaushik Bhattacharya; Division of Engineering and Applied Science, California Institute of Technology, Pasadena, California.

The interest in micro actuation and sensing MEMS devices has given rise to the necessity for studying the mechanical response of thin released films, which are made of active materials such as ferroelectrics, ferromagnetics, and shape memory alloys. The trend to reduce the film thickness in these devices compel the development of new techniques, which enable loading in the sub-mN scale and take into account the fragility of these devices. We have developed a new technique for measuring static and dynamic mechanical response of thin released films, by either load control or displacement control in the range of 0.1mN - 1N. An optical system, which measures the deflection of a Laser beam, allows us to measure displacements in the sub-mum region and a sample holder with 4 adjustable degrees of freedom allows us to test specimens, which are integrated on a silicon wafer. The capabilities of the new technique are demonstrated with results on released films of ferroelectric (Ba1-xPbx)TiO3 and ferromagnetic shape memory alloy Ni2MnGa.

11:15 AM D11.7
Bi-Directional Motion Achieved with a Surfaced Micromachined Electrothermal MEMS Microengine.
Edward S. Kolesar, William E. Odom, Joseph A. Jayachandran, Matthew D. Ruff, Simon Y. Ko, Jeffery T. Howard, Peter B. Allen, Josh M. Wilken, Noah C. Boydston, Jorge E. Bosch, Richard J. Wilks and Justin B. McAllister; Department of Engineering, Texas Christian University, Fort Worth, Texas.

Several microactuator technologies have been investigated for positioning individual elements in large-scale microelectromechanical systems (MEMS). Electrostatic, magnetostatic, piezoelectric and thermal expansion represent the most common modes of microactuator operation. This research is focused on the design and experimental characterization of two types of MEMS asymmetrical electrothermal microactuators. The motivation is to present a unified description of the behavior of the electrothermal actuator so that it can be adapted to a variety of microsensor and microactuator applications. Both microactuator design variants use resistive (Joule) heating to generate thermal expansion and movement. Deflection and force measurements of both microactuator variants as a function of applied electrical power are presented. Also described is the practical integration of the electrothermal microactuators in a monolithic microengine that has been operated in a bi-directional mode to control the position of a mechanical shuttle. Postional control of the mechanical shuttle has been achieved with a resolution of 1 micron.

11:30 AM D11.8
Piezoelectric Actuators for Synthetic Jet Applications.
Karla Mossi[1] and Rob Bryant[2]; [1]Virginia Commonwealth University, Richmond, Virginia; [2]NASA Langley Research Center, Hampton, Virginia.

Synthetic jets have been identified and utilized widely in airflow control applications. These jets of air are usually created by the use of compressed air, or an electromechanically driven vibrating platform. All of these approaches produce desired results in airflow-control such as enhanced lift and increased maneuverability. Despite the results however, system weight, size, response time and force limit their use in aircraft applications where space is a premium. The objective of this study is to characterize the relevant properties for the design of a synthetic jet utilizing three types of piezoelectric actuators as mechanical diaphragms. The limiting parameters of the actuators for this application are shape and volumetric space. Thus, the actuators were circular with a diameter of 6.35 cm, and overall device cavity volume no larger than 147.5 cm3 on a 7 cm x 7 cm areal coverage. The actuators tested were pre-stressed curved metallic unimorphs, bimorphs, and radial field diaphragms. These piezoelectric elements were chosen because of their geometry, quasi-isotatic topography and overall free-displacement. Each actuator was affixed about its perimeter in a cavity, and relevant parameters such as clamped displacement variations with voltage and frequency using applied pressure (load) differentials was measured. Also, the air velocities produced through apertures of varying sizes were measured using the same mounting configuration. Other properties measured include calculated and experimental volume displacement and pressure versus displacement.

11:45 AM D11.9
Design for Compressive or Tensile Strain in Spherically-Deformed Circuit Foils and Electrical Implications. Pai-hui Iris Hsu[1], Min Huang[1], Helena Gleskova[1], Zhichen Xi[2], Zhigang Suo[1], Sigurd Wagner[1] and James C Sturm[1]; [1]Center for Photonics and Optoelectronic Materials, Princeton University, Princeton, New Jersey; [2]Center for Composite Materials, University of Delaware, Newark, Delaware.

Many proposals for future sensors and actuators depend on stretchable and deformable electronics fabricated on thin foil substrates, e.g. smart electronic skins that can be deformed to arbitrary shapes. The most common type of deformation is to roll a thin foil substrate into a cylinder. For such deformation, the strain in any devices on the substrate or foil surface can be minimized by using thin substrates. However, for more complicated shapes, such as a spherical cap shape, deformation of the substrate (from a flat initial state) requires that it be put into considerable tension even for thin substrates. This can cause failure in devices on the substrate surface. In this work, we show that if the devices are placed in hard "islands" on the substrate surface, depending on the choice of structure, not only can the strain in devices be kept small, but also the sign of the strain can be either positive or negative. This is shown by numerical modeling and confirmed by experimental data demonstrating the effects of spherical deformation on the electrical performance of amorphous silicon thin-film transistors (TFTs) on polyimide substrates in such device islands. TFTs were fabricated using a conventional amorphous silicon process in islands on a planar, thin polyimide (Kapton) foil substrate, and the substrate was then plastically deformed into a spherical dome shape subtending up to a 66° field-of-view (one steradian). Planar islands of "hard" device materials were in tension like the substrate, but deep mesas were etched into the substrate underneath the hard device islands. These mesa islands could actually be in compression even if the overall substrate strain was as high as 5% tension. The sign and magnitude of the change in the mobility of a-Si TFTs tracked the sign and magnitude of the island strain, with compression leading to a lower mobility and tension a higher mobility. This work shows that it is possible to fabricate devices with structures less sensitive to the substrate deformation so it endures little or no strain even if the substrate is expanding. The concept of controlling and tuning the strain and its sign (either tension or compression) during deformation will be useful for other smart materials and devices in electronic skins.

SESSION D12: Magnetic and Metallic Based Smart Materials

Chairs: Kanryu Inoue and Eckhard Quandt
Thursday Afternoon, December 4, 2003
Room 202 (Hynes)

1:30 PM *D12.1
The remanence enhancement in magnetically interacting particles. Jiangyu Li and Heliang Qu; Engineering Mechanics, University of Nebraska-Lincoln, Lincoln, Nebraska.

In this talk, we report an effective medium theory on the remanence of magnetically interacting particles to demonstrate the effect of inter-granular magneto-static interactions on the remanence enhancement of materials, which agrees excellently with micromagnetic simulations. A dimensionless parameter lambda measuring the competition between anisotropy energy and magneto-static energy is defined, which completely characterizes the remanence of magnets when the exchange coupling is negligible. Three distinct regimes were observed: lambda<0.1 for hard magnets, where anisotropy energy dominates and little remanence enhancement is observed; 0.1 < lambda < 1 for intermediate magnets where up to 50% remanence enhancement is observed due to the inter-granular magneto-static energy; and lambda>1 for soft magnets, where the dominance of magneto-static energy leads to much reduced remanence in the materials.

2:00 PM D12.2
Repeatable Magnetic-field-induced Deformation and Magneto-mechanical Fatigue of Ni-Mn-Ga Ferromagnetic Martensites. Peter Muellner[1], Volodymyr A Chernenko[2] and Gernot Kostorz[1]; [1]ETH Zurich, Zurich, Switzerland; [2]Institute of Magnetism, Kiev, Ukraine.

Magnetoplasticity, i.e. the magnetic-field-induced deformation of ferromagnetic shape-memory alloys, and its inverse effect, i.e. the deformation-induced change of magnetization, can be used for building actuators and sensors. While magnetic-field-induced strains - and more recently also magnetic-field-induced stresses - have been studied intensively since 1996, information about the repeatability and long-time stability of these properties is very limited. In this study, cyclic magneto-mechanical experiments were performed with Ni-Mn-Ga alloys exhibiting 5-layered modulated tetragonal and 7-layered modulated orthorhombic martensites. The length of the samples was measured in a rotating magnetic field, and the magnetic-field-induced strain was evaluated as a function of the number of field cycles. More than 10^7 cycles were applied. Depending on thermo-mechanical history and the martensite structure, the evolution of magnetic-field-induced strain varies dramatically. For 7-layered orthorhombic martensite, the field-induced strain can increase during cycling up to 9.7%, but it also can decrease rapidly to less than 0.5%. For 5-layered tetragonal martensite, repeatable strains up to 6% were measured. In orthorhombic martensite, the cyclic field-induced strain is less stable than in tetragonal martensite. The different magneto-mechanical long-term stabilities result from differences in crystal symmetry. For tetragonal martensite with uniaxial magnetic anisotropy, there is only one preferred martensite domain (out of three) for a given field direction. Therefore, there is a well-defined deformation path upon a change of magnetic field direction. This results in a stable cyclic field-induced strain. In contrast, there are always two preferred martensite domains (out of six) for orthorhombic martensite. Therefore, the deformation path is not unique; different options are available upon magnetic field variations. This results in a variable cyclic field-induced strain and less reproducible magneto-mechanical fatigue.

2:15 PM D12.3
Magnetization Process Associated with Rearrangement of Martensite Variants in Iron Based Ferromagnetic Shape Memory Alloys. Takashi Fukuda[1], Tatsuaki Sakamoto[1], Tomoyuki Terai[1], Tomoyuki Kakeshita[1] and Kohji Kishio[2]; [1]Department of Materials Science and Engineering, Graduate School of Engineering, Osaka University, Suita, Osaka, Japan; [2]Department of Superconductivity Engineering, Graduate School of Engineering, University of Tokyo, Bunkyo-ku, Tokyo, Japan.

Fe-31.2Pd(at.%) and Fe_3Pt are ferromagnetic shape memory alloys whose transformation temperature is about 230 and 85 K, respectively. Both alloys exhibit a large magnetic field-induced strain (MFIS) in association with rearrangement of martensite variants. Especially, concerning the Fe_3Pt, a part of MFIS recovers spontaneously in the field removing process. In this presentation, we show the magnetization process of Fe-31.2Pd and Fe_3Pt, and discuss the mechanism of MFIS. The magnetization curves measured in the martensite state of these alloys exhibit a hysteresis between field applying and removing processes, whose area corresponds to the energy dissipated during the rearrangement of variants. The area depends on characteristics of MFIS. In case a large MFIS appears in the field applying process but it does not recover in the field removing process, the area is in the order of 100 kJ/m^3. This is the same order as that has been reported for Ni-Mn-Ga alloys. On the other hand, in case the MFIS induced by the magnetic field recovers spontaneously in the field removing process, the area is in the order of 10 kJ/m^3. The uniaxial magnetocrystalline anisotropy constant is also evaluated from their magnetization curve, and it is about -350 kJ/m^3 for Fe-31.2Pd at 77 K, and is about 500 kJ/m^3 for Fe_3Pt at 4.2 K. These values are the same order as that of Ni-Mn-Ga alloys, supporting the macroscopic explanation of MFIS in ferromagnetic shape memory alloys that the MFIS is caused by the magnetic energy difference between adjacent variants under a magnetic field.

2:30 PM D12.4
Microstructure and Magnetostriction of Rapid-Solidified Fe-Ga System Alloys. Chihiro Saito[1], Yasubumi Furuya[1], Teiko Okazaki[1], Takashi Matsuzaki[2], Tadao Watanabe[2] and Manfred Wuttig[3]; [1]Intelligent Machines and System Enginieering, Hirosaki University, Hirosaki, Japan; [2]Graduate School of Engineering, Tohoku University, Sendai, Japan; [3]Materials and Nuclear Engineering, University of Maryland, College Park, Maryland.

It was reported that melt-spun, rapid solidified Fe-Ga ribbon sample exhibited large magnetostriction and good ductility as compared with conventional bulk sample. In order to reconfirm the occurrence of large magnetostriction in Fe-Ga ribbon sample, the correlation between magnetostriction and the crystal grain morphology has been inspected in detail by SEM/EBSP method for Fe-15at%Ga alloy. In comparison with as-spun ribbon sample, short-time (0.5 h) heat treated ribbon has stronger orientation to [100] and it has exhibited larger magnetostriction (130ppm) at 800 kA/m. These phenomena suggest that such a large magnetostriction is caused by the release of considerable large internal stresses in as-spun ribbon as well as the remained strong textures after annealing. The following conclusion was obtained. 1. Fe-Ga rapidly solidified ribbon consists of many low angle grain boundaries and strong texture. 2. Texture is formed in near [100], and that is concentrated by short-time heat treatment. 3. Largest magnetostriction occurs in short heat-treated Fe-15at%Ga ribbon. That results from both released internal stresses and stronger orientation to [100]. Bulky Fe-Ga actuator/sensor materials have been developed by combining the laminated rapid-solidified ribbons with spark plasma sintering method. Reference 1. A.E.Clark, J.B.Restorff, M.Wun-Fogle, T.A.Lograsso, D.L.Schlagel. IEEE Trans. Magn. 36 (2000) 3238 2. Y.Furuya, C.Saito, T.Okazaki, JIM 66 (2002) 901-904 (in Japanease)

2:45 PM D12.5
Magnetostriction of Field-Structured Composites.
James E. Martin[1], Robert A. Anderson[1] and Gerald Gulley[2,1]; [1]1122, Sandia National Labs, Albuquerque, New Mexico; [2]Physics, Dominican University, River Forest, Illinois.

Field-structured magnetic particle composites are an important new class of materials that have great potential as both sensors and actuators. These materials are synthesized by suspending magnetic particles in a polymeric resin and subjecting these to magnetic fields while the resin polymerizes. If a simple uniaxial magnetic field is used, the particles will form chains, yielding composites whose magnetic susceptibility is enhanced along a single direction. A biaxial magnetic field, comprised of two orthogonal ac fields, forms particle sheets, yielding composites whose magnetic susceptibility is enhanced along two principal directions. A balanced triaxial magnetic field can be used to enhance the susceptibility in all directions, and biased heterodyned triaxial magnetic fields are especially effective for producing composites with a greatly enhanced susceptibility along a single axis. Magnetostriction is quadratic in the susceptibility, so increasing the composite susceptibility is important to developing actuators that function well at modest fields. To investigate magnetostriction in these field-structured composites we have constructed a sensitive, constant-stress apparatus capable of 1 ppm strain resolution. The sample geometry is designed to minimize demagnetizing field effects. We have demonstrated field-structured composites with nearly 10,000 ppm strain, and have shown that at large magnetic fields a structural phase transition occurs within the composite. These experimental results are compared to microscopic, self-consistent field simulations of magnetostriction in these complex, disordered materials.

3:30 PM *D12.6

Development of Strain Sensors Based on Magnetostrictive Tunneling Junctions. Markus Loehndorf[1], Stefani Dukupil[1], Manfred Ruehrig[2], Joachim Wecker[2] and Eckhard Quandt[1]; [1]caesar (Center of Advanced European Studies and Research), Bonn, Germany; [2]Corporate Technology, Siemens AG, Erlangen, Germany.

Micrometer-sized highly sensitive strain sensors are presented. The sensors are based on magnetic tunneling junctions (MTJs) incorporating magnetostrictive free layers. Usually, nearly zero-magnetostrictive soft magnetic materials are chosen as free layer, in order to avoid interaction caused by stress or strain. In this study, however, we have intentionally used highly magnetostrictive materials and alloys as free layers to in fact promote and govern strain sensitivity. Results for magnetostrictive Fe50Co50 materials or amorphous Co- or Fe-based alloys serving as sensing (or free) layers are discussed in view of possible applications. In addition MTJ based sensor devices have been prepared on polymer substrates. As a result MTJ with magnetostrictive free layers show gauge factors on the order of 600 which are a factor of 3 better than silicon based strain sensors [1], and due to the small sizes of MTJ they are possible candidates for strain sensing with a very high spatial resolution. Future work will focus on the investigation of highly strain sensitive MTJ's operating at zero magnetic bias fields as well as on the preparation of strain sensor arrays. Financial support was given by the German Ministry for Education and Research (BMBF, grant # 13N7943). [1] M. Loehndorf et al. Applied Phys. Lett . 81(2), 313 (2002).

4:00 PM D12.7

High frequency devices with integrated magnetoelastic materials. Michael Frommberger and Eckhard Quandt; smart materials, caesar, Bonn, Germany.

A wireless mechanical stress sensor based on thin films showing the inverse magnetostrictive effect will be presented. The magnetostrivtive films are incorporated in the inductance L of an electrical LC resonator (LC-tag). Such sensors are interesting for wireless measurements of mechanical quantities such as stress or torque on rotating or hidden objects. Their operating frequencies can be up to several GHz limited by the ferromagnetic resonance frequency of the used material and its eddy current losses. Such a stress sensor based on soft magnetic magnetostrictive FeCoBSi thin films has been fabricated and characterized. In a bending test jig, the sensor shows a high sensitivity exceeding most comercial available strain gauge elements. This sensor can be used in a wide range of different applications of wireless measurements of mechanical values. Besides the optimization of the sensor layout with regard to the desired frequency range future research efforts will also focus on improving the different microstructuring techniques and the continuation of high frequency magnetic material development suitable for those applications. Our current efforts in material design and process development will be presented and their benefits will be discussed.

4:15 PM D12.8

Development of Texture-controlled Bulky Actuator/Sensor Materials by Combining Rapid-solidified Fiber/Ribbon Elements with Spark Plasma Sintering/Joining(SPSJ).
Yasubumi Furuya[1], Masanori Yokoyama[1], Chihiro Saito[1], Teiko Okazaki[2] and Mamori Omori[3]; [1]Intelligent Machines and Systems Engineering, Hirosaki University, Hirosaki 036-8561, Japan; [2]Material Science and Engineering, Hirosaki University, Hirosaki 036-8561, Japan; [3]Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan.

It has been shown that rapid-solidified(RS) melt-spun fiber/ribbon/foil type samples can have mope high performance of the metallic actuator/sensor properties by the authors(1). However,as for the applications of these sample materials in smart material systems, the developed rapid-solidified actuator samples are inevitably too small force to move the machines and structures in engineering applications. Then,in this paper, we propose one novel material processing approach that can produce the bulk type solid-state actuator/sensor materials by combining the rapid-solidified fiber/ribbon with very short time spark plasma sintering/joining (SPSJ) method.(2) First, we made the pre-forms for bulky composites which consist of 1)stacked layers of RS-ribbons or 2)the crashed RS-fibers by ball-milling. Then, the green-compact or stacked layers of RS-material elements are sintered or joined under electric pulse currents with short time within a few minutes in the dies under compressive stresses at high temperature. The produced samples, 1)the disk-type sample from stacked layers of RS-ribbons and 2)the sintered compact from ball-milled RS-fibers of Ti50Ni40Cu10at% alloy, showed clear thermoelastic phase transformation by DSC evaluation as well as shape recovery. These experimental results show us the effectiveness of the proposed material processing method. The same approach was also tried for developing bulky magnetostrictive FeGa alloy at Hirosaki University. References:
(1)Y.Furuya;Proc.MRS,vol.604(2000)pp.108-116
(2)Y.Furuya,H.Yaguchi,M.saito,M.Taya and R.Watanabe; Proc.5thICIM(1998,Tokyo)pp.64-67

4:30 PM *D12.9

The development of active and sensitive structural material systems. Hiroshi Asanuma, Dept. of Electronics & Mechanical Eng., Chiba University, Chiba-shi, Japan.

This paper describes the new concepts the author has proposed and demonstrated to realize metal and polymer based active and sensitive structural material systems suitable for smart structures. The following topics are mainly examined: Fabricating multifunctional composites by using conventional structural materials - 1) an active laminate of CFRP/Al of which unidirectional actuation is realized by electrical resistance heating the CFRP layer and its curvature change can be monitored using optical fiber multiply fractured in the CFRP layer, and 2) a multifunctional aluminum matrix composie where oxidized titanium fiber is embedded for sensing temperature and strain, and generation of heat for actuation.

SESSION D13: Materials and Devices for Smart Systems
Chairs: Yasubumi Furuya and Eckhard Quandt
Friday Morning, December 5, 2003
Room 202 (Hynes)

8:30 AM *D13.1

Ferromagnetic Shape Memory Alloys: Recent Advances.
R. C. O'Handley[1], S. M. Allen[1], C. P. Henry[1,2], M. Marioni[1], M. Richard[1], J. Feuchtwanger[1], B. Peterson[1], D. Bono[1], J. K. Huang[1], D. I. Paul[1], X-J Jin[1,3], Robin Ivester[1], Ryan Wager[1], Catherine Jenkins[1] and Kelli Griffin[1]; [1]Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; [2]Present Address:, Fortis Technologies, Thousand Oaks, California; [3]Present Address:, Shanghai Jiao Tong University, Shanghai, China.

Ferromagnetic shape memory alloys (FSMAs), most notably those based on Ni-Mn-Ga, have received considerable attention because of the remarkable 6% to 9% field-induced strains they have exhibited at room temperature in magnetic fields of order 0.5 T. The stress above which the field-induced strain in FSMAs is blocked is currently 2 to 4 MPa. This is to be compared to blocking stresses of over 100 MPa and several tens of MPa for piezoelectric and magnetostrictive materials, respectively. Recent progress in understanding and using these materials will be reviewed. Ni-Mn-Ga samples that show relatively easy twin boundary motion near room temperture fall in a narrow composition range that includes both tetragonal and orthorhombic martensites. FSMA samples measuring several mm on edge show a bandwidth of approximately 1 or 10 kHz for continuous wave excitation or pulse field excitation, respectively. Application of sub-milli-second field pulses of amplitude greater than the anisotropy field (about 0.6 T) do not induce greater strain, but only increase the speed at which full strain is achieved. While the curves of strain versus alternating field amplitude appear smooth, studies of individual twin boundary motions show them to be highly irregular. Because $actuator applications$ seem appropriate for Ni-Mn-Ga FSMAs given the large values of $d_{ij} = \delta\epsilon_i/\delta \mathrm{H}_j \approx 1$ to 3×10^{-7} /A, significant efforts have been aimed at developing small pumps, valves and underwater transducers. The equally large values of $d_{ij} = \delta \mathrm{M}_i/\delta\sigma_j$ in Ni-Mn-Ga suggest $sensor applications$, but there is no published work directed toward these goals. The large hysteresis associated with twin boundary motion has been exploited to demonstrate superior $vibration energy absorption$ in Ni-Mn-Ga-loaded composites compared to Terfenol-loaded polymers.

9:00 AM *D13.2

Fundamental Investigation Of Ferromagnetic Shape Memory Alloys – *In – Situ* Study Of Temperature Dependent Magnetic And Thermo-Elastic Correlated Behavior. Matthew R. Sullivan[1], Daniel A. Ateya[1], Steven Pirotta[1], Ashish A. Shah[1], G. H. Wu[2] and Harsh Deep Chopra[1]; [1]Mechanical & Aerospace Engineering Department, State University of New York at Buffalo, Buffalo, New York; [2]State Key Laboratory for Magnetism, Institute of Physics, Chinese Academy of Sciences, Beijing, China.

Ferromagnetic shape memory alloys (SMAs) belong to a class of complex correlated systems whose physical properties depend on interaction across two or more energy regimes – magnetic and thermo-elastic energy in the case of ferromagnetic SMAs. This talk will focus on results obtained from experiments in which the micro-magnetic, structural, domain dynamics and thermodynamic properties of these alloys were measured and observed *in – situ* and at the same time. Simultaneous study of these properties has enabled a fundamental understanding of some key issues that govern the properties of these alloys. The talk will focus on results obtained from oriented single crystals of Ni-Mn-Ga and Fe-Pd. Some key results include formation and dynamics of magnetic vortices at the parent (austenite)-product (martensite) phases, which provides clear evidence that the length scales of magnetic domains are a function of the lattice parameters of the martensite phase. Additionally clear evidence is found of a series of magnetic and structural transformations, as well as precursors just above the martensite transformation temperature. Finally, from a practical viewpoint, these results directly pinpoint the origin of high coercivity, an issue of practical concern where high loads/displacements are needed at low switching fields and coercivity. This work was supported by DOE Grant No. DE-FG02-01ER45906, Office of Basic Energy Science, and this support is gratefully acknowledged.

9:30 AM D13.3

Synthesis And Characterization of Sputtered FePt-Based Alloy Thin Films. Jinn P. Chu[1], T. Mahalingam[2,1], S. F. Wang[3] and K. Inoue[4]; [1]Institute of Materials Engineering, National Taiwan Ocean University, Keelung 202, Taiwan; [2]Department of Physics, Alagappa University, Karaikudi-630 003, India; [3]Department of Materials and Minerals Resources Engineering, National Taipei University of Technology, Taipei 106, Taiwan; [4]Department of Materials Science and Engineering, University of Washington, Seattle 98195, Washington.

FePt alloy thin films have attracted significant interest owing to its potential application in magnetic recording. FePt alloys exhibit very high magneto crystalline anisotropy energy, high coercivity and large energy products. In the present study, polycrystalline FePt-based thin films were prepared by an r.f. magnetron sputtering technique onto silicon wafer substrates. The deposited films are annealed between 400 and 800°C in order to transform the soft magnetic fcc phase to the hard magnetic fct phase. The effects of film composition and annealing treatment on the microstructure and magnetic properties of FePt alloy thin films are investigated in detail. X-ray diffraction studies on annealed $Fe_{54}Pt_{46}$ films at 600°C revealed an ordered fct $L1_0$ phase whereas annealed $Fe_{78}Pt_{22}$ and $Fe_{85}Pt_{15}$ exhibited disordered $FePt_3$ and Fe_3Pt phases, respectively. The long-range order parameter for argon-quenched film at 600°C is estimated to be 0.90±0.01. The grain size of $Fe_{54}Pt_{46}$ film is measured to be in the range between 60 and 80nm. A maximum in-plane coercivity of 9.75kOe is obtained for argon-quenched film at 600°C. The saturation magnetization is found to increase with ferrous content in the alloy films. The large values of Ms and $Hc_{\parallel}$ for $Fe_{54}Pt_{46}$ may be attributed to the predominant existence of pure γ_1-FePt phase in the sample. It is observed that argon quenched films exhibited larger saturation magnetization than RTA processed films. Argon quenched and RTA processed films exhibited micro-twins in scanning electron microscopy analysis. The appearance of micro-twins in annealed films may be attributed to the planar defects in these films. Moreover, these micro-twins are found to be beneficial to improve the magnetic hardness and properties of FePt films. The large value of saturation magnetization obtained from M-H hysteresis indicates the predominant existence of hard fct-γ_1 phase. The combined effects of twins and the hard fct-γ_1 phase on the magnetic properties are discussed.

9:45 AM D13.4

Micromagnetic Modeling of the Behavior of Magnetostrictive Films under Stress. Yi-Chung Shu, Institute of Applied Mechanics, National Taiwan University, Taipei, Taiwan.

We have developed a framework based on micromagnetics to explore the effect of stress on the magnetostrictive behavior in ferromagnetic films. Our approach is different from the conventional one which simply replaces the total strain by magnetostrain. Question arises for such an approach because of the loss of strain compatibility. Here, we have included the kinematic constraints in our micromagnetic model and developed a fast algorithm to calculate the intrinsic stress induced by incompatible magnetostrain. We have shown that for small magnetostriction of the order of 10^{-5}, the results predicted by the present approach are slightly different from those predicted by the conventional method. But we have found that for large magnetostriction around 10{-3} order of magnitude such as Terfenol-D, the conventional approach is insufficient to predict magnetic domain patterns and hysteresis precisely, and the effective magnetic field induced by intrinsic stress cannot be neglected.

10:30 AM D13.5

Thermal Analysis And X-Ray Diffraction Study Of Ferromagnetic Fe-Pd-Pt-Based Shape Memory Alloys. Gwon-Seung Yang[1], Reid Jonasson[2], Seung-nam Baek[1], Shozo Inoue[3], Kinzo Murata[4], Keiji Koterazawa[3], Soon-jong Jeong[5], Kiyoshi Mizuuchi[6] and Kanryu Inoue[2]; [1]Materials Science and Engineering, Chosun University, Kwang Ju, South Korea; [2]Materials Science and Engineering, University of Washington, Seattle, Washington; [3]Mechanical and Intelligent Engineering, Himeji Institute of Technology, Himeji, Hyogo, Japan; [4]Kobe Material Testing Lab. Co., Hyogo-ku, Kobe, Japan; [5]Korean Electric and Magnetic Devices Group, Korea Electrotechnology Research Institute, Changwon, South Korea; [6]Osaka Municipal Technical Research Institute, Osaka, Japan.

Ferromagnetic shape memory alloys have received considerable attention in the past several years because of their potentiality as elements of high performance and power actuators due to their ferromagnetic nature of martensite phase. At present, there are only five alloy systems that show shape memory effects when their thermally induced ferromagnetic martensite goes back to its austenite phase upon heating. Magnetically induced ferromagnetic martensite, on the other hand, leads to shape change but no macroscopic shape recovery has been detected upon the removal of magnetic field. Similar shape change also occurs when ferromagnetic martensite variants are rearranged by magnetic field, but no shape recovery occurs when magnetic field is removed. Recently, we have encountered one incident where some shape recovery was observed upon the removal of magnetic field. Hence, we believe there are some conditions under which macroscopic shape memory effects should occur when magnetic field-induced ferromagnetic martensite is reverted to its austenite phase. In the present study, we have investigated ternary Fe-Pt-Pd alloys in an attempt to develop ductile ferromagnetic shape memory alloys appearing at around room temperature. Several alloys with the compositions of Fe-(25-x) at% Pt-x at% Pd and Fe-y at% Pt-(30-y) at% Pd were prepared by arc melting, followed by hot rolling and homogenization. Homogenized alloys were electro discharge machined to make specimens of about 2x2x50mm and heat treated in vacuum for atomic ordering in encapsulated quartz tubes, and quenched into iced water. Ordering treated alloy samples were then thermally analyzed using a DSC unit and x-ray diffracted using a diffractometer. All alloys investigated have martensite and austenite transformation temperatures, which are much lower than Curie temperature. Among those alloys, one Fe-23at%Pt-2at%Pd alloy shows good shape memory effects near room temperature. Structural analyses and thermal analyses will be presented in detail.

10:45 AM D13.6

Compression Properties Of Ferromagnetic Ni_2MnGa-Based Shape Memory Alloy With/Without Magnetic Field Loading. Kinzo Murata[2], Tamotsu Shimada[3], Soon-jong Jeong[4], Takashi Kira[3], Shozo Inoue[3], Keiji Koterazawa[3] and Kanryu Inoue[1]; [1]Materials Science and Engineering, University of Washington, Seattle, Washington; [2]Kobe Material Testing Lab. Co., Hyogo-ku, Kobe, Japan; [3]Mechanical and Intelligent Engineering, Himeji Institute of Technology, Himeji, Hyogo, Japan; [4]Korean Electric and Magnetic Devices Group, Korea Electrotechnology Research Institute, Changwon, South Korea.

In past several years ferromagnetic Heusler-type compound Ni_2MnGa has received considerable attentions for its potential applications as high performance and energy-density actuators. The compound has systematically been studied to clarify the shape change associated with the martensite-to-austenite transition under stress and/or temperature loading at constant magnetic field and under magnetic field loading at constant stress and temperature. In the series of research efforts, the shape change in [221] for a Ni-19.5at.%Mn-27at.%Ga single crystal is found to be extremely small when temperature is either decreased or increased without magnetic field, while it is about 1.3% under a magnetic field of 3 kOe applied along the [672] direction. This magnetic-field-assisted thermally-induced shape change is reversible and associated with the formation of favorably oriented martensite variants under magnetic field. Similar shape change also occurs at a constant compressive stress when magnetic field was applied. The present study was dealt with the effect of compressive deformation behavior of polycrystalline Ni_2MnGa-based compounds at various temperatures with and

without magnetic field. The compound compositions used were around Ni-25at.%Mn-23at.%Ga, where the transition occurred near room temperature. It was found that triggering stress is lowered when deformed under constant magnetic field at temperatures below M_s, while such stress is little affected when deformed at temperatures above A_f. In the former case the compound could be deformed repeatedly without failure, showing ductile behavior, while in the latter it failed after several cycles of deformation. The ductile and brittle behavior appears associated with the readiness of rearrangement of martensite variants. Magnetic field effect on stress was also investigated under constant stress and it was found that there is a threshold stress, above which magnetic-field-induced strain increases linearly with magnetic field when deformed at temperatures near Ms. This magnetic-field-induced stress-assisted strain was irreversible at the experimental conditions used.

11:00 AM D13.7
Coupled Magneto-mechanical Modeling of Ferromagnetic Particle Reinforced Composites. Lizhi Sun and H.M. Yin; University of Iowa, Iowa City, Iowa.

The objective of this work is to investigate effective magneto-mechanical behavior of composites containing aligned ferromagnetic particles. We first solve the local magnetic field, magnetic force and elastic field for pair-wise interacting magnetic particles embedded in the infinite domain subject to applied magnetic and mechanical loads. We further derive the effective (homogenized) magnetoelastic properties and effective magnetostriction of the composites. For magnetorheological elastomer, the configuration of the microstructure rapidly changes with the magnetic and mechanical loading due to the small elastomer's stiffness, so the local magnetic field and elastic field are fully coupled. This model accounts for the coupled magneto-elastic interaction and pair-wise interaction between particles. We finally compare the proposed model with available experimental data and other models (Davis, L.C., 1999, J. App. Phys. 85 3348-3351; Jolly, M.R., et al, 1996, Smart Mater. Struct. 5 607-614).

SESSION D14: Sensor Materials and Devices
Chairs: Yasubumi Furuya and Minoru Taya
Friday Afternoon, December 5, 2003
Room 202 (Hynes)

1:30 PM D14.1
Piezoresistive Properties of Ceramic Strain Gages with Controlled Nanoporosity. Otto J. Gregory and Tao You; Chemical Engineering, University of Rhode Island, Kingston, Rhode Island.

A ceramic strain gage based on reactively sputtered indium-tin-oxide (ITO) thin films is being developed to monitor the structural integrity of components employed in advanced aerospace propulsion systems that operate at temperatures in excess of 1500C. Electrical and chemical stability is particularly critical in these harsh environments, since these ceramic strain gages must survive tens of hours of strain testing at elevated temperatures. SEM micrographs of the surfaces of these strain gages after high temperature exposure revealed a partially sintered microstructure consisting of a contiguous network of nano-sized ITO particles with well defined necks. Electrical conduction along the surfaces of these contiguous ITO particles resulted in a very stable and large piezoresistive response at temperatures as high as 1575C. It appeared that densification of the ITO particles was retarded during high temperature exposure with nitrogen playing a key role in stabilizing the nanoporosity. To prepare this nanoporous ITO, sputtered ITO films were subjected to a post deposition anneal at 700C in nitrogen and subsequently exposed to high temperature. Based on these preliminary results, ITO strain sensors were reactively sputtered in various nitrogen/oxygen/argon environments. SEM and AFM indicated that although the microstructures of these nitrogen-sputtered films were similar in appearance to those produced by a post deposition anneal in nitrogen, the average pore size and particle size were an order of magnitude smaller. It appears that nitrogen was metastably retained in the individual ITO grains during sputtering and diffused out of the bulk grains at elevated temperature, eventually becoming trapped at grain boundaries and triple junctions. Under these conditions, sintering and densification of the ITO particles containing these nitrogen rich grain boundaries was retarded and a contiguous network of nano-sized ITO particles was established. Static strain testing of the nitrogen-sputtered ITO sensors indicated that a similarly stable and responsive strain gage could be reproduced using this approach. The high temperature-piezoresistive behavior of ITO strain gages prepared with controlled nanoporosity is presented within and the potential impact on other types of ceramic sensors will be discussed.

1:45 PM D14.2
Automated Amperometric Immunosensor for Detection of Pathogens in food samples. Sireesha Chemburu and Ebtisam Wilkins; Chemical and Nuclear Engineering, University of New Mexico, Albuquerque, New Mexico.

We describe the development of an amperometric flow-through electrochemical biosensor that can be applied to the identification and quantification of pathogenic bacteria such as E .coli, Listeria and Campylobacter in various food matrices (apple juice, milk and chicken) on the spot and in a short time. The system is able to detect very low concentrations of the bacterial cells in the samples. The sensor is sensitive, rapid, small, inexpensive, disposable, and can be operated by untrained personnel. The prototype has [removed] potential for being used in the food industry as the assay time is only 30 minutes and the results obtained are reliable and repeatable. Point introduction of the sample is done and pre-enrichment of the sample is not required

2:00 PM D14.3
Development of a New Design of Immunosensor for the Rapid Detection of Pathogens. Eric Carnes and Ebtisam Wilkins; Chemical and Nuclear Engineering, University of New Mexico, Albuquerque, New Mexico.

A portable, flow-through amperometric immunoassay system utilizing a newly designed immunosensor has been developed for the detection of pathogens. A sandwich scheme of immunoassay involving two stages of immunoreaction was employed using Escherichia coli as a model. E. coli antibodies were immobilized on Toray carbon paper to create a disposable immunofiltration membrane. Detection of the bacteria was achieved by using horseradish peroxidase-labelled antibodies, which, when coupled with a substrate of hydrogen peroxide and sodium iodide, yielded an electro-reduction which was measured amperometrically at a potential of 0.150 V. The immunosensor is highly sensitive and selective through the use of a disposable immunofiltration membrane that also functions as an immunoelectrode, which enhances the rate of the immunoreaction due to the high surface to volume ratio of the solid to liquid phases. The system was designed to be simple and effective, as well as cost effective and rapid. Comparison of the performance of this system with conventional immunoassays demonstrated a significantly shorter time for the assay. Optimization of the parameters of the system was conducted, resulting in an overall assay time of 17 minutes when a flow of 200 mL/min is used. The first stage of immunoreaction of immobilized antibodies with the analyte was found to saturate in 2 minutes. The second stage of immunoreaction with the enzyme-labelled conjugates exhibited a more complex behavior but became saturated in 6 minutes. The amperometric detection using the substrate gave a steady output signal after 3 minutes. All stages are separated by 2 minutes of washing by a rinsing buffer solution, and the whole analysis is preceded by a 2-minute pre-washing stage in order to eliminate the problem of non-specific binding of immunoagents. The working range for the immunoassay was found to be 50-1000 cells/ml. The disposable immunofiltration membranes were found to retain most of their activity over a period of 7days when refrigerated at 4C. The developed system demonstrated improved analytical characteristics over conventional immunoassay techniques. Potential commercialization of this system was a major objective, so the design highlights simplicity along with functionality. An automated, portable prototype is currently under construction. The enhanced sensitivity and short assay time permit the application of the developed system to near-real time detection of a wide range of analytes in medical diagnostics, biological and chemical analysis, food quality control, and environmental applications.

2:15 PM D14.4
A Study of Ammonia Gas Induced Irreversibility in Polypyrrole Films. Jaewu Choi[1], J. Hormes[2] and Pawan K. Kahol[3]; [1]Department of Electrical and Computer Engineering, Wayne State University, Detroit, Michigan; [2]Center for Advanced Microstructure and Devices, Louisiana State University, Baton Rouge, Louisiana; [3]Department of Physics, Wichita State University, Wichita, Kansas.

Polypyrrole's conductivity as a function of polymerization temperature and its properties on treatment with ammonia gas are studied. Electronic structures of PF6 (hexafluorophosphate) doped polypyrrole (PPY) films grown at -40 °C, 0 °C, and 20 °C, before and after ammonia treatment, are compared using angle resolved photoemission spectroscopy, electron paramagnetic spectroscopy, and semi-empirical calculations. The valence band electronic structures of these polypyrrole films largely depend on polymerization temperature, and ammonia treatment. The light polarization dependent valence band structures reflect ordering of polymer chains with decreasing polymerization temperature. The irreversibility properties of polypyrrole, which can be used as an ammonia gas senor, are shown to possibly arise from detachment of the dopant molecules under pumping.

2:30 PM D14.5

Accelerated Reliability Test inputs in analyzing the device response of MgZnO based UV Detector. Shiva S Hullavarad[1], Ichiro Takeuchi[1], Tirumalai Venkatesan[1], Ratnakar Vispute[2,1] and Sharad Yedave[2,1]; [1]Center for Superconductivity Research, University of Maryland, College Park, Maryland; [2]Blue Wave Semiconductors, Inc, Columbia, Maryland.

Ultra-violet (UV) light sensor is an essential component of UV light detection and monitoring required in a variety of commercial applications like dosimeter, radiation processing of materials, fire detection, lithography, data storage as well as for defense applications. MgZnO is found and shown to exhibit immense potential in this area as a tunable UV sensor due to its desirable optical and electrical properties. In this report, we present results on the fabrication of MgZnO based Metal-Semiconductor-Metal (MSM) UV sensor and address its stability issues. MgZnO is grown by pulse laser deposition (PLD) on three different substrates viz., glass, quartz and sapphire. X-ray diffraction and UV- Visible transmission spectroscopy are used to characterize crystallinity and the optical nature of the films. Standard photolithographic and lift off techniques are used to define MSM patterns on the device quality MgZnO films. Photo-response of the device is monitored by monochromator and lock-in-amplifier combination set up. Device stability and performance is examined under accelerated conditions of prolonged UV exposure.

2:45 PM D14.6

a large grain polycrystalline silicon film for resistive bolometers. Tae-Sik Kim and Hee Chul Lee; EE & CS, KAIST, DAEJEON, South Korea.

Bolometers, uncooled IR detector, based on vanadium oxide (VOx) and amorphous silicon (a-Si) films have been studied all over the world for a long time. VOx films have large temperature coefficient of resistance (TCR) of -3%/K; however, it is difficult to obtain reliable properties and dioxide form without some hysteresis. Although the fabrication process for bolometer made with a-Si film is CMOS compatible and its TCR value is around -2.5%/K for a resistivity of 86Ωcm, a-Si films exhibit large 1/f noise, thereby yielding a decrease of detectivity. In this paper, we have investigated large grain polysilicon films for such resistive materials of bolometers as alternative to VOx and a-Si films. That is because polysilicon films are much more stable material than VOx and a-Si films and CMOS technology can be used to fabricate polysilicon-based bolometers. In comparing with a small grain polysilicon film, a large grain polysilicon film has properties as follows: First, the TCR of the film theoretically increases in proportion to the increase of the grain size of the film. Second, we can obtain a polysilicon film with fewer grain boundary defects, which lead to a decrease of the 1/f noise of the film. Last, the large grain polysilicon film shows less dependence of resistivity on the variation of the doping concentration; therefore, it is easy to obtain required resistivity in the film. The achieved average grain size was as large as 1670Åusing a seed selection through ion channeling technique. Its TCR at 20°C and defect density were investigated to be as high as -2.46%/K for a resistivity of 30Ωcm and to be about 1.752×10^{12}/cm2, respectively. The value of k, 1/f noise parameter, was calculated to be 1.35×10^{-9}. From the above results, the estimated detectivity was found to reach up to 5.6×10^{8}cmHz$^{1/2}$/W.

3:30 PM D14.7

Self-Assembled Membranes Based on Humic Acid for Controlled Detection of Glucose in Implantable Biosensors. Ritesh Tipnis[1], Siddhesh D Patil[2], Diane J Burgess[2], Faquir Jain[3] and Fotios Papadimitrakopoulos[1]; [1]Nanomaterials Optoelectronic Laboratory, Department of Chemistry, Polymer Program, Institute of Material Science, University of Connecticut, Storrs, Connecticut; [2]Department of Pharmaceutical Sciences, University of Connecticut, Storrs, Connecticut; [3]Nanomaterials Optoelectronics Laboratory, Electrical and Systems Engineering, University of Connecticut, Storrs, Connecticut.

Implantable biosensors are an opportune way to continuously monitor blood glucose levels. However, their potential applications remain unexploited due to the negative tissue responses such as biofouling, inflammation, tissue fibrosis, and calcification generated by the implantation of such devices thus leading to device malfunctioning and eventually failure. We report here on development of a semi-permeable membrane, which can prolong the life of glucose sensors by protecting them from mineralization related failures. Our group had previously shown that the layer-by-layer self-assembly of humic acid (HAs), a naturally occurring biopolymer, with ferric ions can provide for the timely growth of a biocompatible, calcification resistant film, over the previously employed Nafion®-based membranes. Miniaturized sensors based on glutaraldehyde-stabilized glucose oxidase were modified with a varying number of self-assembled layers. For this, the pH was carefully monitored to impart the desired control over the thickness and permeability of HAs/Fe^{3+} films. This enabled us to overcome the previous impediment over enzyme deactivation due to the acidic nature of Fe^{3+} ions and obtain long-lived glucosensors while maintaining current linearity over physiological and pathogenic glucose levels. Furthermore, we are currently working towards fabricating a miniaturized, CMOS-based sensor, which would be completely autonomous and can be inserted subcutaneously to eliminate the presence of an open wound for feed-through electrodes. Acknowledgements : Department of Defense, US Army Medical Research Grant# DAHD17-02-1-0713.

3:45 PM D14.8

Merging Micro- and Nano-Technologies: Advanced Gas Sensors for an Intelligent Air Quality Monitoring Network. Marie-Isabelle Baraton[1] and Lhadi Merhari[2]; [1]SPCTS UMR CNRS 6638, University of Limoges, Limoges, France; [2]CERAMEC R&D, Limoges, France.

The growing concern worldwide about the consequences of urban air pollution on public health has generated an increasing demand for outdoor air quality monitoring. In the framework of two European projects, we have proposed a new concept for cost-effective outdoor air quality monitoring based on a dense network of mobile microstations. These microstations mounted on GPS-tracked public transportation vehicles wirelessly communicate with a central computer via the GSM network and are equipped with advanced semiconductor chemical sensors in order to decrease both the cost and the size of the system. The development of our advanced chemical sensors benefited from three major research directions: 1) use of semiconductor nanoparticles in the screen-printing fabrication technology; 2) development of mechanical layer-by-layer deposition technique for further increase of gas sensor sensitivity; 3) control and tailoring of surface chemistry of nanoparticles. We present here some of our consolidated results including the characterization of the nanosized particles and the resulting gas sensors as well by various methods such as XRD, TEM, FTIR, and electrical measurements. We explain how our prototype devices which are capable of detecting O_3 and NO_x concentrations down to 20ppb and 50ppb respectively, and CO concentrations down to 3ppm in air, can be used as sensing elements in an intelligent air quality monitoring network. The SMOGLESS and INTAIRNET projects were financially supported by the European Community (contracts No. BRPR-CT95-0002, and IST-12615).

4:00 PM D14.9

Organically Hybridized SnO2 Sensors for Selective Detection of Gas Molecules. Ichiro Matsubara, Kouta Hosono, Norimitsu Murayama, Woosuck Shin and Noriya Izu; National Institute of Advanced Industrial Science & Technology, Nagoya, Japan.

Sensing gas molecules is critical to environmental monitoring, control of chemical processes, saving energy, and safety in life. Gas sensors using semiconducting metal oxides such as tin oxide has been used practically as home gas leakage monitors. Such the sensors working by combustion mechanism detect any kinds of reducing gases in principle. The lack of gas selectivity, e.g. between CO and H2 gases, has been regarded as a problem to be solved. The improvement of selectivity requires a careful study of the surface reactions at the origin of the gas detection mechanisms. Here we demonstrate gas sensors based on SnO2 films hybridized to organic component with hydroxyl or amino groups. Upon exposure to CO gas, the electrical resistance of the sensor increases (R-increasing response), whereas other reducing gases such as H2 and CH4 gases cause the decreasing in the sensor resistance due to the ordinary combustion mechanism. The SnO2 films hybridized to organic component without hydroxyl or amino groups do not show the R-increasing response to CO gas. Interaction between the hydroxyl or amino groups of the organic component and SnO2 surface could be affected by CO gas, which works as switching to change the sensor resistance. We expect that our approach can provide highly selective gas sensors.

4:15 PM D14.10

Gas- and Bio-Sensors Based On Organic Thin Film Transistors. Zhengtao Zhu[1], Jeff Mason[1], Jamie Schnapp[1], Sanjay Aggarwal[1], Scott Stelick[2], Nate Cady[2], Carl Batt[2] and George Malliaras[1]; [1]Mater. Sci. and Eng., Cornell University, Ithaca, New York; [2]Food Science, Cornell University, Ithaca, New York.

Organic electronic devices have enormous potential for various applications due to their chemical and electronic tenability, mechanical flexibility, processability, and low cost. In this talk, we will discuss the field effects in the organic electronic materials and their applications for gas- and bio-sensing using a transistor configuration. Pentacene thin film transistors with 100 μm channel lengths were fabricated on Si substrates with 300 nm SiO2 as the gate dielectric. Our studies show that the characteristics of these transistors are sensitive to the relative humidity of the surrounding atmosphere. Their sensitivity was found to depend on the thickness of the pentacene film. The origin of the humidity effects is a change of pentacene film mobility. We will also discuss an unusual field effect in conducting polymer poly (ethylenedioxy-thiophene)-polystyrenesulphonate (PEDOT:PSS) and the application of these transistors in DNA sensing. The advantages of the PEDOT:PSS devices include high sensitivity and low gate voltage.

4:30 PM D14.11
Metal-containing diamond-like carbon-silicon nanocomposite films as temperature sensors. Toshiyuki Takagi[1], Alexei Bozhko[2], Takanori Takeno[1] and Mikhail Shupegin[3]; [1]Institute of Fluid Science, Tohoku university, Katahira 2-1-1, Aoba-ku, Sendai, Japan; [2]Moscow State University, Moscow, 119899, Russian Federation; [3]Moscow Power Engineering Institute, Moscow, 105835, Russian Federation.

The transition metal-containing diamond-like carbon-silicon nanocomposites are characterized by the stability to the aggressive environments and possess attractive mechanical properties, such as high hardness and wear resistivity, low friction. The variation of the metal concentration allows to change the conductivity of the nanocomposites in the wide range. The possibility of the application of metal-containing diamond-like carbon-silicon nanocomposite films as advanced temperature sensors with controlled temperature resistive response was studied. W- and Nb-containing diamond-like carbon-silicon nanocomposite films were deposited onto dielectric polycrystalline substrates by PECVD of siloxanes vapours in DC stimulated discharge and DC magnetron co-sputtering of metal target. The RF bias voltage was applied to the substrate holder during deposition of the films. It was shown that the conductivity of diamond-like carbon-silicon nanocomposite films containing both W and Nb decreases with temperature and follows the power law dependence. The decrease of metal concentration leads to rather sharp increase of the power exponent in the range from 0.3 to 0.7. Such temperature dependence of the conductivity corresponds to the electron transport mechanism being described by the inelastic electron tunnelling between metal nanoclusters dispersed in an insulating carbon-silicon matrix with high degree of disorder. The absolute value of the dimensionless sensitivity defined as dlns/dlnT, where s is the conductivity and T - temperature, demonstrates the dependence on the temperature close to the linear one in the investigated temperature range and increases from 0.2 to 1 with metal concentration. The experimental data obtained show the principal possibility of the fabrication of the wide temperature range thermometers, which retain their functionality under extreme environmental conditions.

4:45 PM D14.12
Chemical Sensing Using Surface Photovoltage Measurement of the Si Surface Potential Barrier. K. Nauka, T. I. Kamins and Z. Li; MS 1159, Hewlett-Packard Laboratories, Palo Alto, California.

Reactions between crystalline Si surfaces and chemical species that lead to modification of the Si surface barrier offer an exciting opportunity for chemical sensing [1]. In order to explore this concept, high quality Si wafers were exposed at room temperature to aqueous solutions of inorganic acids and bases, and then the surface potential was measured in a non-contact, non-destructive fashion using the Surface Photovoltage (SPV)[2]. The SPV signal was obtained by illuminating the Si surface with monochromatic light (wavelength > Si bandgap) and it was measured with a transparent, conducting electrode placed above the Si surface. The change of the surface potential barrier was clearly related to the type of chemical species and their concentration. It was also dependent on the Si orientation and the microscopic topography of the Si surface. In addition, the SPV signal was dependent on the wafer cleaning process that preceded the experiment. Relatively small changes in the concentration of chemical species, especially within the low concentration range, caused large changes of the SPV signal, demonstrating high sensitivity. Repeated cycles of acid and base exposure showed that the surface reactions were reversible and that the SPV signal cycled between the base-specific and acid-specific values, thus showing that the sensing device can be reused many times. In order to further enhance the SPV signal, the Si surface was intentionally terminated with a layer of selected organic or inorganic molecules. This termination had two purposes: it provided stable surface termination, and it offered the possibility of selective binding of only specific molecules. Preliminary results indicate that proper choice of the binding layer can make the SPV chemical sensing highly selective with respect to a large number of chemical species. The simplicity of the SPV measurement setup indicates that SPV-based chemical sensors can be easily miniaturized and cheaply produced in a compact form. [1] J.M.Buriak; Chem.Rev.102, 1271 (2002). [2] K.Nauka, T.I.Kamins, J.Electrochem.Soc.146, 292 (1999).