

# SYMPOSIUM V

## Materials and Devices for Smart Systems

November 28 - December 1, 2005

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\* Invited paper

**8:00 AM \*V1.1**

**Piezoelectric Materials for Smart System Applications.**  
Thomas P. Daue<sup>1</sup>, Keats Wilkie<sup>2</sup> and Andreas Schoenecker<sup>3</sup>; <sup>1</sup>Smart Material Corp., Sarasota, Florida; <sup>2</sup>Larc, NASA, Hampton, Virginia; <sup>3</sup>IKTS, Fraunhofer, Dresden, Saxony, Germany.

The concept of smart systems is based on intelligent materials which are changing local mechanical properties on external stimulation. Due to the overall performance, piezo ceramics are considered as the most promising basis for active materials. The use of thin monolithic piezo ceramic wafers and fibers as actuating and sensing material has been discussed in many publications. Piezo ceramic fiber based composites have become available on industrial scale, recently. This has stimulated the development of new applications for piezo fiber composites during the past two years. Examples of piezo fiber composites devices are the AFC - actuators (Active Fiber Composites) and the MFC - actuators (Macro Fiber Composite) actuators and 1-3 type composite transducers. The present paper is giving an update on availability and developments of piezo fiber composite materials for smart systems. Beside the intended use of these devices as actuators, new design forms have been investigated in order to qualify piezo fiber composites for sensor applications, especially as strain gages and pressure sensors as well as for energy harvesting. This paper gives also an update of emerging applications for piezo fiber composites and new devices under development to accommodate these applications.

**8:30 AM V1.2**

**MEMS Flextensional Actuator Using a Lead Zirconate Titanate Thin Film.** Han Geun Yu<sup>1</sup>, Srinivas Tadigadapa<sup>1</sup> and Susan Trolter-McKinstry<sup>2</sup>; <sup>1</sup>Electrical Engineering, The Pennsylvania State University, University Park, Pennsylvania; <sup>2</sup>Materials Research Institute, The Pennsylvania State University, University Park, Pennsylvania.

The design and fabrication of a microelectromechanical systems (MEMS) flextensional actuator based on the cymbal transducer design are reported. Flextensional actuators amplify small axial displacements into large normal displacement by utilizing flexural movement. The flextensional actuator is made up of a PZT unimorph actuator as the active element and a thick aluminum end cap as the flexure element. A fabrication process using four photomasks and a stiction free release step has been developed. The unimorph actuator consists of 0.5  $\mu\text{m}$  thick low stress LPCVD silicon nitride support layer and a 1  $\mu\text{m}$  thick PZT active layer. The PZT film was deposited by a chemical solution deposition process and is actuated in the through-the-film thickness ( $d_{31}$ ) mode. The flexural element consisting of 1  $\mu\text{m}$  thick aluminum element is attached to the unimorph actuator only at the ends and is freestanding along the majority of its length. 2.7  $\mu\text{m}$  thick photoresist was used as the sacrificial layer for the aluminum structure and 1  $\mu\text{m}$  thick polysilicon layer was used as the sacrificial layer for the unimorph actuator. The sacrificial photoresist under the aluminum layer was first removed by dissolution in acetone followed by a methanol rinse to reduce stiction. Finally the sacrificial polysilicon/substrate silicon was etched in  $\text{XeF}_2$  to obtain the freestanding flextensional structure. The resonance frequencies of the actuators were obtained by an Agilent 4294A impedance analyzer in a vacuum ambient of  $5 \times 10^{-4}$  mbar. Displacements in the range of 2.3-12.7  $\mu\text{m}$  at 200 kV/cm (an applied voltage of 20V) with corresponding resonance frequencies in the range of 407- 32.6 kHz have been obtained for these actuators. A preliminary measurement of the blocking force of a 500  $\mu\text{m}$  long actuator yielded 4.5  $\mu\text{N}$ . These measurements were made on actuators with 1  $\mu\text{m}$  thick PZT and 1  $\mu\text{m}$  thick aluminum. Additional measurement for actuators with other configurations will be reported. The ferroelectric properties of the PZT films, along with a detailed analysis of the device design and performance will be presented.

**8:45 AM V1.3**

**Microfabrication of PZT/SiO<sub>2</sub> Piezoelectric microcantilevers for rapid, direct, in-situ biosensing.** Wei-Heng Shih, Zuyan Shen and Wan Y. Shih; Department of Materials Science and Engineering, Drexel University, Philadelphia, Pennsylvania.

Miniaturized piezoelectric cantilever that composed by PZT and non-piezoelectric material such as metal or glass bi-layer structure offers the advantages of simple electrical detection and better capabilities to withstand damping in liquid. It is especially suitable for in-situ aqueous detection of bio or chemical sensing. Binding of antigens to the antibody immobilized on the cantilever surface increases the cantilever's mass, modifies the spring constant of the cantilever, and reduces its resonance frequency, which is detected by monitoring the resonance frequency shift. Our earlier work, both

theoretical and experimental, indicated that piezoelectric microcantilevers less than 100 microns in length will reach femtogram/Hz mass detection sensitivity which provide possibility to detect the mass of a single cell or virus. In this work, we focused on the development and incorporation of micron-thick highly piezoelectric PZT thin films by a sol-gel method on Si/SiO<sub>2</sub>/TiO<sub>2</sub>/Pt substrate and their subsequent patterning using Cl-based Inductively Coupled Plasma (ICP) dry etching. Integrated with the microelectronic fabrication technology for silicon based materials, freestanding PZT/SiO<sub>2</sub> piezoelectric microcantilevers with 50 micron length, 20 micron width dimension were made. In addition, our earlier theoretical and experimental work indicated that a nonpiezoelectric extension of an optimal length at the free end of cantilever further enhanced the mass detection sensitivity. To further enhance the mass detection sensitivity, a SiO<sub>2</sub> extension was incorporated in the PZT/SiO<sub>2</sub> design. Electrical insulation was achieved with parylene coating. Results of the resonance spectra, the mass detection sensitivity, and demonstration of in-situ protein will be presented.

**9:00 AM V1.4**

**Metal Core Piezoelectric complex fiber and Application for smart board.** Hiroshi Sato<sup>1</sup>, Lihua Yu<sup>1</sup> and Masaru Nagamine<sup>2</sup>; <sup>1</sup>AIST, Tsukuba, Japan; <sup>2</sup>Nagamine Manufacturing Co.,Ltd, Nakatago gun, Japan.

Piezoelectric material can be used in sensing and actuating applications. Much attention has recently been paid to the application of piezoelectric materials in smart structures such as health-monitoring devices and vibration-control systems by embedding them into such composite materials as Carbon-Fiber Reinforced Plastic. In embedding piezoelectric materials into composite material, it is essential to minimize the inclusion effect on the mechanical performance of the composite. As one solution, the Active Materials and Structures Laboratory at MIT proposed the use of fiber-shaped piezoelectric material(AFC). Thin PZT fibers were transversely aligned in a polymer matrix, and the fiber laminates were placed between interdigital electrodes(IDT) at a right angle in order to obtain a larger d33 mode strain along the fiber axis.. However, their fiber has disadvantages, as only the part between electrodes can be used as sensors and actuators in this application and the fragility of ceramics may be problematic. In order to solve these problems, we propose the use of a piezoelectric fiber having a metal core fabricated by means of the extrusion method. Our piezoelectric fiber has the following advantages (1)No need of electrodes Generally, a piezoelectric material needs one pair of electrodes for its use in sensor and actuator applications. In our piezoelectric fiber, however, electrodes are not required, since the metal core in the fiber is functions as an electrode and the CFRP matrix can play the role of ground electrode because of the high electrical conductivity of the carbon fiber. (2)High performance Because the electrical field in AFC is supplied by means of an IDT, only an interelectrode in the surface part of the fiber can be used. However in our metal core piezoelectric fiber, the electric field is supplied between an internal metal core and CFRP. As a result, every embedded part of the piezoelectric material can be utilized as an actuator or a sensor. (3) Durability Although piezoelectric ceramics such as PZT are fragile, the metal core materials are quite strong. (4)High resistance to external noise . The sensitivity of the sensor is evaluated by in terms of its S/N ratio. Therefore, it is important to increase the output signal from the sensor and to decrease noise from the sensors surroundings. Since our fiber is embedded in a CFRP composite matrix that which has a high electrical conductivity, external noise is easily suppressed and the clarity of the signal from the sensor is enhanced. In this paper, we describe the fabrication of piezoelectric fibers with metal cores using the extrusion method, and describe the design of a CFRP smart board using these piezoelectric fibers. Additionally, we shows that this smart board can act as both a sensor and an actuator .

**9:15 AM V1.5**

**Electromechanical Response of 1-3 Piezoelectric Composite Materials: Effects of Poling Characteristics, Fiber Geometry and Distribution.** Ronit Kar-Gupta and T. A. Venkatesh; Mechanical Engineering, Tulane University, New Orleans, Louisiana.

Recognizing the potential for the use of piezoelectric materials in a number of applications as sensors and actuators, there has been a continuing research and development effort to synthesize monolithic materials with enhanced coupled properties. Because the sensing or actuating actions of monolithic piezoelectric materials are limited, the composite approach to piezoelectric materials provides a unique opportunity to access a new design space with optimal mechanical and coupled characteristics, hitherto inaccessible through monolithic materials. Through finite-element based numerical modeling, a systematic methodology for predicting the complete set of coupled properties of the piezoelectric composites as a function of the poling characteristics, size, shape, and distribution of the constituent phases is presented. Strategies for designing novel piezoelectric composites

with enhanced sensitivities in multiple directions are also identified. Model predictions are compared with experimental results for select piezoelectric composites.

#### 9:30 AM **V1.6**

**Pressure Loading of Piezo Composite Unimorphs.** Karla Mossi<sup>1</sup>, Poorna Mane<sup>1</sup> and Robert Bryant<sup>2</sup>; <sup>1</sup>Virginia Commonwealth University, Richmond, Virginia; <sup>2</sup>NASA Larc, Hampton, Virginia.

Over the past decade synthetic jets have emerged as a promising means of active flow control. One way to achieve flow control is through the use of piezoelectric actuators since they could decrease the complexity of the aircraft by reducing the number of moving parts. This paper discusses the effects of pressure loading on the displacement of two pre-stressed circular piezoelectric Unimorph composites, Thunder and Lipca. Thunder is composed of a PZT-5A ceramic bonded between stainless steel and copper using a polyimide as an adhesive. Lipca consists of a glass/epoxy top layer, a layer of unidirectional carbon epoxy layer, another glass/epoxy layer, a PZT layer, and another glass/epoxy layer. In this study both actuators are loaded by pressurizing one side of the device. Using a fiber optic sensor, the displacement at the non-pressurized side is monitored at various voltages, frequencies at different pressure loadings.

#### 9:45 AM **V1.7**

**Improvement of Aerodynamic Performance by Using Piezoelectric Actuators.** Min Li<sup>1</sup>, Wei-min Chen<sup>2</sup>, De Guan<sup>1</sup> and Wei Li<sup>1</sup>; <sup>1</sup>School of Aeronautics Science and Engineering, Beijing University of Aeronautics and Astronautics, Beijing, China; <sup>2</sup>Division of Engineering Science Research, Institute of Mechanics, Chinese Academy of Sciences, Beijing, China.

To flight vehicle designer, the ability to adapt air vehicle aerodynamic shape to increase the optimum flight envelope is highly desirable. In this work, by distributing some piezoelectric actuators on the top and bottom surfaces of a rectangular wing, the improvement of aerodynamic performance of flight vehicle is studied. The approach of the Fictitious Control Surface (FCS) is evaluated at variable dynamic pressure and wing stiffness through examining four aspects including the improvement of rolling power, the increase of lift, the decrease of the induced drag and the decrease of the bending moment at the root of wing. The analytical results demonstrate that for the wing with FCS, the aeroelastic effect can be changed into favorable effect unlike the unfavorable effect of traditional fixed wing with control surface. That is to say, compared with the traditional wing, the air performance of flight vehicle can be improved by actively deforming a more flexible wing and using smaller control energy. For the model used in this paper, if  $\pm 800V$  voltage is applied on the actuators, the produced rolling power is equal to that by the aileron deflecting  $5.3^\circ$  angle, or the produced lift is equal to the stiff wing deflecting  $0.55^\circ$  angle of attack, and the induced drag is decreased by 12% around or the root bending moment is decreased by 5% around. At last an experimental model in high-speed wind tunnel is designed in order to validate the results of theoretical analysis. Through ground tests and wind tunnel tests, the approach of obtaining lift and rolling moment by using the favorable aeroelastic effect is validated, and the experimental results agree well with the analytical results. The theoretical and experimental results obtained by this work indicate the feasibility of improving aerodynamic performance by distributing piezoelectric actuators on the surfaces of wing. And for further application into practical flight vehicle, more efficient actuators and more optimal placement need to be developed.

#### 10:30 AM **\*V1.8**

**Piezoceramic/metal Composites and Graded Materials.** Jing-Feng Li and Hailong Zhang; State Key Lab of New Ceramics and Fine Processing, Department of Materials Science and Engineering, Tsinghua University, Beijing, China.

Piezoelectric ceramic actuators are technologically important in the fields of smart materials and microelectromechanical systems (MEMS). Unimorph and bimorph actuators are two well-known types of piezoelectric actuators, which consist of piezoelectric plates and metal shims that are bonded together using an organic agent. However, The inherent defect in such actuators is deterioration of the bonding after a long period of use. Introducing graded microstructures into the interface is a good solution to this problem. For this purpose, we have recently developed FGM actuators with graded compositional profiles from piezoelectric ceramic to metal. For the design of graded actuators, the piezoelectric and mechanical properties including dielectric constants, piezoelectric constant, bending strength and fracture toughness of Pt- or Ag-particle-dispersed PZT composites fabricated by conventional ceramic sintering process were measured and analyzed in relation with the composite microstructures. Using the experimentally determined compositional dependency of elastic and piezoelectric properties in the PZT/Pt and PZT/Ag composites, the modified classical lamination theory and the finite element

method (FEM) were applied to design the optimum FGM compositional profile. The electromechanical bending characteristics of the graded PZT/Pt and PZT/Ag actuators were measured using the conventional method with electronic gauges, and the results showed that the bending displacement is strongly dependent on the compositional distribution across the thickness of the actuator, being in good agreement with the theoretical prediction.

#### 11:00 AM **V1.9**

**Hybrid Actuation System (HYBAS) - A Review.** Ji Su<sup>1</sup>, Tian-Bing Xu<sup>2</sup>, Shujun Zhang<sup>3</sup>, Thomas R. Shrout<sup>3</sup> and Qiming Zhang<sup>3</sup>; <sup>1</sup>NASA Langley Research Center, Hampton, Virginia; <sup>2</sup>National Institute of Aerospace, Hampton, Virginia; <sup>3</sup>Materials Research Institute, The Pennsylvania State University, State College, Pennsylvania.

The recently developed electroactive polymer-ceramic hybrid actuation system (HYBAS) demonstrates significantly-enhanced electromechanical performance by utilizing the advantages of the synergistic contributions of the electromechanical responses of an electrostrictive copolymer and of an electrostrictive single crystal. The HYBAS offers not only an electromechanical device but also a concept to utilize different electroactive materials in a cooperative and efficient method for an optimized electromechanical performance. The HYBAS concept contains most of the advantages of both flexensional and thunder actuators. The application of this concept offers a new mechanism for developing a new generation of electromechanical devices including flexensional and Thunder actuators. In order to optimize the performance of a hybrid actuation system (HYBAS) and make the concept more comprehensively applicable, a theoretical model has been developed. The model is based on the elastic and electromechanical properties of the constituent materials and on the configuration of the designed device. The model can also optimize the performance a HYBAS as a function of geometric parameters, including the length and thickness ratios of the constituent components. The comparison between the model and the experimental results shows a good agreement and validates the model as an effective method for the further development of high performance actuating devices or systems for various applications. The HYBAS concept can support various applications for Navy, Air Force, and NASA missions, as well as, for civil applications. This report reviews and provides a guideline for future applications of the proven concept of the hybrid actuation system.

#### 11:15 AM **V1.10**

**Intrinsic Electrochemical Actuation Strain and Energy Density of Graphite Intercalation Compounds.** Yukinori Koyama, Timothy E. Chin and Yet-Ming Chiang; Dept. of Materials Science & Engineering, MIT, Cambridge, Massachusetts.

Due to their large crystallographic strain (up to 10% linear) and high elastic modulus, solid-state intercalation compounds have exceptionally high theoretical energy densities when used as electrochemical actuators. Although much data exists on the electrochemically-induced strain under no mechanical constraint, the potential of the intercalation compounds for producing mechanical work has not been evaluated. In this work, we have therefore studied the intrinsic actuation properties of graphite including electrochemically-induced strain under high mechanical loads. An array of small posts of highly-oriented pyrolytic graphite (HOPG) bonded on alumina substrates was used as the active material. The small posts, typically 0.2 mm square and 0.4 mm tall, were carved from a HOPG piece by laser micromachining. Lithium foil was used to surround the HOPG posts as a counter electrode. The samples were placed in polymer pouches filled with non-aqueous liquid electrolyte, and cyclically charged and discharged under various mechanical loads applied along the [0001] direction while simultaneously measuring strain. Under 10 MPa stress a linear strain of 6.7% was measured; under 30 MPa stress, the linear strain reached 4.3% to a mechanical energy density of 1,290 kJ/m<sup>3</sup>, more than 10x greater than that of high energy piezoelectrics. The measured strain is nonetheless smaller than the free expansion of single crystal graphite along the [0001] direction (10%). The origin of this behavior, and other results, will be analyzed in the presentation. This work was sponsored by the DARPA Nastic Materials Program under contract no. W911W6-05-C-0013.

#### 11:30 AM **V1.11**

**Texture and Microstructure Design of Electrochemical Actuators.** R. Edwin Garcia<sup>1</sup> and Yet-Ming Chiang<sup>2</sup>; <sup>1</sup>Materials Engineering, Purdue University, West Lafayette, Indiana; <sup>2</sup>Materials Science and Engineering, MIT, Cambridge, Massachusetts.

Electrochemical actuators constitute an emerging technology based on the induced change of shape that arises through the reversible intercalation of ions between two electrochemically active electrodes. For such devices, the local transport and mechanical properties are combined in an effort to obtain the largest possible actuation rate.

Moreover, designs based on crystallographically and morphologically anisotropic electrode materials have the potential of providing performances tailored for specific advanced applications. In this paper, the microstructural details of the elastic and electrochemical fields are spatially resolved for negative and positive electrode materials. Their impact is assessed by systematically exploring the space of fiber texture parameters, for a fixed morphology of the active phase. Design issues, such as device reliability and irreversible plastic deformation are considered in the formulation.

#### 11:45 AM **V1.12**

**A Reversible Molecular Valve.** Thoi Dang Nguyen<sup>1,2</sup>, Hsian-Rong Tseng<sup>1,2</sup>, Paul C. Celestre<sup>1,2</sup>, Amar H. Flood<sup>1,2</sup>, Yi Liu<sup>1,2</sup>, J. Fraser Stoddart<sup>1,2</sup> and Jeffrey I. Zink<sup>1,2</sup>; <sup>1</sup>Chemistry and Biochemistry, UC Los Angeles, Los Angeles, California; <sup>2</sup>California NanoSystems Institute, University of California, Los Angeles, Los Angeles, California.

A macroscopic valve is a device with a movable control element that regulates the flow of gases or liquids by blocking and opening passageways. Construction of such a device on the nanoscale level requires (i) suitably proportioned movable control elements, (ii) a method for operating them on demand, and (iii) appropriately-sized passageways. These three conditions can be fulfilled by attaching organic, mechanically interlocked, linear motor-molecules, that can be operated under chemical, electrical or optical stimuli to stable inorganic porous frameworks, i.e., by self-assembling organic machinery on top of an inorganic chassis. In this presentation, we demonstrate a reversibly-operating nanovalve that can be turned on and off by redox chemistry. It traps and releases molecules from templated nanopores in silica by controlling the operation of redox-activated bistable [2]rotaxane molecules tethered to the pore openings in a nanoscale reservoir. The release of probe molecules and the operation of the valve itself are monitored by fluorescence spectroscopy.

SESSION V2: Novel Devices and Systems  
Chairs: Christian Boller and Yet-Ming Chiang  
Monday Afternoon, November 28, 2005  
Independence W (Sheraton)

#### 1:30 PM **\*V2.1**

**High Displacement, High Authority Actuation Using Electrochemical Storage Compounds.** Yet-Ming Chiang<sup>1</sup>, Steven R. Hall<sup>2</sup>, Yukinori Koyama<sup>1</sup>, Kyung Yeol Song<sup>2</sup>, Timothy E. Chin<sup>1</sup>, Urs Rhyner<sup>1</sup>, Fernando Tubilla<sup>2</sup> and Dimitrios Sapnaras<sup>1</sup>; <sup>1</sup>Department of Materials Science and Engineering, M.I.T., Cambridge, Massachusetts; <sup>2</sup>Department of Aeronautics and Astronautics, M.I.T., Cambridge, Massachusetts.

Piezoelectric, electrostrictive, and magnetostrictive materials are widely used in technology as compact solid-state actuators, transducers, and sensors, but have not to date enabled such applications as shape-morphing structures and vehicles (aircraft, rotorcraft, watercraft). In this work we show that solid-state ion intercalation offers a simple low-voltage (<5V) electrically operated actuation mechanism of extremely high energy density, approaching that of hydraulics and greatly exceeding that of existing field-operated mechanisms. The high elastic modulus (30-150 GPa) and large and reversible crystallographic strains (up to ~12%) intercalation compounds, combined with large electrochemical driving force (0.5-5 eV per atom) even at low operating voltage, allow high operating stresses up to the mechanical failure limit of the active materials and a "set and forget" capability. In addition, low materials densities (2-5 g/cm<sup>3</sup>) compared to other leading solid-state actuators (metal alloys and lead-based ceramics) provide for high weight-based actuation figures of merit. Actuator designs based on single- and polycrystalline graphite, and LiCoO<sub>2</sub>, have been constructed and tested, and demonstrate the potential for a new class of high-strain, high-authority, moderate-frequency actuators suitable for a broad range of applications, including nastic structures. This research was supported by the DARPA Nastic Materials Program under Contract No. W911W6-05-C-0013.

#### 2:00 PM **V2.2**

**Parameterized Approach to MEMS Gear Design.**  
Edward S. Kolesar and Christopher A. Edwards; Department of Engineering, Texas Christian University, Fort Worth, Texas.

The need for mechanical gears in Microelectromechanical Systems (MEMS) technology presents the problem of accurately and efficiently designing gears of the required types and sizes in a short amount of time. Currently, all gears must be hand drawn in commercial MEMS CAD programs, with each new gear requiring the customization of each of its features. Despite this difficulty, mechanical gear design has currently been accomplished to some extent, but the process is time

consuming, it is difficult to render precise gears, and, as a result, their use has been limited. To facilitate rapid and accurate mechanical gear design, a new parameterized approach to Microelectromechanical Systems (MEMS) gear design has been developed. To facilitate the expeditious design of a complicated gear mechanism, a parameterized mathematical design process has been developed. This parameterized approach to gear design facilitates the creation of gears with any practical inner radius, outer radius, tooth number, tooth size, number of spokes, dimples, and other features, simply by specifying the desired attributes. After the MEMS designer specifies these parameters, they are entered into a script file, in this case, a .cif file, which can then be imported into the desired commercial MEMS CAD program. The result is the creation of a customized gear design within minutes.

#### 2:15 PM **V2.3**

**Harmonic Detection of Resonance in Micro- and Nano-Cantilevers.** Jay Gaillard, Razvan Ciocan, Malcom Skove and Apparao Rao; Physics and Astronomy, Clemson University, Clemson, South Carolina.

Integrating micro- and nano- scale mechanical oscillators into circuits has been hampered by the lack of a completely electronic means of measuring their resonant frequency with sufficient sensitivity. This has been the main holdup for nanoelectromechanical systems (NEMS) to be used in practical applications. Optical, magnetic and electrical measurement schemes exist.<sup>2,3</sup> The optical and magnetic techniques would be difficult to apply to an integrated circuit. Electrical detection of motion within NEMS is the next alternative. In silicon-based NEMS, existing methods usually have more than one counter electrode and rely on cantilever to electrode contact or very small tunneling gaps,<sup>2,3</sup> again difficult to apply in an integrated circuit. Most preferred is a capacitive readout method because it can be readily integrated with NEMS devices that are fully compliant with standard CMOS technologies.<sup>2</sup> The main drawback, however, comes from the parasitic capacitance between the electrodes, which gives a large signal at the driving frequency.<sup>4</sup> We completely avoid the effects of this parasitic capacitance by locking into the 2<sup>nd</sup> harmonic of the driving frequency. We exploit a 2 $\omega$  term in the expression for the charge  $q(t)$  of the cantilever when driven by a counter electrode at angular frequency  $\omega$ . We demonstrate that by using this harmonic detection of resonance (HDS) system we can electrically detect the resonant frequencies of micro cantilevers with sensitivity equal or better than optical methods, and can detect the resonant frequency even in cantilevered multi-walled carbon nanotubes, which have diameters on the order of 50 nm. We specifically target the measurements in nanotubes because of their superior mechanical properties to silicon although the HDS system can also be applied to silicon-based NEMS. Ultimately, this new approach may hold promise for high-efficiency transduction with the smallest of NEMS devices.<sup>1</sup>  
1. Roukes, M. L. Nanoelectromechanical Systems Face the Future. *Phys. World* 14, 25 (2001). 2. Datskos, P. G. Thundat, T. Lavrik, and Nickolay V. *Encyclopedia of Nanoscience and Nanotechnology* 5, 551 (2004). 3. Roukes, M. L. Nanoelectromechanical systems. *Tech. Digest. Solid State Sensor and Actuator Workshop (Hilton Head Island, SC, 2000)* 4. J. Teva, G. Abadal, Z.J. Davis, J. Verd, X. Borrise, A. Boisen, F. Perez-Murano, and N. Barniol, *Ultramicroscopy* 100, 225 (2004).

#### 3:30 PM **\*V2.4**

**From the Global Biomimeticism to the Bioinspired and Bioderived Materials.** Pierre Francois Gobin<sup>1</sup>, Michelle Salvia<sup>2</sup>, Philippe Guy<sup>1</sup>, Nathalie Godin<sup>1</sup> and Yves Jayet<sup>1</sup>; <sup>1</sup>GEMPPM, Institut National des Sciences Appliquées de Lyon, Villeurbanne, France; <sup>2</sup>LTDS, Ecole Centrale Lyon, Ecully, France.

Observation of the nature provides many examples of systems with multi-functional components integrated into an efficient and sometimes elegant design. Evidently the direct bio-mimicry is rarely possible or appropriate because these biological systems are optimized following a set of requirements very different of the engineering needs. Nevertheless a lot of lessons can be learned of studying the living world and there is now some interesting and successful engineering solutions that were biologically inspired. For instance, the widely used reversible adhesive Velcro was invented by Georges de Mestral after noting how the burdock seeds attach to the fur of his dog! In fact applications or attempt of applications are far from new, and biomimetic dreams feed for thousand years legends and myths from Icarus to Frankenstein! More seriously the domain of the biologically inspired technology has evolved from static copies of living creatures in the form of statues to the emergence of robots that operate with realistic appearance and behaviour. A good time for globally biomimetic devices was the XVIII century with its androids and automatons as the famous Vaucanson's duck, but evidently the technology evolution led now to more sophisticated robots. Moreover, as was discovered in the last decades, studying nature can lead to some novel approaches. Effectively in the 1980's the concept of 'intelligent' or even 'wise' materials was pioneered in USA and Japan

with the purpose to establish new areas in materials science. This new way takes in account the relationships between materials, natural environment. This concept has been at first proposed in a totally biomimetic frame in which a material or a material-system would possess as a living creature 'nerves' simulated by a network of sensors, 'muscles' or active components and a 'brain' in order to coordinate the whole. This ambitious concept leads naturally to the multifunctionality for the materials and to the most fascinating of the challenges: the self healing behaviour. This type of observations can also provide somewhat of confirmation for some non traditional techniques. For example though the design of composites structures was not directly coming from a biological inspiration, the general and optimized use of natural composites suggest that this now unavoidable approach for structural materials is founded and could be probably deeply improved in the bioinspired way. In this brief talk the active part of 'smart materials' and the physical basis necessary to realize usable 'artificial muscles' will be emphasized first. Then the continuous health monitoring of composite structural parts will be discussed as a major application of this bioinspired approach. Finally in some cases in which it is not possible or very difficult to mimic the nature, it seems more realistic to use some living cells in order to produce 'bioderived' new materials.

#### 4:00 PM \*V2.5

**Smart Systems for Enhanced Integrity of Existing and Future Structures.** Christian Boller, Mechanical Engineering, The University of Sheffield, Sheffield, United Kingdom.

Ageing is a general problem not only in society but also in engineering. Many of our engineering structures are seemingly built ageless although they are not. Others had to be built with higher safety factors in the past due to lack of engineering knowledge at the time, which nowadays allows these structures to be used longer than initially anticipated. As a consequence new design principles may allow future structures to be further built lighter weight. Much of this enhancement has been due to implementing smart systems technologies into design. This paper will give a few examples based on integrated sensing as well as actuation principles into structures.

#### 4:30 PM V2.6

**Carbon Fiber Reinforced Rigidizable Space Structures.** Stephen Andrew Sarles<sup>1</sup>, Todd Bullions<sup>2</sup>, Thompson Mefford<sup>2</sup>, Judy S. Riffle<sup>2</sup> and Donald J. Leo<sup>1</sup>; <sup>1</sup>Mechanical Engineering, Virginia Tech, Blacksburg, Virginia; <sup>2</sup>Macromolecules and Interfaces Institute, Virginia Tech, Blacksburg, Virginia.

The prominence of inflatable gossamer space structures has propelled the need for advances in both the materials used to construct these components as well as the methods for implementing geometric shape-holding and structural stiffening. A powdered prepreg tow was prepared from carbon fibers and a toughened epoxy Unyte<sup>®</sup> powder from Hydrosiz Technologies. The resin was designed with a sufficiently wide processing window separating the consolidation and cure temperatures for these investigations of in-situ rigidization. The rheological parameters were evaluated in the parallel plate mode utilizing both dynamic and isothermal temperature scans. Between 145°C and 180°C the viscosity was below 40 Pa-s, with a minimum viscosity of 14 Pa-s at 170°C. Isothermal rheological scans demonstrated that Unyte Set 201A would remain below 25 Pa-s for 22 minutes and at 10-12 Pa-s for 10 minutes at 175°C but only dropped below 25 Pa-s for 4 minutes at 200°C. Using differential scanning calorimetry to establish the times and temperatures required for cure, the 175°C isothermal cure was achieved within about 60 minutes. Based on the above rheological and thermal data, holding Unyte Set 201A between 160°C and 175°C for 30-60 minutes provides adequate time for the resin to wet out the fibers of the braided tow prepreg and then cure the resin. Rigidization of carbon-fiber reinforced polymer composites occurs by internal resistive (Joule) heating as current flowing through the material generates heat within the composite, initiating the consolidation and cure of the material. The ability to provide spatially distributed heating and accurately reach critical temperatures during the rigidization process is vital for a complete and even cure. Specifically, feedback control of the resistive heating process allows for precise temperature control of the composite as well as indirect forecasting of the cured composite's mechanical properties. Initial experiments indicate that a tuned PID feedback controller can be used to track and maintain a desired temperature profile to within only a few degrees error. Further, prescribing a high temperature across the sample results in a noticeable and measurable increase in the composite stiffness. Combining effective temperature control during the curing process with measurable changes in the physical properties contributes to a model of the resistive heating scheme. Additionally, a predictive model of structural integrity as a function of control accuracy is also available. The performance of the stiffened composite is evaluated with respect to the amount of power supplied and the level of cure completion. The integration of feedback control via resistive heating with a novel polymer thermoset reduces the

energy required for rigidization while concurrently improving the final product.

#### 4:45 PM V2.7

**Effect of SiC Shape on The Crack-Healing Mechanism of Alumina/SiC Composite.** Wataru Nakao, Toshio Osada, Kazuya Yamane, Koji Takahashi and Kotoji Ando; Yokohama National University, Yokohama, Japan.

Crack-healing of structural ceramics is a most valuable technique to overcome their weak point such as surface cracks. Crack-healing ability appeared by admixing SiC to ceramics, because this function is to use the oxidation of SiC. Endowment of crack-healing ability as well as improvement in fracture toughness was expected to be achieved by compositing SiC whiskers. For this purpose, alumina / 30 vol% SiC whiskers and alumina/ 20vol% SiC whiskers/ 10 vol% SiC particles multi-composites were developed. Crack-healing ability and fracture toughness of these composites were investigated. alumina/ 20 vol% SiC whiskers/ 10 vol% SiC particles multi-composite and alumina/ 30vol% SiC whiskers composite were found to have 1.43 times and 1.65 times higher fracture toughness than monolithic alumina, respectively. From the results of the strength recovery by crack-healing, alumina/ 20 vol% SiC whiskers/ 10 vol% SiC particles multi-composite and alumina / 30vol% SiC whiskers composite were found to be able to heal the pre-crack below 250 μm and 200 μm in surface length, respectively. In spite of the same SiC content, SiC particles and SiC whiskers multi-compositing enlarged the limit crack size to be able to crack-heal. However, increasing SiC whiskers content enhanced strength recovery of the specimen with the large crack above limit crack-size.

#### SESSION V3: Poster Session: Piezoelectric Actuators II

Chair: Takayuki Narushima

Monday Evening, November 28, 2005

8:00 PM

Exhibition Hall D (Hynes)

#### V3.1

**High Load Capable Piezoelectric Single Crystal Actuators.**

Tian-Bing Xu<sup>1</sup>, Ji Su<sup>2</sup>, Xiaoning Jiang<sup>3</sup>, Paul W. Rehrig<sup>3</sup> and Wesley S. Hackenberger<sup>3</sup>; <sup>1</sup>National Institute of Aerospace, Hampton, Virginia; <sup>2</sup>NASA Langley Research Center, Hampton, Virginia; <sup>3</sup>TRS Technologies, Inc., State College, Pennsylvania.

Piezoelectric lead magnesium niobate-lead titanate (PMN-PT) single crystal is one of the most promising materials for electromechanical device applications due to its high electrical field induced strain and high electromechanical coupling factor. PMN-PT single crystal-based multilayer stack actuators and multilayer stack-based flextensional actuators have exhibited high stroke and high displacement-voltage ratios. The mechanical load capabilities of the recently developed single crystal stack was determined. These stacks were used in the fabrication of flextensional actuators. The actuator capabilities were evaluated using a newly developed method based upon a laser vibrometer system under various loading conditions. The measured displacement as a function of the mechanical load at different driving voltages indicates that the displacement response of the actuators is approximately constant under broad ranges of frequency and mechanical load. The high load capabilities of these PMN-PT single crystal-based actuators and the advantages of the capability for applications will be presented.

#### V3.2

**Resonances in Micromachined Circular Piezoelectric**

**Unimorph Diaphragms.** Eunki Hong<sup>1</sup>, Susan Trolrier-McKinstry<sup>1</sup>,

Robert Smith<sup>2</sup>, S. V. Krishnaswamy<sup>2</sup> and Carl B. Freidhoff<sup>2</sup>;

<sup>1</sup>Materials Research Institute, Penn State Univ., University Park, Pennsylvania; <sup>2</sup>Sensors System Sector, Northrop Grumman Electronics, Baltimore, Maryland.

When piezoelectric materials are incorporated into diaphragm structures, they can be used to generate actuation and sense external stimuli. To amplify the available response, the structures are driven at resonance. In the same way, resonance is sometimes used in piezoelectric sensing structures to increase sensitivity. In addition, the resonance frequencies of micromachined structures such as cantilevers, bridges and diaphragms can also be used to extract material parameters such as Young's modulus, residual stress (or tension) and density. In this study, the resonances of micromachined circular piezoelectric diaphragms are investigated. The diaphragm structures are piezoelectric unimorphs, which consist of a Pb(Zr<sub>0.52</sub>Ti<sub>0.48</sub>)O<sub>3</sub> film and a silicon oxide (SiO<sub>2</sub>) passive layer. Electrically and mechanically excited resonances in micromachined circular piezoelectric diaphragms have been investigated. For electrical excitation, ring-shaped interdigitated (IDT) electrodes formed on the

top of the PZT layer were utilized to induce strain in the diaphragms. The diaphragm structures behaved much like circular membranes, where the membrane tension was  $\sim 206$  N/m, at the fundamental modes. For higher modes, the resonance frequencies deviated from the theoretical values due to the finite stiffness of the diaphragms. Under mechanical drive, both symmetric and asymmetric modes were excited. However, for electrical excitation, the symmetric modes were dominant due to the symmetry of the driving IDT electrodes. At a pressure of 727 Torr, the quality factor was  $\sim 250$  and this rose to 2000 at pressures below 1 Torr. In the diaphragm structure, the nonlinear vibration was governed by the geometric nonlinearity rather than the material nonlinearity.

**V3.3**  
**Novel Microfluidic Devices and Functional Microfabrications for Sensors or Actuators Applications.** Lisa S. Brown, University of California, Irvine, Irvine, California.

Microfabrication technology offers us integration of patterns since small features fabricated on a variety of substrates have produced numerous active devices to satisfy a set of our demands in miniaturization. There is a growing interest in the pattern integration using functional materials for sensors or detective devices. In this presentation, we will demonstrate a novel approach to the microfabrication and microsynthesis for unconventional functional materials, which can be fabricated on diverse substrates. As an example, we introduce a molecularly imprinted polymer (MIP) as a promising candidate material for functional patterning task to fabricate sensors or devices based on materials with specific functions. MIP is a highly cross-linked macroporous thermoset with both high internal surface areas and specific molecular recognition sites. We also introduced a novel approach to synthesize nano- or micro-sized MIPs polymer particles using a 'microfluidic droplet reactor'. This results in 'monoclonal MIPs particles', which have only high affinity binding sites to achieve specific molecular recognition function for bio/chemical-sensors or detective devices with enhanced sensitivity.

**V3.4**  
**The Optimal Design and Analysis of Piezoelectric Cantilever Beams for Power Generation Devices.** Dongna Shen<sup>1</sup>, Song-Yul Choe<sup>2</sup>, Young-Sung Lee<sup>3</sup>, Jong-Dae Lim<sup>3</sup> and Dong-Joo Kim<sup>1</sup>; <sup>1</sup>Materials Research and Education Center, Auburn University, Auburn, Alabama; <sup>2</sup>Dept. of Mechanical Engineering, Auburn University, Auburn, Alabama; <sup>3</sup>Hyundai Motor Company, Whasung, South Korea.

With the rapid development of wireless sensor technology, battery is becoming the limiting factor in the lifetime of the devices and miniaturization. The conversion of ambient energy has attracted many interests as a way to eliminate battery in the system. The numerous vibration sources easily accessible in our environment can make the conversion of vibration energy into electrical energy more promising. The utilization of piezoelectric materials for such conversion can realize the power generation devices with high density and simple configuration. We fabricated piezoelectric cantilever beams for energy conversion since they can generate large strain and energy density. The design was optimized through numerical analysis and simulation. The parameters influencing the output energy of piezoelectric bimorph cantilevers including the type of piezoelectric materials, dimension of cantilever beams, proof mass and electrical circuits were investigated. The robustness of cantilever structure was also considered for implementing piezoelectric power conversion devices in harsh conditions. The energy density generated by the optimized piezoelectric devices was high enough ( $> 100 \mu\text{W}/\text{cm}^3$ ) to operate microsensors systems. The comparison between experimental results and simulation models will be also extensively discussed with the proposal for miniaturization of piezoelectric power generation devices.

**V3.5**  
**Active CPU Cooling System with Piezoelectric Actuated Droplet Generator.** Chun-Fu Lu<sup>1</sup>, Huang Chi-Ming<sup>1</sup>, Jinn-Cherng Yang<sup>1</sup>, Hung-Liang Chiang<sup>1</sup>, Chiang-Ho Cheng<sup>2</sup> and Chun-Jung Chen<sup>1</sup>; <sup>1</sup>Opto-Electronics & Systems Laboratories, Industrial Technology Research Institute, Chutung,, Hsinchu, Taiwan; <sup>2</sup>Mechanical and Automation Engineering, Da-Yeh University, Changhua, Changhua, Taiwan.

Ink jet technology is applied to the industry application of CPU cooling in this study. The PZT actuated micro-cooling device which generating the micro-scale liquid droplet is used in the CPU cooling of desktop and laptop PCs. It consists the advantages of simple device and easily manufactured of the structure with PZT actuator. Since the high latent heat transfer rate, 2260 J/g, of water droplet for the liquid-vapor phase change cooling system, it removes heat from the CPU surface effectively for over 100 W. In order to design an efficient cooling device, it is important to study the piezoelectric material property varies after the laser cutting process and the dimensional

effects of PZT actuator on the nozzle plate vibration mode. The frequency and amplitude of the voltage used to energize the PZT transducer are also important parameters that should be properly controlled in order to achieve the optimal liquid breakup conditions. It also reveals the low noise and power consumption in the CPU cooling system with some appropriate operational conditions.

**V3.6**  
**Fabrication of Directional Piezoelectric Transducers for Lamb Waves Propagating in Thin Aluminum Plate.** Takashi Iijima, Satoko Osone, Yoshiro Shimojo, Hideki Nagai, Nobuyuki Toyama and Junji Takatsubo; Research Institute of Instrumentation Frontier, National Institute of Advanced Industrial Science and Technology, Tsukuba, Japan.

A structural health monitoring system for identifying damage locations, especially in aircraft structures, has recently been a subject of considerable interest to reduce maintenance costs by allowing for a localized inspection, saving time and expense. The acoustic emission technique is useful for this purpose, since it has the advantage of the source location identification. Piezoelectric transducers operated in the thickness mode are generally adopted as sensor devices for detecting acoustic emissions. The conventional planar source location for a plate-like structure requires three or more receivers. The source location can be calculated using differences in arrival times of the acoustic emission at different receivers and the wave velocity. However, monitoring a large structure requires numerous receivers with different positions, complicating the monitoring system. Therefore, developing a new source location method that can reduce the number of receivers and/or their positions is important. In this study, to identify the planar location and the angle of a source using low-frequency Lamb waves, we develop a directional piezoelectric transducer element. Thin rectangular PZT elements were prepared and bonded on a 1-mm-thick aluminum plate. Lateral oscillation mode was adopted to detect the Lamb  $A_0$  mode propagating from the source. The dimensions of the element were designed so that the element has directional sensitivity for the 50 kHz  $A_0$  mode propagating in a 1-mm-thick aluminum plate. The length, longitudinal dimension, was selected to be sensitive to the 50 kHz  $A_0$  mode. It was therefore determined as 7 mm, which is equal to half the wavelength of the  $A_0$  mode. On the other hand, the width, transverse dimension, and thickness were intentionally selected to be insensitive to the 50 kHz  $A_0$  mode. In the case of bulk PZT transducers, their width was changed from 5 to 1mm, and the thickness was kept as 1mm. With decreasing width of the PZT transducer, the peak-to-peak amplitude of the detected signal in the width direction was decreased, whereas the amplitude in the length direction did not exhibit remarkable difference. This means that the directivity of the transducer is successfully developed and selectivity of the incident angle for the Lamb wave is progressed. To increase the directivity, film type rectangular shaped PZT transducer was prepared. The 10- $\mu\text{m}$ -thick PZT film deposited on Si substrate, and the film was etched rectangular shape using a RIE process. The directivity of the PZT film transducer is undertaken now.

**V3.7**  
**The Effect of the Metal Gate Design on the MIS Sensor's Performance.** Jagdish S. Thakur<sup>1</sup>, Linfeng Zhang<sup>1</sup>, Lajos Rimai<sup>1</sup>, Ratna Naik<sup>2</sup>, K. Y. Simon Ng<sup>3</sup> and Gregory W. Auner<sup>1</sup>; <sup>1</sup>Electrical and Computer Engineering, Wayne State University, Detroit, Michigan; <sup>2</sup>Physics and Astronomy, Wayne State University, Detroit, Michigan; <sup>3</sup>Chemical Engineering and Materials Science, Wayne State University, Detroit, Michigan.

Research activities in hydrogen storage, generation, and fuel cells are gaining a wide spread attention due to their tremendous applications. However, hydrogen becomes explosive when its concentration in air exceed more than 4 vol %, so there is an acute demand for a highly sensitive, selective, and stable sensor which can monitor a small hydrogen concentration fluctuations in a given environment. Metal-insulator-semiconductor (MIS) type sensor with Pd alloy/AlN/Si structure shows high sensitivity and selectivity to hydrogen. This type of sensor works as a capacitor. In the depletion region of the capacitance-voltage (C-V) curve, the voltage shift at certain capacitance is directly related to the device response to hydrogen. Since the composition and structure of the metal gate can greatly affect the operating parameters and the performance of the sensor, it is important to understand the relationship between the nature of metal gate and the electrical characteristics. The objective of this study is to investigate the C-V characteristic as a function of metal gate composition (Pd, PdCr, PdCr/Pd, and Pd/PdCr) of the sensors. The insulator AlN layer was grown on top of n-Si with Plasma Source Molecular Beam Epitaxy (PSMBE), and different metal gates were deposited with magnetron sputtering. The composition of the gate was studied by X-ray Photoelectron Spectroscopy (XPS). The C-V curves were obtained with the AC signal from 100 Hz to 1 MHz. In the C-V curves of these four devices, it is interesting to note that the single layer metal gated (Pd/AlN/Si

and PdCr/AlN/Si) and the double metal layer gated (Pd/PdCr/AlN/Si and Pd/PdC/AlN/Si) devices fall into two distinct groups. The double layer gated samples showed a large shift, about 6 V, as compared to the single layer gated device. In addition, there was a decrease in the measured capacitance for the double layer gated device suggesting an existence of additional positive charges. The interface state densities for single metal layer gate samples are calculated and the plausible relationship between these electrical characteristics and the performance of the sensor is discussed.

### V3.8

#### TiO<sub>2</sub> Anatase Thin Films Based CO Gas Sensor.

Ibrahim A. Al-Homoudi<sup>1</sup>, L. Rimai<sup>2</sup>, R. Naik<sup>3</sup>, R. J. Baird<sup>2</sup>, G. W. Auner<sup>2</sup> and G. Newaz<sup>1</sup>; <sup>1</sup>Mechanical Engineering, Wayne State University, Detroit, Michigan; <sup>2</sup>Electrical and Computer Engineering, Wayne State University, Detroit, Michigan; <sup>3</sup>Physics and Astronomy, Wayne State University, Detroit, Michigan.

TiO<sub>2</sub> anatase thin films with different thickness (100–1200 nm) have been deposited on glass, sapphire and Si(100) substrates using pulsed DC magnetron reactive sputtering. Pt electrodes were deposited on the top of the thin films using DC reactive sputtering. The thin films were exposed to carbon monoxide (CO) gas at different concentrations (20–100 ppm) in a nitrogen carrier, and the resistance was measured as a function of the CO concentration for films of different thicknesses for temperatures in the range of (100–300°C). The film with good crystal structure showed better response than amorphous films. The responses increased monotonically with CO. The thicker film showed higher sensitivity. The anatase films deposited on sapphire had generally the larger response than those deposited on glass or on silicon. The films on sapphire substrate showed good response to temperatures as high as 300°C, while those deposited on glass and silicon had good responses only to 200°C. Furthermore, the addition of different concentrations of O<sub>2</sub> and H<sub>2</sub> showed different response as expected. The response to CO in the presence of N<sub>2</sub> only showed good reversibility which is evidence that complete regeneration on turn off the CO does not require exposure to oxygen or air. Thus, the carbon monoxide (CO) interacts directly with the oxide rather than adsorbed oxygen. The response and the recovery times are fast.

### V3.9

#### Degradation Of Implantable Electrochemical Devices For Glucose Sensing And Ways Of Improving Its Long-Term Stability.

Ritesh Tipnis<sup>1</sup>, SanthiSagar Vaddiraju<sup>1</sup>, Faquir Jain<sup>2</sup>, Diane Burgess<sup>3</sup> and Fotios Papadimitrakopoulos<sup>4</sup>; <sup>1</sup>Nanomaterials Optoelectronics Laboratory, Polymer Program, Institute Of Materials Science, University Of Connecticut, Storrs, Connecticut; <sup>2</sup>Nanomaterials Optoelectronics Laboratory, Electrical and Computer Engineering, University Of Connecticut, Storrs, Connecticut; <sup>3</sup>Department Of Pharmaceutical Sciences, University Of Connecticut, Storrs, Connecticut; <sup>4</sup>Nanomaterials Optoelectronics Laboratory, Polymer Program, Department Of Chemistry, Institute Of Materials Science, University Of Connecticut, Storrs, Connecticut.

The search for long-lived implantable sensors for continuous glucose monitoring has been the focus of research community for more than 30 years. Our group has been actively involved in the development of a Humic Acids (HAs) based semipermeable membrane which can alleviate several of the problems plaguing these devices. By varying the thickness of this membrane, we could introduce a diffusion limiting barrier towards sampling of glucose, thus restricting the amount of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) being produced. We believe the over-sampling of glucose to be the cause of electrode fouling as well change in the redox characteristics of sensor coatings. Herein we report on the enhancement in the sensor design which has led to long-term stability during *in vitro* testing as well as improvement in its sensitivity at higher glucose concentrations. We would also be discussing further miniaturization of the devices enabling us to implant these for *in vivo* studies. Furthermore, we would be proposing a simple model which can assist in simulating an optimum sensor design and also suggest improved testing methodology that can prolong the life of implantable devices whilst maintaining high selectivity and sensitivity.

### V3.10

#### Decrease in Resistance of Ceria Oxygen Sensor Induced by 10 mol% Hf and Zr Doping.

Noriya Izu, Woosuck Shin, Ichiro Matsubara and Norimitsu Murayama; National Institute of Advanced Industrial Science and Technology (AIST), Nagoya, Japan.

Recently, exhaust gas regulations have been strict worldwide. Not only three-way catalysts but also oxygen sensors are indispensable for decreasing exhaust gas emission. Thus, recently, resistive oxygen sensors have been the focus of much attention. A resistive oxygen sensor using cerium oxide has been reported by several teams (3), (4), (6), (7). Since cerium oxide has a good corrosion resistance to corrosive gases (8), it is a promising candidate sensing material for

resistive oxygen sensors. However, ceria has a large resistivity. So a sensing material with a lower resistance is desired. In this study, resistive oxygen sensors using ceria doped with Hf and Zr were fabricated, and its sensing properties were investigated. We found that it had good sensing properties such as a low resistance. Cerium oxide powder doped with hafnium oxide was prepared by the solid-state reaction method. The obtained powder was mixed with an organic binder. The paste was screen-printed on an alumina substrate on which Pt electrodes with interdigital structures had been deposited. The screen-printed sample was fired at 1573 K in air for 2 h. Finally, Ce<sub>0.9</sub>Hf<sub>0.1</sub>O<sub>2</sub> and Ce<sub>0.9</sub>Zr<sub>0.1</sub>O<sub>2</sub> thick films with Pt electrodes were obtained. From XRD analysis, the Ce<sub>0.9</sub>Hf<sub>0.1</sub>O<sub>2</sub> and Ce<sub>0.9</sub>Zr<sub>0.1</sub>O<sub>2</sub> thick films had a single-phase fluorite structure. Thus, it was confirmed that Hf and Zr ions dissolved in the cerium oxide lattice. The peak position of Hf-doped ceria or Zr-doped ceria was higher than that of non doped ceria, that is to say, the lattice constant of Hf-doped ceria or Hf-doped ceria was smaller than that of non doped ceria. The resistance increased in the order of Ce<sub>0.9</sub>Hf<sub>0.1</sub>O<sub>2</sub> sensor, Ce<sub>0.9</sub>Zr<sub>0.1</sub>O<sub>2</sub> sensor, and CeO<sub>2</sub> sensor. At 873 K, the resistances of the Ce<sub>0.9</sub>Hf<sub>0.1</sub>O<sub>2</sub>, Ce<sub>0.9</sub>Zr<sub>0.1</sub>O<sub>2</sub>, and CeO<sub>2</sub> sensors were 0.11, 0.48 and 1.7 M respectively. So it was found that the Ce<sub>0.9</sub>Hf<sub>0.1</sub>O<sub>2</sub> sensor had a much lower resistance than the Ce<sub>0.9</sub>Zr<sub>0.1</sub>O<sub>2</sub> or CeO<sub>2</sub> sensor. We will discuss the decrease in resistance induced by Hf or Zr doping at the presentation of MRS. 1) E. Ivers-Tiffée, K.H. Hardtl, and W. Menesklou, J. Riegel, *Electrochimica. Acta.*, 47, 807 (2001). 2) R. Moos, F. Rettig, A. Hurland, and C. Plog, *Sen. Actuators B*, 93, 43 (2003). 3) N. Izu, W. Shin, I. Matsubara, and N. Murayama, *Sen. Actuators B*, 94, 222 (2003). 4) N. Izu, W. Shin, I. Matsubara, and N. Murayama, *Sen. Actuators B*, 101, 381 (2004). 5) J. Gerblinger, W. Lohwasser, U. Lampe, and H. Meixner, *Sen. Actuators B*, 26-27, 93 (1995). 6) H.-J. Beie and A. Gnorich, *Sen. Actuators B*, 4, 393 (1991). 7) P. Jasinski, T. Suzuki, and H. U. Anderson, *Sen. Actuators B*, 95, 73 (2003). 8) E. B. Varhegyi, I. V. Perczel, J. Gerblinger, M. Fleischer, H. Meixner, and J. Giber, *Sen. Actuators B*, 18-19, 569 (1994).

### V3.11

#### Effect of Temperature on the Resistivity of Polycrystalline 3C-Silicon Carbide Thin Films.

Jingchun Zhang<sup>1,2</sup>, Roger T. Howe<sup>1,3,4</sup> and Roya Maboudian<sup>1,2</sup>; <sup>1</sup>Berkeley Sensor & Actuator Center, University of California, Berkeley, California; <sup>2</sup>Department of Chemical Engineering, University of California, Berkeley, California; <sup>3</sup>Department of Electrical Engineering and Computer Science, University of California, Berkeley, California; <sup>4</sup>Department of Mechanical Engineering, University of California, Berkeley, California.

This work presents a study on the effect of temperature on the resistivity of n-type polycrystalline 3C-silicon carbide (poly-SiC) with various doping levels. The poly-SiC films are deposited at 800 °C on SiO<sub>2</sub> isolated Si(100) substrates by low-pressure chemical vapor deposition (LPCVD) using 1,3-disilabuanane (1,3-DSB) single precursor and in-situ doped by NH<sub>3</sub>. The temperature coefficient of resistivity (TCR) is characterized in the range of 304 to 638 K and is found to increase with temperature from - 2.4 to - 1.3 % K<sup>-1</sup> for undoped and - 0.17 to - 0.10 % K<sup>-1</sup> for most conductive samples. X-ray diffraction and Hall effect probe are performed to characterize the crystalline quality, carrier concentration and mobility of the films. The results are then discussed based on one-dimensional poly-Si model in order to explain the effect of temperature on the resistivity of poly-SiC.

### V3.12

#### Determination of Elastic Constants of Aluminum Nitride.

Soma S. Perooly<sup>1</sup>, Varun Garg<sup>2</sup>, Md. H. Rahman<sup>3</sup>, Leland W. Rosenberger<sup>3</sup>, Ronald F. Gibson<sup>2</sup> and Gregory W. Auner<sup>1</sup>; <sup>1</sup>Electrical Engineering, Wayne State University, Detroit, Michigan; <sup>2</sup>Mechanical Engineering, Wayne State University, Detroit, Michigan; <sup>3</sup>Chemical Engineering and Materials Science, Wayne State University, Detroit, Michigan.

Aluminum Nitride (AlN) is a technologically important wide band gap semiconductor as well as potential piezoelectric material for Biosensor applications [1]. Recently, the vibration characteristics of AlN-based MEMS sensors for the detection of Ultrasound have been studied [2]. In the current work, an attempt has been made to determine the true elastic constants of AlN under high vacuum (to avoid the effect of air damping) using Si micro-cantilevers, employing Micro Scanning Laser Doppler Interferometry. The micro-cantilevers are ideally suited for all applications where extremely small forces have to be detected, such as chemical and biochemical sensing or nanocalorimetry. Modal vibration frequencies for microcantilevers without and with the AlN coating will be used, along with analytical models and knowledge of the Si modulus, to back out the modulus of the AlN. Results of AlN deposited by low temperature Plasma Source Molecular Beam Epitaxy (PSMBE) with thickness of films ranging from 200 nm to 500 nm on micro cantilevers would be presented along with the characterization results. Laminated beam theory incorporating the coupling effect would be employed to evaluate the elastic constants.



So far the earlier studies have been done only using simple beam theory. Since the modulus of Si is known, the modulus of AlN can be evaluated using micro mechanics equations. Results of intentional defects generated, and the attempt to quantify the micro scale defects would also be presented. Reference: 1. J. Xu, J.S. Thakur, F. Zhong and G.W. Auner, *Appl.Phys.Lett*, 96, 212-217 (2004) 2. Ronald F.Gibson, Niranjan Srinivasan, Gregory Auner, Changhe Huang, Qianghua Wang and Soma S Perooly, Annual Conference of Society for Experimental Mechanics, Portland (2005)

### **V3.13**

**White electrophosphorescent devices based on iridium complexes via efficient energy transfer from conjugated polymer.** Tae-Ho Kim and O. Ok Park; Chemical and Biomolecular Engineering, Korea Advanced Institute of Science and Technology, Daejeon, South Korea.

We developed efficient white light-emitting diodes (LEDs) using polyfluorene-type blue-emitting conjugated polymer doped with green and red phosphorescent dyes. The emission spectrum of the conjugated polymer, which has a very high luminescent efficiency, showed a large spectral overlap with the absorbance of green and red iridium complexes. Also, efficient energy transfer from conjugated polymer to iridium complexes was observed. Poly(N-vinylcarbazole) (PVK) was used to improve the miscibility between conjugated polymer and iridium complexes because of their poor chemical compatibility and self-aggregation. The white emission spectrum was easily obtained by varying the contents of the three materials and controlling the phase morphology. Moreover, these white LEDs showed a voltage-independent electroluminescence due to a similar threshold and the driving voltage of the three materials caused by energy transfer.

### **V3.14**

**Amplified Fluorescent Polymers Sensors for the Detection of 2,3-Dimethyl-2,3-dinitrobutane and Hydrazine.**

Samuel William Thomas and Timothy M. Swager; Chemistry, MIT, Cambridge, Massachusetts.

The use of fluorescent conjugated polymer films for the trace detection of highly electron deficient vapors, such as 2,4,6-trinitrotoluene, is well established. Polymers used for this purpose to date, however, are somewhat limited in the scope of analytes to which they respond via fluorescence quenching. This is a result of the photoinduced charge transfer reaction that causes quenching being energetically favorable only for very electron deficient analytes. Here we report the preparation of new, low workfunction conjugated polymers aimed at the detection of analytes with less favorable reduction potentials, such as the plastic explosive taggant 2,3-dimethyl-2,3-dinitrobutane (DMNB). These polymers show interesting and unique optical properties. By manipulating both the ground state oxidation potentials as well as the band gaps of conjugated polymers, vapors of these target molecules can be detected. In addition, the use of these polymers to detect trace hydrazine vapor in a fluorescence turn on scheme will be presented. The introduction of oxidized trap sites into the polymer gives a quenched film. Exposure of the film to hydrazine vapor results in the reduction of the intercalated quencher sites and a turn on of the polymer emission. Sensitivity below 1 part-per-million has been demonstrated with this technique.

### **V3.15**

**Temperature sensor based on sol-gel (SiO<sub>2</sub>-Co) and a holey fiber.** Miguel Angel Basurto<sup>1</sup>, Rosenberg J. Romero<sup>1</sup>, Romeo

Selvas-Aguilar<sup>2</sup>, Ismael Torres-Gomez<sup>2</sup>, Jose Javier Sanchez-Mondragon<sup>3</sup>, Antonio Campero<sup>4</sup> and Celso Velasquez<sup>4</sup>; <sup>1</sup>CIICAp, Universidad Autonoma del Estado de Morelos, Cuernavaca, Morelos, Mexico; <sup>2</sup>Optica, CIO, Leon, Guanajuato, Mexico; <sup>3</sup>Optica, INAOE, Tonantzintla, Puebla, Mexico; <sup>4</sup>Quimica, Universidad Autonoma Metropolitana - Iztapalapa, Mexico, Mexico.

Optical fibers offer many convenient and reliable possibilities of building up sensor, in particular temperature sensors. The convenient architecture of Bragg Fibers, and in particular Holey fibers add to these possibilities by using new composite systems. The holes of Holey fibers have played a rather passive confining role of the radiation, however these can be enhanced by filling up or covering the interior walls of these holes with materials that are sensitive to temperature or other physical parameter, and we may build a new class of sensors. We have designed a temperature sensor by filling up the holey fiber holes with a Solgel (SiO<sub>2</sub>-Co), and designed a reference sensor by applying the same solgel material on both faces of a microscope slide. We characterized the operation of both systems by determining the correlation between the wavelength and the transmission of light, while the capability of temperature sensing of both systems has been determined by the relationship between the light transmission as a function of the temperature. Work supported by CONACyT, Mexico, under grant 45667 and ALFA Project IPECA.

### **V3.16**

**Soliton Propagation on periodic nonhomogenous media.**

Miguel Angel Basurto<sup>1</sup>, Jose Javier Sanchez-Mondragon<sup>2</sup>, D.

Rojano-Guido<sup>2</sup>, A. Alejo-Molina<sup>2</sup>, Romeo Selvas-Aguilar<sup>4</sup> and Miguel Torres-Cisneros<sup>3</sup>; <sup>1</sup>CIICAp, Universidad Autonoma del Estado de Morelos, Cuernavaca, Morelos, Mexico; <sup>2</sup>Optica, Instituto Nacional de Astrofisica Optica y Electronica, Tonantzintla, Puebla, Mexico; <sup>3</sup>FIMEE, Universidad de Guanajuato, Salamanca, Guanajuato, Mexico; <sup>4</sup>Optica, CIO, Leon, Guanajuato, Mexico.

Temporal and spatial solitons are at the core of different applications on optical communication and nonlinear processes. We have relied on propagation in an homogeneous media to study the different aspects of their interaction, decay and multiple amplifications and convergence to a single Soliton eigenvalues spectrum. However, the propagation in periodic inhomogeneous media of length longer than the wavelength, interrupt this convergence to the different solitons spectra corresponding to each media, leading to an oscillatory behavior that is described for a Two level atom atomic system and intensity dependent nonlinearities as those in Kerr, nonlinear optical fibers and Photorefractive materials. Work supported by CONACyT, Mexico, under grant 45667 and ALFA Project IPECA.

### **V3.17**

**Temperature sensing base on Optical Transmission in a Br-Li heat pump.** Miguel Angel Basurto<sup>1</sup>, J. Escobedo-Alatorre<sup>1</sup>,

Margarita Tecpoyotl-Torres<sup>1</sup>, Rosenberg Romero<sup>1</sup>, Jose Javier Sanchez-Mondragon<sup>2</sup> and Aurelio Horacio

Heredia-Jimenez<sup>2</sup>; <sup>1</sup>CIICAp, Universidad Autonoma del Estado de Morelos, Cuernavaca, Morelos, Mexico; <sup>2</sup>Optica, INAOE, Tonantzintla, Puebla, Mexico.

Cooling systems, such as the heat pumps based on lithium bromide, require of special temperature sensors because this liquid is highly corrosive. As an improvement on the current temperature approximation, we have introduced fiber sensors. We present the study of the behavior of the lithium bromide in different concentrations and in a range of temperatures from the room temperature to 70°C, and in a light transmission range that goes from 1000 nm to 4000 nm. The working points were 1330nm y 1550nm, where the communication windows were optical fibers. Work supported by CONACyT, Mexico, under grant 45667 and ALFA Project IPECA.

### **V3.18**

**Interferometer Type Pressure Microsensor.** Miguel Angel Basurto<sup>1</sup>, Rosenberg Javier Romero<sup>1</sup>, Amparo Andrade-Lucio<sup>4</sup>, Aurelio Heredia-Jimenez<sup>2,3</sup>, Jose Javier Sanchez-Mondragon<sup>2</sup> and A. Torres-J<sup>2</sup>; <sup>1</sup>CIICAp, Universidad Autonoma del Estado de Morelos, Cuernavaca, Morelos, Mexico; <sup>2</sup>Optica, INAOE, Tonantzintla, Puebla, Mexico; <sup>3</sup>UPAEP, Puebla, Puebla, Mexico; <sup>4</sup>FIMEE, Universidad de Guanajuato, Salamanca, Salamanca, Mexico.

We aim at the research, manufacturing and testing of an optical type microsensor, by using an electrooptical modulator based on the MEMS technology. The measurement configuration chosen to detect the variation produced in the light properties is an interferometer configuration of the Mach Zender Type (MZI), because of its high sensitivity. One of its arms is positioned on a floating silicon nitride membrane, that becomes deformed under small pressures on the range of mili Pascuales. According to the parameters of the membrane and the waveguide we obtain a maximum deformation of 236 nm, that produces a dephasing of 180° between the two waves, producing therein a null interferometer output for the maximum pressure. The material used for the manufacturing of the waveguide is an alloy of amorphous silicon with Germanium doped with boron, with a refraction index of 4.124 and infrared transmittance of 0.8 to 5 μm at the 80%. The floating silicon nitride membrane has a thickness of 200 nm, a width of 50mm, a length of 1000 mm and a refraction index of 2. The optical waveguides that form the MZI must satisfy two conditions: single mode and high superficial sensitivity; therefore we will use the waveguides of the ARROW type (antiresonant Reflecting Optical Waveguides). It was found that the value of the maximum phase difference of the modulator, for a deformation of the membrane of 236 nm, corresponds to a value of  $1/\{2(n-1)\}$ . This value will allow us to predict a maximum pressure that can be applied on the sensor. Work supported by CONACyT, Mexico, under grant 45667 and ALFA Project IPECA.

SESSION V4: Shape Memory Alloys and Magnetostrictive Devices  
Chairs: A. Flatau and S. Miyazaki  
Tuesday Morning, November 29, 2005  
Independence W (Sheraton)



8:00 AM \*V4.1

**Microactuators Utilizing SMA Sputter-deposited Thin Films.** Shuichi Miyazaki, Institute of Materials Science, University of Tsukuba, Tsukuba, Ibaraki, Japan.

The shape memory characteristics associated with the martensitic transformation of the Ti-Ni, Ti-Ni-Pd and Ti-Ni-Cu and those associated with the R-phase transformation of the Ti-Ni will be shown and the actuator characteristics of microactuators utilizing these thin films will be presented. These alloys can show the stable shape memory effect and superelasticity which are equivalent to those of bulk materials produced by conventional melting methods. Because of the unique microstructures consisting of non-equilibrium thin plate precipitates in the sputter-deposited thin films, they can sometimes show the stable properties even better than the bulk materials. Three types of high-speed diaphragm-type microactuators utilizing the TiNi-based thin films were also developed. One type is a microactuator utilizing the martensitic transformation of a Ti-Ni-Pd thin film which has high transformation temperatures. Cooling rate of a micro-actuator increases with increasing the temperature difference between the transformation and atmosphere temperatures, thus increasing the response of actuation. The second type is a microactuator utilizing the martensitic transformation of a Ti-Ni-Cu thin film which has a transformation temperature hysteresis which is one third of that of the Ti-Ni. The third type is a microactuator using the R-phase transformation of a Ti-Ni thin film which has a narrowest transformation temperature hysteresis. In the case of the R-phase transformation, the forward and reverse transformations occur with such a narrow temperature hysteresis, thus being effective to increase the response of actuation. These three types of actuators are promising for high response applications. The working frequency of the microactuators utilizing the Ti-Ni-Pd and Ti-Ni-Cu thin films reached 100Hz, and that of the microactuator using the R-phase transformation reached 125Hz. More challenges for developing fabrication methods of microdevices are requested for many scientists and engineers to extend the possibility of applications utilizing SMA thin films.

8:30 AM \*V4.2

**Fabrication of Superelastic NiTi Thin Films and Net-Shaped Structures.** Holger Rumpf, Volker Wipperfuhr, Christiane Zamponi and Eckhard Quandt; caesar, Bonn, Germany.

NiTi thin films have been fabricated by magnetron sputtering technique on heated substrates with sub-sequent rapid thermal annealing on the released freestanding films. Differential scanning calorimetry (DSC) as well as tensile testing showed results comparable with bulk material with a slightly adjustable AF temperature at about room temperature, a superelastic strain of more than 6.5% at 400MPa, and an ultimate high tensile strength exceeding 1100 MPa. Composition (nickel and titanium) and contamination (oxygen, carbon) have been determined using Auger Electron Spectroscopy (AES) and energy dispersive x-ray analysis (EDX) which agree with data known for commercially available bulk superelastic NiTi material. Contrary to results for bulk NiTi, electron microscopy investigations revealed, that the microstructure showed significant differences in the grain size and in the presence and size of Ni<sub>4</sub>Ti<sub>3</sub> precipitates. Microfabrication techniques (photolithography and wet etching) have been applied in order to create net-shaped structures in the freestanding films which are potential candidates for vascular implants. Financial support of the German Federal Ministry of Education and Research (BMBF) through 03N4031A and the German Research Foundation through SFB 459 is gratefully acknowledged.

9:00 AM V4.3

**Crystallization Kinetics of Amorphous NiTi Shape Memory Alloy Thin Films.** Xi Wang and Joost J. Vlassak; Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

The crystal structure of NiTi films sputter-deposited at room temperature is amorphous. Consequently, these films need to be crystallized by annealing them at elevated temperature before their shape memory properties can be used in micromachined devices. As will be demonstrated, this annealing process allows unprecedented control over the microstructure of the films. The crystallization kinetics of amorphous sputter-deposited NiTi thin films was studied in the temperature range from 400°C to 465°C. Nucleation and growth rates were determined by annealing SiNx-capped NiTi coatings in a furnace with precise temperature control and by tracking the growth of individual crystallites using optical and atomic force microscopy. Nuclei form homogeneously in the amorphous matrix resulting in films with a random crystallographic texture. The nuclei quickly consume the film thickness, and then transition to a two-dimensional growth mode. Interfaces with the substrate or with the SiNx capping layer do not act as preferential sites for nucleation. This is attributed to a small shift in composition at these interfaces that locally increases the

crystallization temperature. Direct observation of crystals makes it possible to establish for the first time the activation energies of both the nucleation and the growth process. By manipulating the nucleation and growth rates during the crystallization process, the average grain size of an 800nm film was varied from approximately 2 to 60 micron, resulting in near single-crystal films.

9:15 AM V4.4

**A Combinatorial Approach to Identifying "Ideal" Shape Memory Alloys.** Jun Cui<sup>1</sup>, Jason Ryan Hatrick-Simpers<sup>1</sup>, Olugbenga O. Famodu<sup>1</sup>, Yong Chu<sup>2</sup>, Sigurd Thienhaus<sup>4</sup>, Alfred Ludwig<sup>4</sup>, Richard. D. James<sup>3</sup>, Manfred Wuttig<sup>1</sup> and Ichiro Takeuchi<sup>1</sup>; <sup>1</sup>Materials Science and Engineering, University of Maryland, College Park, Maryland; <sup>2</sup>Advanced Photon Source, Argonne National Laboratories, Argonne, Illinois; <sup>3</sup>Department of Aerospace Engineering and Mechanics, University of Minnesota, Minneapolis, Minnesota; <sup>4</sup>Combinatorial Materials Science, CAESAR, Bonn, Germany.

"Ideal" shape memory alloys are a subset of shape memory alloys with superior fatigue properties due to their minimized thermal hysteresis. Recent advent of the geometric non-linear theory of martensites has provided criteria for the search for the ideal shape memory alloys which are solely based on their lattice parameters. The search is therefore reduced to exploring the composition space of candidate systems for appropriate lattice parameters. The combinatorial approach to materials provides a ready tool for the rapid screening of hundreds of samples with systematically varying compositions for properties of interest. We have used the combinatorial approach to search within various ternary systems for potential ideal shape memory alloys. The composition spread samples were synthesized in our combinatorial ultra high vacuum magnetron co-sputtering chamber. Mapping of the crystal structure was performed using synchrotron x-ray microdiffraction at the Argonne Advanced Photon Source. Various fitting programs were used as refinement tools to quickly analyze the diffraction data and screen for compositions that most closely approximate the conditions put forth by the geometric non-linear theory. Thermal hysteresis of the martensites was mapped using a variable temperature 4-point probe measurement scheme. Results on the Ni-Ti-Cu and Ni-Ti-Pd systems will be discussed and compared.

9:30 AM V4.5

**Reversible Shape Change of Shape Memory Composites using TiNi Wire and Elastic Materials.** Hitoshi Yoshida<sup>1</sup>, Hideki Nagai<sup>2</sup>, Ryutaro Oishi<sup>2</sup>, Norio Ami<sup>4</sup> and Ya Xu<sup>3</sup>; <sup>1</sup>Chuo University, Tokyo, Japan; <sup>2</sup>National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Japan; <sup>3</sup>National Institute for Materials Science, Tsukuba, Japan; <sup>4</sup>Chiyoda Maintenance Co. Ltd., Asahi, Kashima-gun, Japan.

This paper describes smart structure composed of TiNi shape memory alloys (SMA) and elastic materials. TiNi-SMA is one of effective materials which possess actuator function and widely used for smart structure. However, in the conventional application of SMA, their shape change is non-reversible. It is known that SMA can have two-way shape memory effect (TWE) and it is well studied. However, two-way SMA is not put into practical use for products because stability or durability of the effect is not enough and strength of force generated by TWE in lower temperature is not sufficient. Furthermore, it is difficult to set the shape of TWE correctly, and it cannot design as industrial products. Also it is not used for smart structure. Composing SMA with elastic materials, the composite structures can respond reversibly depending on temperature. It is a kind of shape memory composites. In this paper, TiNi wires are composed with high rigidity resin or CFRP or metal and ring shape type is investigated. Analytical models of reversible change and generated forces are led based on beam theory. We make both high temperature opening type and closing type and the curvature radius of the composites in each temperature was measured. The validity of proposed analytical models is verified by comparison with experimental results.

9:45 AM V4.6

**Superelastic Shape Memory Behaviour of NiTi Studied with Hard X-Ray Synchrotron Radiation.** Wolfgang W. Schmahl<sup>1</sup>, Mahamudul Hasan<sup>1</sup>, Susanne Gollerthan<sup>2</sup>, Holger Nebel<sup>2</sup>, Alberto Baruj<sup>3</sup>, Jafar Khalil-Allafi<sup>4</sup> and Reinhart Job<sup>5</sup>; <sup>1</sup>Earth and Environmental Sciences, Munich University, Munich, Germany; <sup>2</sup>Institute of Materials, Ruhr-University, Bochum, Germany; <sup>3</sup>Centro Atomico Bariloche, S. C. de Bariloche, Argentina; <sup>4</sup>Faculty of Materials Engineering, Sahand University of Technology, Tabriz, Iran; <sup>5</sup>Electrical Engineering and Information Technology, University of Hagen, Hagen, Germany.

Physically meaningful micromechanical models of polycrystalline

shape memory materials rely on the description of preferred orientation of ferroelastic twin variants in the martensite phase. We investigated the phase transition behaviour of superelastic NiTi in flat tensile test specimens (30 mm gauge length and 1.8mm x 6 mm cross section) by synchrotron hard x-ray diffraction at the wiggler-beamline BW5 at HASYLAB, Hamburg, Germany, using an area detector to be able to reconstruct texture information from the intensity distribution in Debye-Scherrer diffraction cones. The stress-induced transition from the cubic B2 austenite to the monoclinic B19' martensite phase is localized in Luders-like transformation shear bands. Residual austenite is observed to persist in the bands up to the end of the superelastic plateau in the stress-strain curve. The plateau in the superelastic stress-strain curve is associated with an extension of the transformation shear bands, while the austenite/martensite ratio inside the bands remains constant. The original texture of the cold-rolled specimen, the texture formation due to stress-induced twin variant selection in the martensite phase, and the texture of the residual austenite were obtained as a function of applied stress. We use the  $a = 0.289$  nm,  $b = 0.462$  nm,  $c = 0.413$  nm,  $\gamma = 96.7$  degrees crystal lattice setting for monoclinic B19' martensite. To optimize recoverable strain, the length of the flat tensile test sample was cut along the axis of cold rolling, and the austenite texture was such that {110} lattice plane normals of austenite were aligned preferentially along the tensile axis. Martensite variants accommodate to the stress field aligning their (020)M plane normal parallel to the tensile axis, giving a near gaussian distribution with a FWHM of 40 degree angular spread. The (020)M lattice spacing increases by 9.5% at the stress-induced phase transition. The remaining martensite poles (111, 002, 11-1) related to austenite {110} planes show a bimodal orientational probability density accumulation near 70 degree and 110 degree orientation relative to the tensile axis (i.e. +20 degree and -20 degree from the transverse direction).

#### 10:30 AM V4.7

##### Nanoindentation of NiTi Shape Memory Alloys.

Carl Pieter Frick<sup>1</sup>, Travis Lang<sup>1</sup>, Kevin Spark<sup>1</sup> and Ken Gall<sup>2</sup>;

<sup>1</sup>Mechanical Engineering, University of Colorado, Boulder, Colorado; <sup>2</sup>School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia.

Shape memory alloys, such as Nickel-Titanium (NiTi), have the ability to regain their shape prior to deformation through a reversible thermo-elastic phase transformation. Specifically, NiTi can restructure itself from a B2 austenite phase, to a B19' martensite phase through a decrease in temperature or an increase in applied stress. This reversible solid-state phase transformation gives NiTi the ability to recover large strains, either spontaneously (pseudoelasticity) or through an increase in temperature (shape memory effect). It is this unique deformation recovery that allows NiTi to be potentially utilized as a mechanical device at the atomic scale. Already NiTi has applications in MEMS including micro-fluid flow valves and micro-actuators, in part due to the extremely high work output per unit volume the phase transformation provides. However, the basic science related to the nucleation of a martensitic phase transformation is not well understood, and investigation of the martensitic phase transformation at small scales is necessary to derive a fundamental understanding of the phase transformation kinetics. This study investigates the thermoelastic shear-driven martensitic transformation of NiTi at the nano-scale, and illustrates a connection between the martensite phase transformation over a nanometer-sized volume to the local material structure. Specifically, testing was performed on specimens machined from hot-rolled, polycrystalline Ti-50.9 at.%Ni. Utilizing conventional bulk material allows for comparison between standard monotonic stress-strain response and mechanical behavior determined with nanoindentation. In addition, the slightly nickel-rich NiTi was given heat treatments at 350 oC and 450 oC for 1.5 hours in order to create Ti3Ni4 precipitates. These precipitates characterized via transmission electron microscopy, strongly influence transformation temperatures and consequently were used to elicit either pseudoelastic or shape memory behavior. Small-scale mechanical behavior was probed by nanoindentation performed using a Berkovich indenter tip at a constant strain rate of 0.05. Both materials displayed reverse phase transformation upon unloading, specifically the shape memory NiTi showed excursions during both the loading and unloading. This demonstrated the first observations of a reversible martensitic transformation over a discrete nanometer length-scale in a metal. Furthermore, atomic force microscopy measurements subsequent to nanoindentation illustrated increasing deformation recovery upon heating as a function of decreasing indentation depth, for both the shape memory and pseudoelastic NiTi. Finally, a hertzian analysis estimated the transformation stress during nanoindentation to be an order of magnitude larger than that shown macroscopically with monotonic compression testing.

#### 10:45 AM V4.8

##### Structural and Magnetic Properties of Single Crystal Ferromagnetic Shape Memory Ni<sub>2</sub>FeGa Films Grown by

Molecular Beam Epitaxy. T. C. Shih<sup>1</sup>, S. McKernan<sup>1</sup>, R. D. James<sup>2</sup> and C. J. Palmstrom<sup>1</sup>; <sup>1</sup>Chemical Engineering and Material Science, U of Minnesota, Minneapolis, Minnesota; <sup>2</sup>Aerospace Engineering and Mechanics, University of Minnesota, Minneapolis, Minnesota.

Ni<sub>2</sub>FeGa is a recently reported ferromagnetic shape memory material showing modulated structures in bulk martensitic phase, resembling the modulations observed in Ni<sub>2</sub>MnGa. As the modulation in the martensitic phase is considered as a key factor for twin mobility and therefore the ferromagnetic shape memory effect, Ni<sub>2</sub>FeGa draws a special interest for structural studies. For this reason, single crystal ferromagnetic Ni<sub>2</sub>FeGa films were grown on GaAs (001) substrates using molecular beam epitaxy. The growth layout is as follows: Al / Ni<sub>2</sub>FeGa / Sc<sub>0.3</sub>Er<sub>0.7</sub>As / GaAs / Al<sub>0.6</sub>Ga<sub>0.4</sub>As / GaAs (001) heterostructure, with the Al<sub>0.6</sub>Ga<sub>0.4</sub>As served as a etch-stop layer for the processing of Ni<sub>2</sub>FeGa free-standing films, the Sc<sub>0.3</sub>Er<sub>0.7</sub>As served as a growth template and a diffusion barrier, and the Al served as a capping layer. In situ reflection high energy electron diffraction, x-ray diffraction, and transmission electron microscopy suggest that the Ni<sub>2</sub>FeGa films grown at 300 ° C form a tetragonally distorted L2<sub>1</sub>-like single crystal with  $a = 5.653$  Å and  $c = 5.941$  Å. The plan-view transmission electron micrographs indicate the free-standing Ni<sub>2</sub>FeGa films transformed into twin structures at room temperature. The saturation magnetization of the Ni<sub>2</sub>FeGa films is 510 emu/cm<sup>3</sup> and the Curie temperature is above 400 K. Effects of growth temperatures on the resulting crystal properties will also be addressed in this presentation.

#### 11:00 AM V4.9

##### Magnetostrictive Vibration Sensor based on Iron-Gallium

Alloy. Supratik Datta and Alison B. Flatau; Aerospace Engineering, University of Maryland, College Park, Maryland.

Structural health monitoring of moving parts is a challenging task. There has been a growing need to develop non-contact strain, force and torque sensors for use in real time health monitoring. Fleming and Garshelis have shown promising work in this area using magnetostrictive materials and such sensors have been developed for application in racing cars. Wuttig has discussed the usage of magnetostrictive thin films in MEMS to produce small, low-cost yet sensitive sensors. A good magnetostrictive sensor should have high sensitivity to strain, large bandwidth for dynamic application, linear characteristics over a large range, low sensitivity to temperature and low hysteresis. Iron-Gallium alloys (Galfenol, Fe<sub>1-x</sub>Ga<sub>x</sub>, 0.13 < x < 0.21) appear to be a promising new magnetostrictive material for such applications and may offer improvements in performance and ease of use because of its high stiffness, machinability and ductility. This work will discuss the results obtained from a prototype sensor which uses a single crystal Fe<sub>84</sub>Ga<sub>16</sub> patch (25 x 8.44 x 1.88 mm<sup>3</sup>) as the sensor material. The prototype vibration sensor has a Galfenol patch attached on the top surface of an Aluminum beam (317.5 x 24.85 x 1.6 mm<sup>3</sup>), with its <100> crystal axis along the length of the beam. A DC magnetic bias is provided to the Galfenol patch. The beam is attached to a magnetic shaker through its center of mass. An accelerometer placed on top of this attachment measures the input signal. Another accelerometer placed at the same distance from the attachment as the Galfenol patch provides a benchmark for the output signal. The change in magnetic induction in the Galfenol patch is recorded using a Hall-effect sensor attached to it. Sweep-sine tests are run and results are obtained for different maximum amplitude of shaker tip displacement. Transfer functions are obtained for the Hall sensor output signal and the accelerometer output signal with respect to the accelerometer input signal and their frequency responses are compared. This work provides information about the effect of loading magnitude and frequency on a magnetostrictive Galfenol sensor. This information can serve as basic design parameters for fabricating magnetostrictive MEMS sensor which can find application as wireless strain gages or non-contact torque sensors.

#### 11:15 AM V4.10

**Free-Energy Model for Actuation and Sensing Behavior of Magnetostrictive Materials and Its Application to Single Crystal Iron-Gallium Alloys.** Jayasimha Atulasimha and Alison B. Flatau; Aerospace Engineering, University of Maryland, College Park, College Park, Maryland.

MOTIVATION: Iron-Gallium alloys (Fe-Ga) exhibit moderate magnetostriction ( $\lambda \sim 350$  ppm) under very low magnetic fields ( $H \sim 400$  Oe), large  $d_{33}^*$  (sensitivity  $\sim 30$  Tesla/GPa) and very low hysteresis, making them promising materials for both actuation and sensing applications [1]. The use of Fe-Ga in these applications, particularly in controls and optimized transducer design, can be greatly enhanced by modeling of its magneto-mechanical behavior, viz., its actuation characteristics ( $\lambda$ -H and B-H curves) and sensing characteristics (B vs. stress). Previous models for actuators have employed both the Jiles-Atherton [2] approach and the free-energy

based methods [3]. More recently, attempts have been made to extend the Jiles-Atherton approach to model sensing behavior [4]. However, a comprehensive free-energy approach which can model the actuator behavior over a wide range of compressive and tensile stresses as well as predict the sensing behavior of that material (at various applied bias fields) using model parameters estimated from the actuation behavior is lacking. It is the objective of this work to both develop such a model and test it with data obtained from characterization of single-crystal FeGa samples. MODELING APPROACH: The free energy is constructed to include the magnetocrystalline, external field (stray field energy is accounted for by a demagnetization factor) and magnetoelastic energy terms. The sample is assumed to be composed of a large number of domains whose magnetization vector is free to rotate (or flip) to an orientation which minimizes this free energy. The magnetostriction and magnetization are calculated from the distribution of the orientations of these domains. The response of these domain orientations to varying field (at constant stress) determines the actuation behavior of this material while the response to varying stress (at constant field) determines its sensing behavior. In summary, this approach has the framework to model both actuation and sensing behavior in various crystallographic directions. REFERENCES: 1. Kellogg, R.A., "Development and Modeling of Iron-Gallium alloys", PhD Thesis, Engineering Mechanics, Iowa State University, Ames, Iowa, 2003. 2. Jiles, D.C., and Atherton, D.L. "Theory of the Magnetization process in Ferromagnets and its application to the Magneto mechanical effect", J. Phys. D: Appl. Phys., 17 (1984) 1265-1281. 3. Smith, R.C, Dapino, M. and Seelecke, S., "Free Energy model for Hysteresis in Magnetostrictive Transducers", J. of Applied Physics, January 2003. 4. Dapino, M., Smith, R.C., Calkins, F.T. and Flatau, A.B., "A Magnetoelastic model for Villari-Effect in Magnetostrictive Sensors", Journal of Intelligent Material Systems and Structures, 13(11), pp. 737-748, November 01, 2002.

#### 11:30 AM V4.11

**Bimorph-type Magnetostrictive Actuator/Sensor Thin Films.** Teiko Okazaki<sup>1</sup>, Norimasa Okanisi<sup>1</sup>, Yuuki Miura<sup>1</sup>, Yasubumi Furuya<sup>1</sup>, Ichiro Takeuchi<sup>2</sup> and Manfred Wuttig<sup>2</sup>; <sup>1</sup>Hirosaki Univ., Hirosaki, Japan; <sup>2</sup>Materials Science and Engineering, University of Maryland, College park, MD20742.

Giant magnetostrictive Fe-Pd and Fe-Ga thin films are expected as actuator/ sensor materials with high respective velocity and huge stress created by the magnetostriction. We have reported that rapid-solidified melt-spun Fe-29.6at%Pd and Fe-17at%Ga thin foils show large magnetostriction of 1100 and 270 ppm, respectively. However, in order to achieve saturation magnetostriction, large magnetic field of 10 kOe is required. In order to take magnetostrictive material induced by low magnetic field, we investigated bimorph-type magnetostrictive (positive magnetostriction / substrate / negative magnetostriction) thin films, that is, Fe-Pd(1, 3micrometermeter) or Fe-Ga(1, 3micrometermeter) / Al(50micrometermeter) / Ni(1, 3micrometermeter) by magnetron sputtering system. Magnetostriction of these films reached about 300 ppm under applying of low magnetic field of 300Oe and exhibits little hysteresis. Moreover, the magnetostriction of the Fe-Ga bimorph-type film rapidly increases in lower field range under 100 Oe, on the other hand, the magnetostriction of the Fe-Pd bimorph-type film increases lineally in the field of 500 Oe. These magnetostrictive properties are maintained under low alternating magnetic field. The magnetoelastic coefficient of these thin films is about 30 MPa, which is comparable to the coefficient of the PbTiO<sub>3</sub>-PbZrO<sub>3</sub> system. The bimorph-type magnetostrictive thin film is useful for application in micro-pump and magnetostrictive 2D-scanners.

#### 11:45 AM V4.12

**Micromagnetic Modeling of Released/Non-released Magnetostrictive Films.** Yi-Chung Shu and Jui-Hen Yen; Institute of Applied Mechanics, National Taiwan University, Taipei, Taiwan.

We study the magnetostrictive behavior of two types of films: one is released from the substrate but constrained from its lateral boundary and the other is attached to the substrate. The former is investigated using the recently developed micromagnetic model which is suitable for films subjected to realistic magneto-mechanical boundary conditions. It is different from the phase-field model which is typically formulated with periodic boundary conditions. The stress-induced magnetic field is computed in the sense of average which is consistent with the demagnetization field evaluated also in the sense of average in the conventional computational micromagnetics. In addition, this average field is derived analytically so that the simulation of domain evolution is able to be performed at a larger scale with few iteration steps as well as no extra numerical truncations. The second case is modeled within a framework extended from our recent theoretical paper studying rough alloy films with composition modulation. The stress-induced magnetic field for an attached film depends not only on the changes of spontaneous magnetostrain but also on the elastic

interaction of the film with the substrate. We compare these two different cases by using various computer simulations and conclude with several discussions.

### SESSION V5: Nanometer Scale Processing and Properties

Chairs: Yasubumi Furuya and P.F. Gobin  
Tuesday Afternoon, November 29, 2005  
Independence W (Sheraton)

#### 1:30 PM \*V5.1

**Characterization of calcium phosphate films prepared by RF magnetron sputtering.** Takayuki Narushima<sup>1</sup>, Kyosuke Ueda<sup>2</sup>, Takashi Goto<sup>3</sup>, Tomoyuki Katsube<sup>1</sup>, Hiroshi Kawamura<sup>4</sup>, Chiaki Ouchi<sup>2</sup> and Yasutaka Iguchi<sup>2</sup>; <sup>1</sup>Tohoku University Biomedical Engineering Research Organization (TUBERO), Sendai, Japan; <sup>2</sup>Department of Materials Processing, Tohoku University, Sendai, Japan; <sup>3</sup>Institute for Materials Research, Tohoku University, Sendai, Japan; <sup>4</sup>Graduate School of Dentistry, Tohoku University, Sendai, Japan.

Calcium phosphates such as hydroxyapatite (HAp) and tricalcium phosphate (TCP) have been used as ceramic biomaterials with osteoconductivity and bioresorbability. One of their applications is coating for metallic implants. In the present work, calcium phosphate films were prepared on commercially pure titanium (CP-Ti) substrates by RF magnetron sputtering using beta-TCP targets, and phases, preferred orientation and reactivity of the films were investigated. Hot-pressed beta-TCP plates with a high density (>99.6 %) were used as a sputtering target. The size of the Cp-Ti substrate was 10 mm x 10 mm x 1 mm. The substrate was not intentionally heated. The total gas flow rate was kept at a value of 20 sccm and the oxygen gas concentration in the sputtering gas (argon-oxygen mixture gas) was controlled between 0 and 95 %. The total pressure in the chamber was varied from 0.1 to 15 Pa, and RF power was changed from 75 to 150 W. XRD and FTIR analyses revealed that the films consisted of amorphous calcium phosphate and oxyapatite phases. The oxyapatite phase was detected in the conditions at higher RF powers and oxygen gas concentration in the sputtering gas and lower total pressures. The preferred orientation of a specific plane for the oxyapatite phase in the films was evaluated by Lotgering factor, which was calculated using the ratio of intensity summation for an experimental data and that of JCPDS data in XRD patterns. The (002) preferred orientation of oxyapatite phase was observed. It is known that HAp exhibits the anisotropy of reactivity and mechanical properties. Since the structure of oxyapatite is close to that of HAp, it will be effective that the preferred orientation of oxyapatite films can be controlled for the biomedical applications. Significantly (002) oriented oxyapatite films were obtained under the process conditions at lower total pressure and oxygen gas concentration in the sputtering gas. The surface reactions of the calcium phosphate films in simulated body fluids were studied. In PBS(-), the amount of calcium ion eluted from amorphous calcium phosphate film was larger than that from oxyapatite film. The CP-Ti plates coated with the calcium phosphate films were placed on mandible of male Japanese white rabbits (2.5-3.0 kg). The CP-Ti plates were in close contact with bone at some parts of the interfaces between the bone and the implant, compared with non-coated titanium plates.

#### 2:00 PM V5.2

**Nonlinear Optical Dynamics During Phase Transition in Vanadium Dioxide.** Sergiy Lysenko, Valentin Vikhain, Guangjun Zhang, Armando Rua, Felix Fernandez and Huimin Liu; Department of Physics, University of Puerto Rico, Mayaguez, Puerto Rico.

Design and development of light-controlled solid-state optical switchers, storage devices, infrared laser cavity mirrors based on metal-oxide films is upcoming branch of modern optoelectronic technology. In this work the laser induced insulator-to-metal (I-M) phase transition in VO<sub>2</sub> thin films was explored by femtosecond optical pump-probe spectroscopy. The 400 nm wavelength pump beam provides sufficient energy for interband transition in VO<sub>2</sub> between occupied lower 3d<sub>||</sub> level and π\* band hybridized with upper 3d<sub>||</sub> one. The 130 femtosecond laser pulses were applied for measurements of transient reflection and relaxation processes, and to characterize the carrier dynamics during I-M transition in 10<sup>-13</sup>-10<sup>-9</sup> temporal-scale. Optical pumping of free carriers in VO<sub>2</sub> initiates extremely fast changes in transmittance and reflectivity, which behavior is strongly dependent on excitation pulse energy and structural properties of the sample. To analyze the ultrafast change of spectral properties of the VO<sub>2</sub> sample the ultrashort white light pulses were used as a probe. White light continuum was generated by focusing of 800 nm femtosecond pulses on the sapphire plate and then focused on the sample. The reflected light was delivered into spectrophotometer. The spectral records in optical region are conducted at different fixed time delays between pump and probe pulses as well as at different

temperatures of the sample. Comparison of the VO<sub>2</sub> transient reflectivity for both VO<sub>2</sub> temperatures corresponded to insulator and metallic phases gave information about light induced ultrafast structural transformation of VO<sub>2</sub>. The experimental data shows that the complete lattice transformation can be observed at the 10<sup>-12</sup> temporal-scale for ≤50 nm thick film as depended on optical pump pulse energy. Upon a laser illumination an ultrafast (in ~10<sup>-13</sup> sec) change in reflectivity and its following pump-dependent relaxation process (in ~10<sup>-9</sup> sec) are observed for VO<sub>2</sub> at different temperatures, in insulator and metallic VO<sub>2</sub> phases. The study of transient reflectivity for VO<sub>2</sub> in metallic state has allowed proposing the model for excited state dynamics at temperatures above insulator-metal phase transition.

### 2:15 PM V5.3

**Transformation of Helically Structured Nanofibers into Linearly Oriented Nanofibers.** M. K. Shin, S. J. Park, S. G. Yoon, M. S. Kim, B. K. Gu, I. Y. Kim, S. I. Kim and S. J. Kim; Dept. of Biomedical Engineering, Hanyang University, Seoul, South Korea.

Micro- and nano-scale helical shapes have been fabricated because of their unique characteristics and potential applications such as in one-dimensional nanoscale sensors, transducers, and resonators. Helically coiled carbon fibers [1] and metal-oxide helical nanostructures [2] have been produced for these purposes. Moreover, since coils of carbon nanotubes were made, the mechanism for their formation has been investigated. [3] However, unlike for inorganic helical or coiled structures, investigation and fabrication of polymeric helical fibers have not been performed actively in spite of their importance as organic helical structures. As it is difficult to produce polymeric helices by inorganic methods, electrospinning is recommended due to convenient conversion of polymer solution into polymeric nanofibers and whipping instability to produce helical structures. In this work, helically structured nanofibers were fabricated from a solution of poly(2-acrylamido-2-methyl-1-propane sulfonic acid) in water and ethanol by whipping instability of electrospinning. In addition to whipping instability, the effect of tensional force by electrostatic interactions on shapes of nanofibers was also elucidated by converting helical structures into linearly oriented nanofibers using parallel sub-electrodes perpendicular to a main electrode with a trench. The effect of electrostatic interactions allowing the charged nanofibers to stretch was strengthened by sub-electrodes and linearly oriented nanofibers were formed across the whole electrode. References 1. L. J. Pan, M. Zhang, Y. Nakayama, J. Appl. Phys. 91, 10058 (2002). 2. S. Motojima, Q. Q. Chen, J. Appl. Phys. 85, 3919 (1999). 3. X. Y. Kong, Z. L. Wang, Nano Lett. 3, 1625 (2003).

### 3:30 PM V5.4

**A High-Performance Smart System Based on Nanoporous Technologies.** Yu Qiao, Xinguo Kong and Deepam K. Panchal; Department of Civil Engineering, University of Akron, Akron, Ohio.

While the application of nanoporous materials such as charcoal can be traced back to the ancient Egypt era, the more searching modern studies became active at the beginning of the 20th century. The most dominant characteristic of a nanoporous material is its ultrahigh specific surface area often in the range of 100-2000 m<sup>2</sup>/g. However, although the importance of these materials has been well appreciated, currently their applications are limited to chemical, environment, and bioscience fields. The studies on using them in mechanical devices, particularly for intelligent materials/systems, are rare. Recently, in an experimental study a novel application of nanoporous technology is evaluated. When a nanoporous material, e.g. a zeolite, is immersed in an effectively wetting liquid, the liquid can be absorbed into the nanopores. As the temperature changes, the solid-liquid interfacial energy can vary significantly, and as a result the liquid can become effectively nonwetting. Consequently, the liquid can "flow" out of the nanopores, accompanied by the release of excess interfacial energy and a large increase in system volume. If the temperature is changed back, the liquid becomes wetting again. The "inflow" would occur and thus the system exhibits a volume memory characteristic. The attractions of such systems include: (1) the high energy density associated with the large surface areas of nanoporous materials; (2) the large displacement; (3) the compatibility with both large scale and small-sized electro/thermo-hydraulic devices, as well as microelectromechanical systems (MEMS); (4) the simplicity in fabrication; and (5) the high controllability, reliability, and repeatability.

### 3:45 PM V5.5

**Application of Gd And Pd Nanoparticles in a 'New Generation' Switchable Mirror.** Aruna Ivaturi, Bodh Raj Mehta and Lalit Kumar Malhotra; Department of Physics, Indian Institute of Technology Delhi, New Delhi, Delhi, India.

Since the remarkable discovery of hydrogen induced optical and

electronic properties, switchable mirrors based on rare earth metals (RE) have been the topic of intense research. A switchable mirror consists of a RE layer covered with a palladium (Pd) over layer deposited on to a transparent substrate. Upon hydrogenation, RE metal (for example Gd) changes from a reflecting metallic state to a weakly transparent metallic dihydride state and finally to a transparent semiconductor in the trihydride state. The Pd over layer protects the underlying RE layer against oxidation and catalyzes hydrogenation. Color neutrality, kinetics, mechanical stability and reversibility are the important issues addressed in 'first and second generation' switchable mirrors consisting of RE layer and RE-Mg alloy or RE-Mg multilayer structures, but with limited success.<sup>1)</sup> The central objective of this study is to use 'nanoparticle route' to improve the switchable mirror characteristics. The present work investigates the effect of reducing the nanoparticle size in both Gd layer and catalytic Pd over layer on the switchable mirror characteristics. Inert gas evaporation technique has been used to deposit Gd and Pd nanoparticle layers. The important nanoparticle characteristics of size-induced blue shift in the optical band gap and enhanced surface area result in achieving better color neutrality, faster response time, good optical contrast, and enhanced stability in case Gd nanoparticle layer are used.<sup>2)</sup> The presence of Gd<sub>2</sub>O<sub>3</sub> shell around Gd nanoparticles results in the absence of deteriorating effect of alloy formation at the Pd-Gd interface.<sup>2,3)</sup> The improvements in optical and electrical switching characteristics have been observed to be directly related to large changes in H concentration during loading-deload cycles in case of Gd nanoparticle based switchable mirrors.<sup>4)</sup> It has been shown that a uniformly deposited Pd nanoparticle over layer is crucial for improving hydrogen recovery time. Increase in surface area, lattice contraction and shift in Pd d-band centroid are observed to play an important role in improving the catalytic properties of the nanoparticle Pd over layer.<sup>5)</sup> The above results show that by employing both the active components (Gd and Pd) in nanoparticle form, the combined effects of better colour neutrality, stability against oxidation, faster switching and recovery time and higher optical and electrical contrast can be effectively harnessed in a 'new generation' switchable mirror device.<sup>1)R. Griessen Europhys. News, 32, 42 (2001). 2)I. Aruna, B. R. Mehta, L. K. Malhotra, and S. M. Shivaprasad, Adv. Mater. 16, 169 (2004). 3)I. Aruna, B. R. Mehta, L. K. Malhotra, and S. M. Shivaprasad, Adv. Func. Mater. 15, 131 (2005). 4)I. Aruna, B. R. Mehta, L. K. Malhotra, S.A. Khan and D. K. Avasthi, J. Nanoscience and Nanotechnology (accepted for publication). 5)I. Aruna, B. R. Mehta, and L. K. Malhotra, Appl. Phys. Lett. (accepted for publication).</sup>

### 4:00 PM V5.6

**Optical and Electrical Properties of Metal Nanoclusters Embedded in a Dielectric Medium.** Frederic Dumas-Bouchiat<sup>1</sup>, Syed Salman Asad<sup>1</sup>, Corinne Champeaux<sup>1</sup>, Alain Cathelinot<sup>1</sup>, Aurelian Crunteanu Stanescu<sup>2</sup> and Pierre Blondy<sup>2</sup>; <sup>1</sup>SPCTS UMR CNRS 6638, University of Limoges, Limoges, France; <sup>2</sup>IRCOM UMR CNRS 6615, University of Limoges, Limoges, France.

For the last two decades different scientific communities have focused on the fabrication, processing and characterization of nanostructured materials. The non-linear electrical and optical properties of such composites are much larger than those of their bulk counterparts and they can be tuned by controlling the volume fraction and morphology of constituents [1,2]. Such nanostructured materials are considered of great importance to the development of a number of promising technologies in areas like semiconductor physics, optics and optoelectronics, chemistry or biology. Our paper deals with the design, fabrication and characterization of smart, intelligent materials containing metal nano-clusters with improved properties and functionalities in the optical and high frequencies domains. The concept is based on generation of nm-sized particles and their insertion in a thin layer host (typically gamma-alumina). We developed a PLD-based method for controlled synthesis of nanostructured materials based on a nano-cluster generator coupled with a conventional PLD system for host medium co-deposition. This approach offers a large flexibility on the choice of aggregates and matrix materials as well as of cluster organization within the matrix. We have fabricated amorphous thin films nanocomposites of Co- and Ag-doped Al<sub>2</sub>O<sub>3</sub>. The films were subjected to a detailed structural study carried out using HRTEM and AFM. The clusters are metallic, well crystallized and possess an fcc structure with an average diameter centered at 3 nm. FTIR and UV-vis spectrometry investigations performed on amorphous Al<sub>2</sub>O<sub>3</sub> and nano-clusters-doped Al<sub>2</sub>O<sub>3</sub> films reveals specific optical absorption bands related to surface plasmon resonance of metallic clusters. The appearance of this absorption band is related to the metallic character of the clusters and is also an indicator of their structural properties (size, shape, concentration). This characteristic absorption can be exploited for certain practical applications like optical switching or non-linear filtering. The dielectric properties of Al<sub>2</sub>O<sub>3</sub> layers doped with Co clusters in different concentrations (5 - 17 vol.%) were studied by integrating

them in MIM capacitive structures and in RF MEMS capacitive switches. The results show an increase of the dielectric constant of the doped-films compared to the pure alumina. In the 50 MHz -20 GHz regions the permittivity grows linearly with the nano-cluster concentration but its value remains rather low (between 10% and 30% increase) while at low frequencies, in the 5 Hz - 1 MHz domain, we obtain values as high as 150 for the dielectric constant. The electrical conduction mechanisms in the doped layers (I(V) and C(V) measurements) are under investigation but preliminary results seem to indicate a 3D field assisted hopping- type conduction. [1] U. Kreibitz and M. Vollmer, Optical Properties of Metal Clusters, Springer, Berlin (1995) [2] V. M. Shalaev, Physics Reports 272, 61, 1996

4:15 PM **V5.7**

**Novel Ferromagnetic Aerogel Composite Materials with Nanoparticles Formation and Chemistry in Response to Light.** Chunhua Yao, Xipeng Liu and William Risen, Jr.; Chemistry, Brown University, Providence, Rhode Island.

Smart composites based on ferromagnetic hybrid aerogels have been prepared. These novel materials change chemistry in response to light to form nanoparticles that react with compounds that are present in the environment of the aerogels. Thus, high surface area silica-functional polymer hybrid aerogels have been formed via sol-gel synthesis. They also can contain coordinated metal-ion species. They are reacted with Fe-precursors to form air stable ferromagnetic aerogels, which then are transferred into active particles themselves or by incorporation of additional metal ions. These active materials, which adhere to tissue, can be positioned magnetically within their environment. Once they are positioned, they can be transformed by UV light to form Au(0) nanoparticles which react with the molecules in their environment. The preparation and properties of these aerogels, their photochemistry and reaction with biotinylated species will be presented.

4:30 PM **V5.8**

**Reversible Transitions on Electrically-Tunable Nanostructured Superhydrophobic Surfaces.** Tom N. Krupenkin, J. Ashley Taylor, Paul Kolodner and Marc Hodes; Bell Labs, Lucent Tech., Murray Hill, New Jersey.

Recently demonstrated electrically tunable nanostructured superhydrophobic surfaces provide a promising new way of manipulating liquids at both micro and macro scales. Dynamic control over the interaction of liquids with the solid substrate is of great interest to many research areas ranging from biology and chemistry to physics and nanotechnology. In this work the reversibility of the electrically induced superhydrophobic - hydrophilic transition on nanostructured surfaces is addressed. A new approach based on superheat explosion of a very thin layer of liquid adjacent to the solid-liquid interface is discussed. The dependence of the hydrophilic - superhydrophobic transition on the topography of the nanostructured layer, as well as on the energy and duration of the electrical pulse is investigated. Several emerging applications of these surfaces, including lab-on-a-chip, chemical microreactor, and skin drag reduction are discussed.

4:45 PM **V5.9**

**Modification of Ge<sub>2</sub>Sb<sub>2</sub>Te<sub>5</sub> by the addition of SiO<sub>x</sub> for improved operation of phase change random access memory.** Jin-Seo Noh<sup>1</sup>, Yoonho Khang<sup>1</sup>, Dong-Seok Suh<sup>1</sup>, Sang Mock Lee<sup>1</sup>, Kijoon H. P. Kim<sup>1</sup>, Woong-Chul Shin<sup>1</sup>, Ju-Cheol Park<sup>2</sup> and Kihong Kim<sup>2</sup>; <sup>1</sup>Materials Lab, Samsung Advanced Institute of Technology, Yongin-Si, Gyeonggi-Do, South Korea; <sup>2</sup>AE Center, Samsung Advanced Institute of Technology, Yongin-Si, Gyeonggi-Do, South Korea.

Ge<sub>2</sub>Sb<sub>2</sub>Te<sub>5</sub> (GST) is universally used for emerging phase change random access memory (PRAM) applications, due to its attractive properties. However, some material issues are yet to be resolved for the successful launch of the memory. Writing current reduction and improved data retention are on top of the list. We modified GST by adding up a small amount of SiO<sub>x</sub> to it, using co-sputtering technique from multiple targets. The SiO<sub>x</sub> content was gradually increased by increasing the power applied to SiO<sub>x</sub> target, up to 8 volume percent. The sheet resistance of SiO<sub>x</sub>-containing GST exponentially increases for samples annealed at 300 °C after deposition at room temperature. Transmission electron microscopy images reveal that no SiO<sub>x</sub> particulates were formed, which is confirmed by Gatan image filtering. It is indicated by x-ray diffraction patterns that the grain size of SiO<sub>x</sub>-containing GST is smaller than normal GST with lattice locally distorted at its crystalline state, suggesting molecular SiO<sub>x</sub> is homogeneously distributed throughout the GST matrix. We observed that the crystallization temperature of SiO<sub>x</sub>-containing GST is gradually elevated by increasing the SiO<sub>x</sub> content, while the melting point decreasing. These promising aspects led to the reset current reduction and the improved retention, which are core requirements for

a reliable high density PRAM.

SESSION V6: Poster Session: Shape Memory Alloys and Magnetostrictive Devices II

Chair: Ichiro Takeuchi  
Tuesday Evening, November 29, 2005  
8:00 PM  
Exhibition Hall D (Hynes)

**V6.1**

**Fabrication and Characterization of Cu-Al-Ni Shape Memory Alloys by Ingot Metallurgy.** Prateek Maheshwari<sup>1</sup>, K. Jai Ganesh<sup>2</sup> and Arunya Suresh<sup>2</sup>; <sup>1</sup>Department of Metallurgy, Punjab Engineering College, Chandigarh, Punjab, India; <sup>2</sup>Department of Metallurgy, National Institute Of Technology, Jaipur, Rajasthan, India.

Shape Memory Alloys (SMAs) are versatile functional materials with an I.Q. of their own. This class of smart materials exhibit unique properties like superelasticity and shape memory effect (SME) which have made them suitable for numerous applications. Even though Ni-Ti SMAs have attracted attention ever since their advent in 1962, Cu based SMAs due to their ease in fabrication, cost effectiveness and high temperature properties are gaining immense popularity. The objective of this research was the fabrication of Cu-13 Al-3.5 Ni (wt%) shape memory alloy, its characterization and structure property correlation. The alloy was melted in an Electric Resistance Furnace at 1473 K and cast in a metallic mould. Homogenisation was carried out at 1123 K for twenty four hours followed by composition analysis by Optical Emission Spectroscopy. Transformation temperatures of the alloy were determined using Differential Scanning Calorimetry. Heat treatment was carried out at 1273 K for one hour followed by quenching in different media. Optical Micrographs were taken at various magnifications and the formation of self accommodating martensite was observed which was further confirmed by X-Ray Diffraction technique. Mechanical properties of the alloy were tested and further improvements by grain refinement have also been cited. Finally, SME was observed in a rolled strip of the alloy, thus concretizing the obtained results.

**V6.2**

**Correlated Acoustic Emission and Shape Memory Effect.** Subbalakshmi Sreekala<sup>1</sup> and Ananthakrishna Garani<sup>1</sup>; <sup>1</sup>Materials Research Centre, Indian Institute of Science, Bangalore, Karnataka, India; <sup>2</sup>materials research Center, Indian Institute of Science, Bangalore, Karnataka, India.

Martensites are known to exhibit unusual features like repetitive nature of acoustic emission signals when the system is cycled repeatedly in a restricted range of temperatures. This is correlated with the growth and shrinkage of martensitic domains and is directly linked to the shape memory effect. We report a recently introduced model for square-to-rectangle martensitic transformation to understand the shape memory effect. The model includes inertial effects, long-range transformation induced strain field between the transforming domains, a dissipative term that mimics the acoustic emission signals, and an inhomogeneous stress field to describe the effect of defects which serves as nucleation centers. The model reproduces the repetitive nature of the acoustic emission signals when the system is cycled repeatedly in a restricted domain of temperatures after a few training cycles. Interestingly, the associated morphology shows a complete reversal of the martensite domains during a stabilized cycle. The role of training cycles is shown to create a deeper set of metastable states for the system to circulate. The model provides insight into the shape memory effect.

**V6.3**

**Abstract Withdrawn**

**V6.4**

**Material Properties of Rapid-solidified Ni-free Shape Memory TiNb Alloy.** Yasubumi Furuya<sup>1</sup> and Yasuko Kubo<sup>2</sup>; <sup>1</sup>Department of Intelligent Machines and System Engineering, Hiroso University, Hiroso, Japan; <sup>2</sup>Institute for Material Research, Tohoku University, Sendai, Japan.

Ti-Ni shape memory alloy is used for bio-medical and industrial fields, however, even if Ni is a substance which has strong allergic nature and carcinogenicity to a human body, then, Ti-Ni alloy system is in a prohibition tendency in the medical field in Europe in recent years. Therefore, Ni-free shape memory alloy is desired in the near future medical field. In this research, we pay our attention to the Ti-Nb alloy which has shape memory as well as superelastic effects. The TiNb fiber was fabricated by using our arc melting-nozzleless rapid solidification method. The fiber samples were used to investigate the following characteristics relating with shape memory effect, that is to

say, (1)cross-sectional microstructure observation, (2)DSC(phase transformation temperature changes),(3)temperature-shape recovery relationship(the degree of recovery-temperature relationship). Moreover,heat-treated effect on the shape recovery effect in the produced samples was observed in comparison with the starting bulk material. Finally,the fundamental discussion to correlate the rapid solidification process condition with the characteristic of Ti-Nb shape memory alloy for bio-engineering application was conducted.

#### V6.5

**Laser Annealing of Amorphous NiTi Shape Memory Alloy Thin Films: Experiment and Modeling.** Yves Bellouard<sup>2</sup>, Xi Wang<sup>1</sup>, Zhenyu Xue<sup>1</sup>, Fadila Khelfaoui<sup>2</sup> and Joost J. Vlassak<sup>1</sup>; <sup>1</sup>Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts; <sup>2</sup>Center for Automation Technologies, Rensselaer Polytechnic Institute, Troy, New York.

We present the results of a crystallization study on NiTi shape memory thin films in which amorphous films are annealed by a scanning laser. Laser annealing makes it possible to spatially distributed shape memory properties in thin-film devices, e.g., amorphous or passive elements can be created in parallel with crystalline or active elements to effect in-plane actuation. A kinetics study shows that nucleation of the crystalline phase occurs homogeneously in the films. Consequently, the laser annealing process produces polycrystalline films with a random crystallographic texture. The crystallized films have a uniform microstructure across the annealed areas. The material in the crystalline regions transforms reversibly to martensite on cooling from elevated temperature and stress measurements show that a significant recovery stress is achieved in the films upon transformation. Temperature profiles induced in the film by the scanning laser were calculated using a full three-dimensional finite element model. The model takes into account oxidation of the NiTi surface and allows prediction of the size of the crystallized regions as a function of laser annealing parameters.

#### V6.6

**Temperature dependent properties of Ni-rich Ni<sub>2</sub>MnGa.** Tilmann Hicke<sup>1</sup>, B. Grabowski<sup>1</sup>, J. Neugebauer<sup>1</sup>, K. Froehlich<sup>2</sup>, K.-U. Neumann<sup>2</sup> and K. R. A. Ziebeck<sup>2</sup>; <sup>1</sup>Computational Materials Design, MPI fuer Eisenforschung, Duesseldorf, Germany; <sup>2</sup>Dept. of Physics, Loughborough University, Loughborough, United Kingdom.

The ferromagnetic shape memory compound Ni<sub>2</sub>MnGa has a Curie temperature of ~360 K and undergoes a martensitic phase transition at 200 K. With increasing Ni content in Ni<sub>2+x</sub>Mn<sub>1-x</sub>Ga compounds the martensitic transition temperature increases while the ferromagnetic transition decreases only slightly. These properties make Ni-rich Ni<sub>2+x</sub>Mn<sub>1-x</sub>Ga interesting for applications as sensors/actuators. We have investigated the crystallographic properties of these materials both experimentally and theoretically, with a special focus on its temperature dependence. Neutron scattering experiments have been performed for x=0.05 and 0.10 as a function of temperature. The low temperature martensitic phase is found to be consistent with the martensitic phase of pure Ni<sub>2</sub>MnGa. The modulation along the cubic (110) direction is given by a 7-fold increase in the martensitic phase. The temperature dependence of lattice parameters is presented for both cooling and heating. In order to understand the effect of excess Ni on the transition temperature the free energy of the stoichiometric and non-stoichiometric physics has been calculated employing density-functional theory.

#### V6.7

**Effect of Boron Addition on Magnetostrictive and Mechanical Properties in Rolled Polycrystalline Fe-18.7%Ga Alloy.** Suok-Min Na and Alison B. Flatau; Aerospace Engineering, University of Maryland, College Park, Maryland.

It has been shown that single crystal Fe-Ga alloys with near 19% Ga exhibit large magnetostrictive behavior (~400 ppm) at low applied magnetic fields when rapidly cooled so as to achieve a disordered bcc structure. Fe-Ga solid solutions with 15~20% Ga may be useful in magnetostrictive actuators and sensors due to their large saturation magnetostriction potential, high mechanical strength, and good ductility. Properly textured polycrystalline Fe-Ga alloys obtained through deformation and recrystallization may provide even more desirable mechanical properties and at a lower cost than single crystal. The authors recently reported that textured polycrystalline Fe-17%Ga with a somewhat dispersed cube texture exhibited magnetostriction of ~160 ppm. Other researchers have reported that texture annealing of Fe-15%Ga with 1 mol% NbC changed the texture to a predominant texture that was close to {100}<001> and the anneal at 1150°C produced a [001] preferred orientation along the rolling direction. In order to achieve maximum performance in polycrystalline form, we initially tried rolling a Fe-18.7%Ga alloy, targeting a peak value in single crystal Fe-Ga magnetostriction. The hot-rolled Fe-18.7%Ga alloy at 1000°C, however, cracked along the grain boundaries and

consequently fractured. It was determined that mechanical properties at this composition were too brittle to make thin sheet. In order to improve the ductility, boron (0.5~1.0 %) was introduced to Fe-18.7%Ga alloy, where small addition of B is known to improve the ductility in other alloys by enhancing grain boundary cohesion. It was reported that thin sheets having a final thickness of 0.28~0.30 mm for boron doped Fe-6.5 wt.%Si alloy were successfully fabricated using a conventional rolling processes. Thin sheets of Fe-18.7%Ga plus B alloys were successfully fabricated to thickness of 0.30~0.35 mm using a rolling process. It was observed that the fracture surface of the B-free Fe-18.7%Ga alloy clearly appeared to typical of a transgranular fracture mode and that of the B-added alloys was changed to an intergranular fracture mode with grain refinement. Boron has a great effect on improvement of ductility due to a strong tendency to segregate to the grain boundaries. The sheets with boron content of 0.5 and 1.0 %, which were annealed at 1200°C under flowing argon and then quenched in water, exhibited maximum magnetostrictions of 156 and 184 ppm, respectively. In the case of (Fe-18.7%Ga)+1.0 %B alloy, x-ray diffraction analysis of samples indicated the presence of a Fe<sub>2</sub>B phase throughout the A2 matrix in small amount. They were very dispersed with an average grain size of 15µm and the amount of 1 vol% determined by EBSD analysis. Therefore, inhibition of primary grain growth by Fe<sub>2</sub>B particles promote secondary recrystallization of the (110)[001] structures during texture annealing.

#### V6.8

**A Magnetomechanical Hysteresis Model for Magnetic Transducer Materials.** Ralph C. Smith<sup>1</sup> and Marcelo Dapino<sup>2</sup>; <sup>1</sup>Mathematics, North Carolina State University, Raleigh, North Carolina; <sup>2</sup>Mechanical Engineering, The Ohio State University, Columbus, Ohio.

This talk addresses the development and validation of a fully coupled magnetomechanical hysteresis model for polycrystalline ferromagnetic transducer materials. The theory is based on a homogenized energy framework which characterizes hysteretic phenomena through a combination of energy principles at the lattice level and macroscopic homogenization techniques. The proposed model incorporates stress effects by extending the framework to include both 90 and 180 degree domain wall switching as a function of input fields and stresses. Properties of the model are illustrated through comparison and prediction of experimental steel data.

#### V6.9

**Multi-functional micromachine driven by small magnetic field.** Harunobu Tomita, Teiko Okazaki and Yasubumi Furuya; Hirosaki University, Hirosaki, Japan.

Rapidly solidified ferromagnetic Fe-Pd thin foil exhibits two way shape memory effect in temperature range of 300K to 420K. We fabricated a multi-functional micromachine driven by small magnetic field, which swims in a tube (a few mm in diameter) filled with water. The micromachine is composed of a fin and a grip made from a piece of the Fe-Pd foil only. The speed and the moving direction depend on the frequency of exchanging magnetic field. In addition, the grip opens by heating. Consequently, these unique features of this micro-machine system will be applicable in the near future for the minimal invasive medical cure such as a wireless catheter in hyperthermia, blood vessel extension treatment and drug delivery system, moreover, the non-destructive inspection micro-robot in the industrial pipes.

#### V6.10

**Magnetostriction and Crystallographic Texture in Rolled and Annealed Fe-Ga Based Alloys.** Suok-Min Na and Alison B. Flatau; Aerospace Engineering, University of Maryland, College Park, Maryland.

Textured polycrystalline Fe-Ga alloys have the possibility of being useful in magnetostrictive actuators and sensors due to their large saturation magnetostriction potential (~400 ppm) in low applied fields (~200 Oe). In order to achieve a <001> crystallographic texture, we examine the surface-energy-induced selective grain growth in a controlled sulfur environment during final annealing. We build on results from selective development of Goss and Cube texture in Fe-Si alloys, an  $\alpha$ -iron alloy that is similar to Fe-Ga. Köhler suggests that although {110} growth occurs under very clean surface conditions, {100} growth occurs under slight surface contamination with sulfur due to adsorption of the sulfur. However, there is a threshold above which the selective growth of {100} grains is changed to that of {111} grains by the interfacial segregation of sulfur which is contained in the bulk interior. For the survival of only {100} grains, the adsorption/segregation of sulfur in the surface should be appropriately controlled by final annealing conditions such as time, temperature and atmospheres. We present results from introduction of sulfur additions to Fe-Ga arc-melted, rolled and annealed specimens and report on resulting texturing. Polycrystalline Fe-18.7%Ga plus B (0.5~1.0%) and S (0~0.05%) buttons prepared by arc-melting are



investigated. Specimens with a final thickness of 0.30 mm were produced by hot, warm and cold rolling. The canned specimen was hot rolled to give a 65% reduction to a thickness of 2.4 mm over 82 passes at 1000°C. Next warm rolling at 400°C provided an 83% reduction over 53 passes to give a sheet 0.4-mm thick. After the stress relief annealing step, cold rolling were undertaken until the thickness of 0.30 mm was achieved. A subsequent annealing was conducted under various temperature of 900~1200°C for 0.5~24 h and various atmospheres. As-rolled (Fe-18.7%Ga)+0.5%B+0.005%S sheets with large amounts of sub-grain which most likely have been deformed by the rolling process have some {100} and {111} grains parallel to the rolling direction. While texture annealing appears to eliminate the majority of the sub-grains present to below 1% of sub-structured or deformed area in the sheet annealed at 1200°C for 2h. From a texture standpoint, the some clustered {100} poles of the wholly recrystallized sheet with a magnetostriction of 200 ppm were found and ~25° away from the rolling direction. And the {100} poles are centered right on the rolling direction for the highly textured subset. The textured sheet, which was annealed at 1000°C for 6h and 1200°C for 1h under flowing argon and then quenched in water, exhibits a maximum magnetostriction of 220 ppm. This value in magnetostriction is double as large as that of the sulfur-free sheet. This is probably due to the formation of partly {100}<001> crystallographic texture through surface-energy-induced selective grain growth controlled by segregated sulfur on the surface.

#### V6.11

**Bending of Iron-Gallium (Galfenol) Alloys for Sensor Applications.** Patrick Downey and Alison Flatau; Aerospace Engineering, University of Maryland, College Park, Maryland.

Galfenol alloys (Fe<sub>100-x</sub>Ga<sub>x</sub>) have been shown to combine significant magnetostriction (~400 ppm) with strong mechanical properties (tensile strengths ~500 MPa), making them well suited for use in robust actuators and sensors as an active structural material. Efforts are underway to grow Galfenol nanowires as small as 20 nm in diameter through the process of electrochemical deposition. This project investigates Galfenol behavior in bending at the macroscale to facilitate the design concepts for use of Galfenol nanowires in biologically inspired sensors, mimicking the hair-like cilia of the inner ear. In order to properly design the sensor, the magnetomechanical bending behavior of Galfenol must first be characterized. To this end, a series of experiments are conducted on the magnetic response of cantilevered beams to dynamic bending loads. Mechanical excitation was applied to the tip of each beam, with tests performed with sinusoidal and broadband inputs. Measuring the magnetic response of the samples were a giant magnetoresistive (GMR) sensor located behind the beam and a pickup coil wound directly on each rod. Permanent magnets were used to provide dc magnetic fields to bias the samples up to 100 Oe. Results show that this novel operating regime of transverse bending produces significant change in magnetic induction that can be measured with both the GMR sensor and the pickup coil. These results are verified by a system model incorporating classical continuum mechanics for beam bending and the constitutive magnetostriction equations, with the various coefficients determined from experimental magnetization vs. stress data. Initial research has begun into the effect of scaling the system down to nanometer lengths for the proposed biologically inspired nanosensors. This work includes the experimental characterization of individual nanowires in bending as well as the acoustic excitation of entire cantilevered nanowire arrays.

#### V6.12

**Thermal stability of Hf-silicate films on Si (100) grown by atomic layer deposition.** Kwun Bum Chung<sup>1,2</sup>, Young Kuk Kim<sup>1</sup>, Chan Joong Lim<sup>3</sup>, Chung Nam Whang<sup>1</sup>, Dae-Hong Ko<sup>3</sup>, Dae Won Moon<sup>2</sup> and Mann-Ho Cho<sup>2</sup>; <sup>1</sup>Institute of Physics and Applied Physics, YONSEI UNIVERSITY, Seoul, South Korea; <sup>2</sup>Nano surface group, Korea Research Institute of Standards and Science, Daejeon, South Korea; <sup>3</sup>Ceramics Engineering, YONSEI UNIVERSITY, Seoul, South Korea.

The thermal stability and physical characteristics of Hf-silicates grown by atomic layer deposition (ALD) were investigated. The Hf-silicate films were grown on Si(100) by alternant ALD process for HfO<sub>2</sub> and SiO<sub>2</sub> using TEMA<sub>4</sub>Hf and TDMAS, respectively. In order to study the thermal stability, Hf-silicate films with different amounts of HfO<sub>2</sub> were annealed by means of rapid thermal process in an ambient of N<sub>2</sub> from 700°C to 900°C for 2min. The physical characteristics and chemical state of Hf-silicate films were examined by medium energy ion scattering (MEIS), x-ray photoelectron spectroscopy (XPS), x-ray absorption spectroscopy (XAS), and high resolution transmission electron microscopy (HRTEM) as a function of the amount of SiO<sub>2</sub> incorporated in HfO<sub>2</sub>. The ratio of HfO<sub>2</sub> and SiO<sub>2</sub> affected to the structural stability in Hf-silicate films. In the Hf-silicate films with high fraction of HfO<sub>2</sub>, the phase between HfO<sub>2</sub> and SiO<sub>2</sub> was separated during post-annealing above the temperature of 800°C.

However, as the fraction of HfO<sub>2</sub> was reduced, the phase separation was decreased. Moreover, the interdiffusion of Si from Si substrates is closely related to the fraction of SiO<sub>2</sub>, affecting the phase separation process. Thus, it was concluded that the structural and interfacial stabilities are closely dependent on the ratio between HfO<sub>2</sub> and SiO<sub>2</sub>.

#### V6.13

**Study for photoconduction of a single ZnO nanowire using the modulated illumination of light.** Kihyun Keem<sup>1</sup>, Jeong-Min Kang<sup>1</sup>, Byungdon Min<sup>1</sup>, Dong-young Jeong<sup>2</sup> and Sangsig Kim<sup>1</sup>; <sup>1</sup>Electrical Engineering, Korea University, Seoul, South Korea; <sup>2</sup>Nano Science, Korea University, Seoul, South Korea.

Photoconduction has been investigated by many research groups for nanowires including ZnO, In<sub>2</sub>O<sub>3</sub>, SnO<sub>2</sub>, InP, GaP. In spite of such vigorous researches, photocurrent of ZnO nanowires has not been widely studied yet. Recently, photoconduction of a single ZnO nanowire under UV light illumination was reported. Nevertheless, the photoconduction mechanism of ZnO nanowires has not been currently understood well. It has been well known that the photoresponse of n-type ZnO nanowires show a slow decay process in conduction controlled by surface effects. To study the photoresponse spectrum of ZnO nanowires, light of excitation source was modulated by chopper. The photoresponse spectrum of a single ZnO nanowire using the modulated illumination of light was measured by Lock-in amplifier. Current-voltage, PL(photoluminescence), and photoresponse-voltage studies were performed for the investigation into photoconduction mechanism in this nanowire. The photoresponse spectrum of a single ZnO nanowire was changed by oxidizing ZnO nanowires. The photoresponse spectrum showed that the photoresponse in UV region was increased, in non-UV region was decreased.

#### V6.14

**Epitaxial growth of metals on semiconductors: ZnO / Pt heteroepitaxy.** Shivaraman Ramachandran, Amit Chugh, Ashutosh Tiwari and Jagdish Narayan; NCSU, Raleigh, North Carolina.

Growth of metallic epitaxial layers / composites on semiconductors has gained importance over the years due to the various applications ranging from microelectronics packaging to spintronics. Of particular interest is improving the electrical conductance of an optoelectronic semiconductor without losing the optical properties. In addition, epitaxial layers of metals can also be combined with diluted magnetic semiconductors to find use in novel spintronic applications. Here, we report the growth of epitaxial ZnO-Pt-ZnO trilayer structures and ZnO-Pt bilayer and composite structures on sapphire (0001) substrate by using pulsed laser deposition technique where heteroepitaxial growth was accomplished by domain matching epitaxy. These structures were characterized using X-ray diffraction, High resolution transmission electron microscopy, STEM (Scanning Transmission Electron microscopy-Atomic number) Z-contrast, optical transmittance, photoluminescence and electrical resistivity measurements. X-ray diffraction and TEM experiments revealed the epitaxial nature of these structures, the orientation relationship being: <111><sub>Pt</sub>—\*dvertb\*—<0001><sub>ZnO</sub>—\*dvertb\*—<0001><sub>Sapphire</sub> (out of plane) and <110><sub>Pt</sub>—\*dvertb\*—<2 -1-10><sub>ZnO</sub>—\*dvertb\*—<01-10><sub>Sapphire</sub> (In Plane) for the trilayer structure and <111><sub>Pt</sub>—\*dvertb\*—<0001><sub>Sapphire</sub> (out of plane) and <110><sub>Pt</sub>—\*dvertb\*—<01-10><sub>Sapphire</sub> (In Plane) for the bilayer structure. Electrical and optical measurements showed that these heterostructures exhibit very high electrical conductivity and at the same time possess quite interesting optical transmittance spectra and exhibit room temperature photoluminescence characteristics.

#### V6.15

**Electronic Structure of VO<sub>2</sub> in Phase Transition by Tunneling Spectroscopy.** Changman Kim<sup>1</sup>, Yasushi Oikawa<sup>1</sup>, Takashi Tamura<sup>1</sup>, Jae-Soo Shin<sup>2</sup> and Hajime Ozaki<sup>1</sup>; <sup>1</sup>Electrical Engineering and Bioscience, WASEDA UNIV., Tokyo, Japan; <sup>2</sup>Advanced Materials Engineering, DAEJEON UNIV., Daejeon, South Korea.

The temperature-dependent phase transition on vanadium dioxide (VO<sub>2</sub>), firstly observed by Morin in 1959, is one of the attractive functions applied for switching devices such as “Thermistor”, “Thermochromic Smart Windows”, and the extensive studies have been made for understanding its physical properties. VO<sub>2</sub> is a metal above the phase transition at T<sub>c</sub> ≈ 340 K, while it is an insulator with energy gap, about 0.65 eV, below T<sub>c</sub>. In the present study, the density of states (DOS) has been firstly observed by tunneling spectroscopy which is one of the useful methods for analyzing the DOS directly with high resolution, at the onset of phase transition toward the low-temperature phase. The single crystals were grown from liquid in a quartz ampoule. The typical size of the single crystal was 3×1×1 mm<sup>3</sup>. The ratio of electrical resistivity for both sides of phase transition attained more than 10<sup>4</sup> with hysteresis width of about 2 K. In the tunneling spectra, small dips were observed in the dI/dV vs. V characteristics around 0.03 eV above and 0.06 eV below the Fermi



level, at temperature well above  $T_c$ . When the temperature was decreased from the high-temperature phase side toward  $T_c$ , DOS in the energy region between the two dips was diminished. This behavior indicates the occurrence of the phase transition from metal to insulator. The electrical resistivity of pure  $\text{VO}_2$  in low-temperature phase was too high to obtain temperature-dependent tunneling data continually. From the previous consideration, the tunneling study on W substituted  $\text{VO}_2$ , which realizes lower electrical resistivity in the insulator phase, will be reported for both sides of phase transition.

#### V6.16

**Ferromagnetic Co-Doped SnO<sub>2</sub> Nanowires.** Zuqin Liu<sup>1</sup>, Daihua Zhang<sup>2</sup>, Chongwu Zhou<sup>2</sup>, Alex A. Puzetky<sup>4</sup>, David B. Geohegan<sup>1,3</sup> and Douglas H. Lowndes<sup>1,3</sup>; <sup>1</sup>Center for Nanophase Materials Sciences, Oak Ridge National Lab, Oak Ridge, Tennessee; <sup>2</sup>Department of EE-Electrophysics, University of Southern California, Los Angeles, California; <sup>3</sup>Condensed Matter Sciences Division, Oak Ridge National Lab, Oak Ridge, Tennessee; <sup>4</sup>Department of Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee.

We have successfully synthesized ferromagnetic Co-doped SnO<sub>2</sub> nanowires by depositing an epitaxial shell of Co-doped SnO<sub>2</sub> onto single crystal SnO<sub>2</sub> nanowires. The materials composition and stoichiometric ratio have been carefully examined and confirmed with a variety of characterization techniques. These novel structures have rendered unique opportunities to investigate the transport behavior and spintronic property of SnO<sub>2</sub> in its one-dimensional form.

#### V6.17

**Hot Carrier Effects in Self-aligned and Offset-Gated Polysilicon Thin-Film Transistors.** Argyrios Hatzopoulos<sup>1</sup>, Dimitrios Tassis<sup>1</sup>, Nikolaos Arpatzani<sup>1</sup>, Charalambos Dimitriadis<sup>1</sup> and Georges Kamarinos<sup>2</sup>; <sup>1</sup>Aristotle Univ. of Thessaloniki, Department of Physics, 54124, Thessaloniki, Greece; <sup>2</sup>IMEP, 23 rue des Martyrs, B.P. 257, 38016 Grenoble Cedex, Grenoble, France.

Polysilicon thin-film transistors (TFTs) have been widely used for applications in active-matrix liquid-crystal displays and three-dimensional integrated circuits. For such applications, hot-carrier induced instability is one of the major problems of the polysilicon TFTs. It has been pointed out that the instability of polysilicon TFTs is more serious than that of single-crystalline silicon MOSFETs, associated with the high density of defects which enhance the local electric field near the drain region and with the poor polysilicon/oxide interface quality. In this work, we investigate the effects of hot carriers on the performance of intrinsic self-aligned n-channel polysilicon TFTs with channel length  $L = 10 \mu\text{m}$  and similar offset gated devices with offset length  $\Delta L = 2 \mu\text{m}$ . From the evolution of the transfer characteristics during electrical stressing, the transistor aging is deduced. The investigated devices were fabricated on fused quartz glass substrates, covered by 200 nm thick  $\text{SiO}_2$ , with the standard low temperature process. The active polysilicon layers (50 nm thick) were prepared by solid phase crystallization of amorphous silicon in a conventional furnace at 600°C for 24 h in nitrogen ambient, followed by KrF excimer laser ( $\lambda = 248 \text{ nm}$ ) irradiation with energy density 320  $\text{mJ}/\text{cm}^2$  in air ambient. Transmission electron microscopy (TEM) analysis has shown that the average grain size is about 2.5  $\mu\text{m}$  with relatively low in-grain defect density. A standard n-channel metal-oxide-semiconductor process was used to fabricate self-aligned devices of gate width  $W = 10 \mu\text{m}$  and gate length  $L = 10 \mu\text{m}$ . As gate insulator, 120 nm thick  $\text{SiO}_2$  was deposited by electron cyclotron resonance - plasma enhanced chemical vapor deposition at 100°C. Offset gate devices were also fabricated with offset length  $\Delta L = 2 \mu\text{m}$ . In self-aligned devices, the on-state current is substantially reduced whereas the subthreshold slope remains unaffected, which are attributed to interface states generation. In the offset gated device, the transfer characteristics are shifted first positively after short stressing time and then negatively, the on-state current is still substantially reduced and well-defined kinks are formed in the subthreshold region. The overall results show that the device degradation becomes worse in the offset gated device due to the enhanced electric field in the reverse-biased  $n^+p$  junction near the drain, since the as-grown polysilicon layer is p-type. The results suggest that the self-aligned device provides an advantage for hot-carrier reliability compared to offset gated devices, due to gate modulation of the p-type as-grown polysilicon near the drain in the on-state region of operation.

#### V6.18

**A Novel Fabrication Technique By Composite Material Processing: Metal Insulator Semiconductor Fiber Components And Fabric Systems.** Mehmet Bayindir, Ayman F. Abouraddy and Yoel Fink; Research Laboratory of Electronics, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Fabrication of functional devices on fibers by thermal drawing requires an empirical material identification scheme and challenging composite material processing. A macroscopic preform rod containing metallic, semiconducting and insulating constituents in a variety of geometries and close contact leads to kilometer-long novel optoelectronic, thermo-optic, thermal and acoustic microscopic devices: I. A spectrally tunable fiber photodetector comprising an amorphous semiconductor core contacted by metallic micro-wires, and surrounded by a cylindrical-shell resonant optical cavity, is thermally drawn from a macroscopic preform. Each fiber is sensitive to illumination along its entire length. A grid of such fibers was constructed that enables the identification of the location of an illumination point and the direction of the incoming light. II. Thermal imaging using a fabric woven out of flexible, thermally sensitive fibers is demonstrated. The fiber core is made of a thermo-electric semiconductor glass, and the cladding is made of a mechanically tough, yet flexible, insulating polymer. The thermal functionality of the fiber is enabled by means of metal electrodes that are co-drawn with the core and cladding materials and run along the entire fiber length. Arranging such fibers in an array, and contacting them to external electronic circuitry, allows one to locate local temperature changes over a large area in real time. III. A hybrid fiber having dual optical and thermal functionalities is designed and demonstrated. The fiber is comprised of a hollow air core surrounded by an omnidirectional photonic band gap structure for optical guidance. Thermal functionality is provided by a metal-semiconductor-metal distributed heat sensor embedded in the cladding and encompassing the core and the dielectric layers. A quaternary amorphous semiconductor chalcogenide glass is synthesized and used as the heat-sensing component. The dual functionalities are demonstrated by transmitting high-power carbon dioxide ( $\text{CO}_2$ ) laser beam through the hollow-core while simultaneously monitoring the temperature of fiber in a straight and a bent configuration. This new fabrication technique, along with novel material systems will pave the way for future development of fibers and fiber-based devices with unique optical, electrical and thermal properties.

#### V6.19

**Forward Active and Blocking Performance 4H-SiC Bipolar Junction Transistors.** Santhosh Balachandran<sup>1</sup>, Paul Chow<sup>1</sup> and Anant Agarwal<sup>2</sup>; <sup>1</sup>Center for Integrated Electronics, Rensselaer Polytechnic Institute, TROY, New York; <sup>2</sup>Cree Inc., Durham, North Carolina.

In previous demonstrations of 4H-SiC BJTs, many aspects that affect the performance of BJTs during forward conduction, including geometry and temperature effects have been discussed [1]. In this paper, we have examined the relationship between  $BV_{ceo}$  and  $BV_{cbo}$  of high-voltage BJTs with lightly doped collector in 4H SiC and qualified the analytical dependence between the two which can be used as a device design guideline. We have also analyzed the key material and processing related issues that affect the forward active performance of the device. The relationship between  $BV_{ceo}$  and  $BV_{cbo}$  can be expressed in a simplistic form as follows:  $BV_{ceo} = BV_{cbo} / (1 + (\beta))^{(1/n)}$  Using the impact ionization coefficients of electrons and holes the ionization integrals for both NPN and PNP BJTs have been evaluated for two different epitaxial layer thicknesses and dopings: 1) 12  $\mu\text{m}$  thickness, doped to  $4 \times 10^{15} \text{ cm}^{-3}$  and 2) 45  $\mu\text{m}$  thickness, doped to  $1 \times 10^{15} \text{ cm}^{-3}$ , based on a zero-carrier solution of the Poisson's equation, a 2D device simulator, from which the 'n' factor is extracted to be equal to 9.9 and 7.8 for NPN and PNP BJTs respectively [2]. The results are also verified experimentally for 900V and 4kV blocking voltage devices. An important feature of the forward I-V characteristics of the 4kV BJT is the collector-emitter offset voltage ( $V_{ceo}$ ) of 0.5V, which is attributed to the finite emitter contact resistance which results in a higher forward drop in the base-emitter junction than in the base-collector junction during forward bias conditions. Also from the simulated current and electron concentration distribution for the device at 100  $\text{A}/\text{cm}^2$  we obtain a  $R_{on,sp}$  value of 53  $\text{m}\Omega\text{-cm}^2$  (56  $\text{m}\Omega\text{-cm}^2$  measured experimentally) when the lifetime of electrons and holes ( $\tau_{no}$  and  $\tau_{po}$ ) in the drift region is assumed to be 0.5  $\mu\text{s}$  and 0.05  $\mu\text{s}$  respectively and a surface recombination velocity (S) of  $1 \times 10^5 \text{ cm/s}$  is assumed along the interface. Thus non-uniform current distribution due to the recombination of injected electrons at the interface and poor lifetime in the drift region results in a  $R_{on,sp}$  value above the unipolar value. An improvement in the  $R_{on,sp}$  number (to 30  $\text{m}\Omega\text{-cm}^2$ ) can be achieved by increasing the lifetime in the collector and by reducing recombination at the interface. Acknowledgments: This work was supported primarily by the ARL Collaborative Technology Alliance in power and energy, Cooperative agreement No. DAAD19-01-2-0010 and NSF Center for Power Electronic Systems (# EEC-9731677). References: [1] S. Balachandran, T.P. Chow, A. Agarwal, C. Scozzie, K.A. Jones, "4kV 4H-SiC Epitaxial Emitter Bipolar Junction Transistors", in Proc. Int. Symp. Power semiconductor Devices and ICs, May 2005 [2] S. Balachandran, T.P. Chow, A. Agarwal, S. Scozzie and K. A. Jones, "BVCEO vs BV CBO for 4H and 6H polytype SiC

SESSION V7/W6: Joint Session: Polymer Actuator III  
Chairs: Z-Y Cheng and J. Su  
Wednesday Morning, November 30, 2005  
Independence W (Sheraton)

**8:00 AM \*V7.1/W6.1**

**High Dielectric Constant Nano-Phase Polymers and Nano-Composites.** Qiming Zhang, Penn State, University Park, Pennsylvania.

High dielectric electroactive polymers have applications such as MEMS, artificial muscles, and capacitors. One challenge is how to significantly raise the dielectric constant of polymeric materials. By exploiting the delocalized electron organics and polymers, we show that nano-dielectric composites can have high dielectric constant, approaching that of the inorganic materials. Consequently, high electromechanical response can be realized with relatively low fields. Further more, experimental results show that in nano-composites, the interface effects such as the exchange coupling can have marked effect in enhancing the dielectric and electromechanical responses. Nano-composites, prepared using grafting, self-assembly, and nano-phase fully functionalized polymers will be discussed.

**8:30 AM \*V7.2/W6.2**

**Carbon Nanotube Reinforcement of Conducting Polymers and Hydrogels for High Strength Actuators.**

Geoffrey M. Spinks<sup>1</sup>, Vahid Mottaghtalab<sup>1</sup>, Binbin Xi<sup>1</sup>, Philip Whitten<sup>1</sup>, Gordon Wallace<sup>1</sup>, Seon Jeong Kim<sup>2</sup>, Su Ryon Shin<sup>2</sup> and Sun Il Kim<sup>2</sup>; <sup>1</sup>School of Mechanical, Materials and Mechatronic Engineering, University of Wollongong, Wollongong, New South Wales, Australia; <sup>2</sup>Hanyang University, Seoul, South Korea.

Practical devices for electromechanical actuators will only be realised when the actuator materials can generate sufficient movement (stroke) and forces within a suitable time frame. Low voltage actuators based on conducting polymers such as polypyrrole (PPy) appear attractive choices because of the relatively large strains (up to 26%) and stresses (to 5-10 MPa) that have been previously reported. However, the small size of the actuator materials used mean that the actual stroke (few mm) and forces (<1 N) are quite small. Many more applications become feasible if low voltage actuators could produce >10mm stroke and >1 N force- preferably within one second. A serious limitation to the maximum force that can be generated by actuator materials is their breaking strength. Several previous studies have shown that the breaking strength can be an order of magnitude lower than the static strength when tested under actuator conditions - that is, when the sample is being cycled between the contracted and expanded states. The practical maximum stress for conducting polymer actuators is then only a few tens of MPa and is much less in some cases. The maximum stress that can be applied to hydrogels is also very small. In our recent work, we have considered the use of carbon nanotubes as strengthening reinforcement for conducting polymers and hydrogels. These high aspect ratio fibers are capable of significantly increasing the strength and stiffness of polymers when added in only small quantities. Although an increase in stiffness may reduce the amount of movement produced by the actuator, this deficiency may be compensated by the increase in load-carrying capacity. We report in this paper the effect of carbon nanotube additions on the actuation behaviour of both polyaniline and chitosan actuator materials. In both cases a significant increase in actuation strength was observed. In addition, the effect of the carbon nanotubes on the modulus and, therefore, actuation strain was also considered. A small reduction in strain was noted for the polyaniline, but little change in actuation strain was observed for the chitosan hydrogels. The results are further analysed in terms of actuation mechanisms occurring in conducting polymers and hydrogels.

**9:00 AM V7.3/W6.3**

**Synthesis and Electronic Properties of Individual Single-Walled Carbon Nanotube/Polypyrrole**

**Heterostructures.** James V. Ly<sup>1</sup>, Xiaolei Liu<sup>3</sup>, Song Han<sup>2</sup>, Alexander Lee<sup>5</sup>, Daihua Zhang<sup>3</sup>, Mark E. Thompson<sup>2,1</sup>, Ari Requicha<sup>4</sup> and Chongwu Zhou<sup>3,2</sup>; <sup>1</sup>Materials Science, University of Southern California, Los Angeles, California; <sup>2</sup>Chemistry, University of Southern California, Los Angeles, California; <sup>3</sup>Electrical Engineering/Electrophysics, University of Southern California, Los Angeles, California; <sup>4</sup>Computer Science, University of Southern California, Los Angeles, California; <sup>5</sup>Chemical Engineering, University of Southern California, Los Angeles, California.

Heterostructures of individual single-walled carbon nanotubes (SWNT) coated with conducting polymer polypyrrole (PPy) were

synthesized by electrochemical polymerization and its electrical properties were analyzed. Growth conditions were explored to controllably produce SWNT/PPy nanostructures. The resultant SWNT/PPy has a SWNT core and a PPy cladding ranging from 10-150 nm in thickness, as measured by AFM and electron microscopy. The PPy thickness has shown to be, among other factors, both voltage and time dependent. In a series of PPy deposition cycles performed on the same individual SWNT, it is found that the electronic properties are more complex than the simple additive effect of both components. Even with similar diameters, whether the heterostructure was synthesized by a single or multiple deposition cycles, resulted in different conductances and further underlines the complexity of this system. What has been discovered, however, is that the thickness of the PPy layer plays a critical role in dominating the conductance of the heterostructure itself. The high surface-to-volume ratio of SWNT/PPy heterostructures also provides an advantage over traditional films as potential chemical gas sensors. Electronic measurements have demonstrated an increased conductance when SWNT/PPy are exposed to NO<sub>2</sub> and, in contrast, a decreased conductance when exposed to NH<sub>3</sub>.

**9:15 AM V7.4/W6.4**

**Carbon Nanocones and Nanocapsules with Metallo-Tetrapyrrol Tips.** Petr Kral and Stanislav Stoyanov; Department of Chemistry M/C 111, University of Illinois at Chicago, Chicago, Illinois.

We predict the existence of novel types of nanocones and nanocapsules that can be realized by a combination of metallo-tetrapyrroles, with their unique binding properties, and carbon nanotubes, possessing a large mechanical rigidity and electric polarizability. Our ab-initio calculations demonstrate that the novel structures are stable and have many interesting features, with potential applications in material sciences, chemistry, molecular mechanics and electronics.

**9:30 AM V7.5/W6.5**

**Suspended Carbon Nanotube Tunneling Switch for Wireless Communication.** Youngsik Song and Jaewu Choi; Electrical and Computer Engineering, Wayne State University, Detroit, Michigan.

We fabricated carbon nanotube tunneling switch by direct and lateral growth of suspended carbon nanotubes and studied its characteristics and performance for future nano-scale wireless communication systems. Laterally suspended carbon nanotubes are very straight, ~3 μm long and strongly anchored to metal electrodes. The physical structure of carbon nanotubes was investigated by Raman spectroscopy and transmission electron microscopy. The onset of tunneling turn-on voltage is as low as 2.6 V. The switching behavior was studied by modulating back gate bias and impedance spectroscopy.

**10:15 AM V7.6/W6.6**

**Effect of CNT on dipole orientation and electro-active properties of polymer nanocomposite.** Jinho Kang<sup>1</sup>, Cheol

Park<sup>1</sup>, Kristopher E. Wise<sup>1</sup>, Steven J. Gaik<sup>2</sup>, Nancy M. Holloway<sup>3</sup>, Sharon E. Lowther<sup>4</sup> and Joycelyn S. Harrison<sup>4</sup>; <sup>1</sup>National Institute of Aerospace, Hampton, Virginia; <sup>2</sup>Department of Materials Science and Engineering, Penn State University, Philadelphia, Pennsylvania; <sup>3</sup>Technology Development and Integration Branch, NASA LaRC, Hampton, Virginia; <sup>4</sup>Advanced Materials and Processing Branch, NASA LaRC, Hampton, Virginia.

High performance electro-active materials are often required for aerospace vehicles and astronaut suits. Investigations of the dipole orientation at the microscopic and macroscopic levels are essential for understanding electro-activities of nano-structured materials. In this presentation, the effect of carbon nanotube (CNT) on dipole orientation and electro-active properties of a polymer matrix will be addressed as a function of type of CNT, CNT content, electric field, poling profiles and temperature. The relaxation of electric-field induced dipole orientation and dielectric relaxation strength were investigated by thermally stimulated current measurement (TSC) technique and an impedance analyzer. The preliminary study showed that the remanent polarization (P<sub>r</sub>) was retained at temperatures close to the glass transition temperature (~200 °C) of the polymer. The high temperature stability of the remanent polarization is one of the keys of developing high performance electro-active materials for aerospace applications in ultra-extreme aerospace environments. Out-of-plane (d<sub>33</sub>) and in-plane (d<sub>31</sub>) electro-active responses were characterized using a fiber optic sensor and a modified Rheovibron. Other sensing characteristics under various stimuli will be also introduced. Analytical modeling and numerical simulation were performed to aid in understanding the experimental results.

**10:30 AM V7.7/W6.7**

**Electro-Mechanical Actuation of Carbon Nanotube Yarns and**

**Sheets.** Mikhail Kozlov<sup>1</sup>, Vignesh Seker<sup>1</sup>, Jiyoung Oh<sup>1</sup>, Mei Zhang<sup>1</sup>, Shaoli Fang<sup>1</sup>, Ryan Capps<sup>1</sup>, John Madden<sup>2</sup>, Von Howard Ebron<sup>1</sup>, John Ferraris<sup>1</sup> and Ray Baughman<sup>1</sup>; <sup>1</sup>NanoTech Institute, University of Texas at Dallas, Richardson, Texas; <sup>2</sup>Department of Electrical and Computer Engineering, University of British Columbia, Vancouver, British Columbia, Canada.

We report preparation of highly conductive SWNT and MWNT yarns and sheets. The materials aim at such applications as electronic textiles, electro-mechanical actuators, and ESD anti-static coatings. The electro-mechanical response of the specimens was measured using custom made force transducer operating in an isometric mode. The measurements were carried out at room temperature in aqueous and organic electrolytes; square-wave potential of variable amplitude was applied with a potentiostat. It was found that the maximum isometric stress generated by nanotube actuators could be as large as 12 MPa. This approaches the stress generation capability of commercial ferroelectrics and is significantly larger than that of natural muscles. A variety of applications of the materials is discussed.

#### 10:45 AM V7.8/W6.8

**Are the Possibilities Infinite for the Molecular Conductivities in Real Measurement?** Anirban Bandyopadhyay<sup>1</sup>, S. Yagi<sup>2</sup>, K. Nittou<sup>2</sup>, K. Miki<sup>2</sup> and Y. Wakayama<sup>3</sup>; <sup>1</sup>International Center of Young Scientists, National Institute of Material Science, Tsukuba, Ibaraki, Japan; <sup>2</sup>Nanoarchitecture Group, National Institute of Material Science, Tsukuba, Ibaraki, Japan; <sup>3</sup>Nanoassembly Group, National Institute of Material Science, Tsukuba, Ibaraki, Japan.

Conductivity of a molecule measured in STM changes continuously due to change in molecular conformation under electric bias, thermal drift and change in the coupling factor between molecule and electrode as a change of the other two mentioned parameters. An experimentalist ends up with measuring several unique current voltage measurements with a single molecule in the same environment. But normally the response that resembles with theoretical prediction is considered others left as an artifact evolved from experimental systems. To find answers to degree of unpredictability of such phenomenon we considered the whole response as a stochastic event and went on measuring several current voltage spectrums under specific environmental conditions of a molecule. Instead of isolated single molecule we have considered cluster of molecules and targeted randomly as interaction of enthalpy and entropy would allow them to create infinite possible dimensional distribution of quantum wells while incubation to the atomically flat gold surfaces. Variation of conductivities has been classified into 10 different natures considering current voltage measurements for Xanthene dye molecules such as Rose Bengal, Eosin Y and Fluorescein Sodium. The result showed that the probabilities in the ionic states controlled by resonance structures of ions while neutral state seems to follow unique conductivity spectrum. The Xanthene dyes have already been reported to show molecular switching<sup>1</sup>. It was also reported that multiple states exist for these molecular systems<sup>2</sup>. As previously it was not possible to detect multiple states of the molecular systems, now we have succeeded in detecting each possible states of such molecular systems. Our results have shown that single molecules can behave as semiconductors, switches, rectifiers and even showing negative differential resistance depending on which molecular state it exists. Further challenge in controlling a particular molecular state on gold substrate is being carried out by tuning supramolecular structures. So statistical dominance of a typical quantum well during growth of supramolecular structures under various pHs might be possible. 1. Large conductance switching and binary operation in organic devices: Role of functional groups. A. Bandyopadhyay and A. J. Pal, J. Phys. Chem. B. 107, 2531 (2003). 2. Multilevel conductivity and conductance switching in supramolecular structures of an organic molecule. A. Bandyopadhyay and A. J. Pal, Appl. Phys. Lett. 84, 999 (2004)

#### 11:00 AM V7.9/W6.9

**The Impact of Interfacial Modification on the Electrical Responses of In Situ Polymerized Polyimide / Carbon Nanofiber Composites.** Michael J. Arlen<sup>1,2</sup>, David Wang<sup>3</sup>, Marlene Houtz<sup>3</sup>, Gary Price<sup>3</sup>, Loon-Seng Tan<sup>1</sup> and Richard Vaia<sup>1</sup>; <sup>1</sup>Polymer Science and Engineering, University of Akron, Akron, Ohio; <sup>2</sup>Materials and Manufacturing Division, Air Force Research Laboratory, Dayton, Ohio; <sup>3</sup>University of Dayton Research Institute, Dayton, Ohio.

The electrical response of in situ polymerized polyimide (PI) / carbon nanofiber (CNF) composites were investigated for use as high temperature ESD and EMI films and as thermal electric switches. The preparation of PI/CNF nanocomposites were conducted via a two-stage polymerization of 2,2-bis(phthalic anhydride)-1,1,1,3,3,3-hexafluoroisopropane (6FDA) and 1,3-bis(3-aminophenoxy)benzene (BAPB) in the presence of 0.1-5 wt%

CNF or amine functionalized CNF (H2N-CNF) in DMAc at room temperature, followed by thermal imidization. This results in 2 PI/CNF systems, differing in the strength of association at the tube-polymer interface. The amine groups on the H2N-CNF participate in the polymerization leading to surface grafted PI chains to the CNFs as illustrated by SEM results. AC impedance results illustrate PI/H2N-CNF composites at 5 wt.% loading exhibit DC conductivity three orders magnitude lower than PI/CNF composites. In addition, PI/CNF with 5 wt.% loading demonstrate the negative temperature coefficient (NTC) effect of resistivity, while PI/ f-CNF composites of equal loadings exhibit the positive temperature coefficient (PTC) effect of resistivity. The difference in the conductivity temperature dependence between composites was attributed to the grafted PI chains formed on the H2N-CNF during the in situ polymerization process.

#### 11:15 AM V7.10/W6.10

**Investigation of the Physics of Transduction in Nafion / Ionic Liquid Composite Membranes.** Matthew Bennett, Barbar Akle and Donald Leo; Mechanical Engineering, Virginia Tech, Blacksburg, Virginia.

Ionic polymer actuators are a class of electroactive polymers (EAPs) that exhibit large bending motion (1%) under the application of small voltages (1-5 V). The most common polymer membrane used for these actuators is Nafion. The actuation mechanism in these materials is believed to arise from the field-induced motion of mobile charges when a voltage is applied. In order for this charge motion to occur, the material must be swollen with a solvent. Typically, the solvent used has been water. However, the use of water imposes several limitations on the application of these actuators. Recently, Bennett and Leo have shown that these limitations can be overcome by replacing the water in these actuators with an ionic liquid (Sensors and Actuators A, 115, pp. 79-90, 2004). In the current paper, we investigate the physics of transduction in these ionic liquid-swollen Nafion membranes. Small-angle X-ray scattering reveals that the structure and properties of the ionic liquid have a strong influence on the morphology of the composites. Infrared spectroscopy is used to probe the ion associations within the films and shows that the ionic liquids are able to effectively mobilize the counterions of the Nafion membrane. Nuclear magnetic resonance spectroscopy is also used to investigate the composites and reveals that the mobility of the counterions increases as the content of ionic liquid within the membrane is increased. The results of these characterizations are compared to an experimental investigation of transduction in Nafion / ionic liquid composites to form an interpretation of the mechanisms of actuation. This comparison reveals that the counterions of the Nafion membrane are the primary charge carriers and that it is the motion of these mobile charges that gives rise to the actuation behavior of the films. Additionally, the use of ionic liquids has facilitated the development of a new type of electrode fabrication technique. This new "direct assembly" method allows for much larger strains to be generated than with previous electroforming processes.

#### 11:30 AM V7.11/W6.11

**Modeling Chemomechanical Devices Based on a Reactive Polymer Gel.** Victor V. Yashin and Anna C. Balazs; Chemical & Petroleum Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania.

We model polymer gel based miniature chemomechanical devices such as pumps, actuators and sensors. The key element for these devices is a reactive polymer gel, which exhibits swelling-deswelling behavior in response to a chemical reaction, thus performing chemical-to-mechanical energy conversion. We consider a theoretical model of a reactive polymer gel, in which the reaction can proceed in an oscillatory regime and generates traveling chemical waves accompanied by waves of local swelling-deswelling. This type of gel could be used for fabricating chemomechanical devices with self-sustained rhythmic action, and gel-based pumps. We assume that the Belousov-Zhabotinsky (BZ) reaction takes place in the reactive gel. The BZ reaction generates periodic redox changes of a metal catalyst covalently bonded to a hydrogel soaked in a solution containing the rest of the BZ reagents. The metal redox changes affect the polymer-solvent interactions, resulting in variations in the gel volume. The self-oscillation of gel volume, and the traveling waves of local swelling in a hydrogel with the BZ reaction have been experimentally observed by Yoshida and co-workers. To describe the system theoretically, we employ the Oregonator model of the BZ reaction, and the two-fluid model of the gel dynamics. Propagation of one-dimensional wave trains through the reactive gel is simulated. Structure of the traveling swelling-deswelling waves, and transport of solvent through the gel induced by the wave propagation are studied.

#### 11:45 AM V7.12/W6.12

**A Novel Glucose Sensor Based on Deflection of a Thin Hydrogel Membrane.** Sandeep Mariserla<sup>1</sup>, Zayd Leseman<sup>2</sup> and

Thomas J. Mackin<sup>2</sup>; <sup>1</sup>Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>2</sup>Mechanical and Industrial Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois.

Self-monitoring of blood glucose has become an important and critical tool for effective management of patients with diabetes. We describe here a novel MEMS sensor that can continuously measure, in real time, the concentration of glucose in a solution. The device utilizes a glucose-sensitive hydrogel membrane, which swells reversibly in the presence of a glucose containing solution. The amount of swelling is related to the concentration of glucose. The hydrogel is composed of 2-hydroxyethyl methacrylate (HEMA) functionalized with 3-acrylamidophenylboronic acid (AAPBA) groups as the glucose-sensitive moiety. Phenylboronic acid (PBA) derivatives interact with glucose by forming a charged complex, causing the hydrogel to swell by solvent intake. The resultant swelling in turn buckles the membrane and deflects a silicon island that is coupled to the hydrogel membrane. Signal transduction is performed by measuring the change in capacitance between the silicon island and a fixed plate. The presence or variation in the concentration of glucose can be observed as a change in capacitance of the device. The device was fabricated using standard microfabrication techniques. Initially, a Si wafer is processed to yield dies with a trench-like opening. Si islands are patterned from the bottom side of the die using photoresist. Subsequently, a coating of PDMS is applied to the bottom surface of the device as a barrier film. In the remaining steps the Si is etched away leaving behind released islands of Si attached to the PDMS membrane. In the final step, the glucose-sensitive hydrogel monomer is then processed onto the opposite surface of the PDMS membrane and UV-polymerized. We will present preliminary results detailing the fabrication of the device, the swelling properties of the AAPBA-containing HEMA hydrogel membranes, their adhesion to the PDMS protective membrane and the deflection caused by their expansion.

SESSION V8: Piezoelectric Materials  
Chairs: Jing-Feng Li and S. Wada  
Wednesday Afternoon, November 30, 2005  
Independence W (Sheraton)

#### 1:30 PM V8.1

**Enhanced Piezoelectric Properties of Barium Titanate Single Crystals by Domain Wall Engineering using Patterning Electrodes.** Satoshi Wada, Koichi Yako, Tomomitsu Muraishi, Song-Min Nam, Hirofumi Kakemoto and Takaaki Tsurumi; Dept. of Metallurgy & Ceramics Science, Tokyo Institute of Technology, Meguro, Tokyo, Japan.

For the [111] poled tetragonal barium titanate (BaTiO<sub>3</sub>) single crystals with the engineered domain configuration, it was clearly observed that the piezoelectric properties increased with increasing 90 deg domain wall densities. To explain the phenomenon, the multidomain single crystals were regarded as the composite of (a) a distorted 90 deg domain wall region and (b) a normal tetragonal domain region. Using a 2-phases model, the piezoelectric properties from the 90 deg domain wall were estimated. As a result, ultrahigh piezoelectric constants over 8,000 pC/N as a minimum value were expected from the 90 deg domain wall region. Moreover, this study suggested that it is possible to obtain the BaTiO<sub>3</sub> crystals with the d<sub>31</sub> and d<sub>33</sub> over 1,000 pC/N, when the domain sizes can decrease below 1 μm. However, the control of (1) temperature and (2) electric-field along [111] direction resulted in the formation of the BaTiO<sub>3</sub> crystals with a minimum 90 deg domain size around 5.5 μm. Therefore, in this study, to induce the much finer 90 deg domain sizes into the [111] BaTiO<sub>3</sub> crystals, the patterning electrodes with sizes from 10 μm to 1 μm were applied. The patterning electrodes were prepared on the crystals using photolithography technique. Using patterning electrodes, the poling treatment was performed with the control of temperature and electric-field along [111] direction. As the results, much finer 90 deg domain sizes was induced into the [111] BaTiO<sub>3</sub> crystals. Moreover, their piezoelectric properties were measured, and this crystals with the 90 deg domain sizes of 3 μm exhibited the d<sub>31</sub> over 250 pC/N. Moreover, the induction of much finer 90 deg domain sizes was discussed.

#### 1:45 PM V8.2

**Synthesis and microwave dielectric properties of 'twinned' and 'shifted' hexagonal perovskites in the system Ba<sub>5+m</sub>Nb<sub>4</sub>Ti<sub>m</sub>O<sub>15+3m</sub>.** Ritesh Rawal, Antonio Feteira and Derek Sinclair; Department of Engineering Materials, The University of Sheffield, Sheffield, United Kingdom.

Among hexagonal perovskites, B-site deficient perovskites of general formula A<sub>n</sub>B<sub>n-1</sub>O<sub>3</sub> present two kinds of structural types[1]

depending on the stacking sequences of the AO<sub>3</sub> layers (i.e. hexagonal (h) or cubic (c) packing). Structures with (hh..c)-type sequences present successive perovskites, which are shifted from one another by a 1/3<01-10>H vector, while (hc..c)-type sequences show twin plane boundaries. The (Ba<sub>5</sub>Nb<sub>4</sub>O<sub>15</sub>+ nBaTiO<sub>3</sub>) Ba<sub>5+m</sub>Nb<sub>4</sub>Ti<sub>m</sub>O<sub>15+3m</sub> system belong to an A<sub>n</sub>B<sub>n-1</sub>O<sub>3n</sub> series of hexagonal perovskites with m=0 and 1 demonstrating shifted structures with the sequences 5H (hhccc) and 18R (hhccc)3, respectively. The sequence 7H (hhccc) for m=2 has not been observed as a stable compound; however, for m=3 a twinned structure with a sequence (hhcc)2 has been observed. It has been reported that twinned structures are favored by a high value of n (number of octahedral layers within a perovskite block) and t (Goldschmidt tolerance factor), while shifted structures are stabilized by the periodicity of the cation sub-lattice. Although a lot of structural work has been reported on the Ba<sub>5+m</sub>Nb<sub>4</sub>Ti<sub>m</sub>O<sub>15+3m</sub> system, very little has been reported on the dielectric properties of this system at microwave frequencies. This system combines relatively high permittivity ranging from (39< ε< 48) and low dielectric loss (Q.f ≈ 23,000 GHz). Although Q.f did not show any substantial change with an increase in 'm', the value of relative permittivity increases from 38 for Ba<sub>5</sub>Nb<sub>4</sub>O<sub>15</sub> [2] to 43 for Ba<sub>6</sub>Nb<sub>4</sub>TiO<sub>18</sub> and 48 for 8-layered Ba<sub>8</sub>Nb<sub>4</sub>Ti<sub>3</sub>O<sub>24</sub>. This increase in relative permittivity can be associated with the increase in the cubic layers of BaTiO<sub>3</sub> within the perovskite blocks. References: [1] G. Trolliard, N. Teneze, Ph. Boullay and D. Mercurio, Journal of Solid State Chemistry, Vol. 177, pp. 1188-1196, 2004. [2] C. Vineis, P.K. Davies, T. Negas and S. Bell, Material Research Bulletin, Vol. 31, No. 5, pp. 431-437, 1996.

#### 2:00 PM V8.3

**Microstructure-Controlled Multilayer PZT Actuators: Effects of Cyclic Actuation on Crystallographic Structure.**

Jens Mueller<sup>1</sup>, Stephanie Hooker<sup>1</sup> and Davor Balzar<sup>1,2</sup>; <sup>1</sup>Materials Reliability 853, NIST, Boulder, Colorado; <sup>2</sup>Department of Physics and Astronomy, University of Denver, Denver, Colorado.

Many new applications are emerging for piezoelectric ceramics, including adaptive structures, vibration isolation, and nano-robotics. In these instances, actuators will likely be operated at higher electric fields than in conventional applications in order to achieve maximum displacement. However, under such relatively harsh driving conditions, there is an increased possibility of property degradation and fatigue. Tailoring the ceramic microstructure through sintering control offers one possible route to improve fatigue resistance. In this work, we studied the influence of both sintering temperature and cyclic actuation on the microstructure of lead zirconate titanate, PbTi<sub>x</sub>Zr<sub>1-x</sub>O<sub>3</sub> (PZT) actuators, using X-ray diffraction and SEM. Specimens were prepared under identical conditions by multilayer fabrication and sintered for 24 minutes at temperatures ranging from 1175 to 1325 °C. The resulting devices had average grain sizes ranging from 2-3.5 μm, with grain size increasing with processing temperature. X-ray diffraction measurements (θ-2θ) were performed on as-grown, poled and electrically fatigued specimens. The observed Bragg reflections could be attributed to the tetragonal phase of PZT, with minor amounts of rhombohedral, monoclinic, and pyrochlore phases also detected. Coherently diffracting domain sizes were evaluated from line broadening in the [001] and [100] directions. SEM analysis of polished and etched cross-sections was used to determine grain size. To separate poling and fatigue effects from processing changes, the dependence of integrated diffraction-line intensities on sintering temperature was first examined for the as-grown material. A transition in ferroelectric domain orientation was observed in the region of 1250 °C. Below this temperature, the domain structure was random, whereas above 1250 °C a decrease in c-oriented domains was observed. In comparison to the as-grown material, the fatigued specimens exhibited a strong decrease in the amount of rhombohedral phase below 1250 °C, with the material becoming almost completely tetragonal at 1225 °C. An increase in the ratio of domain size to SEM grain size, D<sub>D</sub>/D<sub>G</sub>, was observed with increasing sintering temperature. This parameter proved critical for explaining the changes connected with domain switching and pinning effects, which take place during poling and operation. A low D<sub>D</sub>/D<sub>G</sub> indicates a lower interaction of domain walls with grain boundaries, which enables domains to switch easier between orientations and phases. These data support corresponding measurements of changes in remanent polarization with cumulative exposure to high electric fields, where specimens processed below 1250 °C showed greater resistance to polarization fatigue, while specimens processed above 1250 °C fatigued faster. Evaluation of the changes in peak intensities further support these results, with the observed trend in the rhombohedral-to-tetragonal phase transition changing significantly below 1250 °C.

#### 2:15 PM V8.4

**Electrophoretic deposited PZT films for embedded components: particle morphology and surface charge effects.** Aiyng Wu<sup>1</sup>, Paula Maria Vilarinho<sup>1</sup> and Angus Kingon<sup>2</sup>; <sup>1</sup>CICECO, Dep. Ceramic and Glass Eng., University of Aveiro, Aveiro, Portugal;

Functional ferroelectric oxide films are of particular interest for microelectronic applications. Fabrication of ferroelectric films on metal foils is of relevance for devices integrated into the electronic package, an approach that is currently receiving considerable attention due to its cost and space advantages. Copper is attractive substrates for ferroelectric film deposition due to low cost and high conductivity. High-permittivity material on Cu is promising for embedded capacitor applications in printed wiring boards. Besides cost reduction, the required wiring board real estate is greatly reduced by replacing the surface-mount components with pre-embedded capacitive layers, offering advantages for electronic miniaturization. Compared to Pt the low cost and high conductivity of Cu makes it an appealing candidate to replace Pt for ferroelectric oxide film-based devices. However, the utilization of Cu foils as substrates for films requires control of oxygen partial pressure to avoid the metal oxidation and compatibility with the high annealing temperature in oxygen atmosphere needed to obtain the desired crystalline phase. By finding conditions that fulfil both requirements the preparation of functional ferroelectric thin films on Cu foils with optimized electric properties was recently reported. The annealing temperature for these thin films on Cu does not exceed 650°C. However, for thick films deposited on Cu the sintering temperature should be much higher than the one used in thin film processing. Our previous work on electrophoretic deposited (EPD) >5 µm PZT films on metal foils (Cu and Pt) showed that good electrical properties could be obtained on Pt foil after sintering at 1150°C. But PZT films deposited on Cu sintered at 950°C showed phase decomposition due to the Cu-Pb alloy formation and, consequently, deteriorated dielectric properties. These results pointed to the importance of processing thick PZT films on Cu at low temperatures. To decrease the sintering temperature (<900°C), current work is focused on the study of the effect of particle size and surface charge of PZT powders on the deposition and sintering behaviour of PZT thick films prepared by EPD. The effect of milling time on the particle size, particle size distribution and surface charge (zeta-potential) of precursor powders towards optimization of EPD deposition conditions is reported. Ferroelectric PZT films in the thickness range from 5 to 50 µm were deposited by EPD on flexible metal foils (Cu, Ni coated Cu, Pt) and sintered at various temperatures using PZT powders with different morphological characteristics and surface charge states. The structure, microstructure and the interface reaction region of the films were inspected by x-ray diffraction, scanning electron microscopy, transmission electron microscopy and Rutherford backscattering. The improved electrical properties of the films were correlated with the particle size and surface charge of the precursor powders.

### 3:30 PM V8.5

**Ring Resonator Method for Evaluation of Dielectric Property of Thin Layers in Microwave Region.** Takashi Teranishi, Kentaro Tajima, Takakiyo Harigai, Song-Min Nam, Hirofumi Kakemoto, Satoshi Wada and Takaaki Tsurumi; Dept. of Metallurgy & Ceramics Science, Tokyo Institute of Technology, Meguro, Tokyo, Japan.

Recently, the accurate measurement technique of microwave dielectric properties of thin layers has been required for the development of multilayered high frequency modules. Especially it is essential to evaluate dielectric property of low-loss dielectric layers for substrate materials in the module. The objective of this study is to develop a ring-resonator method applicable to dielectric substrates and thin films. In this method, a sample of dielectrics thin layers is pressed with constant stress on a ring resonator formed on a sapphire substrate, and the dielectric permittivity and the loss tangent of the sample are evaluated from the change of the resonance frequency and resonance peak width. The shape of the ring resonator was first designed by electromagnetic simulation using finite integrate time domain method. The ring resonator of Cu was formed on the substrate by a photo-lithography technique. Dielectric permittivity was determined by fitting the resonance frequency observed with that simulated using the electromagnetic-field analysis, while the loss tangent was determined from the half bandwidth of the observed and simulated resonance peaks. Dielectric property of some dielectric thin layers, such as strontium titanate, alumina ceramics and new polymers for module substrates, could be determined accurately by this method, which indicated that the ring resonator method developed in this study would be a powerful tool to evaluate microwave dielectric properties of dielectric thin layers.

### 3:45 PM V8.6

**Structural Characteristics and Piezoelectric Properties of Electrospun Piezoelectric nanofibers.** Yong Shi<sup>1</sup>, Shiyu Xu<sup>1</sup>, Sang-Gook Kim<sup>2</sup> and Matthew Libera<sup>3</sup>; <sup>1</sup>Mechanical Engineering, Stevens Institute of Technology, Hoboken, New Jersey; <sup>2</sup>Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; <sup>3</sup>Chemical, Biomedical and Materials Engineering, Stevens Institute of Technology, Hoboken, New Jersey.

Lead Zirconate Titanate (PZT), an important piezoelectric material, has been used both as actuators and sensors at macro scale for a long time. PZT nano-fibers provide the capability to make nano scale sensors and actuators. There are a few ways to fabricate PZT fibers, such as dicing and extrusion. Extruding of PZT sol through a spinneret can generate PZT fibers with diameters of 10 to 80 µm (average 30 µm). It was also reported that electrospinning had been used to obtain PZT fibers with diameter of less than 10 µm. This paper reports the development and characterization of PZT nanofibers with average diameters of about 150 nm and possible further down to 50 nano-meters for various sensing and actuation applications. PZT nano fibers have been developed by sol-gel electrospinning process. Both randomly distributed and uniaxially aligned PZT fibers were obtained from the sol-gel solution with viscosity modified by polyvinyl pyrrolidone (PVP). The diameters of the nano fibers can be further reduced or controlled for different applications. The morphology and structure of the nanofibers were examined with SEM, TEM and XRD. We used two-step process to anneal the electrospun fibers and XRD confirmed that pure perovskite phase was formed after the as-spun fibers being annealed at about 650°C. TEM results showed that the grain size of the fibers was about 10 nm. Microelectromechanical (MEMS) based micro-fabrication technologies were used to assist the development of the nano-fibers in designing the test samples, depositing and patterning the electrodes, and also testing the performance of the nano fibers. Different approaches have been explored to fabricate the uniaxially aligned nano fibers. SEM results showed that partial aligned PZT nano-fibers were obtained on the pre-patterned substrates. Interdigitated Electrodes were evaporated on the partial aligned fibers by using shadow mask. The typical hysteresis curve of the nano piezoelectric fibers was also obtained. These properties have shown that the PZT nanofibers have promising application potentials in designing and enabling micro and nano devices.

### 4:00 PM V8.7

**Abstract Withdrawn**

### 4:15 PM V8.8

**Piezoresponse Force Microscopy of Lead-Free Relaxors: Size Dependence of Electromechanical Properties.**

Andrey Soukhojak, Ram Chandra Tiruvalam and Animesh Kundu; Materials Sci & Eng, Lehigh University, BETHLEHEM, Pennsylvania.

In this work we experimentally address the question of how small a sample of a normally ferroelectric material has to be to have an unstable spontaneous polarization direction, i.e. to be in a superparaelectric state. We report on the results of piezoresponse force microscopy (PFM) study of nano-size samples of lead-free relaxor ferroelectric: sodium bismuth titanate (NBT) co-doped with Ba and Zr (BNBZT system). The BNBZT has been shown to exhibit a rich variety of phases and substantial compositional sensitivity of electromechanical properties: from well-pronounced ferroelectric to electrostrictive. The BNBZT samples of both ferroelectric and electrostrictive phases ranging in size from a few to a few hundred nanometers were prepared by a novel solution chemistry (citrate gel) route. We present both compositional and size dependences of the electromechanical properties.

### 4:30 PM V8.9

**Studies of Bi<sub>4-x</sub>Gd<sub>x</sub>Ti<sub>3</sub>O<sub>12</sub> Bifunctional Material.**

Maharaj S. Tomar<sup>1</sup>, Ricardo E. Melgarejo<sup>1</sup>, Rolando P. Guzman<sup>1</sup> and Ram S. Katiyar<sup>2</sup>; <sup>1</sup>Physics, University of Puerto Rico, Mayaguez, Mayaguez, Puerto Rico; <sup>2</sup>Physics, University of Puerto Rico, San Juan, San Juan, Puerto Rico.

Bi<sub>4-x</sub>Gd<sub>x</sub>Ti<sub>3</sub>O<sub>12</sub> is a material where Gd-ion substitutes for Bi-ion in Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> without changing the crystal structure and it appear to be bifunctional material - ferroelectric and ferromagnetic. In order to look at this possibility, we synthesized Bi<sub>4-x</sub>Gd<sub>x</sub>Ti<sub>3</sub>O<sub>12</sub> powder by sol-gel process and thin films were deposited by spin coating. Thin films were characterized by x-ray diffraction and Raman spectroscopy for structural properties. Films showed single phase materials up to the compositions: x < 1. The films were also investigated for ferroelectric and ferromagnetic response. Results indicate the presence of ferroelectric memory and weak ferromagnetism at lower temperatures. Composition dependent properties will be presented.

SESSION V9: Poster Session: Piezoelectric Materials II

Chair: S. Wada

Wednesday Evening, November 30, 2005

8:00 PM

Exhibition Hall D (Hynes)

### V9.1

**Fabrication of Microwave Dielectric Ceramics by Aerosol**

**Deposition Method.** Mihoko Momotani<sup>1</sup>, Hirofumi Kakemoto<sup>1</sup>, Satoshi Wada<sup>1</sup>, Takaaki Tsurumi<sup>1</sup>, Song-Min Nam<sup>2</sup> and Jun Akedo<sup>2</sup>; <sup>1</sup>Dept. of Metallurgy & Ceramics Science, Tokyo Institute of Technology, Meguro, Tokyo, Japan; <sup>2</sup>National Institute of Advanced Industrial Science and Technology, Tokyo, Japan.

Recently, miniaturization and high-frequency-operation of the electric devices have been in great need. Although the surface mount technology has been effective as a way of solving the influence of wiring resistance or crosstalk noise, it has a limit of reducing the wiring length. Therefore, the development of integrated RF modules is required, in which passive components, such as capacitors, resistors and inductors, are built in one printed wiring board. As conventional fabrication technologies of the integrated RF modules, LTCCs (low temperature cofired ceramics) and polymer composites have been intensively researched. However, low temperature fabrication process of dielectric ceramics with a high performance in microwave region are still required to form a microwave modules. Essential characteristics for microwave dielectric ceramics may include no temperature dependence, a high quality factor and a relatively high dielectric constant. If microwave dielectric ceramics, which satisfy these characteristics, could be fabricated at low temperature, embedded capacitors and integrated RF modules would be realized. Therefore, we focused on the aerosol deposition method, which enables us to fabricate ceramic thick films at room temperature. In order to fabricate microwave dielectric ceramic films, high purity BaCO<sub>3</sub>, Sm<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> powder were mixed to be the composition of BaO·Sm<sub>2</sub>O<sub>3</sub>·5TiO<sub>2</sub>, calcined at 900° and then fired at 1350~1380°. This BaO·Sm<sub>2</sub>O<sub>3</sub>·5TiO<sub>2</sub> powder was deposited on Cu substrate by aerosol deposition method at room temperature. Dense films could be obtained by optimizing deposition conditions. The microwave dielectric properties were measured by a newly developed ring resonator method.

#### **V9.2** **Microstructure-Controlled Multilayer PZT Actuators: Effects of Cyclic Actuation on Polarization Fatigue.**

Stephanie Anne Hooker<sup>1</sup>, Jens Mueller<sup>1</sup> and Clayton Kostelec<sup>2</sup>; <sup>1</sup>Materials Reliability, NIST, Boulder, Colorado; <sup>2</sup>Synkera Technologies, Longmont, Colorado.

Piezoelectric ceramics are desirable actuator materials due to their fast response, wide bandwidth, high force, compact size, and ease of motion control. Many new applications are emerging for these materials, including adaptive structures, vibration isolation, and nanorobotics. In these instances, the materials may be required to perform continuously over long durations, while also achieving maximum physical displacement. These demands translate into relatively harsh driving conditions, where an increased possibility of property degradation and fatigue exist. Microstructure control offers one possible route to improve fatigue resistance. To determine the relationship between microstructure and fatigue, we examined the effect of continuous cyclic actuation on multilayer lead zirconate titanate (PZT) actuators. These devices were produced from sub-micron particles and processed over a 150 C sintering range. The resulting actuators possessed average grain sizes from 2-3.5 microns, with grain size increasing with increasing processing temperature. Components were fabricated in a surface mount configuration (0.30 cm x 0.15 cm) with ten active (PZT) internal layers, each 50 microns thick. Fatigue resistance was determined by monitoring the change in ferroelectric properties (remanent polarization, PR, saturation polarization, PS, and coercive field, EC) during cumulative exposure to switching electric fields. Measurements were performed using a modified Sawyer-Tower circuit by applying a sinusoidal wave approximating the PZT-5A coercive field (EC ~ 15 kV/cm). Microstructure evolution affected both the initial and fatigued properties of the different devices. Initial values for dielectric constant and polarization reached maxima at 1275 C. Below this temperature, properties increased linearly with processing temperature, with PR increasing nearly 50 % over 100 C. This increase in polarization is likely due to the presence of fewer grain boundaries, which can clamp domain wall motion. Above 1275 C, dielectric and ferroelectric properties also decreased, likely due to secondary phase (pyrochlore) formation. Similar results have been observed by other authors for PZT as a function of grain size. However, unique effects were observed during cyclic actuation tests. Devices processed at low temperatures fatigued far less than those processed at higher temperatures (< 20 % vs. > 50 %), with fatigue rate increasing with increasing sintering temperature up to 1250 C. In all cases, most degradation occurred during the first 100,000 cycles, which is markedly different than behavior previously observed for other PZT devices. All parts remained functional beyond 10 million cycles, with no microcracking observed. Because of the dependence of the initial ferroelectric properties on sintering temperature, final polarization values were quite similar among all parts, indicating the retention of a minimum polarization regardless of microstructure.

**V9.3**  
**Mechanosynthesized Pb(Sc<sub>0.5</sub>Nb<sub>0.25</sub>Ta<sub>0.25</sub>)O<sub>3</sub> Relaxor Ferroelectrics.** Margarita Isabel Correa, Ram Naresh Prasad Choudhary and Ram S. Katiyar; Physics, University of Puerto Rico, San Juan, Puerto Rico.

Though a large number of ferro/antiferroelectric oxides of different structural families (i.e. perovskite, tungsten bronze, etc), synthesized by various routes, are now-a-days available, only a few of them are useful for commercial applications. Because of the simplicity in structure, ferroelectric properties at room temperature, high permittivities, and high piezo/pyro-electric coefficients, some of the Pb-based perovskite (i.e.ABO<sub>3</sub>-type) compounds/composites have been found potentially useful for smart/intelligent devices. Out of these compounds, Pb(Fe/Sc,Nb/Ta)O<sub>3</sub> has drawn more attention because of (i) high dielectric constant and low loss, (ii) high piezo and electro-restrictive coefficients (iii) diffuse phase transition, (iv) frequency dependent dielectric relaxation behavior (i.e. relaxor property), and (v) co-existence of ferroelectric-ferromagnetic properties (i.e. multiferroic behavior) of the material which are required for multilayer capacitors, actuators, pyroelectric detectors, piezoelectric transducers, sensors, multifunctional devices etc. Unfortunately, synthesis of the material in a single phase (at low cost) with optimum properties (required for devices) is still a big challenge. Further, it has been established that the particle size reduction has a dramatic effect on the material properties. In view of the above, we have synthesized single-phase nanoceramics of Pb(Sc<sub>0.5</sub>Nb<sub>0.25</sub>Ta<sub>0.25</sub>)O<sub>3</sub> through a novel processing techniques (mechanochemical route) using high-energy ball milling (speed 300-400 rpm, duration 0-90 hours in 10 hour-step). Detailed analysis of the prepared powders using X-ray and TEM techniques show the formation of material in perovskite phase (starting from 10 hours of milling), and particle size (30-10nm) decreases with milling times. Extensive studies of spontaneous polarization using hysteresis loop tracer (M/S Radiant Technologies) and dielectric properties (dielectric constant, tangent loss and impedance) as a function of temperature (77-600K) and frequency (100Hz to 1MHz) have shown that the material has relaxor ferroelectric behavior and diffuse phase transition. The maximum dielectric constant (at T<sub>m</sub>) was found decreasing with increase in frequency (400 at 100Hz to 3400 at 1 MHz for temperatures T<sub>m</sub>=312 to 317 K). Our detailed studies of particles size effect on the multiferroic properties of the materials for multifunctional application will also be discussed.

**V9.4**  
**Dielectric Properties and Microstructure Stability of Alloyed HfO<sub>2</sub> Films for Gate Dielectric Applications.** Karthik Raman, Rajiv K. Singh and V. Craciun; Materials Science and Engg, University of Florida, Gainesville, Florida.

We have investigated the material and electrical properties of hafnium oxide alloyed with Ti for future gate dielectric applications. Hafnium oxide is a promising high permittivity material but its implementation in CMOS processing is hindered by its low re-crystallization temperature. Alloying has been shown as an alternative to delay the onset of crystallization. Hafnium oxide has been alloyed with aluminum and the films have shown high thermal stability but the overall capacitance achieved is normally around 12-17 owing to a lower dielectric constant of Al<sub>2</sub>O<sub>3</sub>. In this study, we report on the electrical and material properties of Ti doped hafnium oxide films deposited by pulsed laser ablation technique. XPS, GIXD and XRR measurements were done to characterize the chemical bonding, composition, crystallinity, interfacial density and roughness. Pt electrodes were sputter deposited for electrical measurements and to extract the EOT values. High temperature anneal studies were performed by GIXD analysis on samples annealed at 6000 C and 9000 C for 1 minute in flowing nitrogen.

**V9.5**  
**Abstract Withdrawn**

**V9.6**  
**Symmetry Change in Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> by adding Eu<sup>3+</sup>.** Fernanda Adriana Camacho Alanis<sup>1</sup>, F. Gonzalez<sup>1</sup>, M. E. Villafuerte Castrejon<sup>1</sup>, A. Ibarra Palos<sup>1</sup>, G. Gonzalez<sup>1</sup>, R. Sato<sup>2</sup> and J. M. Saniger<sup>2</sup>; <sup>1</sup>Metálicos y Cerámicos, Instituto de Investigaciones en Materiales, Mexico, D.F., Cd. Mexico, Mexico; <sup>2</sup>CCADET, Universidad Nacional Autónoma de México, México, D.F., México.

Bismuth layer-oxide is currently a material of high technological interest, which is proposed as a candidate to develop ferroelectric random access memory. Bismuth titanate (BIT), leading-free ferroelectric materials, has a Curie temperature of 675C and belongs to Aurivillius family. When La<sup>3+</sup> forms solid solutions with BIT, the electrical properties are improved because of lattice cell distortion. Thus, it is possible to assume that any other rare earth could modify the crystalline structure in the same way. This work is focused on to



analyze the symmetry distortion of BIT due to the addition of  $\text{Eu}^{3+}$ . Solid solutions,  $\text{Bi}_{4-x}\text{Eu}_x\text{Ti}_3\text{O}_{12}$   $0 < x < 1.2$ , have been prepared by coprecipitation route. The effect of  $\text{Eu}^{3+}$  in the structure was characterized by X ray diffraction, scanning electron microscopy and Raman spectroscopy. Preliminary results let us identify that symmetry changes occurs as a function of the europium amount incorporated in the lattice, until the solubility limit of  $x=1.2$  is reached. X ray diffraction patterns show a coalescence of the Bragg reflections  $[h00]$  and  $[0k0]$ , indicating modifications in the lattice symmetry. The observed changes in the vibrational modes analyzed by Raman are consistent with the XRD diffraction results. Finally, effects on microstructure by doping ion were included.

#### V9.7

**Dielectric Behavior of High-Pressure Sintered  $(1-x)(\text{Na}_{1/2}\text{Bi}_{1/2})\text{TiO}_3 - x\text{La}(\text{Mg}_{1/2}\text{Ti}_{1/2})\text{O}_3$  Ferroelectric Ceramics.** Andrei Salak<sup>1</sup>, Nikolai Vyshatko<sup>1</sup>, Andrei L. Kholkin<sup>1</sup>, Victor M. Ferreira<sup>2</sup>, Nikolai Olekhovich<sup>3</sup>, Yury Radyush<sup>3</sup>, Ivan Maroz<sup>3</sup> and Anatoly Pushkarev<sup>3</sup>. <sup>1</sup>Department of Ceramics and Glass Engineering / CICECO, University of Aveiro, Aveiro, Portugal; <sup>2</sup>Department of Civil Engineering / CICECO, University of Aveiro, Aveiro, Portugal; <sup>3</sup>Institute of Solid State and Semiconductor Physics / NAS, Minsk, Belarus.

Ferroelectric relaxors belong to the class of smart materials owing to the co-existence of several functional properties: high electromechanical strain, stress-dependent dielectric constant, tunable piezoelectric coefficients and extremely broad temperature range, where these useful properties are maintained. Sodium-bismuth titanate,  $(\text{Na}_{1/2}\text{Bi}_{1/2})\text{TiO}_3$  (NBT), is a promising parent material for the fabrication of relaxors and its solid solutions with low-loss microwave dielectric  $\text{La}(\text{Mg}_{1/2}\text{Ti}_{1/2})\text{O}_3$  (LMT) are expected to display a multifunctional properties useful for smart devices. Ceramics of the  $(1-x)\text{NBT}-x\text{LMT}$  system ( $0 \leq x \leq 0.25$ ), sintered in air by the conventional mixed oxide method, were recently found to be a single-phase perovskite. XRD spectra of all the compositions were successfully refined with the  $R3c$  space group, which allows accounting for both antiphase oxygen octahedra tilting and A-site cation displacements. As LMT content increases, a gradual reduction in dielectric permittivity of  $(1-x)\text{NBT}-x\text{LMT}$  ceramics is observed. Dielectric peaks are flattened, whereas the temperature location of their anomalies is nearly unchanged. This behavior is attributed to the chemical contraction effect upon LMT substitution promoting the antiparallel (AFE-type) cation displacements. As has been shown in a model relaxor system lead magnesium niobate (PMN), the dielectric properties of high-pressure sintered ceramics can be tailored by their processing conditions and are quite different from those of the ceramics obtained by the conventional methods [1]. There are several evidences that high-pressure sintering promotes an AFE-type ordering in PMN. At the same time, the effect of high pressure on NBT has turned out to have features different from that of PMN [2]. This work reports on structure and dielectric characterization of  $(1-x)\text{NBT}-x\text{LMT}$  ceramics ( $0 \leq x \leq 0.25$ ) sintered under high pressure and high temperature conditions. Dielectric behavior of these ceramics is discussed in terms of their composition and effect of high pressure on chemical and dipole ordering in the system. The work is supported by the Foundation for Science and Technology (FCT-Portugal, grants SFRH/BPD/14988/2004 and SFRH/BPD/15004/2004) and the Belarusian Republican Fund for Fundamental Researches ( $\Phi 05\text{MC}-013$ ). [1] N.M. Olekhovich, N.P. Vyshatko, Yu.V. Radyush, A.N. Salak, and V.M. Ferreira, J. Phys.: Condens. Matter 15 (2003) 6879. [2] J. Kreisel, P. Bouvier, B. Dkhil, P.A. Thomas, A.M. Glazer, T.R. Welberry, B. Chaabane, and M. Mezouar, Phys. Rev. B 68 (2003) 014113.

#### V9.8

**Fabrication and Characterization of PZT-Based Coatings on Fibers.** N. Vyshatko<sup>1</sup>, E. Joanni<sup>2,3</sup>, J. R. A. Fernandes<sup>2,3</sup>, R. Savu<sup>3</sup>, P. M. Vilarinho<sup>1</sup> and Andrei L. Kholkin<sup>1</sup>; <sup>1</sup>Departamento de Engenharia Ceramica e do Vidro / CICECO, Universidade de Aveiro, Aveiro, Portugal; <sup>2</sup>UOSE, INESC-Porto, Porto, Portugal; <sup>3</sup>Departamento de Fisica, Universidade de Tras-os-Montes e Alto Douro, Vila Real, Portugal.

Piezoelectric thin and thick film coatings on optical fibers are extremely attractive for a wide range of applications in fast telecommunication lines and optical processing devices [1, 2]. Several attempts to integrate piezoelectric layers on fibers resulted in optical phase modulators and tunable Bragg gratings. In these applications, the piezoelectric coating was used to generate mechanical stress and the consequent optical path length modulation. However, the piezoelectric coating can be used to physically display the fiber by means of asymmetrically induced mechanical stress. Bending displacement of the fiber may provide mechanical scanning of the fiber tip, opening new possibilities for the scanning microscopes and fiber alignment systems. In this work, we report deposition of piezoelectric  $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$  (PZT) thick film onto optical fibers and

their microstructural characterization by a variety of analytical techniques (SEM, TEM, Raman scattering). The films were also electrically characterized in terms of their dielectric constant, polarization and conductivity. PZT thick films were deposited onto a standard single-mode optical fiber of a 0.125 mm diameter by dip-coating method. Sol-gel precursor solution was prepared with dissolution of Ti-isopropoxide and Zr-propoxide in methoxyethanol. Lead acetate trihydrate was used as a Pb-containing source. Fine PZT powder (TRS Ceramics) was then used as ceramic filler dispersed in the solution. This technique is referred as a hybrid sol-gel route. [3] For improving the film quality, the infiltration and intermediate sintering procedures were used. The final sintering temperature of the films was less than 600°C. Deposition procedure was developed for both bare fibers and for those with metal coating (Pt). Several buffer layers between the PZT films and the fiber were used to relieve the mechanical stress due to different thermal expansion coefficients of silica and PZT. Bending vibrations of the fiber driven with external electric field were investigated by the simple Michelson interferometer using various configurations of electrodes. Piezoelectric properties of the films were evaluated based on the measured displacements. Authors are thankful to Portuguese Foundation for Science and Technology (FCT) for the financial support through the grants SFRH/BPD/15004/2004 and POCTI/CTM/44732/2002. 1. H. Ky, H. G. Limberger, R. P. Salathe, and G. R. Fox, J. Lightwave Tech. 14, 23 (1996). 2. G. R. Fox, C. A. P. Muller, C. R. Wuethrich, A. L. Kholkin, N. Setter, D. M. Costantini, N. H. Ky, and H. G. Limberger, Mat. Res. Soc. Proc. 459, 25 (1997). 3. A. L. Kholkin, A. Wu, P. M. Vilarinho, Recent Res. Devel. Mat. Sci. 5, 1, 2004, Research Signpost, Kerala (India).

#### V9.9

**The Crystalline and Dielectric Properties of  $\text{BiScO}_3\text{-PbTiO}_3$  Ferroelectric Ceramics Prepared by Different Processes.** Yi Chen, Dingquan Xiao, Dejun Lan, Qiang Chen, Xi Yue and Jianguo Zhu; Materials Science, Sichuan University, Chengdu, Sichuan, China.

The  $0.36\text{BiScO}_3\text{-}0.64\text{PbTiO}_3$  (BSPT) ferroelectric ceramics were synthesized using the conventional oxide electric ceramics synthesis methods including one-step-sintering method and two-step-sintering method. The structural properties and morphology of the BSPT ceramics were characterized by XRD, SEM analysis techniques. The dielectric and piezoelectric properties of the BSPT ceramics were measured. The results of XRD showed that the BSPT ceramics with perovskite structure could be prepared by using one-step-sintering method and two-step-sintering method, respectively. The highest percent of the perovskite phase of the BSPT ceramics was 92% and 95%, prepared by using one-step-sintering method and two-step-sintering method, respectively. The SEM observation showed that the crystalline grains of the BSPT ceramics prepared by two-step calcination and fired at 1150-1180 degree were well-stacked and the grain boundaries were well clear. It was found that with the increase of sintering temperature, mechanical quality factor ( $Q_m$ ) of the BSPT ceramics increases apparently. And the  $Q_m$  of the sample prepared by two-step calcination and sintering temperature of 1180 degree is 305.1

#### V9.10

**Investigation of the Dielectric, Ferroelectric Properties of  $(1-x)\text{Pb}(\text{Sc}_{0.5}\text{Ta}_{0.5})\text{O}_3\text{-}x\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$  Relaxor Ceramics.** Dejun Lan, Dingquan Xiao, Yi Chen, Qiang Chen, Xi Yue and Jianguo Zhu; Materials Science, Sichuan University, Chengdu, Sichuan, China.

The  $\text{Pb}(\text{Sc}_{1/2}\text{Ta}_{1/2})\text{O}_3$  (PST) relaxor ferroelectric ceramics were investigated greatly in the world for its high pyroelectric, ferroelectric and dielectric properties and comprehensive application on uncooled focal plane arrays infrared detector and high-density dynamic random access devices. However, some other ferroelectrics were added into the PST ceramics can have better properties above. In this paper, complex  $(1-x)\text{PST}-x\text{PZT}$  (PSTZT) relaxor ferroelectric ceramics was prepared respectively by wolframite precursor process (named two-step-sintering method, TSSM) and common oxides mixing method (named one-step-sintering method, OSSM). The experiment results demonstrated that the PSTZT ceramics with pure perovskite structure could be acquired by OSSM and its crystal particle size was smaller than those of the samples prepared by TSSM. The temperature dependence of permittivity and dielectric loss tangent of PSTZT were investigated in detail, which indicated that PSTZT is not a complete diffusive phase transition ferroelectric ceramics, with a little frequency dispersion and partly coordination with Curie-Weiss law when  $T > T_m$ . The pyroelectric property of PSTZT ceramics were also investigated and discussed.

#### V9.11

**The Influence of Sintering Temperatures on the Properties of Lithium Sodium Potassium Niobate Ceramics.** Qiang Chen, Dingquan Xiao, Xiaolu Shi, Long He and Jianguo Zhu; Materials



Science, Sichuan University, Chengdu, Sichuan, China.

Lead-Free piezoelectric ceramics were paid more attentions in the past years because of the needs for environment protection. Lithium sodium potassium niobate ( $\text{Li}_x\text{Na}_{1-x-y}\text{NbO}_3$  (LNKN) ceramics as one of promising candidates of lead-free-based piezoelectric materials have been widely investigated because of their attractive properties such as high Curie temperature and high planar coupling coefficient. In this paper, doped lithium sodium potassium niobate ceramics were prepared by conventional oxide method. The crystalline properties of LNKN ceramics were characterized by X-ray diffraction. The dielectric and piezoelectric properties were also measured. The experiment results show that the doped LNKN ceramics have high Curie temperature (above 450°C) and excellent dielectric property. The piezoelectric constant  $d_{33}$  of LNKN ceramics is about 200 pC/N. The properties of doped LNKN ceramics are affected strongly by the sintering temperature. The dependence of the piezoelectric property of doped LNKN ceramics on composition has also been studied.

#### V9.12

##### Synthesis and Electrical Properties of $\text{Pb}(\text{Sn}_{0.45}\text{Ti}_{0.55})\text{O}_3$

**Ferroelectrics.** Bhakti Das<sup>1</sup>, Ram N. P. Choudhary<sup>2</sup> and P. K. Mahapatra<sup>1</sup>; <sup>1</sup>Physics, Vidysagar University, Midnapur, India; <sup>2</sup>Department of Physics, Indian Institute of Technology, Kharagpure, India.

The polycrystalline samples of  $\text{Pb}(\text{Sn}_{0.45}\text{Ti}_{0.55})\text{O}_3$  is synthesized by a solid-state reaction technique. A tetragonal crystal system with its unit cell parameters  $a = 3.9563 \text{ \AA}$ ,  $c = 4.0636 \text{ \AA}$  was selected from the X-ray diffraction profiles of the sample at room temperature. However, a small extra diffraction peak is observed around  $27^\circ$ , this peak suggests the presence of a very small (less than  $2\%$ ) amount of second (pyrochlore) phase in the compound. An analysis of the real and imaginary parts of the impedance and dielectric constant with frequency at different temperature is performed. The impedance spectroscopy reveals the range of frequencies where the different polarization mechanisms are effective. The convincing evidence of the existence of the grain, grain boundary and electrode effects in  $\text{Pb}(\text{Sn}_{0.45}\text{Ti}_{0.55})\text{O}_3$  have been observed in a complex impedance study. Substantial decrease in impedance with rise in temperature is observed from the impedance loss spectrum for  $\text{Pb}(\text{Sn}_{0.45}\text{Ti}_{0.55})\text{O}_3$  compound. The slope of  $\ln \sigma_{ac} \sim 1/T$  shows distinct variations for temperature below 400 K, 400K to  $T_c$  and for  $T > T_c$  of the compound. The room temperature ac conductivity at 10 kHz lies in the range of  $10^{-5}$  to  $10^{-6} (\text{ohm-cm})^{-1}$ . The ac conductivity at room temperature increases almost linearly with frequency and follows the well known Joncher power law  $\sigma_{ac}(\omega) = A \omega^s$  where frequency exponent  $s$  is a constant which can vary between 0 and 1.

#### V9.13

Abstract Withdrawn

#### V9.14

**Preparation of 3D Colloidal Sphere Array using Barium Titanate Nanoparticles and its Dielectric Property.** Michiyasu Nishiyama, Takuya Hoshina, Hirofumi Kakemoto, Takaaki Tsurumi and Satoshi Wada; Dept. of Metallurgy & Ceramics Science, Tokyo Institute of Technology, Meguro, Tokyo, Japan.

Recently, nm-sized particles have been very attractive materials in various fields. It is known that the nm-sized particles have some special properties derived from their nm size. In this study, we focus on the dielectric properties of barium titanate ( $\text{BaTiO}_3$ ) nanoparticles. Then, we proposed new method for ultrafine  $\text{BaTiO}_3$  particles below 10 nm. In this method, a mixture between barium titanate oxalate aqua solution with low concentration below  $10^{-3}$  mol/l and ethanol was used as the starting material. First, the droplets with a size below  $3 \mu\text{m}$  were atomized with an ultrasonic vibrator, dried at low temperature below 300°C, and nm-sized barium titanate oxalate particles were prepared. Next these particles were thermally decomposed at low temperature below 500°C in air, and intermediate products were prepared. At last, intermediate products were thermally decomposed in a vacuum, as a result, nm-sized  $\text{BaTiO}_3$  particles with diameters of around 10 nm were prepared. These particles were characterized by XRD, SEM, and TEM. However, the dielectric properties of  $\text{BaTiO}_3$  nanoparticles cannot be measured. Therefore, 3D colloidal sphere array of barium titanate nanoparticles were produced, and dielectric property of this colloidal sphere array was measured.

#### V9.15

**Crystal Growth of Large-Scale Silver Lithium Niobate Single Crystals and Their Piezoelectric Properties.** Akiko Saito, Hirofumi Kakemoto, Takaaki Tsurumi and Satoshi Wada; Dept. of Metallurgy & Ceramics Science, Tokyo Institute of Technology, Meguro, Tokyo, Japan.

To date, there has been no lead-free piezoelectrics with the higher piezoelectric performances than that for PZT ceramics. On the basis of environmental problems, the development of new lead-free piezoelectrics with the high piezoelectricity is required. In this study, in silver lithium niobate ( $\text{Ag}_{1-x}\text{Li}_x\text{NbO}_3$ ,  $0 < x < 0.1$ ) single crystals, their crystal growth and piezoelectric property were investigated.  $\text{Ag}_{1-x}\text{Li}_x\text{NbO}_3$  single crystals were grown by a slow cooling method without flux under oxygen flow. The sizes of the grown crystals were strongly dependent of (1) atmosphere and (2) cooling rate. When the crystal was grown under conventional atmosphere, the deposition of a lot of silver metal and the formation of small crystals with sizes below 2 mm were observed. On the other hand, when the crystal was grown under oxygen flow, no silver metal deposition was observed while formation of the middle-size crystals with sizes over 2 mm was observed. Moreover, the relationship between cooling rate and crystal size was investigated in detail. As the result, it was found that the crystal sizes increased with decreasing cooling rate, and finally, the while the  $\text{Ag}_{0.9}\text{Li}_{0.1}\text{NbO}_3$  crystals with sizes over  $20 \times 10 \times 10 \text{ mm}^3$  were successfully grown at the cooling rate of  $2^\circ\text{C/h}$ . The color of the crystals was changed from light green to yellow with increasing Li contents. These crystal structures were assigned to perovskite-type orthorhombic phase. The dielectric properties were measured from 20°C to 600°C. As a result, with increasing Li contents, the phase transition at around 60°C was shifted to lower temperatures below 20°C while the phase transition around 360°C was shifted to higher temperatures. The dielectric loss tangent of the crystals was always below 2% from 20°C to 400°C. P-E hysteresis measurement revealed that pure silver niobate crystals were weak ferroelectric with Pr of  $0.095 \mu\text{C/cm}^2$  while  $\text{Ag}_{0.9}\text{Li}_{0.1}\text{NbO}_3$  crystals were normal ferroelectrics with Pr of  $10.68 \mu\text{C/cm}^2$ . Moreover, these piezoelectric properties were measured, and for  $\text{Ag}_{0.9}\text{Li}_{0.1}\text{NbO}_3$  crystals, high electromechanical coupling factor were observed.

#### V9.16

##### Ionic-polymer-metal composite membrane with

**gradient-metal-electrodes.** Ren-Jei Chung<sup>1</sup>, Li-Chun Chen<sup>1</sup>, Ming-Fa Hsieh<sup>2</sup> and Tsung-Shune Chin<sup>1</sup>; <sup>1</sup>Materials Science and Engineering, National Tsing Hua University, Hsin-Chu, Taiwan; <sup>2</sup>Biomedical Engineering Center, Industrial Technology Research Institute, Chu-Tung, Taiwan.

A typical ionic-polymer-metal composite consists of a thin perfluorinated ionomer membrane and noble metal electrodes plated on both surfaces. It undergoes a large bending motion when an electric field is applied hence exhibits deformation by a certain amount of cation. With proper arrangement and package, a great number of "smart devices" are anticipated. In this study, a solution-cast route was used to prepare the electro-active polymer membrane and Pt electrodes were fabricated onto the membrane through electro-less plating. The optimized processing conditions, membrane properties and electrodes behaviors were investigated. The ionic polymer used in this study is the commercial Nafion™, the perfluoro-sulfonated ionomer membrane, developed by DuPont Co. Nafion™ film was cast by the solution-casting route and then loaded with different weights simultaneously. The effect of weight on the crystallinity of the solution-cast films was investigated through FTIR, Wide Angle X-ray Diffraction and Small Angle X-ray Scattering. DSC was used to study the thermal behavior of prepared films. The mechanical properties were tested through Instron. A new method to fabricate the Pt electrodes onto IPMC by a reverse electro-less plating was also developed in this study. The resistance was measured. The distribution of Pt atoms was ensured through EPMA. The surface morphology of the electrodes was recorded by FESEM and AFM. At last, a vibration test was carried out using a laser-PSD device. The results showed that weights facilitate the crystallinity of the solution-cast films. The  $\text{Imor.}(778\text{cm}^{-1})/\text{Isymm}(1156\text{cm}^{-1})$  in FTIR increased with loadings. The shift in WAXD peaks also supported this fact. The number and size of the crystalline domains of solution-cast film decrease as studied by SAXS. The Young's Modulus of solution-cast film decreases as increasing weight because of the loss of crystallinity (180~140 MPa). A finely dispersed platinum particle deeper and gradient penetrating within the near-boundary region with a smaller average particle size and more uniform distribution could be obtained through a reverse electro-less plating. Its surface roughness is 3nm comparing to 52nm of a traditional process. But its surface resistance is too high (3.5 ohm) to activate the bending. To solve this problem, we coated the second Pt electrodes by a typical electro-less plating, and the resistance decreased to 0.7 ohm. The results depicted that the fabricated IPMC shows longer bending lifetime than typical IPMC. In a 0.09% NaCl solution, the device was able to vibrate for 8 hours under a 5V, 0.1 Hz actuation. In conclusion, we prepared Nafion™ thick membranes through a solution-cast route. The post mechanical treatments decreased the crystallinity as well as the Young's Modulus of the films. A uniform, fine, and deep-going gradient Pt electrodes could be achieved using a combined reverse and traditional electro-less plating. The device was able to long-term vibrate.

### V9.17

#### Porous methylsiloxane gel thick film for millimeter-wave antenna substrate prepared by gap filling method.

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Small and lightweight antennas which operate in millimeter-waves are in demand to realize the next generation high-speed wireless communications. The aim of this study is to apply porous methylsiloxane gel with low dielectric loss (low dielectric constant and low loss tangent ( $\tan\delta$ )), for the antenna substrate. The authors have developed a gap filling method which is a novel process to fabricate a porous methylsiloxane gel film over 100 $\mu\text{m}$  thick on a metal substrate by the sol-gel method accompanied by phase separation. Methyltrimethoxysilane (MTMS) was processed to sol-gel reaction by using methanol and nitric acid as the solvent and the catalyst, respectively. The mixed solution was introduced into a confined gap between two parallel plates, i.e., a metal conductor substrate and a cap plate, followed by inducing spinodal decomposition during sol-gel transition to form a porous methylsiloxane matrix. The thickness of the film was determined by the gap distance between the two plates. Aluminum was used as a metal conductor considering its high electric conductivity and chemical resistance to nitric acid. To improve the adhesion between the porous methylsiloxane gel and the aluminum surface, a SiO<sub>2</sub> thin film was deposited on the aluminum substrate by a chemical vapor deposition (CVD) method. Since the cap plate has to be released after the gel formation, the cap plate was covered with an amorphous carbon thin film to prevent the gel film adhering to it. The above method successfully realized over 100 $\mu\text{m}$  thick porous films on a metal substrate. A thin continuous layer less than 1 $\mu\text{m}$  was formed at the interface between the porous film and the cap plate, which gave the film a smooth surface preferable for patterning metal circuits of the antenna. Mercury penetration measurement revealed the porosity of the porous films exceeded 70%. The observed dielectric constant at 60GHz was 1.7. These results show that the gap filling method is applicable for the preparation of porous thick film that is needed for antenna applications. This research was supported by grants from NEDO of Japan.

### V9.18

#### New Materials for Solvent- and Chemically-Resistant Microfluidic Devices with Tailored Surface Functionalities.

Junhoe Cha<sup>1</sup>, Jason P. Rolland<sup>1</sup>, Claus R. Poulsen<sup>1</sup>, Zhilian Zhou<sup>1</sup>, J. Michael Ramsey<sup>1</sup> and Joseph M. DeSimone<sup>1,2</sup>; <sup>1</sup>Chemistry Department, University of North Carolina at Chapel Hill, Chapel Hill, North Carolina; <sup>2</sup>Chemical Engineering, North Carolina State University, Raleigh, North Carolina.

Microfluidic devices developed in the early 1990s were fabricated from hard materials such as glass and silicon using photolithography and etching techniques. These processes do not lend themselves to low cost high throughput manufacturing and have materials properties that limit their utility. Soft materials make possible cost effective method for large scale fabrication. In addition, soft materials enable actuation of devices containing valves, pumps, and mixers. Crosslinked poly(dimethyl siloxane) (PDMS) has become the material of choice for many microfluidic devices because of its small Young's modulus, low surface energy for easy surface patterning, and is non-toxic which makes it useful for medical implantations. Despite the advantages of PDMS for microfluidic devices, this material suffers from serious drawbacks. PDMS swells significantly upon exposure to organic solvents e.g., hexane, toluene, acetone. In addition, PDMS is very susceptible to degradation by many different organic reagents because of the highly polarized silicon-oxygen bond. As such, PDMS-based microfluidic devices are not compatible with a microchemistry platform which would involve the use of common reactive reagents. We have recently demonstrated the use of photocurable perfluoropolyethers (PFPEs) as alternatives to PDMS materials for soft lithographic applications for use in microfluidic devices. In this conference, we will show that PFPE microfluidic devices have superior solvent- and chemically-resistance compared to PDMS. Moreover we will present preliminary results on electrokinetic transport in PFPE microchannel devices modified with amine or sulfonate containing monomer.

### V9.19

#### Modeling of the photochemical trans-cis isomerization of azobenzene confined between montmorillonite sheets.

Hendrik Heinz<sup>1,2</sup>, Richard A. Vaia<sup>1</sup> and Barry L. Farmer<sup>1</sup>; <sup>1</sup>Materials and Manufacturing Directorate, Polymer Branch, Wright Patterson

AFB, WPAFB, Ohio; <sup>2</sup>Department of Mechanical and Materials Engineering, Wright State University, Dayton, Ohio.

Novel photoresponsive materials with switchable gallery spacing between layered silicate sheets would provide an important mechanism for actuator or sensor applications. Molecular dynamics simulation has been shown to be a useful tool in predicting such basal plane spacings [1] and we apply this technique for the design of azobenzene-based actuators. Using the recently extended polymer consistent force field and a novel technique to model the trans-cis isomerization of azobenzene through semiempirical classical atomistic simulation, we explore different ways to generate a significant, controllable change in basal plane spacing upon irradiation. 4,4'-diammonium-azobenzene ions, which are simultaneously bonded to the upper and lower silicate sheet and stand upright to the silicate surface (reinforced through hydrogen bonds to the surface oxygen), yield a reversible 15% reduction in basal plane spacing ( $\pm 3$  Angstroms) upon trans-cis conversion. In contrast, 4-(6-ammonium-n-hexyl)-azobenzene ions attached to the montmorillonite surface provide only a negligible change in gallery spacing after photoisomerization, as previously demonstrated experimentally [2]. The different behaviour is explained with the presence of flexible alkyl chain spacers that help to accommodate the photoactive azobenzene unit in either configuration, and details of the organic-inorganic interface as well as inclination angles of the benzene rings relative to the surface during the reaction are discussed. Rigid, pillar-like structures between the clay sheets, whose height can be controllably switched through a laser pulse, may be useful in the design of bistable actuators. [1] Heinz, H.; Suter, U. W. *Angew. Chem. Int. Ed.* 2004, 43, 2239-2243. [2] Ogawa, M.; Ishii, T.; Miyamoto, N.; Kuroda, K. *Adv. Mater.* 2001, 13, 1107-1109.

### V9.20

#### Luminescent Conjugated Polyelectrolytes for DNA

Biosensing. Wayne George<sup>1</sup>, Joachim H. G. Steinke<sup>1</sup>, Mark Giles<sup>2</sup> and Iain McCulloch<sup>2</sup>; <sup>1</sup>Department of Chemistry, Biological and Biophysical Section, Imperial College London, London, United Kingdom; <sup>2</sup>Polymer Electronics, Merck New Business Chemicals, Southampton, United Kingdom.

The ability to identify DNA or RNA strands with minimal treatment represents a challenge that has generated significant scientific interest.[1] Rapid techniques for the detection of small amounts of DNA/RNA are important for many applications including identifying genetic mutations and diseases and monitoring gene delivery and as a result, new techniques that can identify target analytes in a highly sensitive and selective manner are eagerly sought-after.[1] Current techniques for biological molecule analysis are selective, but expensive and time-consuming.[1-3] The use of electronic materials, specifically Conjugated Polymers (CPs), is rapidly emerging as an innovative approach for the development of a new class of highly sensitive biosensor.[3] Here we describe the synthesis and analysis of novel water-soluble conjugated polymers based on a poly(phenylene ethynylene) (PPE) backbone, as sensitive probes for DNA detection. The sensing scheme involving such materials assumes that small molecule quenchers ion-pair with the CPs allowing rapid energy transfer.[2-3] The sensitivity of these polymers was quantified by conducting fluorescence quenching experiments. The strength of the ion-pairing observed encouraged us to explore the direct effect of combining a water-soluble CP with single-stranded oligonucleotides. The results of this and further experiments will be disclosed. In other work we sought to monitor the activity of an enzyme, using the fluorescence emission from a CP. Enzymes represent an attractive target for monitoring due to the sheer variety of processes that they impact.[4-5] This assay scheme consisted of a phosphate-based small molecule quencher, a CP and a phosphatase enzyme in a buffered solution. Data regarding the transfer of the sensing scheme to the solid state will also be presented. References 1. L. Chen, D. W. McBranch, H.-L. Wang, R. Helgeson, F. Wudl, and D. G. Whitten, *Proc. Natl. Acad. Sci. U. S. A.*, 1999, 96, 12287. 2. C. Y. Tan, M. R. Pinto, and K. S. Schanze, *Chem. Commun.*, 2002, 446-447. 3. Q.-H. Xu, S. Wang, D. Korystov, A. Mikhailovsky, G. C. Bazan, D. Moses, A. J. Heeger, *Proc. Nat. Acad. Sci. USA.*, 2005, 102, 530-535. 4. M. R. Pinto, K. S. Schanze, *Proc. Nat. Acad. Sci. USA.*, 2004, 101, 7505-7510. 5. F. Riminsland, W. Xia, S. Wittenburg, X. Shi, C. Stankewicz, K. Achyuthan, D. McBranch, D. Whitten, *Proc. Nat. Acad. Sci. USA.*, 2004, 101, 15295-15300.

### V9.21

#### Novel Fluorinated Poly(Arylene Ether Ketone/Sulfone)s Containing Cross-linkable Tetrafluorostyrol Units for Optical Waveguide Applications.

Yinghua Qi<sup>1</sup>, Jianfu Ding<sup>1</sup>, Michael Day<sup>1</sup>, Jia Jiang<sup>2</sup> and Claire L. Callender<sup>2</sup>; <sup>1</sup>Institute for Chemical Process and Environmental Technology, National Research Council Canada, Ottawa, Ontario, Canada; <sup>2</sup>Communications Research Centre Canada, Ottawa, Ontario, Canada.

Organic polymers are emerging as promising alternatives to inorganic

materials for photonic applications due to their good processability, low cost for device integration and structure-property tunability. However, for practical waveguide device applications, challenges still remain in developing polymers that meet stringent material requirements such as low optical loss at telecommunication wavelengths, low birefringence, good chemical and physical stability, and good thin-film forming capability. Novel cross-linkable, highly fluorinated poly(arylene ether ketone/sulfone)s have been prepared by copolycondensation reactions of perfluorinated aromatic ketone/sulfone with 4,4'-(hexafluoroisopropylidene) diphenol / 4,4'-isopropylidene bis(2,6-dibromophenol) and a tetrafluorostyrol-containing bisphenol at low temperatures in the presence of cesium fluoride. These polymers have demonstrated promising waveguide properties such as high glass transition temperatures (> 150 degree), good thermal stabilities (up to 480 degree), low optical losses (0.4 - 0.6 dB/cm at 1550 nm) and small birefringence (0.002) at 1550 nm. In addition they can form tough, transparent and flexible thin-films by both solution casting and spin-coating techniques. Due to the presence of the reactive tetrafluorostyrol units in the polymer structure, these polymers can undergo rapid thermal and photochemical cross-linking reactions. The direct photo-patterning of these polymers can be achieved by the use of a suitable photo acid initiator. The bromo content in these polymers can be precisely controlled by varying the feed ratio of the bisphenols, which allows for excellent tuneability of refractive index within a wide range of 0.07. Consequently, these polymers are potentially useful for both the core and cladding materials in optical waveguiding applications. Prototype waveguide devices such as straight waveguides, splitters and arrayed waveguide gratings have been fabricated using these materials and have been found to exhibit good device properties.

#### **V9.22**

##### **Intelligent Materials for Biomolecular Diagnostics.**

K. RyAnne Noss<sup>1</sup>, Aleksandr L. Simonian<sup>2</sup> and Mark E. Byrne<sup>1</sup>;

<sup>1</sup>Biomimetic & Biohybrid Materials, Biomedical Devices, and Drug Delivery Laboratories, Department of Chemical Engineering, Auburn University, Auburn, Alabama; <sup>2</sup>Biosensor Laboratory, Department of Material Engineering, Auburn University, Auburn, Alabama.

Synthetic intelligent polymeric materials that can recognize biomolecules have a tremendous potential in micro/nano scale applications such as sensors, biomolecular valves, actuators and point of care diagnostics. Non-covalent complexation between template or "guest" biomolecules and functional monomers during polymerization can create networks with selective binding sites. The concept of macromolecular recognition manifests itself from two major synergistic effects, (i) shape specific cavities that match the template biomolecule, which provide stabilization of the chemistry in a crosslinked matrix, and (ii) chemical groups oriented to form multiple complexation points with the template. Such biorecognitive polymeric gels can be grafted onto sensor based platforms such as surface plasmon resonance (SPR), to produce robust sensors and to elucidate network structural properties on binding affinity and sensitivity. This work illustrates the use of acrylate and methacrylate copolymer networks with a biocompatible crosslinking agent poly (ethylene glycol-n-dimethacrylate) (n = 1, 4.5, 13.6) with imprinting template molecules such as testosterone, phenylalanine, progesterone and homocysteine. Parameters such as the crosslinking percentage in the feed, the length of the crosslinking monomer, functional monomer to template ratio, polymerization reaction and kinetics, double bond conversion and the reaction rate versus time were studied from the bulk material to optimize sensor affinity, selectivity, and response time. The material was patterned onto SPR sensors using a sulfur based chemistry and photolithography technique. The thickness of the thin film material could be varied to determine optimum binding kinetics and diffusional mass transfer. The thin film produce a quick template response time on the SPR sensors and the equilibrium binding parameters matched the bulk material data. Results also demonstrated that a decrease in the amount of crosslinking monomer in the feed as well as an increase in the linear size of the crosslinking monomer resulted in a significant decrease in equilibrium association constants for the template studied.

SESSION V10: Sensor Materials and Devices  
Chairs: Eckhard Quandt and V.K. Varadan  
Thursday Morning, December 1, 2005  
Independence W (Sheraton)

#### **8:30 AM \*V10.1**

**Functional passive sensor system using reflective SAW delay line.** Tooru Nomura<sup>1</sup>, Atushi Saitoh<sup>2</sup> and Tomoe Kosaka<sup>2</sup>; <sup>1</sup>Faculty of Engineering, Shibaura Institute of Technology, Tokyo, Japan; <sup>2</sup>Shibaura Institute of Technology, Tokyo, Japan; <sup>3</sup>Shibaura Institute of Technology, Tokyo, Japan.

A new passive and remote SAW sensor system having ID tag function is presented. Surface acoustic wave (SAW) devices have been widely used as a chemical and physical sensor for measuring the environment. SAW devices have been also used as identification tags (ID tags). The majority of the work reported on SAW sensors to date has used Rayleigh mode SAW and are concerning a delay line. The other hand, it has been shown that the sensors utilizing the shear horizontal mode SAW (SH-SAW) can be designed to sense the liquid properties. The SH-SAW has the unique characteristic of complete reflection at a 90 degree edge normal to the direction of the propagation. This characteristic makes it possible to construct a reflective delay line using the edge reflection. The reflective delay line can be used for as passive and remote sensing. In this paper, a passive and remote SAW sensor with a function of identification tags has been designed by using the reflective SH-SAW delay line. The reflective delay line was constructed on the 36 YX LiTaO3 substrate having a 90 degree edge normal to the direction of the SAW propagation. Passive and remote SAW sensor consists of several reflective delay lines having different propagation length. The passive sensor and ID tags were made by the multi-channel delay line. The responses of the sensor and ID tags using the edge reflection of SH-SAW are obtained in time domain. Several experiments were performed to verify the performances of the multi-channel edge reflection sensor. The basic characteristics of the reflective SAW sensor were obtained by measuring the amplitude and phase of reflected wave. The obtained results showed that the reflective SH-SAW sensor was very effective for sensing the physical and chemical properties. Wireless systems are also suggested for the effective operation of the passive SAW sensor. A novel and simple electronic circuit system for accurately measuring the phase characteristics of responses form multi-channel is proposed. In the application, the reflective delay line has been mounted on cantilever to monitor the strain experimentally. The variation of the SH-SAW velocity on the delay line caused by a strain was measured as the passive SAW sensor response. The velocity varied in proportion to the strain applied to the propagation surface. The results showed that the passive sensor was very effective to measure the strain in a wireless mode and it was found that the system is very suitable to health and safety monitoring of building or airplane wing. Finally it will be shown some applications of the wireless sensor with ID tags on the measurement of the conductivity and viscosity of same liquids.

#### **9:00 AM V10.2**

##### **Optical Resonator Based Bio-Chemical Sensors.**

Sudhaprasanna Kumar Padigi<sup>1</sup> and Shalini Prasad<sup>1,2</sup>; <sup>1</sup>Electrical and Computer Engineering, Portland State University, Portland, Oregon; <sup>2</sup>Bio-medical Engineering, Oregon Graduate Institute, Hillsboro, Oregon.

With increasing number of applications for bio-chemical sensing in the area of environmental monitoring and homeland security, there is a need for robust, compact, ultra-sensitive bio-chemical sensors. We demonstrate a unique platform where we have integrated the high Q technology with functional chemistry for real-time detection of multiple bio-chemical agents. We have designed and fabricated micro disk and micro cylinder resonators on the silica-on-silicon material system with diameters on the order of 25 micro meters to 50 micro meters. The top surfaces of the resonators are coated with specific capture layers that serve as receptors for specific bio-chemical agents. The resonators are travelling wave resonators and work on the principle of whispering gallery modes (WGM), which are excited by the evanescent coupling of light from the tapered optical fiber. The coupled light propagates along the inner periphery of the resonator through total internal reflection leading to the large field enhancements. On resonance, the light from the resonator is coupled out into another tapered optical fiber. The interaction of a bio-chemical agent with the functionalized surface of the resonator is on a nano-scale, similar to the enzyme-substrate based interaction, leading to a change at a specific frequency in the transmission spectra of the resonator, which is characteristic of the bio-chemical agent. This results in the real-time detection of multiple bio-chemical agents on-a-chip.

#### **9:15 AM V10.3**

**Diamond FET biosensor to actualize the detection of SNP based its charge detection.** Kwangsup Song<sup>1,2</sup>, Goujung Zhang<sup>1,2</sup>, Kei Furukawa<sup>1,2</sup>, Takahiro Hiraki<sup>1,2</sup>, Takahashi Ohiki<sup>1,2</sup>, Junghoon Yang<sup>1,2</sup>, Iwao Ohdomari<sup>1,2</sup> and Hiroshi Kawarada<sup>1,2</sup>; <sup>1</sup>Science and Engineering, Waseda University, Tokyo, Japan; <sup>2</sup>Institute for Biomedical Engineering, Waseda University, Tokyo, Japan.

The detection of specific DNA relies on the sensing of hybridization between functionalized probe DNA and its complementary target DNA. Although many methods are available to detect the hybridization of DNA, fluorescent labeling-based microarrays are normally used. However, eliminating the labeling steps has been required to produce the 'Gene chip' providing a simple, accurate,

stable and inexpensive platform for patient diagnosis. On the other hand, surface plasmon resonance (SPR), quartz crystal microbalance (QCM), and mechanical cantilever array, which are label-free detection methods achieve the highest sensitivity and decrease the analytical process (without labeling process), these methods have been required highly precise and expensive instrumentation. Here we show diamond solution-gate FET (SGFET) for highly sensitive and reliable FET to detect DNA hybridization. The time response and detection limit is better than existing Si-FET type DNA sensor. The DNA immobilizes directly on semiconducting diamond surface where the charge redistribution due to hybridization can be detected swiftly and effectively. The FET operates within the large potential window of diamond ( $> 3.0$  V) where the oxidation and reduction do not happen. Thus the semiconductor surface does not need to be covered by protective layer for the ion attack from the solution. It is greatly advantageous for the miniaturization and integration of the FET type biosensors and the realization of label-free DNA chip.

#### 9:30 AM V10.4

##### **Detection of Cancer Antigens Using a Self-Referencing Surface Plasmon Resonance Biosensor.** Fengyu Su<sup>1</sup>, Chunye

Xu<sup>1</sup>, Minoru Taya<sup>1</sup>, Kazuko Handa<sup>2</sup> and Senitiroh Hakomori<sup>2</sup>;  
<sup>1</sup>Mechanical Engineering, University of Washington, Seattle, Washington; <sup>2</sup>Division of Biomembrane Research, Pacific Northwest Research Institute, Seattle, Washington.

Surface plasmon resonance (SPR) has attracted intense attention as a biosensor in the past two decades because it can provide label-free, real-time detection with high sensitivity. SPR measures refractive index changes on the sensor chip. The refractive index changes due to the specific bindings cause a shift in the SPR signal, however, non-specific binding or environmental change such as solution or temperature change can also cause a shift in SPR signal. Therefore, it is important to prepare accurate referencing for SPR biosensors. In this work, on-chip referencing has been implemented by using microfluidic devices of poly(dimethylsiloxane) which contain a group of 200  $\mu\text{m}$  channels. The referencing materials flow through the microchannels and are adsorbed onto the gold surface. Through multi-functionalization, sensing materials of anti-CEA antibodies and referencing materials of mouse IgG are immobilized on gold surface in a striped pattern. When an analyte solution of CEA antigen in buffer passes through the patterned surface, CEA antigens will bind to antibodies only. The signal changes on both anti-CEA antibodies and mouse IgG surfaces are monitored by SPR simultaneously, therefore, by eliminating the background refractive index fluctuations on mouse IgG surface from binding events between antibodies and antigens, accurate information of SPR signal shift have been obtained.

#### 9:45 AM V10.5

##### **Enhanced Excitation of Surface Acoustic Waves by Coincident Resonance.** Jonathan Raymond Petrie and Robert Bruce van Dover; Materials Science and Engineering, Cornell University, Ithaca, New York.

The high-frequency magnetostrictive response of magnetic actuators can be enhanced by engineering the magnetic material so that the inherent precession frequency of the magnetic moments is coincident with the external excitation frequency. This magnetoacoustic resonance effect was previously demonstrated in bulk in platelets of garnet, such as Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> (YIG), using a custom-built single turn permeameter. Increased dissipative peaks on the ferromagnetic resonance (FMR) curve were observed when the excitation frequency was adjusted to yield an integral number of acoustic wavelengths through the sample. We have now found that a similar enhancement can be achieved using a parallel array of magnetostrictive lines to excite surface acoustic waves (SAW). The characteristic frequency for this system depends on the pitch of the array. This technique could enable the design of improved magnetically-transduced SAW devices for use as sensors and/or electronic filters.

#### 10:30 AM \*V10.6

##### **Applications of Smart Materials in the Development of High Performance Biosensors.** Z.-Y. Cheng, Auburn University, Auburn, Alabama.

High performance biosensors that are capable of detecting single bacterium or a few viruses are urgently needed in areas such as medical diagnosis, food safety/security and the war on bio-terrorists. Biosensors based on microcantilevers have the sensitivity to satisfy these needs. However, there are some drawbacks in the employment of current microcantilevers. Three new types of high performance sensor platforms have been recently developed in our laboratory using piezoelectric and magnetostrictive thin films. The principle and advantages of these sensors as well as the characterization techniques are discussed. The fabrication of these sensors using microelectronic process is presented. The performance of these sensors in air and liquid was determined and compared with current microcantilevers.

The experimental results demonstrate the high sensitivities and many other advantages of these devices over current microcantilevers. The detection of foodborne pathogen *Salmonella typhimurium* and bio-threaten agent *Bacillus anthracis* using these devices has been conducted. The results are reported in this talk.

#### 11:00 AM V10.7

##### **Spore Detection in Air and Fluid Using Microcantilever Sensors.** Angelica Paola Davila<sup>1,2</sup>, Amit Gupta<sup>1,2</sup>, Tom Walter<sup>3</sup>, Demir Akin<sup>1,2</sup>, Arthur Aronson<sup>3</sup> and Rashid Bashir<sup>1,2,4</sup>; <sup>1</sup>Birck Nanotechnology Center and Bindley Biosciences Center, Purdue University, West Lafayette, Indiana; <sup>2</sup>Electrical and Computer Engineering, Purdue University, West Lafayette, Indiana; <sup>3</sup>Department of Biological Sciences, Purdue University, West Lafayette, Indiana; <sup>4</sup>Weldon School of Biomedical Engineering, Purdue University, West Lafayette, Indiana.

In this work, we will report on the development of a real-time monitoring device that uses micro-cantilevers for the gravimetric detection of biological organisms in air and fluid. The biological agent used was *Bacillus anthracis* spore. The experiment was conducted using a laser Doppler vibrometer (LDV) to measure the thermal noise and determine the resonant frequency of the cantilever and the corresponding decrease in frequency as a result of the added mass. Moreover, the added mass attributed to the spores was quantified and compared in the different media, which were air and deionized water. The silicon cantilevers used in this study were of lengths from 20  $\mu\text{m}$  to 50  $\mu\text{m}$ , 200nm thick and a width of approximately 9  $\mu\text{m}$ . The first part of the experiments consisted of suspending spores onto the cantilevers in fluid, drying the cantilevers, performing measurements in air and extracting the mass of the added spores. The resonant frequency and quality factor were obtained by fitting the thermal spectra to amplitude response of a simple harmonic oscillator. The Sader method was used to obtain the spring constant of the cantilevers. Resonant frequencies ranged from 600 kHz to 150 kHz for the corresponding lengths mentioned above, with quality factors ranging from 35 to 12. The mass of a dry spore in air was measured and ranged between 80 fg to 100 fg. The second part of the experiment utilized antibody and BSA (bovine serum albumin) physically adsorbed onto the cantilevers for the purpose of specifically attaching the spores on the surface during the measurements in deionized water. The resonant frequencies in liquid of the corresponding lengths mentioned above ranged from 125 kHz to 18 kHz with quality factors ranging from 2.5 to 0.8. The decrease of the resonant frequency and quality factor is attributed to the medium in which the measurements are being performed. The extracted mass of a spore in fluid was measured to be an average of 1.8 pg. This study demonstrated the ability to detect biological samples not only in air but also in a liquid environment.

#### 11:15 AM V10.8

##### **Development of a Micro fluidic Nanoscale Protein Sensor Device for Improving Vascular Surgical Outcomes.** Shalini Prasad, ECE, Portland State University, Portland, Oregon.

One hundred thousand patients undergo vascular surgery daily in the United States, 33 million annually, which is 10% of the population. This costs \$ 450 billion annually, averaging \$ 13,000 per treatment, which is 40% of the healthcare budget. One million patients have adverse events after surgery annually, costing an additional \$ 45 billion. Within the next two decades, vascular surgical patients will increase by 25%, costs by 50%, and complications by 100% as our population ages. The present surgical burden may become a surgical crisis. The best chance to allay this crisis is to improve outcomes after vascular surgery. The normal physiologic response of surgery is one of acute and intense inflammation and thrombosis. Preliminary data indicates drugs that can affect perioperative inflammation and thrombosis such as statins improve clinical outcomes as well, but mechanistic data explaining such benefit, namely the protein markers indicating the perioperative effects of statins are lacking. Micro and Nanotechnology has advanced to the point that enhanced protein detection is possible. A microfluidic based nanoscale device is fabricated that identifies the pro-inflammatory proteins. The protein markers of inflammation from perioperative serum samples and post operative serum samples are correlated based on the electronic signatures of the relevant proteins. The effect of the statins on the pro-inflammatory proteins from perioperative patients serum samples are also determined by developing electronic signatures. Based on the three sets of electronic signatures a finding is made regarding the similarity of the pro-inflammatory proteins in the perioperative and surgical cases and the suitability of the statins for improving surgical outcomes will be postulated. The rationale for this study is that if the protein markers of the perioperative state were linked with clinical outcomes, then it may be possible to both identify patients at high risk before surgery, and to make an intervention to improve the patients clinical outcome.

### 11:30 AM V10.9

**Assembly and Packaging of Wireless Sensor Systems by Directed Three-Dimensional Self-Assembly.** Wei Zheng and Heiko O. Jacobs; Department of Electrical and Computer Engineering, University of Minnesota, Minneapolis, Minnesota.

Self-assembly offers a powerful tool to integrate individual micro and nanometer sized devices into functional systems. One of the grand challenges in self-assembly is the realization of heterogeneous systems that require integration of components that cross traditional length scales and material boundaries. Here we describe a directed self-assembly process for the fabrication of heterogeneous microsystems that contain non-identical parts: a silicon carrier element with a simple passive circuit, a sensor chip for sensing and amplification, an III/V light emitting diode for optical communication, and a Pyrex encapsulation unit to package and connect the different components. The self-assembly process uses geometrical shape recognition to identify different components and subsequent bond formation between liquid solder and metal-coated areas to form mechanical and electrical connections. The self-assembly of four-component assemblies is demonstrated by sequentially adding device segments to the assembly solution. Photodetector sensor chips and 200 micrometer sized light-emitting diodes have been assembled onto silicon carrier elements during the first two assembly steps. During the third step we added encapsulation units to the assembly solution. The encapsulation units protect the assembled components and more importantly completes the electrical connections without the need of a wirebonder. In all steps the minimization of the free surface area of the liquid solder drove the assembly into a stable, aligned position. The functionality of the final structure was tested by hand mounting the assembled sensor modules on a printed circuit board. The reported procedure provides a new route to the creation of heterogeneous microsystems that contain sensors, actuators, microfluidic elements, and other electronic and photonic devices.

### 11:45 AM V10.10

**A Smart Medical Diagnostic tool using thin film SMO Sensor technology.** K. K. Iyer and P. I. Gouma; Department of Materials Science and Engineering, State University of New York at Stony Brook, Stony Brook, New York.

This paper reports on the development of a smart sensor array consisting of selective gas sensing elements for use in disease diagnosis based on the fact that gaseous species in exhaled human breath can serve as biomarkers for specific diseases (for example-isoprene is a biomarker for blood cholesterol, NO for bronchial diseases, pentane for breast cancer etc). Utilizing the polymorphic selectivity of semiconducting metal oxides towards gases we have developed a smart gas selective sensor array able to identify and discriminate between isoprene, NOx, alcohols/ketones and amines/NH3. The sensing elements are thin films based on the various polymorphs of molybdenum trioxide, tungsten trioxide, titanium dioxide-and their hybrids. A range of analytical techniques have been used to characterize these sensors (HRTEM, XRD and FTIR) and the results are discussed in the paper. A breath analysis system based on our smart sensor array offers the promise of non-invasive monitoring and differential diagnosis of diseases.

SESSION V11: Rheological Systems  
Chairs: John Ulicny and N.M. Wereley  
Thursday Afternoon, December 1, 2005  
Independence W (Sheraton)

### 1:30 PM \*V11.1

**Transient Rheology and Structure Evolution in ER and MR Suspensions.** Daniel Klingenberg<sup>1</sup>, David Kittipoomwong<sup>1</sup>, Yannis Pappas<sup>1</sup>, John Ulicny<sup>2</sup>, Mark Golden<sup>2</sup>, Chandra Namuduri<sup>3</sup> and Jeffrey Morris<sup>4</sup>; <sup>1</sup>Chemical and Biological Engineering, University of Wisconsin, Madison, Wisconsin; <sup>2</sup>Materials & Processes Lab, General Motors R&D and Planning, Warren, Michigan; <sup>3</sup>Electrical & Controls Integration Lab, General Motors R&D and Planning, Warren, Michigan; <sup>4</sup>Benjamin LeVich Institute and Department of Chemical Engineering, City College of New York, New York, New York.

Electro- and magnetorheological (ER and MR) suspensions are well known for their changes in rheological properties when external electric or magnetic fields, respectively, are applied. The ability to rapidly and reversibly control the apparent suspension viscosity over orders of magnitude has been exploited in the development various torque transfer and semi-active damping devices. Some applications have recently been commercialized. One of the major challenges in the development of certain applications is that the rapid increase in suspension viscosity caused by the application of an external field is often followed by a slow, transient increase of the viscosity. The viscosity can continue to increase for minutes to hours, depending on

the flow conditions. Experiments and particle-level simulations suggest that this transient evolution of the apparent rheological properties is associated with the formation and coarsening of lamellar structures within the fluid. In this presentation, we will review the experimental and simulation evidence for lamellar pattern formation and the transient evolution of the rheological properties of ER and MR suspensions. We will then discuss the mechanisms that drive the field-induced change in the suspension structure and control the slow coarsening of the lamellar structures, as well as the mechanisms by which these changes alter the rheological properties. We will also describe a continuum model for the long-time evolution of the lamellar structures, and compare predictions with experimental observations.

### 2:00 PM \*V11.2

**Magnetorheological Fluids as Herschel-Bulkley Materials and their Application in Shock Absorbers.** Norman M. Wereley, S. R. Hong and Y. T. Choi; Aerospace Engineering, University of Maryland, College Park, Maryland.

A magnetorheological (MR) fluid, modeled as a Bingham-plastic material, is characterized by a field dependent yield stress, and a (nearly constant) postyield plastic viscosity. Based on viscometric measurements, such a Bingham-plastic model is an idealization to physical magnetorheological behavior, albeit a useful one. A better approximation involves modifying both the preyield and postyield constitutive behavior as follows: (1) assume a high viscosity preyield behavior over a low shear rate range below the yield stress, and (2) assume a power law fluid (i.e., variable viscosity) above the yield stress that accounts for the shear thinning behavior exhibited by MR fluids above the yield stress. Such an idealization to the MR fluid's constitutive behavior is called a viscous-power law model, or a Herschel-Bulkley model with preyield viscosity. This study develops analytical quasi-steady analysis for such a constitutive MR fluid behavior applied to a flow mode MR damper. Closed form solutions for the fluid velocity, as well as key performance metrics such as damping capacity and dynamic range (ratio of field on to field off force) are developed. Also, specializations to existing models, such as the Herschel-Bulkley, Biviscous, and Bingham-plastic models, are shown to be easily captured by this model when physical constraints (idealizations) are placed on the rheological behavior of the MR fluid. This class of quasi-steady models can be used to analyze magnetorheological dampers that exploit Poiseuille flow, or pressurized flow through an orifice. A key aspect of this presentation will be to apply these models to magnetorheological dampers and shock mounts developed at the University of Maryland to show the applicability of each type of constitutive model: Bingham-plastic, biviscous, Herschel-Bulkley, or viscous Herschel-Bulkley. This experimental validation will utilize viscometric or rheological data to develop parameters of the above constitutive models. The fluids are then used in the shock absorber device, and the damper models are then also validated. Applications of MR fluids currently under development to aerospace applications are outlined.

### 2:30 PM V11.3

**Photo-fluid and anisotropic photo-fluidity.**

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An increase in stochastic molecular vibrations of a solid due to heating above the melting point leads to a decrease in its long-range order and a loss of structural symmetry. Therefore conventional liquids are isotropic media. We report on a light-induced isothermal phase transition of a polymer film from an isotropic solid to an anisotropic liquid state in which the degree of mechanical anisotropy can be controlled by light. Whereas during irradiation by circular polarized light the film behaves as an isotropic viscoelastic fluid, it displays considerable fluidity only in the direction parallel to the light field vector under linear polarized light. In this case the film behaves visually as a liquid along one direction and as a solid in others (one-direction-liquid-state), demonstrating thus extraordinarily anisotropic mechanical properties. The fluidisation phenomenon is related to photoinduced motion of azobenzene-functionalized molecular units, which can be effectively activated only when their transition dipole moments are oriented close to the direction of the light polarization. Since the molecular motion can be induced also by spatially confined non-propagating optical fields, we are able to fluidise nanoscopic elements of the film surface and to trap and transfer a nano-drop of the polymer using the optical near-field of an illuminated metallic tip. We also show how the light induced motion allows mechanical detection of the light polarization using micro-cantilevers system.

### 2:45 PM V11.4

**Structural Anisotropy of a Novel Modulus-Switching Electrorheological Networks.** Byung Doo Chin<sup>1</sup> and H. Henning

Winter<sup>2</sup>; <sup>1</sup>Optoelectronic Materials Research Center, Korea Institute of Science and Technology, Seoul, South Korea; <sup>2</sup>Chemical Engineering, University of Massachusetts, Amherst, Massachusetts.

We studied the anisotropic network of electrorheological (ER) material in a crosslinked polymer matrix. Electrorheological networks (ERNs) are model systems in which the particle connectivity is locked by the crosslinked polymer network with relatively low storage modulus. Microstructure formation of random and aligned ERN was made by simultaneous field-induced alignment of semiconducting particles with crosslinking polydimethylsiloxane prepolymer. The particle alignment can be controlled by applying an electric field. The alignment can be probed with shear (loading perpendicular to field direction) and with compression (loading in field direction) rheometry. In small-amplitude oscillatory shear and compression, the storage shear modulus was found to be a function of applied electric field, particle volume fraction, and the pre-alignment electric field strength during the crosslinking reaction of matrix, which governs the thickness of particle columns and column-to-column distance. Throughout this study, we fabricated the ERN with controllable modulus-switching effect (700% increase of storage modulus) acting in a shear-mode operation. Moreover, we have presented detailed understanding of rheology and mechanical behavior of ERN probed at linear and nonlinear viscoelasticity. As a novel smart material, ERN can provide controllable anisotropy in an electric field, improved stability and mechanical strength compared to fluid-type ER materials, and faster response time compared to that of conventional charged polymer gels.