SYMPOSIUM LL

Smart Materials

November 30 – December 2, 1999

Chairs

Rolf Gotthardt
IGA-Physics Dept
EPFL
Lausanne, CH 1015 SWITZERLAND
41-21-693-3392

Yukio Ito
Ceramic & Materials Engr
Rutgers Univ
Piscataway, NJ 08855
732-445-5567

Kenji Uchino
Materials Research Lab
Pennsylvania State Univ
University Park, PA 16802
814-863-8035

Marilyn Wun-Fogle
Carderock Div
Naval Surface Warfare Center
Code 684
West Bethesda, MD 20817-5700
301-227-5968

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* Invited paper

SESSION LL1: PIEZOELECTRICS I
Chair: Subra Suresh
Tuesday Morning, November 30, 1999
Room 309 (H)

8:30 AM *LL1.1
TEMPLATED GRAIN GROWTH OF PIEZOELECTRIC CERAMICS. S. Trolier-McKinstry, B. Brahmaroutu, C. Duran, P. Rehrig, E. Sabolsky and G.L. Messing, Materials Research Laboratory, The Pennsylvania State University, University Park, PA.

Most ferroelectric actuators utilize randomly oriented polycrystalline ceramics. This, of necessity, leads to averaging of the single crystal piezoelectric properties. That factor, in combination with the recent work on high strain single crystal materials makes development of new methods for preparing highly textured piezoelectrics interesting. This paper describes the use of templated grain growth (TGG) to prepare ferroelectric single crystals as well as fiber and sheet textured ceramics. TGG of oriented ceramics relies on the texturing which occurs when aligned single-crystal template particles are dispersed in a dense, fine-grained matrix. It does not require a high temperature-high pressure processing step. TGG has been used to prepare highly textured (>90%) $Sr_2Nb_2O_7$ and $Bi_4Ti_3O_{12}$ polycrystalline ceramics for high temperature piezoelectric applications, and $(Sr,Ba)Nb_2O_6$ tungsten bronzes for pyroelectrics and capacitors. Using this approach it has been possible to develop over 75% of the single crystal dielectric and piezoelectric response. In addition, TGG can be used to grow single crystals by contacting a seed crystal to a polycrystalline matrix and propagating the boundary into the ceramic body. This has been used to prepare $BaZr_{1-x}Ti_xO_3$ single crystals for actuators. Orthorhombic compositions oriented along the [001] pseudocubic axis show high field piezoelectric d_{33} values of 350 pC/N, which is significantly larger than is available in other non-lead systems, even though the grown crystals entrap some of the original matrix porosity. Finally, extension of TGG to the lead based relaxor ferroelectric perovskites will be discussed.

9:00 AM LL1.2
PHASE PURITY AND MICROSTRUCTURE OF EPITAXIAL $Pb(Mg_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ THIN FILMS GROWN BY MOCVD. Susanne Stemmer, N.D. Browning, Department of Physics, University of Illinois at Chicago, Chicago, IL; G.R. Bai, S.K. Streiffer, Materials Science Division, Argonne National Laboratory, Argonne, IL.

The relaxor ferroelectric lead magnesium niobate (PMN) and its solid solution with lead titanate (PMN-PT) are smart materials that have attracted much attention because of their large piezoelectric and electrostrictive responses. These properties make thin films of PMN-PT very interesting for applications in microelectromechanical systems. Here, we use transmission electron microscopy techniques to study the microstructure of PMN-PT films grown under different conditions by metalorganic chemical vapor deposition onto $SrTiO_3$ substrates. Selected area diffraction and high-resolution transmission electron microscopy show that the films are epitaxial with a cube-on-cube orientation relationship. The microstructure of lead-poor films consists of sub-grains with a perovskite core and a Mg-rich shell, possibly $Mg_4Nb_2O_9$. The mechanisms for the formation of this microstructure are discussed. Chemically sensitive, atomic resolution Z-contrast images show that the interface between the perovskite phase and the substrate is atomically abrupt, and that the film-substrate lattice mismatch is relaxed by misfit dislocations with a $< 100 >$ Burgers vector. Phase pure PMN-PT films that are obtained under different processing conditions, and are imaged in Z-contrast along the [110] zone axis, are used to identify the presence of local order and disorder, which influences the dielectric properties of the films.

9:15 AM LL1.3
DIELECTRIC AND PIEZOELECTRIC PROPERTY DEPENDENCE ON HIGHLY TEXTURED (100), (111) AND RANDOM THIN FILMS GROWN BY RF SPUTTERING. Sriram Kalpat, I.R. Robin[1], A.Akiba[2], H.Goto[2], S.Trolier McKnistry and Kenji Uchino, Materials Research Laboratory, The Pennslyvania State University, University Park, PA; [1]Institute of Materials Research and Engineering, SINGAPORE, [2]OMRON Corporation, JAPAN.

Abstract: Recently there has been a great interest in PZT thin films because of their potential as piezoelectric actuators capable of being integrated with MEMS devices. Previously our group presented theoretical calculation predicting the enhancement of piezoelectric d33 coefficient and electromechanical coupling factors along the (001) direction in the rhombohedral composition near the morphotrophic phase boundary. Highly oriented PZT thin films have been grown along the (100) and (111) directions on Pt/Si and Pt/MgO substrates by reactive rf sputtering using multielemental metallic targets. The films were grown at a substrate temperature of 300oC followed by rapid thermal annealing (RTA) at 700oC for 30 secs. The dielectric permittivity of the (100) and (111) oriented PZT films were around 1000 and 900 respectively. The PZT(100) films exhibited sharper hysteresis loops with remnant polarization $20\mu C/^2$ and coercive field 30 kV/cm compared to the PZT(111) which had its remnant polarization and coercive field values at $25\mu C/^2$ and 45 kV/cm. Piezoelectric properties of these films deposited on various substrates were also studied in the rhombohedral composition.

9:30 AM LL1.4
ALUMINUM NITRIDE THIN FILMS FOR MICROWAVE FILTER AND MICROSYSTEM APPLICATIONS. Marc-Alexandre Dubois, Paul Muralt, Laboratoire de Ceramique, Swiss Federal Institute of Technology (EPFL), Lausanne, SWITZERLAND.

Aluminum nitride thin films have been grown by reactive magnetron sputter technique using a pulsed power supply. The higly (002)-textured columnar films deposited on platinized silicon substrates exhibited quasi single crystal piezoelectric properties. The effective d_{33} was measured as 3.4 to 3.9 pm/V, the effective e_{31} as 1.0 C/m2. The pyroelectric coefficient turned out to be positive (4.8 $\mu Cm^{-2}K^{-1}$) due to a dominating piezoelectric contribution. Thin film bulk acoustic resonators (TFBAR) with a fundamental resonance at 3.6 GHz have been fabricated to assess resonator properties. The material parameters derived from the thickness resonance were a coupling factor k=0.23 and a sound velocity vs=10'900 m/s. With a Q-factor of 300 the TFBAR's proved to be apt for filter applications. The temperature coefficient of the frequency could be tuned to practically 0 ppm/K. Simulation calculations have been performed in order to predict loading effects of electrodes for even higher frequency ranges.

10:15 AM *LL1.5
HIGH VIBRATION-LEVEL CHARACTERISTICS FOR PIEZOELECTRIC CERAMICS. Sadayuki Takahashi, Yasuhiro Sasaki, NEC Corporation, Kawasaki, JAPAN; Mikio Umeda, Niigata Polytechnic College, Niigata, JAPAN; Kentaro Nakamura, Sadayuki Ueha, Tokyo Institute of Technology, Yokohama, JAPAN.

One of the application fields of piezoelectric actuators is a mechanical power source. Elastic waves with high vibration-levels based on the piezoelectric resonance are utilzed for the mechanical power source. It has been shown that a great amount of heat generation as well as a marked change in electromechanical properties occurs in piezoelectric ceramics when the ceramics are continuously driven under a high vibration-level. A practical upper limit of an available vibration-level has been restricted by these phenomena. It seems, therefore, very useful for designing piezoelectric actuators and important for developing materials capable of withstanding high vibration-level drive to study vibration-level characteristics in piezoelectric ceramics. This paper deals with the vibration-level characteristics of electromechanical properties in various type piezoelectric ceramics such as soft and hard PZT. Measurements were carried out using both burst and continuous voltage wave drive methods.
In the case of burst voltage wave drive, no temperature rise of the ceramics is observed, since the vibration duration time is very short (<100 ns). As a result, pure effect of a vibration stress on the electromechanical properties of the ceramics were measured, separating from a temperature rise effect. An elastic, dielectric and piezoelectric properties are almost independent of the vibration stress below 30 MPa. A mechanical loss factor, however, increases with the vibration stress. In the case of continuous voltage wave drive, all of the electromechanical properties are changed with the vibration-level. These changes are caused mainly by the temperature rise accompanied by a heat generation of the ceramics. It is also found that the temperature rise and property change in the hard PZT ceramics is suppressed comparing with in the soft PZT ceramics.

10:45 AM LL1.6
LOSS MECHANISMS IN PIEZOELECTRICS. Kenji Uchino, Jiehui Zheng, Yun-Han Chen and Xiaohong Du, International Center for Actuators and Transducers, Materials Research Laboratory, The Pennsylvania State University, University Park, PA; Seiji Hirose, Faculty of Engineering, Yamagata University, Yonezawa, JAPAN.

Losses in piezoelectrics are considered in general to have three different mechanisms: dielectric, mechanical and piezoelectric losses. This paper deals with the phenomenology of losses, first, then how to measure these losses experimentally. We will discuss two methods for measuring the electrical, mechanical and piezoelectric coupling losses separately: (1) strain vs. stress and field, and displacement vs. stress and field curves, and (2) a resonance/antiresonance technique. Also, one can measure heat generation at an off-resonance frequency to obtain a total loss. By combining the above methods, we can investigate the loss mechanisms in practical piezoelectric materials and their consequences in devices. It will be shown that heat generation is caused mainly by the dielectric loss, not by the mechanical loss. Furthermore, a drastic decrease in mechanical Q with an increase of vibration level is observed in resonant piezoelectric ceramic devices, a result not observable using conventional impedance analyzers at low power. Based on this loss knowledge, we will discuss the differing situations between piezoelectric resonance and antiresonance states. In particular, since the mechanical quality factor at an antiresonance frequency is larger than that at a resonance frequency, the antiresonance mode seems to be superior for high power applications.

11:00 AM LL1.7
MICROSTRUCTURAL EFFECTS ON PIEZOELECTRIC CRACKING. Chandler C. Fulton and Huajian Gao, Division of Mechanics and Computation, Stanford University, Stanford, CA.

The successful development of smart structures using piezoelectric sensors and actuators depends on a thorough characterization of the mechanical limits of these materials. However, fundamental discrepancies between theoretical predictions and empirical observations of their cracking behavior hinder attempts to provide appropriate design guidelines. The complex microstructure of a piezoelectric ceramic leads to severe nonlinear effects operating at the length scale of the crack tip, motivating a physics-based investigation of the fracture mechanics. Specifically, the switching and saturation that occur at the level of individual grains and polar domains destroy the path-independence of the J-integral needed to calculate a crack driving force. Therefore, a local viewpoint is required to evaluate the conditions which control the bond-breaking associated with crack advance. We introduce a model for domain switching based on discrete electric dipoles superimposed on a homogeneous medium with the macroscopic material properties. Each dipole then represents the deviation of a given domain's polarization vector from the linear constitutive law. Within this framework, we develop a relationship between the apparent loads applied to a cracked sample and the local energetic forces driving the crack. Shrinking the length scale down to the crack tip reveals a "singularity conversion", from the apparent combination of stress and electrical intensity factors to purely mechanical effective opening forces. Using the energy release rate derived from these local stress singularities to predict the dependence of failure load on applied electric field, we are able to reproduce the trends observed in the laboratory.

11:15 AM LL1.8
COMMONALITIES OF THE INFLUENCE OF LOWER VALENT AND HIGHER VALENT A-SITE AND B-SITE MOFIFICATIONS ON LEAD ZIRCONATE TITANATE FERROELECTRICS AND ANTIFERROELECTRICS. Dwight Viehland, Naval Undersea Warfare Center, Newport, RI.

A comparative study of the influence of both mobile and randomly quenched impurities and their associated defect complexes on ferroelectric phase transformations in lead zirconate titanate ceramics has been performed by dielectric spectroscopy and transmission electron microscopy. These investigations have shown a strong dependence of the structure-property relations on the mobility of impurities and/or defect complexes in the temperature range near and below the phase transformation. Impurities-defects that are mobile until temperatures below the transformation are believed to preferentially locate near domain boundaries, resulting in polarization pinning. For these compositions, no evidence of relaxor ferroelectric behavior was observed. However, for the compositions whose impurities-defects were essentially immobile from temperatures above the ferroelectric phase transformation, relaxor behavior and polar nanodomains were found. Studies of the influence of electrical and thermal histories on properties provided additional insights into the influence of impurity-defect mobility. Internal dipolar fields were evidenced by strong asymmetries in the polarization electric field behaviors for materials containing mobile impurities that became quenched on cooling within the ferroelectric phase. A model is proposed to explain domain evolution in systems containing randomly quenched or mobile impurities and defects.

11:30 AM LL1.9
STUDIES ON SWITCHING CURRENT IN RELAXOR BASED (1-X)PZN - X PT SINGLE CRYSTALS. Uma Belegundu, Hideaki Aburatani, X.H. Du, Kenji Uchino, International Center for Actuators and Transducers Materials Research Laboratory, The Pennsylvania State University, PA.

Single crystals of 0.91Pb(Zn1/3 Nb2/3)O3 - 0.09PbTiO3 (0.91PZN - 0.09PT)show very high values of electromechanical coupling factor k33 of 94% and piezoelectric constant d33 of 1500pC/N. In this system, PZN is a relaxor material having a diffuse phase transition around 140 C and has rhombohedral symmetry. Lead Titanate PT is a normal ferroelectric materials with a sharp phase transition at 490C and has tetragonal symmetry. They form a morphotropic phase boundary at 0.91 PZN - 0.09PT. The high values of k33 and d33 are shown by crystals with rhombohedral composition and poled along [001] direction. Switching current measurements have been carried out on single crystals in order to study the polarization reversal processes. Crystals with rhombohedral and tetragonal compositions were grown and the switching current measured along crystallographic directions [001] and [111]. It was found that the tetragonal compositions show an exponential dependence for both the directions. However, for similar applied fields rhombohedral compositions show linear dependence along [001] and power dependence along [111]. These experimental details and the possible polarization reversal mechanisms involved will be discussed in the presentation.

11:45 AM LL1.10
COMPOSITE PIEZOELECTRIC MATERIALS FOR HEALTH MONITORING OF COMPOSITE STRUCTURES. Panagiotis Blanas, US Army Research Laboratory, Weapons and Materials Research Directorate, Aberdeen Proving Ground, MD; Dilip K. Das-Gupta, University of Wales, School of Electronic Engineering and Computer Systems, Bangor, UNITED KINGDOM.

To reduce operation and maintenance costs, future structural systems are likely to employ sensors and actuators to monitor their state of structural health. Polymer composite materials are the leading candidates for incorporating such sensor/actuator networks. Composite structures can become smart with the addition of embedded active materials, such as piezoelectric materials and shape memory alloys, which can provide the sensor and actuator functions needed. However, currently employed active materials do present some disadvantages, such as high density and stiffness and incompatibility to the host polymer matrix materials. Furthermore, current processing methods do not easily facilitate the incorporation of distributed sensors and actuators within the composite structure. For this reason, the need exists for active materials that are more flexible with regards to material compatibility and processing. Research efforts have been undertaken towards the development of polymer based active materials that can provide sensing and actuating functions for fiber reinforced polymer composite structures. Piezoelectric composite materials consisting of ferroelectric ceramics within a polymer matrix can be fabricated to take advantage of the favorable properties of their constituents while limiting the detrimental ones. The mechanical, electroactive and acoustic properties of such materials can be tailored to enhance their performance for specific applications. In this study, ferroelectric ceramic/polymer composites have been developed and employed as embedded sensors in composite structures. The ability of these composite piezoelectric materials to detect and differentiate between transient elastic waves, generated within composite plate structures, has been investigated. In addition, the response of the composite sensors to low energy impact and cyclic dynamic loading has been determined. The present paper reports on results obtained from these studies.

SESSION LL2: PIEZOELECTRICS II
Chair: Susan Trolier-McKinstry
Tuesday Afternoon, November 30, 1999
Room 309 (H)

1:30 PM *LL2.1
ELECTROSTRICTIVE POLY(VINYLIDENE-FLUORIDE-TRIFLUOROETHYLENE) COPOLYMER. Q.M. Zhang, Materials Research Laboratory and Electrical Engineering Department, The Pennsylvania State University, University Park, PA.

The paper reports the massive electrostrictive response, both in longitudinal and transverse directions, in the high energy electron irradiated P(VDF-TrFE) copolymer discovered recently. It will be shown that the electrostrictive strains of the copolymer can be tuned over a wide range by different processing and treatment conditions. Because of the high elastic modulus of the irradiated copolymer, the material also possesses a high elastic energy density and high mechanical load capability as demonstrated by the results from experiments under different load conditions. To understand the underlying mechanism for the observed phenomena, a series of studies were conducted including the dielectric, polarization, X-ray, FT-IR, DSC, and SEM. The results reveal that the high energy electron irradiation has converted the macro-polar domains in the normal ferroelectric P(VDF-TrFE) copolymer into nano-polar regions embedded in a non-polar matrix. The transformation from non-polar to polar regions under electric fields produces the massive electrostrictive strain. The existence of the local polar regions is also responsible for the observed dielectric dispersion and ferroelectric relaxor like behavior in the material.

2:00 PM LL2.2
Abstract Withdrawn.

2:15 PM LL2.3
SCALE UP TO LARGE DIAMETER, HIGH AUTHORITY LEAD MAGNESIUM NIOBATE COFIRED ACTUATORS. Roger B. Bagwell, Paul A. Davis, and Mark A. Ealey, Xinetics Inc, Devens, MA.

Cofired ceramic actuators based on lead magnesium niobate (PMN) have been successfully used in a variety of adaptive optics applications based on deformable mirror technology. Typically an array of actuators deforms a thin facesheet to provide wavefront correction for a primary optic. Some applications require increased facesheet thickness and correspondingly larger forces to deform them, dictating an increase in the diameter and therefore force, or authority, needed from the actuator. The focus of this investigation is the scale up to actuators in excess of 15 mm in diameter and some of the critical process parameters that must be understood when producing larger cross sections of PMN. Particularly important is the increased tendency to form the parasitic pyrochlore phase in the core of the actuator during thermal cycles. The interaction of the decomposition products of the binder system with the PMN has been linked to pyrochlore formation on the inside of the piece. The pyrochlore formation degrades both the displacement and strength characteristics of the actuator and can impact reliability. Process changes and the impact of binder chemistry on the production of large diameter actuators will be discussed.

2:30 PM LL2.4
GROWTH AND CHARACTERIZATION OF BARIUM TITANATE BASED MATERIALS FOR HIGH STRAIN APPLICATIONS. Ytshak Avrahami, Harry L. Tuller, MIT, Dept. of Materials Science and Engineering, Cambridge, MA.

Ferroelectric single crystals combine high frequency response and high strain actuation. Current materials for high strain actuation are largely based on lead containing relaxor perovskites. Materials such as $Pb(Mg_{1/3}Nb_{2/3})O_3$ and $Pb(Zn_{1/3}Nb_{2/3})O_3$ in solid solution with $PbTiO_3$ show high strain levels but are not easily fabricated due to the high vapour pressure of lead at elevated temperatures. Investigation of Barium Titanate based perovskite systems (BT) as candidates for alternative, lead-free materials, will be presented. These systems show promise for high strain electromechanical actuation and ease of single crystal growth. Results from electromechanical testing of both polycrystalline and single crystal BT will be shown. Piezoelectric coefficient and strain levels of polycrystalline BT were found to be comparable to those of PZT. Measurements of single crystal BT are presently in progress. The effects of single crystal growth parameters and composition on structure and properties will be discussed.

3:15 PM LL2.5
INDENTATION OF PIEZOELECTRIC MATERIALS: PART I. THEORY. Subra Suresh, Antonios E. Giannakopoulos, S. Sridhar, U. Ramamurty, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA.

A new method is proposed for the characterization of properties of piezoelectric materials using depth-sensing indentation involving both mechanical and electrical measurements. First, a rigorous general theory is presented for axisymmetric indentation of piezoelectric solids with anisotropic properties. Explicit expressions connecting the indentation load with the depth of penetration or contact radius, as well as the indentation-induced surface charge. Various indentor geometries of practical interest, i.e. sphere, cone and flat circular punch, are analyzed. The theoretical results facilitate the analysis of coupled electrical/mechanical response for different electrical boundary conditions involving conducting as well as insulated indentors. All aspects of the theory have also been confirmed with finite element computations. The theory is compared with experiments in Part II.

3:30 PM LL2.6
INDENTATION OF PIEZOELECTRIC MATERIALS: PART II. EXPERIMENTS AND APPLICATIONS. Subra Suresh, Antonios E. Giannakopoulos, S. Sridhar, U. Ramamurty, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA.

The response of polycrystalline lead zirconate titanate and barium titanate to spherical and conical microindentation was investigated. Force versus depth of penetration curves obtained from instrumented indentation reveal that the indentation stiffness depends on material poling and the type of electrical boundary conditions employed (e.g., conducting indentor or insulated indentor). The current induced into the indentor due to the polarized layer on the contact surface of the piezoelectric specimen was found to increase with time as the contact area increased. Used in conjunction with instrumented indentation experiments at the nanoscopic, microscopic or macroscopic size scales, the theory leads to the prediction of some of the elastic, dielectric and piezoelectric constants as well as the activation energy for depolarization. The theoretically predicted force-displacement and surface charge versus time relations for piezoelectric solids have also been checked with finite element analyses and experiments. The implications of the proposed method for the design and characterization of piezoelectric materials and for quality control in commercial production are also addressed. The application of the present methods to piezoelectric composites are also examined.

3:45 PM LL2.7
BENDING STRENGTHS OF COMPOSITE-PIZOELECTRIC-COMPOSITE SANDWICH LAMINATES UNDER COMBINED MECHANICAL AND ELECTRICAL LOADING. Jinquan Cheng, Tong-Yi Zhang, Ricky S.W. Lee, Ping Tong, Hong Kong University of Science and Technology, Department of Mechanical Engineering, Clear Water Bay, Kowloon, Hong Kong, CHINA.

Lee et al. have successfully employed composite-piezoelectric (PZT)-composite sandwich laminates to fabricate motors. The present work studies the bending strength of composite-piezoelectric-composite sandwich laminates under combined mechanical and electrical loading. Three-point bending tests are carried out under an applied electric field and with in-situ acoustic emission measurements. An acoustic scanning microscope is used to examine damage features at different loading stages. At each level of the applied electric field strength, 30 samples are tested to ensure the experimental data reliable. The experimental results show that the load-deflection curve deviates slightly from a straight line when the middle PZT plate starts to fragment. However, the load continues to increase with deflection during the course of fragment of the PZT plate, until delamination between the PZT plate and the bottom composite laminate occurs. The load drops quickly as the interfacial crack propagates. Applying a positive electric field increases the bending strength. A high negative electric field reduces the bending strength, while a low negative electric field enhances little the bending strength. Supported by the Hong Kong Research Grants Council.

4:00 PM LL2.8
PIEZOELECTRIC MATERIALS FOR HIGH TEMPERATURE NANOBALANCES. Holger Fritze, Günter Borchardt, Technische Universit"at Clausthal, Department of Physics, Metallurgy and Materials Science, Clausthal-Zellerfeld, GERMANY; Harry L. Tuller, Massachusetts Institute of Technology, Department of Material Science & Engineering, Cambridge, MA; Tsuguo Fukuda, Institute for Materials Research, Tohoku University, Sendai, JAPAN.

Piezoelectric materials such as langasite ($La_3Ga_5SiO_{14}$) and related compounds were investigated with respect to their potential application in high temperature nanobalances. Langasite does not exhibit phase transformations up to the melting point of 1470°C and is thus a promising candidate material for high temperature resonator devices. The properties necessary for stable operation of langasite as a high temperature bulk acoustic wave resonator are high resistivity and low oxygen diffusivity. The electrical properties of langasite single crystals were investigated at temperatures of up to 900°C as a function of oxygen partial pressure. The oxygen diffusivity was determined by ^{18}18O tracer diffusion and subsequent SIMS measurements. Based on these data, a model for electronic and ionic transport in langasite was developed. At elevated temperatures, the bulk resistivity of the resonator devices cannot be neglected due to attenuation of the resonance signal. In contrast to current resonator materials, we have observed bulk oscillations at temperatures of up to 750°C in langasite devices. Optimization of the electrical properties may lead to oscillations at even higher temperatures. The temperature dependence of the electrical properties of langasite resonator devices, such as bulk capacity and resonance frequency, were measured and are presented. At 700°C the mass load response for relatively thick resonators is approximately 10 Hz/μg.

4:15 PM LL2.9
CRYOGENIC TESTING OF ELECTROSTRICTIVE ACTUATORS FOR THE NEXT GENERATION SPACE TELESCOPE (NGST). M.L. Mulvihill, M.A. Ealey Xinetics Inc., Devens, MA.

The transition to space-based adaptive optics for wavefront correction requires an evolution to electroactive materials that function in cryogenic environments. Single crystal and polycrystalline (ceramic) materials that exhibit electrostrictive properties at cryogenic temperatures (between 30 and 60 K) are the emphasis of the present research and development project funded by NASA at Xinetics. The primary application of these materials with tailored electrical and thermal properties is the actuator material to be used in the deformable mirrors (DMs) of the Next Generation Space Telescope (NGST). These materials must exhibit sufficient electrostrictive response at the device temperature to deform the mirror surface with negligible electrical hysteresis and aging. The goal is also to tailor the materials properties to be stable over a temperature range rather than at an individual temperature. The candidate materials selection procedure and cryogenic testing results will be discussed.

4:30 PM LL2.10
DESIGN AND CONSTRUCTION OF A ROTARY AND A LINEAR ULTRASONIC MOTORS WITH FREE STATORS. Philippe Bouchilloux, Kevin Craig Active Materials and Smart Structures Laboratory, Department of Mechanical Engineering, Aeronautical Engineering and Mechanics, Rensselaer Polytechnic Institute, Troy, NY; Burhanettin Koc, Kenji Uchino, International Center for Actuators and Transducers, Materials Research Laboratory, The Pennsylvania State University, University Park, PA.

This paper presents two original piezoelectric motors for application in space and robotics. One motor is for rotary motion and the other for linear actuation. These motors use two friction surfaces to transmit motion, which eliminates the need for elastic mounts to hold the stator. This design is expected to reduce mechanical losses and dampening of the transmitted mechanical vibrations that are usually observed with such mounts. The proposed motors offer the usual characteristics found in piezoelectric motors: large torque or force to weight ratio, high holding torque or force, high positioning resolution, short response time, low input voltage, operation independent of the magnetic environment, and compact and gearless design. This paper describes the concept underlying the design of the motors: the rotary version consists of a ring-shaped vibrator excited in a non-axisymmetrical mode; the linear version of the motor is based on a longitudinal vibrator using a L1B4 excitation. Both motors are bi-directional. The paper also gives experimental results obtained with the motors in terms of speed and torque. These results are used to discuss the strong and weak points of the proposed design. These motors are being developed for applications in space and robotics. In this paper, we explain how they can be implemented in adaptive truss members and used to deploy geometry variable structures in space.

4:45 PM LL2.11
CONSIDERATIONS IN THE DEVELOPMENT OF A PIEZO-ELECTRIC TRANSDUCER COCHLEAR IMPLANT.
Niloy Mukherjee, Rodney Dean Roseman, Department of Materials Science and Engineering, University of Cincinnati, Cincinnati, OH.

The mammalian inner ear cochlea is an organ that performs the vital function of transduction of incoming sound energy into electrical signals in the nerve fibers terminating within it. Sensorineural hearing loss stems from the inability of the cochlea to perform this transduction task, due to the absence/destruction of cellular structures within the cochlea. Present cochlear implants invoke the principle of direct electrical stimulation of surviving nerve fibers in the cochlea to partially alleviate the profound deafness caused by sensorineural hearing loss. This research was aimed at studying the scope of, and ultimately developing, a piezoelectric cochlear implant, which would transduce the sound energy into electrical charge/ voltage, which would then stimulate the neural structures. Such a device offers the possibility of greatly simplifying the process of electrical stimulation of the cochlea. Aspects of feasibility, the issues involved in device design, the materials science challenges that need to be overcome, and results from in vitro tests of developed transducers will be presented.

SESSION LL3: SHAPE MEMORY

Chairs: Rolf Gotthardt and Manfred Wuttig
Wednesday Morning, December 1, 1999
Room 309 (H)

8:30 AM *LL3.1
SHAPE MEMORY ALLOYS - THEIR CHALLENGE TO CONTRIBUTE TO SMART STRUCTURES. Christian Boller, DaimlerChrysler Aerospace, Military Aircraft, Muenchen, GERMANY.

The transformation process between martensite and austenite is known for decades. So far the commercial use of the resulting shape memory alloys (SMA) has been limited to switching between these two phases only. Practical applications have resulted in clamps, switches, springs or any type of actuators allowing robots to work. Smart structures consider more than just this 'on-off' stage. Briefly spoken it includes sensing, actuation, and control which allows the structure to smoothly adapt to various conditions. It is the control aspect which requires a much more in depth knowledge. One of the key issues in that regard is related to the description of SMAs' constitutive behaviour. Structural engineers are generally used to consider materials in a two-dimensional linear form with stress and strain being the two dimensions. With SMAs as a structural material, temperature has to be included as a third dimension. Furthermore non-linearity in SMAs' constitutive behaviour is unavoidable. This all-together increases complexity of these materials by more than 50%. Handling of this complexity can be made attractive if the behaviour of SMAs can be well understood and workload downloaded to a computer through numerical modelling as a consequence. Various models for describing SMA constitutive behaviour have been proposed over the past years. However the next step comes when SMAs have to interact as actuators within a whole structural system. An initial understanding is given with simple beams being actuated by SMA elements. The ultimate goal is however SMAs becoming an integral part of the structural material (e.g. SMA reinforced composites). This paper intends to give an overview on state of the art and ways allowing to determine SMAs' potential of implementation into smart structures for commercial application. It will start with applications used today, describe the industrial needs and outline options for possible future applications.

9:00 AM LL3.2
CHARACTERIZATION OF NiTi MATERIALS USING A NOVEL ACES METHODOLOGY. Shivananda P. Mizar, M.I. Pech-Canul, Ryszard J. Pryputniewicz, WPI/ME-CHSLT, Worcester Polytechnic Institute, Mechanical Engineering Department, Center for Holographic Studies and Laser MicromechaTronics, Worcester, MA.

Shape Memory Alloys (SMAs) exhibit complex thermomechanical behavior and have characteristics unique in comparison to commonly used engineering materials. One of these characteristics is related to Shape Memory Effect (SME) which is attributed to a phase transformation resulting in a variation in the modulus of elasticity as a function of temperature. Effective application of SMAs depends on an accurate knowledge of this variation and hence the modulus of elasticity should be well characterized as a function of temperature. Equi-atomic NiTi is the particular SMA studied in this paper. The study concentrates on detailed characterization of the modulus of elasticity of the NiTi as a function of temperature. More specifically, this characterization is made using Analytical, Computational, and Experimental Solutions (ACES) methodology. This novel ACES methodology facilitates solution of problems that would not be possible, or, at the best, difficult to obtain, using either analytical, or computational, or experimental solutions, alone. In this paper, analytical investigations are primarily based on the Euler-Bernoulli theory. Finite Element Method (FEM) is used for computational investigations. Time-average Opto-Electronic Holography (OEH) is used for experimental investigations. The experimental results obtained from OEH are complemented by such other experimental methods as X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy Disperssive X-ray spectroscopy (EDX). The time-average OEH results indicate a change in the modulus of elasticity of the NiTi samples, used in this study, from 38 GPa to 72 GPa as the temperature changed from -15°C to 190°C. The XRD/EDX revealed material composition of the samples as 57% Ni and 43% Ti. The change in the modulus of elasticity, as a result of the change in temperature to which the samples were subjected, corresponds to the phase transformation from martensite to austenite. Comparison of analytical, computational, and experimental results show good correlation.

9:15 AM LL3.3
NEUTRON DIFFRACTION STUDY OF THE EVOLUTION OF PHASE FRACTION, TEXTURE AND STRAIN DUE TO STRESS-CYCLING IN SUPERELASTIC NiTi. Raj Vaidyanathan, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA; Mark A.M. Bourke, Los Alamos National Laboratory, LANSCE/MST, Los Alamos, NM; David C. Dunand, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA.

Superelastic NiTi exhibits upon mechanical loading strains as large as 8% through the formation of stress-induced martensite. During unloading, the martensite becomes unstable and reverts to austenite, with a concomitant recovery of all the accumulated macroscopic strain. In engineering applications, superelastic alloys are subjected to repetitive loading and unloading accompanied with forward and reverse martensitic transformations of the austenitic phase. In the present study, we carry out simultaneous stress-cycling and neutron diffraction to monitor changes with cycling of the stress-induced transformation in polycrystalline NiTi. Rietveld refinement of the spectra allow for quantitative evaluation of the phase fraction, strain and texture. From these in-situ neutron diffraction measurements, the following trends are noted with increasing cycle numbers: (i) the volume fraction of martensite remains almost unchanged at a given stress; (ii) the average phase strain in austenite remains mostly unchanged at intermediate loads but changes for unloaded austenite; (iii) the isotropic and anisotropic components of the strain in austenite redistribute themselves; (iv) the texture in martensite and austenite under load evolve significantly, leading to changes in macroscopic stress-strain behavior. This texture evolution suggests that different initial textures may reduce the number of training cycles (mechanical cycles that stabilize the transformation) and increase the total engineering fatigue life of such materials. This study connects for the first time the microstructural evolution of internal strains, volume fraction and texture with the macroscopic evolution of mechanical properties upon mechanical cycling.

9:30 AM LL3.4
STRESS-INDUCED MARTENSITE IN NiTi CORRUGATED FILMS. Kiyotaka Mori, Yun Zheng, J.S. Slutsker and Manfred Wuttig, Department of Materials Science and Engineering University of Maryland, College Park, MD.

NiTi Films deposited on corrugated Si surfaces develop non-uniform stresses when cooled from the deposition temperature. A disclination model shows that the stresses have rhombohedral and tetragonal components which can preferentially stress induce martensite variants. The deflection of NiTi/Si bimorphs caused by the formation of martensite should thus be different if the NiTi/Si interface is corrugated or not. Experimental results confirm this expectation and show that the corrugation enhances the bimorph's (MEMS) actuation performance by as much as 100%.

10:15 AM *LL3.5
RAPID-SOLIDIFIED METALLIC ACTUATOR MATERIALS DEVELOPED BY ELECTROMAGNETIC NOZZLELESS MELT-SPINNING METHOD. Yasubumi Furuya, Hirosaki Univ, Dept of Intelligent Machines and Systems Engineering, Faculty of Science and Technology, Hirosaki, JAPAN.

Recently, the demand for development of higher performance solid-state actuator materials having large strain, rapid response time and long durability has been increasing for fabricating the advanced mechatronics systems such as micromachine and intelligent/smart structures. Most metallic actuator materials (i.e. magnetostrictive, shape memory alloy etc.) experience the crystallographic phase transformation accompanied with energy conversion between two physical quantities such as magnetic, thermal and mechanical energy. In the process of phase transformation, the enhancement of following two factors generally become important, i.e.(1) crystal anisotropy (grain texture) and , (2) mobility and growth of the so-called domain having almost same physical property in the microscopically local region where domain walls have to move across the interfaces with potential energy gap such as grain boundaries, precipitates etc. in the material. Therefore, artificial interface design of actuator material becomes essentially important to develop high performance solid-state actuator materials. As one of the material processing method for controlling metallurgical microstructure as well as material interface design, the originally developed electromagnetic nozzleless melt-spinning technique was used in this study. This method is characterized by combining the control of the flow down of the metal melts after electromagnetic float-melting (i.e., levitation) with rapid solidification by high-speed rotating roll. First, my works on iPjdevelopment of high performance shape memory alloys is reviewed for the conventional SMAs, TiNi and NiAl alloys having stronger crystal anisotropy, larger shape recovery deformation and higher ductilities. Second,(2) the work on developing a new equiatomic, super high temperature shape memory Ru-Ta alloy system having its inverse transformation temperature over 1000, and lastly, (3) the very recent work on ferromagnetic shape memory alloy, Fe-Pd alloy system fabricated by this method are introduced. The mechanisms of these new types of SMAs are discussed from the view point of metallurgical microstructures, especially, crystal anisotropy and grain boundary interface morphology in the rapid-solidified SMAs.

10:45 AM LL3.6
SHAPE MEMORY THIN FILM COMPOSITES. Eckhard Quandt, Bernhard Winzek, Stiftung Caesar, Bonn, GERMANY.

The hysteresis of thin film shape memory actuators affects the frequency of actuators switching between martensite and austenite. Therefore, the hysteresis properties of thin films of TiNi, Ti(Ni,Cu) and Ti(Ni,Pd) were investigated, which were deposited onto metallic substrates by DC-magnetron sputtering. The substrates have different expansion coefficients in order to establish biaxial tensile and compressive film stresses, respectively. The results show, that the hysteresis width is significantly affected by the stress state of the shape memory film and, in principal, depends on the temperature range of measurement whereas the difference Af-Mf remains unchanged and is only influenced by the composition of the shape memory films. In addition, bimorphs consisting on shape memory films, passive metallic films, and sacrificial layers were completely deposited by magnetron sputtering in compatibility to batch-process technology for MEMS and microelectronics. Applications of these shape memory thin film composites for use in microactuators (e.g. microfluidic and thermal protection devices) will be addressed.

11:00 AM LL3.7
INFLUENCE OF DEFORMATION SPEED ON FATIGUE AND TENSILE PROPERTIES OF A Ti-Ni-Cu SHAPE MEMORY ALLOY. Yoichi Kishi, Zenjiro Yajima, Kenichi Shimzu, Kanazawa Institute of Technology, Advanced Materials Science Research and Development Center, Ishikawa, JAPAN; Makoto Asai, The Furukawa Electric Co., LTD., Materials Research Center, Yokohama, JAPAN.

The mechanical fatigue life and tensile property of a Ti-41at.%Ni-8.5at.%Cu alloy were investigated at 370±1 K as a function of deformation speed. After some thermo-mechanical treatments, the alloy was solution-treated and its martensitic transformation start temperature (B2 $\rightarrow$ B19), M_s, was determined by a differential scanning calorimetry. M_s of the solution-treated alloy was 338 K, while that of the as-rolled alloy could not be determined. Fatigue tests were carried out by using plate-shaped specimens with 3.5 mm width and 1.5 mm thickness, a sinusoidal waveform stress of 20 Hz and 0.5 Hz frequency being applied. The fatigue life obtained at 20 Hz was superior to that at 0.5 Hz, the life of the former being about 2 times longer than the latter at the same stress value. Tensile tests were performed with three tensile speeds of 8.3×10^{-6} m/sec, 8.3×10^{-5} m/sec and 8.3×10^{-3} m/sec. In all the stress - strain curves obtained, an apparent yielding point was observed after the liner elastic deformation. The apparent yielding point is due to the martensitic transformation. The critical stress for inducing martensite and tensile fracture stress increased with increasing the tensile speed. Therefor, it is clear that fatigue and tensile properties of Ti-Ni-Cu SMAs are strongly affected by deformation speed.

11:15 AM LL3.8
HIGH Hf CONTENT NITIHF SHAPE MEMORY FILMS. Chen Zhang, Ralph H. Zee, Auburn University, Materials Research and Education Center, Auburn, AL; Paul E. Thoma, Johnson Controls, Inc., Corporate Technology, Milwaukee, WI.

Polycrystalline NiTiHf films with 10at% Hf have been successfully deposited from a single NiTiHf target using a DC magnetron sputtering system. Free standing films were obtained by depositing the films on single crystal silicon substrates. Thickness of the films was controlled at around 10 μm. In this investigation, the effects of deposition temperature on the microstructure and transformation temperatures of the films were studied. Substrate temperature during deposition was varied between 300°C to 600°C at 100°C intervals. The influence of post heat treatment temperature on the properties of the films was also investigated. The post heat treatment temperature examined was between 300°C to 800°C at 100°C intervals. Transformation temperatures of these films were determined by differential scanning calorimetry (DSC). The surface microstructure of the films was studied using a scanning electron microscope (SEM), and the crystallinity was determined using x-ray diffractometry. It was found that both martensite and austenite transformation termperatures increase with increasing substrate temperature and increasing heat treatment temperature. All the as-deposited films were found to be crystalline even when the substrate temperature was as low as 300°C. The microstructure of the film deposited on a hot substrate showed a fine grain size. The grain size increased with increasing substrate temperature during deposition. The effect of post deposition heat treatment on grain size was minimal.

11:30 AM LL3.9
SUPERELASTIC SHAPE MEMORY ALLOY FOR SEAL APPLICATIONS PRODUCED BY LOW PRESSURE THERMAL SPRAY TECHNOLOGY. Albert Sickinger, ProMet Technologies, Inc., Laguna Hills, CA; J.P. Teter, Naval Surface Warfare Center, West Bethesda, MD.

Recent developments in low pressure thermal spray technology (LPTS) show that shape memory alloy (SMA) materials can be successfully created with the thermal spraying process. Thin, near net shape foils (100 μm thick) were produced using the LPTS method. Using this process no significant change of their transition temperatures (max. 5°C) in the sprayed and heat treated condition could be found. Tensile strength and elongation of 2,100 MPa (300 ksi) and 3% respectively, were measured. Thin (0.02cm) and thick (0.076cm) wall tubes have also been produced in the 1.25 cm diameter range. These samples were heat treated and subjected to Hot Isostatic Press (HIP). Metallographic analysis, SEM and shape memory parameter determinations will be presented. The objective of this paper is to demonstrate the feasibility of producing superelastic NiTi shape-memory alloy (SMA) foil material by the low pressure thermal spraying (LPTS) technique for the development of robust, corrosion resistant and long life sealing technology. This development may solve a long-standing Naval problem of reliably preventing green water and other fluids or air born contaminants from entering a vessel. Conventional elastomer seals are prone to leakage due to wear and environmental degradation and unrecoverable strain, which reduces sealing force (compression set). The superelastic shape memory seal concept provides large recoverable strains, which maintains high sealing force (no compression set) and no environmental degradation and wear.

11:45 AM LL3.10
ADAPTIVE COMPOSITES WITH EMBEDDED SHAPE MEMORY ALLOY WIRES. J.A. Balta, V. Michaud, J-A.E. Manson, Laboratoire de Technologie des Composites et Polymeres, Ecole Polytechnique Federale de Lausanne, Lausanne, SWITZERLAND; M. Parlinska, R. Gotthardt, Institut de Genie Atomique, Ecole Polytechnique Federale de Lausanne, Lausanne, SWITZERLAND.

Pre-strained martensitic Shape Memory Alloy (SMA) wires embedded into a composite material act against the stiffness of the host material if they are heated above their transformation temperature, biasing their strain recovery. As a result, recovery stresses are generated in the composite, leading to a shift in resonance vibration frequency if the wires are placed along the neutral axis of a composite beam. Guidelines for quantification of the effects produced, as a function of SMA wire composition, volume fraction, level of pre-strain, and of host material stiffness are not available yet. In order to investigate the governing mechanisms of activation, adaptive composite materials based on Kevlar fiber reinforced epoxy matrices have been produced by embedding thin Shape Memory Alloy wires, 150 microns in diameter, during processing in an autoclave. A mold was specially designed to pre-strain the SMA wires and prevent their recovery during the cure cycle, allowing various geometrical arrangement of the wires and levels of pre-strain. Additionally, unit cell composites consisting of a single wires embedded in an epoxy resin disk were produced in order to test the shear strength of the SMA-resin interface as a function of the type of wire, the level of pre-strain and the number of activation cycles. Experimental results concerning the interfacial properties will be presented and discussed in light of micro-mechanical models under development. Values of the degree of activation in the composite materials, in terms of maximal recovery force and of the corresponding maximal resonance vibration frequency shift will be presented as a function of the stiffness of the host material and SMA volume fraction. Preliminary guidelines for the optimization of these materials will thus be given.

SESSION LL4: ACTUATOR MATERIALS
Chair: Kenji Uchino
Wednesday Afternoon, December 1, 1999
Room 309 (H)

1:30 PM *LL4.1
INTELLIGENT GELS-THEIR DYNAMISM AND FUNCTIONS. Yoshihito Osada, Jiaping Gong, Division of Biological Sciences, Graduate School of Science, Hokkaido University, Sapporo, JAPAN.

The polymer system which undergoes shape change and produce mechanical energy in response to environmental stimuli is called a chemomechanical system. This system can transform chemical free energy directly into mechanical work under isothermal condition in a similar manner to living organisms and might be used wherever power for conventional devices is limited or difficult to obtain: underwater, in space or in the human body. We have developed chemomechanical systems which show quick responses and worm-like motions. The principle of motion is based upon an the molecular assembly reaction of surfactant molecules on the hydrogel.[1,2] Behaviors and principle have been analyzed both from thermodynamic and kinetic viewpoints. A hydrogel with a shape memory function have also been developed using acrylic acid(AA) and n-stearyl acrylate(SA).[3] The shape memory behavior is associated with the reversible order-disorder transition of the layered structure of alkyl side chains.[4]Using this, a thermo-responsive smart artificial sphincter capable of spontaneously opening and closing the valve was introduced. Spontaneous motion by spreading fluid on water have been demonstrated using (SA-CO-AA) gel.[5] When these gels undergo motion in water or on solid, the friction appears at the interface of the gel and the surroundings. The sliding friction of the various kinds of hydrogels has been investigated, and has been found that the frictional behaviors of hydrogels do not conform to Amonton's law $F=\mu W$, which well describes the friction of a solid. Instead, the friction force F of the gel shows slight dependence on the load W, while it strongly depends on the sliding velocity and area. The frictional coefficient μ of the gel reached a value as low as $\sim 10^{-3}$, which is often smaller than those observed in cartilage of the animal joints.[6] Mechanism[7,8,9] and biomedical applications of these smart gels will also be described. References [1] Y. Osada, H. Okuzaki and H. Hori, Nature, 355, 242 (1992) [2] Y. Osada and Simon Ross Murphy, Scientific American, 268, 82 (1993) [3] Y. Osada and A. Matsuda, Nature, 376, 219 (1995) [4] T. Tanaka, Y. Kagami, A. Matsuda and Y. Osada, Macromol., 28, 2574 (1995) [5] J. P. Gong, S. Matsumoto, M. Uchida, N. Isogai and Y. Osada, J. Phys. Chem., 100, 11092 (1996) [6] J. P. Gong, Y. Osada et al, J. Phys. Chem. B, 101, 5487 (1997) [7] J. P. Gong, Y. Osada et al, J. Chem. Phys., 109, 8062 (1998) [8] J. P. Gong, Y. Osada et al, J. Phys. Chem. B, 103, in press (1999) [9] J. P. Gong, Y. Osada et al, J. Phys. Chem. B, 103, in press (1999)

2:00 PM LL4.2
NOVEL SENSING AND ACTUATION SYSTEM INVOLVING SECONDARY PHASE MATERIALS WHICH ARE RELEASED AND ACTIVATED IN RESPONSE TO A CHANGE IN THE ENVIRONMENT. Carolyn Dry, University of Illinois, School of Architecture, Champaign, IL.

Smart materials tend to be expensive and difficult to add to matrices. The addition of secondary phase chemicals which are distributed as part of the composite system, either as fibers or particles, are integral to the system and relatively inexpensive, off the shelf materials. The release is in response to sensation of a change in the environment. Several examples are presented such as encapsulated corrosion inhibitors which are released by the onset of corrosion in the fiber encapsulators or another example is the release of crack repair chemical in response to the matrix cracking and then the fibers crack open. Several of these systems have been tested for years now and some have bneen tested in the field. These results will be reported as well as other applications currently under development.

2:15 PM LL4.3
SHAPE MEMORY CERAMICS. Takanobu Matsumura, Takanori Nakamura, Masaki Tetsuka, Kentarou Takashina, Kazuo Tajima and Yoshitake Nishi, Tokai Univ, Dept of Materials Science, Hiratsuka, Kanagawa, JAPAN.

The shape memory ceramics is developed to apply the hard environments. The glasses generally show the volume changes at different glass transition temperatures. To contact the different glasses each other, bi-ceramics has been prepared. The heating expands the molar volume of the glass rod A at the glass transition temperature (Tg^A), whereas the glass rod B doesn't change at Tg^A. The glass rod B is the supporting rod to be back to the size of glass rod A bellow Tg^A. Namely, the reversible shape memory effect should be induced by the supporting rod B as well as the expanding glass rod A at Tg^A. We studied the shape memory effect of bi-ceramics made for pyrexglass and sodiumglass. The shape memory effect was found from 473K to 623K. We conclude that the bi-ceramics is a new concept of the actuator, which can be operated at high temperature.

3:00 PM *LL4.4
PROPERTIES AND APPLICATIONS OF MAGNETO-RHEOLOGICAL FLUIDS. Mark R. Jolly, Thomas Lord Research Center, Lord Corporation, Cary, NC.

Magnetorheological (MR) fluids are materials that respond to an applied magnetic field with a change in rheological behavior. Typically, this change is manifested by the development of a yield stress that monotonically increases with applied field. Interest in MR fluids derives from their ability to provide simple, quiet, rapid response interfaces between electronic controls and mechanical systems. In this presentation, the underlying mechanisms of magnetorheology will be briefly reviewed. Rheological and magnetic properties of several commercial MR fluids will be presented and discussed. These fluids will be compared using appropriate figures of merit based on conventional design paradigms. The presentation will conclude with some contemporary applications of MR fluids.

3:30 PM LL4.5
DOPANT OPTIMIZATION IN CRYOGENIC ACTUATOR MATERIALS FOR THE NEXT GENERATION SPACE TELESCOPE. Ryan J. Shawgo, Maureen L. Mulvihill, Mark A. Ealey, Xinetics Inc., Research & Decelopment Dept., Devens, MA

Electroactive materials that function at cryogenic temperatures are needed for applications such as the Next Generation Space Telescope (NGST). The temperature at which the maximum dielectric constant and displacement are generated is the Curie temperature of the material. The Curie temperature can be tailored for a particular application by varying the dopant type and concentration in the ceramic. Development of cryogenic actuator material processing techniques to be used in a deformable mirror for NGST are currently under investigation by Xinetics. Studies include effects of dopant levels to produce maximum actuator displacement in the temperature range for NGST (30-60 K). Effects of process parameters on density, particle size, capacitance and displacement of the actuator will also be discussed.

3:45 PM LL4.6
MONOLITHIC SHAPE-MEMORY-ALLOY ACTUATOR: A NEW CONCEPT FOR DEVELOPING SMART MICRO-DEVICES. Yves Bellouard, Ecole Polytechnique Fédérale de Lausanne, Institut

de Systèmes Robotiques, Lausanne, SWITZERLAND; Thomas Lehnert, Ecole Polytechnique Fédérale de Lausanne, Institut de Génie Atomique, Lausanne, SWITZERLAND; Thomas Sidler, Ecole Polytechnique Fédérale de Lausanne, Institut d'Optique Appliquée, Lausanne, SWITZERLAND; Rolf Gotthardt, Ecole Polytechnique Fédérale de Lausanne, Institut de Génie Atomique, Lausanne, SWITZERLAND; Reymond Clavel, Ecole Polytechnique Fédérale de Lausanne, Institut de Systèmes Robotiques, Lausanne, SWITZERLAND.

Shape Memory Alloys (SMA) have strong potential in the field of micro-systems: they are easy-to-actuate and have the highest force/weight ratio among all known actuators. Unfortunately, the shape memory effect is not intrinsically reversible: if no special treatment has been done on the material, an external force must be applied to deform the material after the shape recovery. In actuator applications, a pullback spring is often used to deliver this external force. Moreover, to initiate the shape transformation, a mechanical pre-straining is also required. In micro-robotics and for micro-systems, forces such as friction, electrostatic and adhesion (whose effects can be negligible for larger systems) become more important. In addition, the assembly of small components is difficult and sometimes impossible to carry out. Therefore, the smaller the device, the harder it is to introduce a simple pullback spring or to pre-strain the structure. We have developed a new concept for SMA micro-devices: this concept consists of considering the SMA not only as the actuating part of a system but rather as a more complete system. Therefore, the active device will only be one piece of material, which we call Monolithic SMA Actuator. In order to obtain a reverse motion, specific solutions such as the use of the Two-Way Shape Memory Effect, push-pull design, local heating and local annealing has been developed. The latter is the most promising and the most powerful method. In this case, we use a laser to create active and passive parts within the same piece of material as well as a pre-strain can be added to the structure without any mechanical interaction. In this paper, this new concept and three applications developed at EPFL are presented: a micro-gripper for optical element assembly, a one-axis translation stage and a high-speed micro-switch.

SESSION LL5: POSTER SESSION

Chair: Yukio Ito
Wednesday Evening, December 1, 1999
8:00 P.M.
Exhibition Hall D (H)

LL5.1
HYPERSONIC AND DIELECTRIC STUDIES OF RELAXOR FERROELECTRIC SINGLE CRYSTALS. Chi Shun Tu, C.-H. Yeh, F.-C. Chao and C.-L. Tsai, Department of Physics, Fu-Jen University, Taipei, Taiwan, REPUBLIC OF CHINA.

Both the longitudinal (LA) Brillouin back-scattering spectra and dielectric permittivities along the [001] direction have been measured as a function of temperature for relaxor ferroelectric single crystals $0.68(Pb(Mg_{1/3}Nb_{2/3})O_3)$-$0.32(PbTiO_3)$ (PMN-32%PT), and $0.915(Pb(Zn_{1/3}Nb_{2/3})O_3)$-$0.085(PbTiO_3)$ (PZN-8.5%PT). A sharp ferroelectric phase transition (which is associated with a Landau-Khalatnikov-like phonon damping maximum) was observed in the temperature range 440-460 K for both PMN-32%PT, and PZN-8.5%PT. As temperature decreases, a diffuse phase transition was detected in the low temperature region. In addition, the nature of the thermal hysteresis for the dielectric permittivity confirms that these transitions of both crystals are first-order type. The frequency-dependent dielectric data prove the existence of an electric dipolar relaxation process in the low temperature region. The activation energy, the Vogel-Fulcher temperature and attempt frequency corresponding to this relaxation process are also calculated for both crystals.

LL5.2
RADIAL BRAGG GRATING PACKAGE FOR TEMPERATURE COMPENSATION IN INTERFEROMETRIC HYDROPHONE ARRAYS. Louis G. Carreiro, Gregory H. Ames, Paul D. Curry and Thomas S. Ramotowski, Naval Undersea Warfare Center, Newport, RI.

A new method of packaging fiber Bragg gratings to stabilize their wavelength over temperature variations was developed using a grating mount fabricated from zirconium tungstate, ZrW_2O_8, a ceramic with a negative coefficient of thermal expansion (CTE). The principle of operation is based on the differences in CTE of the Bragg grating and grating mount, with the mount compensating for the thermo-optic effects of the fiber. As the temperature is elevated the refractive index of the grating increases, shifting the reflection wavelength to longer wavelength. At the same time the ceramic grating mount contracts relieving some of the tension on the grating causing it to shift to shorter wavelength. Since these two effects offset each other, the thermal shift of the grating can be either eliminated or minimized. In addition to a description of the temperature-compensating device, the properties of zirconium tungstate are briefly discussed with emphasis on the processing of the ceramic grating mount. Thermal analysis data are given for zirconium tungstate with 5% polymer binder added. It was found that addition of the polymer caused the magnitude of the CTE of zirconium tungstate to decrease slightly from its literature value of $8.8 \times 10^{-6}/°C$ to $7.8 \times 10^{-6}/°C$.

LL5.3
PREPARATION OF (001)-ORIENTED PZT THICK FILMS ON SILICON WAFER BY PULSED LASER DEPOSITION. J.-M. Liu, Institute of Materials Research & Engineering, National University of Singapore, SINGAPORE and Department of Physics, Nanjing University, CHINA; S.Y. Xu, W.Z. Zhou, C.K. Ong, Department of Physics, National University of Singapore, SINGAPORE; L.C. Lim, Department of Mechanical & Production Engineering, National University of Singapore, SINGAPORE.

Completely (001)-oriented $Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT) films up to $\sim 10\mu m$ thick, deposited on (100) silicon wafers with Y-stabilized ZrO_2 (YSZ) as buffer and YBCO as electrode, are prepared by using pulsed laser deposition. The X-ray rocking curve scanning with respect to (001) reflection of $6.0\mu m$-thick films exhibits the FWHM of only 0.6-0.7°. Small grain size and smooth surface of the as-prepared films were identified. The performance of YSZ as excellent resisting layer against silicon diffusion was confirmed by the SIMS measurements. The electrical property evaluations demonstrated quite good ferroelectric property. A piezoelectric coefficient $d_{31} \sim$-300pC/N, acceptable for piezoelectric applications, was measured.

LL5.4
APPLICATION OF PIEZOELECTRIC MATERIALS FOR USE AS ACTUATORS AND SENSORS IN HARD DISK DRIVE SUSPENSION ASSEMBLIES. Jason R. Heffelfinger and Dennis A. Boismier, Hutchinson Technology Inc., Hutchinson, MN.

Track density, the number of recording lines per inch as measured along the radius of a disk, continues to increase at a rate of $\sim$ 30% per year for the hard disk drive industry. In order to maintain this rate of growth, dual stage actuators will need to be implemented in the next 2 to 3 years or by the end of 2001. Suspension based actuators that use piezoelectric ceramic materials pose the most economical and manufacturable option for dual-stage actuation. In particular, lead zirconium titinate (PZT) ceramics that have high piezoelectric coefficients and high Curie temperatures are required for this application. In this investigation the electrical and mechanical properties of a commercially available PZT material are studied as a function of processing and disk drive operation conditions. The coupling coefficients, dielectric constant and piezoelectric coefficients are measured as a function of time, voltage exposure (cyclic and DC) and temperature exposure. The fracture strength is measured by a 3-point bend technique as a function of material flaw size. Since the piezoelectric material is in direct contact with the suspension, this material acts as an active sensor inside the disk drive. The electrical response of the PZT is measured for shock events from 500 to 2000 G and during mechanical resonance of the suspension assembly. Tests indicate that the piezoelectric material is effective in detecting shock and resonance events that occur to the suspension assembly.

LL5.5
ELECTRICAL PROPERTIES AND POWER CONSIDERATIONS OF A PIEZOELECTRIC ACTUATOR. Thomas Jordan, Zoubeida Ounaies, ICASE, John Tripp and Ping Tcheng, NASA Langley Research Center, Hampton, VA.

This paper assesses the ability of piezoelectric wafers to meet design requirements in aeronautical applications such as active control of aircraft. Determination of capacitive behavior and power consumption is necessary to optimize the system configuration and to provide efficient driving electronics. Empirical relations are developed from experimental data to predict the capacitance and loss tangent of a PZT5A ceramic as nonlinear functions of both applied peak voltage and driving frequency. Power consumed by the PZT is the rate of energy required to excite the piezoelectric system along with power dissipated due to dielectric loss and mechanical damping. Overall power consumption is thus quantified as a function of peak applied voltage and driving frequency. In addition, energy conversion efficiency was investigated. The mechanical power generated by a PZT under load was experimentally determined. It was demonstrated that by incorporating the variation of capacitance and power loss with voltage and frequency, satisfactory estimates of power requirements can be obtained. These relations allow general guidelines in selection and application of piezoelectric actuators and driving electronics for active control applications.

LL5.6
TEMPERATURE DEPENDENCE OF HIGH FIELD CHARACTERISTICS IN PMN-PT-BT CERAMICS CONTAINING CHEMICAL ADDITIVES. Yong S. Cho*, Chang H. Yoon, Steven M. Pilgrim, New York State College of Ceramics at Alfred Univ, Alfred, NY; Keith Bridger, Active Signal Technologies, Cockeysville, MD. *Now with DuPont Photopolymer and Electronic Materials, Wilmington, DE, Supported by the Office of Naval Research under Contract No. N00014-97-C-0285.

A particulate coating process incorporating small amounts of several additives such as Ti, Zn, Ba, Sr and Fe was used to modify the composition and resultant properties of commercially available lead magnesium niobate based ceramics. Low- and high-field characteristics including dielectric properties, induced strain and polarization, and associated hystereses were evaluated for the samples sintered at 1200C for 4 hrs. All properties were found to depend on the chemical additives and temperature. Specifically, the addition of small quantities of Ti and Ba tended to increase strain and effective Q coefficient while maintaining a low dielectric constant–a promising result for room temperature transducer applications. The addition of Ti with Zn or Fe raised dielectric constant, strain, and polarization with significant increases in the temperature of maximum permittivity. On the other hand, the electromechanical properties of the samples containing Ba or Zn with Ti did not exhibit a transition to piezoelectric behavior at the temperature expected from the dielectric measurements. The main concern of this work is to study the high field characteristics according to changes in temperature and additives.

LL5.7
DETERMINATION OF ENERGY LOSS IN PIEZOELECTRIC ACTUATORS UNDER OFF-RESONANCE DRIVING. Jiehui Zheng and Kenji Uchino, International Center for Actuators and Transducers, Materials Research Laboratory, The Pennsylvania State University, University Park, PA; Bertram Sugg, Robert Bosch BmbH, Stuttgart, GERMANY.

Piezoelectric actuators are used in industries, automobiles, and aerospace, etc.. During the applications, it has been found that heat generation occurred when the actuators were driven cyclically. The heat generation is due to the internal loss mechanisms in the actuators. This work aims at determining energy loss in piezoelectric actuators from the view points of driving conditions and different materials used. Heat generation in piezoelectric actuators under different driving waveforms, rise times, and frequencies, etc. has been investigated. Through monitoring the temperature rise in the actuators, it has been found that the heat generation varied by changing the driving conditions, and the results can be discussed in terms of frequency dependence of dielectric and mechanical losses. Furthermore, different actuator materials (hard, soft piezoelectrics and electrostrictor) exhibit different internal loss mechanisms, and thus leading to different heat generation. The dielectric loss was determined from polarization vs. electric field hysteresis measurement under a constant stress level, and the mechanical loss was determined from strain vs. stress hysteresis measurements under a constant electric-field level. The strain vs. electric-field measurements under a constant stress and polarization vs. stress measurements under an open-circuit condition provide information on piezoelectric loss. Through the heat generation and loss mechanism studies, different sources of energy loss in piezoelectric actuators and their contribution to the total loss was investigated. Means of reducing energy loss in actuators will be discussed.

LL5.8
DISK TYPE PIEZOELECTRIC TRANSFORMER DESIGN EMPLOYING HIGH POWER PIEZOELECTRIC CERAMIC MATERIAL. Burhanettin Koc, Yongkang Gao and Kenji Uchino, International Center for Actuators and Transducers, Materials Research Laboratory, The Pennsylvania State University, University Park, PA.

Previously, we proposed a new disk type piezoelectric ceramic transformer design, which has crescent shape electrodes at the input part and focused poling direction at the output side on a commercially available hard PZT disk (APC841, APC International Ltd., USA). The electrode and poling configurations on the transformer were designed so that a planar or shear mode electromechanical coupling constant become effective rather than a transverse mode coupling constant. The voltage step-up ratio of the designed transformer was dramatically improved in comparison with an equivalent rectangular type transformer. We made transformer using pseudo-ternary solid solution ceramic with the composition of $xPb(Mn_{1/3}Sb_{2/3})O_3$-$yPbZrO_3$-$zPbTiO_3$ (xPMS-yPZ-zPT) as the high power piezoelectric ceramic material. In this study, the characteristics such as step-up ratio, power transformation efficiency and temperature rise of the transformers were compared for conventional hard PZT and newly developed high power piezoelectric materials.

LL5.9
DOPING EFFECTS IN Pb(Mg1/3Nb2/3)O3-PbTiO3 CERAMICS FOR HIGH POWER TRANSDUCTION APPLICATIONS. Yun-Han Chen and Kenji Uchino, Material Research Laboratory, The Pennsylvania State University, University Park, PA; Senji Hirose, Yamagata University, Yonezawa-shi, Yamagata, JAPAN; Dwight Viehland, Naval Undersea Warfare Center, Newport, RI.

Piezoelectric ceramics are potential high-power electro-acoustic sources, and have been studied for many years. However, when these devices are driven under high level vibration, the electromechanical characteristics depart significantly due to the loss and nonlinear behavior in terms of elastic and dielectric properties. In this paper, we present results concerning the development of modified Pb(Mg1/3Nb2/3)O3-PbTiO3 (PMN-PT) ceramics for high-power applications. We have focused efforts on base PMN-PT compositions close to the morphotropic phase boundary. Different doping have been studied to understand doping effects on the properties of PMN-PT ceramics and, moreover, to improve properties for the high-power applications. Evidence will be presented which indicates that the improved mechanical quality factor results from the introduction of hard characteristics into PMN-PT by the substituents. Investigations of the nonlinearity of the electromechanical properties on the vibrational amplitude were performed using a constant displacement method. The degradation of the mechanical quality factor with increasing drive amplitude also presents thermal stability problems for the use of these materials in high power transducer applications. This work is sponsered by the Office of Naval Research.

LL5.10
THE EFFECT OF HAFNIUM AND THERMAL CYCLING ON THE TRANSFORMATION TEMPERATURES OF NiTi BASED SHAPE MEMORY ALLOYS. Paul E. Thoma, John J. Boehm, Johnson Controls, Inc., Corporate Technology, Milwaukee, WI.

The effect of thermal cycling on the austenite to martensite (A to M) and martensite to austenite (M to A) transformation temperatures (TTs) of $Ni_{49.8}Ti_{50.2}$ and $Ni_{49.8}Ti_{40.2}Hf_{10}$ shape memory alloys is investigated. Test specimens are from arc melted buttons homogenized at 900°C for 100 hours. The results show that the A to M and M to A TTs for both alloys decrease with increasing number of thermal cycles through the phase transformations. The A to M TT of the NiTiHf alloy stabilizes more quickly than the A to M TT of the NiTi alloy when cycled. However, the M to A TT of the binary NiTi alloy stabilizes more quickly than the M to A TT of the NiTiHf alloy when cycled.

LL5.11
COMPARISON BETWEEN STRUCTURAL AND NÉEL TRANSITION TEMPERATURES IN Fe-Mn-Si SHAPE-MEMORY ALLOYS OBTAINED BY DIFFERENT TECHNIQUES. M.I.N. da Silva[1], G.J. de Arruda [1], P.E.F. Côrtes [1], M.S. Andrade [1], R. Paniago[2] and N.L. Speziali[2]; [1]Setor de Tecnologia Metalurgica SDT, Fundacao Centro Tecnologico de Minas Gerais, Belo Horizonte-MG, BRAZIL; [2] Departamento de Fisica ICEx, Universidade Federal de Minas Gerais, Belo Horizonte-MG, BRAZIL.

Fe-Mn-Si based alloys exhibit shape memory effect depending on their composition. Upon cooling, it has a martensitic transformation, gamma (FCC) and epslon (HCP) phases, and a magnetic transition from antiferromagnetic to paramagnetic, Néel temperature. It is said that this temperature increases the stability of the gamma phase, depressing the start martensitic transformation temperature to quite lower values. In this work, the phase and magnetic transition temperatures were measured on a Fe-27Mn-2.5Si (%wt.) shape-memory alloy by using differential scanning calorimetry, dilatometry, electrical resistivity, Mössbauer spectroscopy and X-ray diffraction. For all techniques employed, the results showed that transition temperatures (structural and magnetic) are in the same order of magnitude. It was observed by calorimetry and electrical resistivity that the magnetic transition temperature was near and before the start of martensitic transformation. When the difference between these temperatures being smaller, the thermo-induced gamma to epslon transformation is suppressed and the quantity of pre-existing epslon-martensite becomes smaller. A Néel temperature around 320K was confirmed through by Mössbauer spectroscopy; also, only 20% of the gamma-phase transformed in epslon-phase. With this last technique and X-ray diffraction, it was also possible to quantify the amount of martensite induced by temperature lowering. X-ray diffraction analysis showed that the alpha-phase segregates to surface on heating.

LL5.12
HIGH TEMPERATURE THERMOELECTRIC PROPERTIES OF SILICON BORIDE CERAMICS AS A SMART MATERIAL.
Takashima Noriyuki, Azuma Yasuo, Matsushita Jun-Ichi, Faculty of Engineering, Tokai University, Hiratsuka, JAPAN.

Several silicon boride phases such as SiB_4, SiB_6, SiB_{6-x}, SiB_{6+x}, and $Si_{11}B_{31}$, were previously reported. Among them, SiB_6 has proved to be a potentially useful material because of its excellent electrical conductivity, high degree of hardness, moderate melting point, and low specific gravity. The sintering conditions and thermoelectric properties of silicon boride (SiB_6) ceramics produced by hot pressing were investigated in order to determine the suitability of this material for high-temperature thermoelectric applications as a smart material. The relative density increased with increasing sintering temperature. With a sintering temperature of 1923 K, a sintered body having a relative density of more than 99% was obtained. X-ray diffraction analysis showed no crystalline phase other than SiB_6 in the sintered body. The specimens were prepared for measurement of the electrical conductivity and Seebeck coefficient by the D.C. four-terminal method. The thermal conductivity of SiB_6 was obtained by calculation from the thermal diffusivity and specific heat capacity of the specimen. The electrical conductivity of SiB_6 increased with increasing temperature. The electrical conductivity of the polycrystalline SiB_6 (99% dense) was 1.0 to 1.1×10^3 S/m at 298 to 1273 K. The thermal conductivity decreased with increasing temperature in the range of room temperature to 1273 K. The thermal conductivity was 9.1 to 2.5 W/mK in the range of room temperature to 1273 K. The Seebeck coefficient of SiB_6 increased with increasing temperature. The Seebeck coefficient of SiB_6 was showed 140×10^{-6} V/K at 1273 K. The figure of merit Z of SiB_6 increased with increasing temperature. The Z of SiB_6 reached 8.1×10^{-6} /K at 1273 K. The ZT value is useful to evaluate the ability of the thermoelectric materials. The ZT value reached 0.01 at 1273 K. Based on the results, SiB_6 showed very good thermoelectric material at high temperature.

LL5.13
$PbZr_{0.52}Ti_{0.48}O_3$ FERROELECTRIC THIN FILMS ON SILICON BY KrF EXCIMER LASER ABLATION.
Kenji Ebihara, Fumiaki Mitsugi, Masaaki Yamazato, Tomoaki Ikegami, Department of Electrical and Computer Engineering, Kumamoto University, Kurokami, Kumamoto, JAPAN; Jagdish Narayan, Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC.

$Pb(Zr_xTi_{1-x})O_3$ (PZT) ferroelectric thin film on the silicon substrate has been required for the integration into semiconductor devices such as nonvolatile memory and ferroelectric field effect transistor. The YSZ buffer layer is needed to prevent reaction and diffusion between the $YBa_2Cu_3O_{7-x}$ (YBCO) bottom electrode and the silicon(100) substrate. The KrF excimer laser ablation technique is employed to fabricate the capacitive structure Au/PZT/YBCO/YSZ on the silicon substrate. The ferroelectric properties of the capacitive hetero-structures also deposited on the MgO substrate are compared to those of the PZT films on Si substrate. Optimal deposition condition and ferroelectric properties such as remanent polarization, and fatigue were reported here.
The X-ray diffraction patterns showed that the PZT films prepared on YBCO/YSZ/Si(100) at 550°C, O_2, 100mTorr and with a laser fluence of 2 J/cm^2(5Hz) are pure perovskite and highly oriented with the (00n) orientation. The polarization-electric field characteristics shows the remanent polarization of about 23 μC/cm^2 and coercive field of 60 kV/cm (applied voltage 30V,1kHz). Remanent polarization of PZT films on a YBCO/YSZ/Si substrate was one-half of initial value after about 10^{10} switching cycles.

SESSION LL6: MAGNETOSTRICTIVE MATERIALS
Chair: Marilyn Wun-Fogle
Thursday Morning, December 2, 1999
Room 309 (H)

8:30 AM *LL6.1
SMART STRUCTURE APPLICATIONS FOR MAGNETO-STRICTIVE MATERIALS.
Alison Flatau, Marcelo J. Dapino, Iowa State University, Aerospace Engineering and Engineering Mechanics Department, Ames, IA; Frederick T. Calkins, The Boeing Company, Phantom Works, Seattle, WA.

Magnetostrictive materials convert magnetic energy to mechanical energy and vice versa. As a magnetostrictive material is magnetized, it strains, and as a force or torque produces a strain in a magnetostrictive material, the material's magnetic state is changed. This bi-directional coupling between the magnetic and mechanical states of the material provides a transduction capability that can be used in smart structures a variety of ways, both for actuation and sensing. The current interest in design of adaptive and/or smart structures, coupled with the advent of giant magnetostrictive materials such as Metglass and Terfenol-D has lead to a renewed interest in the engineering of optimized magnetostrictive transducer designs and the need for advances in the ability to model the behaviors of these materials under dynamic applied magnetic fields and mechanical loads. An overview of recent applications for giant magnetostrictive materials in smart applications as both sensors and actuators will be presented along with a brief discussion of some of current tools and needs for modeling of magnetostrictive devices.

9:00 AM LL6.2
FORCE CAPABILITY OF A PLANAR PERISTALTIC TER-FENOL-D INCHWORM MOTOR.
Michael J. Gerver, James H. Goldie, Won Jong Kim, Jerome Kiley, John R. Swenbeck, SatCon Technology Corp., Cambridge, MA.

A magnetostrictive peristaltic inchworm motor was built and tested, using Terfenol-D as the active element. Unlike the Kiesewetter inchworm, which uses a rod of Terfenol-D in a closely fitting cylindrical channel, in our design the Terfenol-D is in the form of a flat slab, held between two spring-loaded plates. With this configuration the motor is not affected by wear of the Terfenol-D surface. In order to avoid eddy currents, we used laminated Terfenol-D. Operating at 1600 Hz with a peak field of 430 oersteds, the speed with no load was observed to be 12 mm/s, which is 65% of the theoretical value (the peak strain times the phase velocity of the magnetic field), probably because the surface flatness of the Terfenol-D was only a few times smaller than the height of the peristaltic bumps. The measured stalling force was 90 newtons, corresponding to 350 kPa in the Terfenol-D, only 3.5% of the peak strain times the stiffness of the Terfenol-D. This was in almost exact agreement with the stalling force predicted by a model which took into account the force from the spring-loaded plates (needed to avoid slippage), the transverse stiffness of the Terfenol-D slab, and the finite bending stiffness of the stator. This model can be used to design optimized inchworm motors with improved force capability. We also tried using composite Terfenol-D, consisting of Terfenol-D particles in an epoxy binder, but the performance was worse, perhaps because it did not have a very uniform distribution of particles, and could not be machined as flat as the laminated Terfenol-D. The motor was driven by a specially designed 3-phase power inverter and digital controller, and the large reactive power inherent in this kind of motor was reduced by putting it in series with resonant capacitors.

9:15 AM LL6.3
ANISOTROPIC MAGNETISM AND MAGNETOSTRICTION IN FIELD-STRUCTURED COMPOSITES: BASIS FOR A FAST ARTIFICIAL MUSCLE?.
James E. Martin, Robert A. Anderson, Eugene Venturini and Judy Odinek, Sandia National Laboratories, Albuquerque, NM.

There is currently quite a bit of interest in developing elastomers or gels that respond mechanically to an imposed field. Such materials would have a wide range of applications as electromechanical actuators and sensors, including robotics applications. We have investigated the properties of field-structured composites consisting of magnetic particles in a polymeric host that have been structured into chains or sheets by applied uniaxial or biaxial magnetic fields, and have found that these have very anisotropic magnetic properties that should give rise to a large magnetostrictive effect. In particular, when a polymeric material containing particles structured into chains is exposed to a magnetic field aligned along the direction of these chains, the induced magnetic dipoles give rise to large local fields that are many times greater than those in a random particle composite. We have shown that these large local fields greatly increase the magnetic permeability of the composite, and that this in turn increases the magnetostrictive effect, which we have shown depends on terms quadratic in the permeability. In addition to the large stress response of these materials, the response time should be very short, on the order of the particle magnetization time. Finally, we present extensive experimental data of magnetic hysteresis curves in structured magnetic particle systems that show that field-structuring can create a magnetic remanence anisotropy of eight in some systems, but that the coercive field is unaffected.

9:30 AM LL6.4
ORIGIN OF COERCIVITY IN GIANT MAGNETOSTRICTIVE TbFe-FeCo EXCHANGE SPRING MAGNETS FOR APPLICATIONS IN MEMS.
Harsh Deep Chopra and P. Wilson, Thin Films and Nanosynthesis Laboratory, Mechanical and Aerospace Engineering Department, State University of New York at Buffalo, Buffalo, NY.

Giant magnetostrictive rare-earth transition metal (RE-TM) thin films are currently being studied for micro-actuation in micro-electromechanical systems (MEMS). Due to limited driving fields available in MEMS, high magnetostrictive susceptibility is an important parametric requirement. Low switching fields can be achieved using the spring-magnet concept in which RE-TM films such as TbFe or SmFe are sandwiched between high saturation magnetization films such as FeCo. Appropriately configured, such multilayers exhibit a collective magnetic behavior which is an average of the individual layers. In the present study, the role of magneto-elastic interactions in controlling the magnitude of switching field and coercivity is investigated using sputter-deposited TbFe/FeCo multilayers. In the absence of long-range anisotropy in amorphous TbFe layers, along with negligible magneto-crystalline anisotropy in FeCo layers, reversal behavior in these multilayers is largely determined by stress-induced anisotropy. Results show that magneto-elastic constraints at the TbFe-FeCo interfaces arising due to different values of saturation magnetostriction in adjacent layers lead to biaxial stresses. This manifests itself as highly square and similar M-H loops measured in different directions in the plane of the films. The measured values of coercivity in these films (between 5.5 to 7.0 mT) are found to be in close agreement with theoretical coercivity values predicted on the basis of stress-induced anisotropy. Biaxial stresses resulting in corresponding stress-induced anisotropy were found to have interesting consequences vis a vis magnetization reversal. In particular, self-organized domain patterns are formed during reversal which are mediated by local stress state in the films. The authors acknowledge Drs. E. Quandt and A. Ludwig at Center of Advanced European Studies and Research at Bonn, Germany, for providing the samples used in this study. This work is supported by the National Science Foundation, Grant No. DMR-97-31733, and this support is gratefully acknowledged.

10:15 AM LL6.5
EVALUATING THE RESPONSE OF MAGNETOSTRICITVE COMPOSITE SYSTEMS ANALYTICAL AND EXPERIMENTAL.
Greg P. Carman, Geoff Mcknight and Terrisa Duenas, University of California, Los Angeles, CA.

This presentation will describe analytical and experimental results for a wide range of magnetostrictive composite systems fabricated at UCLA. Magnetostrictive composites contain Terfenol-D and other magnetic particulate embedded in a polymer matrix resin. The composites are cured in the presence of a magnetic field to align the particles into continuous chains representative of continuous fiber composites (i.e. 1-3 composites). Based on a comparison between stiffness measurements and analytical predictions, we conclude that the composite behaves similar to a continuous fiber composite system rather than a particulate composite sysetm. Advantages associated with 1-3 composite when compared to monolithic magnetostrictive materials include, higher frequency response, automatic alignment of domains, durable system, net shape fabrication, and impedance matching. A detailed study on the response of the composite system as a function of volume fraction will be presented. It will be demonstrated that for a specified polymer resin system an optimal volume fraction exists to maximize the magnetostriction of the composite. For one thermoset resin the optimal volume fraction is 20% and produces strains similar to the monolithic material. Increasing the value for the optimum volume fraction requires choosing resins that have either a larger cure temperature or a larger coefficient of thermal expansions. To demonstrate this concept results from a polyimide magnetostrictive composite study will be described.

10:30 AM LL6.6
DEVELOPMENT OF HIGH FREQUENCY TERFENOL-D TRANSDUCER MATERIAL.
E.A. Lindgren, J.C. Poret, Industrial Quality, Inc., Gaithersburg, MD; J.J. Whalen, M. Rosen, Department of Materials Science, Johns Hopkins University, Baltimore, MD; M. Wun-Fogle, J.B. Restorff, Naval Surface Warfare Center, Carderock Division, W. Bethesda, MD; A.E. Clark, Clark Associates, Adelphi, MD.

The goal of this research and development program is to develop production methods to obtain thin strips of oriented crystalline Terfenol-D and to develop multi-layered driver rods for transducers that will operate over a broad spectrum into the MHz frequency range. To reach this goal, special methods must be used to minimize the amount of mechanical processing, such as machining, as the material is both brittle and expensive. A modified rapid solidification method has been developed to process Terfenol-D to obtain the desired geometric configuration of long thin strips. Currently, sections that are 500 mm long, 2 mm wide and 0.1 mm thick can be produced. In addition, this method enables the crystallographic orientation of the strips of Terfenol-D to be controlled in order to obtain the preferred orientation that will maximize the magnetostrictive displacement of the material. Magnetostrictive measurements have demonstrated that the samples prepared by this approach have a 62 percent improved magnetostrictive performance when compared to non-oriented Terfenol-D. Parallel to this effort is the development of a procedure to consolidate the strips of material into transducer driver rods. This includes maintaining the improvements in the magnetostrictive performance of the Terfenol-D while providing a greater cross sectional area for the active surface of the driver rods. This will require matrix materials that have sufficient elasticity to maintain electrical isolation between the ribbons when subjected to the cyclical loading required for operation. In addition, the matrix material must have sufficient rigidity to minimize the loss of magnetostrictive displacement through absorption of the magnetostrictive deflection. This development is currently underway and magnetostrictive displacement values of several different driver rods are expected to be available for this presentation. The Office of Naval Research Small Business Innovative Research Program is acknowledged for funding this progam, with Jan F. Lindberg as the Program Monitor.

10:45 AM LL6.7
MODELING AND EXPERIMENTS FOR DEFORMATION UNDER LOAD IN Ni-Mn-Ga FERROMAGNETIC SHAPE MEMORY ALLOY.
S.J. Murray, S.M. Allen and R.C. O'Handley Massachusetts Institute of Technology, Cambridge, MA.

Ferromagnetic Shape Memory (FSMA) materials are actuator materials that deform under magnetic field by twin boundary motion in martensite. The maximum strain seen in FSMA materials has increased recently to 1.3%[1], and then 5%[2] in Ni-Mn-Ga. A model is described here which recognizes a fundamental difference in the mechanism of energy input between magnetostrictives and ferromagnetic shape memory alloys. In a magnetostrictive, the magneto-crystalline anisotropy (Ku) is minimized to allow for easy rotation of magnetization within the crystal. The mechanism of strain in an FSMA, twin boundary motion, causes the magnetic direction to change only when the magnetization is strongly coupled to the crystal structure by a large Ku. This model equates strain energy with magnetic energy for twin boundary motion. As this twin wall moves through a tetragonal FMSA, the area where the twin has passed undergoes a strain of (1-c/a), yielding a total strain energy of (1-c/a)*stress. As well, magnetic energy (MsH) of rotating the moment of the material will only be coupled into the sample in this area where the magnetization has been rotated by twin motion, the rest of the sample can be ignored. Putting these terms together and solving for stress, we get the relation stress=(Ms/[1-c/a])H, where H is the only adjustable parameter. This suggests that a critical stress exists. Below this stress, magnetic energy dominates and twin boundaries will move freely until the material is transformed to a single variant, above this stress mechanical energy dominates and there will be no strain whatsoever. This idea is applied to both axial and shear actuation. The predictions of this theory are compared with the predictions of theories from O'Handley[3] and James and Wuttig[4], and with data from Tickle et. al.[1]. Experiments are performed in the case of axial loading to validate the ideas of the theory. REFERENCES 1. R. Tickle, R. D. James, T. Shield, P. Shumacher, M. Wuttig, and V.V. Kokorin, Ferromagnetic shape memory in the NiMnGa system Submitted for Publication. 2. Steven J. Murray, Ryogi Hayashi, Miguel Marioni, Samuel M. Allen, Robert C. O'Handley, Magnetic and mechanical properties of FeNiCoTi and NiMnGa magnetic shape memory alloys SPIE proceedings 1999 3. R. C. O'Handley, Model for Strain and Magnetization on Magnetic Shape Memory Alloys Journal of Applied Physics vol. 83, No. 6 (1998) 3262-3270. 4. R.D. James and M. Wuttig, Magnetostriction of Martensite, Phil. Mag A, (1998)

11:00 AM LL6.8
MODELING AND CONTROL ISSUES CONCERNING SMART MATERIALS WITH HYSTERESIS.
Ralph Smith, Dept. of Mathematics, North Carolina State University, Raleigh, NC; Marcelo Dapino, Alison Flatau, AEEM, Iowa State University, Ames, IA; Craig Hom, Lockheed Martin, Palo Alto, CA; Zoubeida Ounaies, ICASE, NASA Langley Research Center, Hampton, VA.

Modeling and control issues concerning certain smart material actuators utilized in nonlinear regimes will be presented. Piezoceramic, electrostrictive and magnetostrictive materials all exhibit various degrees of hysteresis and nonlinear dynamics at high drive levels. The accurate and efficient quantification of these effects and their incorporation in control design are necessary to attain the full capabilities of the materials. Various modeling techniques and their impact on control design will be presented. Linear control methods prove ineffective at high drive levels and certain nonlinear control laws and inverse compensation techniques for smart material applications will be discussed.

11:15 AM LL6.9
A MECHANISM FOR THE MAGNETICALLY DRIVEN SHAPE-MEMORY EFFECT.
P.J. Fereira and J.B. Vander Sande, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA.

The recent development of magnetically driven shape memory alloys, for which the deformation processes are controlled by the application of a magnetic field, is attracting a great interest for engineering purposes, in particular for the development of fast actuators. The integration of these actuators with sensing and control capabilities is at the heart of smart systems, which are becoming relevant in modern machine design. Thus far, it is believed that the magnetic field induced shape memory effect involves reorientation of certain twin variants of the martensite phase. In other words, if two adjacent variants have their easy axis of magnetization oriented at different angles with respect to the applied magnetic field, an extra driving force for the translation of the twin plane will arise. However, the exact mechanism by which reorientation of the twin variants under an applied magnetic field occurs has not been discussed. The present work attempts to describe this effect based on the argument that the twin structure of a martensite phase may be reoriented by the magnetic pressure exerted at the twin dislocations and their subsequent motion. Reorientation of the twin structure is controlled by the strength and orientation of the magnetic field, relative orientation between the twin variants and the friction stresses imposed on the dislocations. Ultimately, if the magnetic field is strong enough, all the martensite plates will consist of one single twin variant. In the present work, it was determined that a magnetic field of 100 kOe applied to a ferrous alloy containing twinned martensite plates may result in stresses of about 35 MPa acting on twin dislocations. Despite this level of stress, which is comparable to the Peierls stress at 0 K, this value may be insufficient to initiate dislocation motion for cases where strong dislocation pinning exists.

11:30 AM LL6.10
FORMATION AND CHARACTERIZATION OF SINGLE CRYSTAL Ni_2MnGa THIN FILMS. J.W. Dong, L.-C. Chen, C.J. Palmstrom, Department of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, MN; R.D. James, Department of Aerospace Engineering and Mechanics, University of Minnesota, MN.

Ni_2MnGa is a ferromagnetic shape memory alloy with a Curie temperature of ~376 K for the stoichiometric compounds. When the temperature is above the martensitic transformation temperature (~202 K), the stable austenitic phase has a cubic Heusler ($L2_1$) crystal structure with a lattice constant of a = 5.825 Å. Below 202 K, the martensitic phase is stable, which has a tetragonal crystal structure with a = b = 5.92 Å, c = 5.57 Å. We have recently observed magnetoelastic strain as large as 4.3% in bulk single crystals. This property makes Ni_2MnGa a promising candidate for magnetic field driven actuator materials. For the applications in microelectro-mechanical systems, single crystal thin films are desirable. We have successfully grown single crystal (001) Ni_2MnGa thin film on (001) GaAs substrates by molecular beam epitaxy. X-ray diffraction and transmission electron microscopy show that the Ni_2MnGa film grows pseudomorphically on GaAs and has a tetragonal structure with a = b = 5.65 Å, c = 6.12 Å. Preliminary magnetic measurements reveal that the Ni_2MnGa film (on GaAs substrates) is ferromagnetic with a Curie temperature of ~320 K. The properties of the Ni_2MnGa thin film before and after the selective removal of the GaAs substrates, as well as the influence of stress, will be discussed.

11:45 AM LL6.11
NEW MAGNETOELASTIC MATERIALS. Volodymyr Chernenko, Institute of Magnetism, Kiev, UKRAINE.

Some ferromagnetic intermetallic compounds like Fe-Rh, MnAs and Ni2MnGa exhibiting magnetostructural transitions at ambient temperature have been found to produce large magnetic field induced strains (more than 1%). Due to the physical mechanisms responsible for these large strains aforementioned materials tend to be considered separately from other known high magnetostrictive materials. These intermetallics are characterised by the two typical features: (1) an existence of the large dilatational (Fe-Rh , MnAs) or deviatoric (Ni2MnGa) components in a spontaneous transition strain tensor, from one hand and (2) pronounced coupling of those strains with the magnetization through magnetoelastic interactions, from the other. The dilatometric measurements of polycrystalline MnAs processed by ball-milling under applied magnetic field have been performed at constant temperatures by means of VSM -3001 magnetometer. The reversible magnetic field induced strains up to 0.7% have been found in this compound as result of a large spontaneous volume magneto-striction accompanying field induced magnetostructural transition above Curie temperature. The magnetoelastic response and magnetic field influence on martensitic transition has been studied experimentally in some single crystalline ferromagnetic shape memory Ni-Mn-Ga alloys. The theoretical treatment of available experimental results has been performed by the phenomenological modelling within the Landau theory of phase transitions.

SESSION LL7: SENSOR AND OTHER MATERIALS
Chair: Alison Flatau
Thursday Afternoon, December 2, 1999
Room 309 (H)

1:30 PM LL7.1
CHEMICAL SENSING WITH Co-DOPED $SrFeO_{2.5+x}$. Mike Post, Dongfang Yang, Xiaomei Du, Jim Tunney, Zakiah Kassam, National Research Council Canada, Ottawa, CANADA.

The oxygen non-stoichiometric perovskite $SrFeO_{2.5+x}$ changes its physicochemical properties when it is exposed to different partial pressures of oxygen. It also contains iron in the unusual +IV oxidation state and shows strongly catalytic reactivity towards volatile organic vapors. These material properties have been exploited in chemical sensor applications for detecting gases such as oxygen, volatile organic vapors, hydrogen and carbon monoxide. Doping $SrFeO_{2.5+x}$ with Co results in an increase in oxygen-ion conductivity and modification of catalytic reactivity. We have prepared materials with an orthorhombic intergrowth phase $Sr_4Fe_{6-x}Co_xO_{13+\delta}$, a perovskite phase $SrFe_{1-x}Co_xO_{2.5+\delta}$, and a mixed intergrowth and perovskite phase $SrFeCo_{0.5}O_y$ by a solid-state reaction method at high temperature in oxygen atmospheres. The crystal structures of these materials were characterized by powder X-ray diffraction. Thin films of the Co-doped $SrFeO_{2.5+x}$ materials have been deposited onto sapphire substrates using the pulsed laser ablation technique. X-ray diffraction, atomic force microscopy and ICP-MS have been used to characterize the texture, morphology and chemical stoichiometry of the films. The changes in electrical conductivity that occur when films of different thickness were exposed to oxygen and volatile organic vapors were measured at temperatures ranging from 300 to 450 °C. The relationship between the film thickness and the kinetics of the gas-solid reaction has permitted distinguishing whether the bulk diffusion process or the surface exchange reaction is the rate-determining step for the sensor reaction. The sensor responses of various Co-doped $SrFeO_{2.5+x}$ single-phase and mixed phases toward oxygen and volatile organic vapors have been compared and these reveal the material structure-property relationship and the relative chemical sensitivities and selectivities.

1:45 PM LL7.2
$Sr(Ti,Fe)O_3$: MATERIAL FOR A TEMPERATURE INDEPENDENT RESISTIVE OXYGEN SENSOR. Wolfgang Menesklou, Hans-Jürgen Schreiner, Karl Heinz Härdtl, Ellen Ivers-Tiffée, Universität Karlsruhe (TH), Institut für Werkstoffe der Elektrotechnik, GERMANY; Ralf Moos, Daimler Chrysler Research, Friedrichshafen, GERMANY.

The metal oxide strontium titanate is a promising material for resistive high temperature oxygen sensors (T > 1000 K), which can applied to control combustion engines. However, a disadvantage of this and of many other metal oxides is the strong temperature dependence of conductivity. In this work, we show that the temperature dependence of $Sr(Ti_{1-x}Fe_x)O_3$ can be adjusted by the iron content. For x = 0.35 the thermal activation of the conductivity is near zero for a limited temperature and oxygen partial pressure range (T= 1000 - 1200 K, pO_2 = 1 to 10^5 Pa). Short response times in the range of 10 ms can be realised with a sensor in thick film technology.

2:00 PM LL7.3
PEROVSKITE MATERIAL $ZnSnO_3$ - AS POSSIBLE ALCOHOL SENSOR. G. Sarala Devi, B. Sreedhar, Materials Science, Inorganic Chemistry Division, Indian Institute of Chemical Technology, Hyderabad, INDIA.

Sensors containing either one or two oxide phases, have been synthesized with controlled architectures. The biphasic sensor composed of $ZnSnO_3$ is active for detection of alcohol vapour(C_2H_5OH). The introduction of cheap, compact digital signal processing in form of microprocessor has revolutionalised the approach for sensor development. The availability of compact digital processing means that intelligence can be moved across interface boundary into sensor itself and such sensors are called smart sensors. Semiconducting oxide based gas sensors have been in use over the past few years for the detection of various gases which operate on the principle of change in surface conductance when in contact with gases. This renders the detection and estimation of toxic gases affected by number of oxygen vacancies in the bulk. We have been investigating how to upgrade the receptor function of a semiconductor gas sensor to a particular gas. As part of this we tried to improve the alcohol sensing properties by use of perovskite material $ZnSnO_3$ and thus contributing to development of good alcohol (C_2H_5OH) sensor for use as a breath alcohol checker(BAC) which is said to be well correlated with ethanol concentration in the blood. Thick films of sensor material were fabricated and its electrical resistance measurements were carried. The output voltage across the sensor was used to evaluate the electrical resistance of the element in presence and absence of test gas and the sensitivity calculated using the formula $S=(R_a - R_g)/R_a$

where R_a and R_g are resistance in air and sample gas. The structural and electrical response of sensor element towards alcohol vapours showed a good sensitivity towards low concentration of about 100ppm. at 250°C. The sensor had a response time of about 160sec. and returned to its original value quickly when the air flow was resumed. Based on the experimental results and gas chromatographic(G.C) studies a possible sensing mechanism is proposed.

2:15 PM LL7.4
SMART - AND ACTIVE - SURFACE NANOCOMPOSITES FORMED BY ION IMPLANTATION AND ANNEALING.
Lynn A. Boatner, Shin-ichi Honda, Frank A. Modine, Tony E. Haynes, Alkiviathes Meldrum, John D. Budai, Kyujeong Song and James R. Thompson, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

Ion implantation and thermal processing have been used to create smart surfaces consisting of active nanophase precipitates embedded in the near-surface region of an otherwise inactive substrate. The precipitated active phase produced by the implantation/annealing process frequently consists of single-crystal nanoparticles that are often faceted, physically isolated from each other by the host matrix, and in the case of single-crystal hosts, aligned crystallographically with respect to the host lattice. Near-surface nanocomposites of this type have a number of significant advantages over conventional thin films or homogeneous bulk materials. These include: the ability to optimize the surface nanocomposite properties by controlling the particle size, the ability to combine the functions of two or more embedded phases into a single integrated surface structure, and the protection of the active embedded nanophase particles from the environment by the host material. The ion-beam/annealing approach to the creation of smart, near-surface nanocomposites was initially applied to the formation of embedded nanocrystals of VO_2 and V_2O_3 in a single-crystal sapphire host. This approach has now been extended to the creation of magnetically and magneto-optically active surfaces through the formation of nanocrystalline precipitates of Co, Fe, Ni, and Fe_3O_4 embedded in the near surface of single crystals of cubic zirconia (Y-stabilized) and Al_2O_3. Additionally, we have recently shown that ion implantation subsequent to the formation of an active ferromagnetic precipitated phase can be used to significantly alter the magnetic properties of the nanophase particles. Due to the ability to form crystallographically oriented precipitates of smart materials, any anisotropic characteristics of the smart material are retained in the surface nanocomposite. The ferromagnetic surface nanocomposites formed using ion-beam methods have been characterized using x-ray, TEM, magnetic circular dichroism, and magnetization methods, and the results of these studies will be described.

2:30 PM LL7.5
MOLECULAR RECOGNITION OF ORGANIC COMPOUNDS WITH IMPRINTED SILICA.
Xiaoyi Gong, Jun Liu, William D. Samuels, Li-Qiong Wang and Zimin Nie, Northwest National Labs, Dept of Materials and Chemistry, Richland, WA.

Molecular recognition of organic compounds by imprinted silica was studied to understand the specific interactions between template and silica when no covalent bonding is involved in the imprinting process. Silica imprinted by p-methyl red or methyl red was found to be able to recognize its imprinting molecules. The same results were obtained from silica imprinted by methyl orange and ethyl orange, as well as silica imprinted by his-ala or his-leu. No molecular recognition was observed in the silica samples imprinted with methyl red or ethyl red, diaminoflourene or diaminodiphenylmethane, and histidineL or histidineDL.The molecular recognition capabilities were discussed regarding their molecular structures. The conclusions include: 1) More than two strong interaction sites on the imprinting molecules are necessary to endow silica with recognition capability. 2) Position of functional groups is more important than size of the functional groups to achieve the molecular recognition. 3) Chiral recognition requires stronger interaction and/or more interaction sites. References 1 Frank H. Dickey, J. Phys. Chem. 1955, 59, 695. 2 Borje Sellergren, ACS Symposium Series 703, 1998, 251. 3 Ha Seon Park, et al, J. Phys. Chem. B, 1999, 103, 2355. 4 Huaiqiu Shi, et al, Nature, 1999, 398, 593.

3:15 PM LL7.6
ELECTRICAL CONDUCTIVITY OF THE PIEZOELECTRIC STRONTIUM BISMUTH TITANATE UNDER CONTROLLED OXYGEN PARTIAL PRESSURE.
Cyril Voisard, Pedro Duran-Martin, Dragan Damjanovic, Nava Setter, Ceramics Laboratory, Materials Science Department, EPFL, Lausanne, SWITZERLAND.

Hysteresis free piezoelectric behavior of $SrBi_4Ti_4O_{15}$ (SrBIT) is very promising for precise sensors/actuators devices. Despite a quite low longitudinal piezoelectric coefficient (around 15 pC/N), its elevated ferroelectric phase transition temperature (540°C) make its use attractive at quite high temperature. Electrical conductivity at such temperatures should be kept as low as possible in order to avoid loss of piezoelectric properties or charge drifts. Under reducing conditions, however, the electrical conductivity may change significantly. The electrical conductivity of SrBIT has been measured under controlled oxygen partial pressure at elevated temperature (600-1000°C) from 1 atm down to 10^{-15} atm using a DC current method. At low temperature (100-220°C) the conductivities have been measured in air using a DC voltage method. Least square curve fittings were used to estimate the equilibrium conductivities in both methods. From 1 atm down to 10^{-5} atm pO_2, above 700°C, the conductivity of SrBIT exhibits a -1/4 slope in log-log scale indicating n-type conductivity and an impurity controlled oxygen vacancy concentration. A conductivity minimum is observed around 0.2 atm at 800°C. Acceptor doping (Mn) raises the minimum and flattens the conductivity curve with slope around -1/10 at 700°C, and -1/6 at 900°C. Ionic conductivity and defect ionization are discussed to account for this. Preliminary results indicate the possibility of a large, pO_2 independent, region, down to 10^{-15} atm pO_2. The ionic transport number was found to be 0.42 at 800°C for undoped SrBIT and 0.75 for Mn doped SrBIT. The activation energies of undoped (1.35 eV) and Mn doped (1.44 eV) samples are close to each other as expected for a common mechanism. Both acceptor (Mn) and donor (Nb) doping indicate a p-type conductivity at low temperature.

3:30 PM LL7.7
TRIBOLUMINESCENCE OF RARE-EARTH-DOPED ALUMINO-SILICATES AND ITS APPLICATION TO SENSING OF STRUCTURAL DAMAGE.
Katsuhisa Tanaka, Kyoto Institute of Technology, Dept of Chemistry and Materials Technology, Kyoto, JAPAN; Tsuguo Ishihara, Hyogo Prefectural Institute of Industrial Research, Kobe, JAPAN; Koji Fujita, Kazuyuki Hirao, Kyoto Univ, Dept of Material Chemistry, Kyoto, JAPAN.

The phenomenon that emission of photons is caused by fracture of a solid is known as triboluminescence and has been observed in many kinds of solids such as ionic crystals, semiconductors, metals, minerals, glasses, and organic crystals. This phenomenon is attracting considerable attention recently from a standpoint of application to sensing of structural damage and fracture. In such a sense, a solid which exhibits intense triboluminescence can be categorized as a smart material. In this study, we report successful observation of intense triboluminescence in rare-earth-doped aluminosilicates such as $SrAl_2Si_2O_8$ and $BaAl_2Si_2O_8$. A comparison between tribo-luminescence and photoluminescence spectra leads to the following conclusions. First, the triboluminescence is caused by the excitation of and emission from rare-earth ions doped in the crystals. Secondly, a discrepancy in the wavelength of maximum emission intensity between triboluminescence and photoluminescence spectra is observed for the 4f-5d transition, whereas the peak positions of triboluminescence and photoluminescence spectra are the same as each other for the 4f-4f transitions. For instance, although both triboluminescence and photoluminescence spectra of $BaAl_2Si_2O_8{:}Eu^{2+}$ manifest a very broad band due to the $4f^65d$-$4f^7$ transition of Eu^{2+}, the wavelength of maximum emission intensity is about 520 and 480 nm in the triboluminescence and photoluminescence spectra, respectively. In other words, the peak position of emission band is shifted to a longer wavelength side in the triboluminescence spectrum. In the case of triboluminescence, rare-earth ions located in the vicinity of the tip of a growing crack and/or on the fractured surface presumably contribute to the emission of photons. The difference in local environment around the rare-earth ions between on the fractured surface and within the bulk brings about the discrepancy between triboluminescence and photoluminescence spectra observed for the 4f-5d transition.

3:45 PM LL7.8
DE-ICING LAYERS OF INTERDIGITATED MICRO-ELECTRODES.
Zoe Courville, Victor F. Petrenko, Dartmouth College, Thayer School of Engineer, Hanover, NH.

We report here development of special active coatings that are capable to de-ice various solid surfaces vulnerable to icing such as airplane wings, helicopter blades, road signs, superstructures and hulls of ships etc. The coating consist of a thin web of metal interdigitated micro-electrodes formed using photolithography. The electrodes are made of very thin (5 micrometer) copper-clad or titanium-clad laminate on thin and flexible kapton substrate. Typical inter-electrode spacing and electrode width varied from 10 to 100 micrometers. After etching the electrodes were electroplated with Pt to increase their resistance to electro-corrosion. The de-icing action of the coating is based on the phenomenon of ice electrolysis [1,2]. Namely, when ice is growing over electrodes a small DC bias of 5 V to 25 V is applied to the electrodes generates DC current through the ice. The ice adjacent to the electrodes then decomposed in gaseous hydrogen (on the cathode) and gaseous oxygen (on the anode) thus eliminating bonding between the ice and the metal. Moreover, gas bubbles rapidly growing on the interface spread as interfacial cracks thus breaking ice. We present a theory of such active grids and describe their preparation procedure. We also demonstrate a video record of de-icing action of the coating. Results of mechanical and electrical tests obtained at different temperatures and on ice of different electrical conductivity will be shown. 1. V. F. Petrenko and N. N. Khusnatdinov, J. Chem. Phys., 100 (12), 9096 (1994). 2. V. F. Petrenko and S. Qi (1999) Reduction of ice adhesion to stainless steel by ice electrolysis, submitted for publication in J. Appl. Phys.

4:00 PM LL7.9
THE DESIGN OF ORGANIC GELATORS BASED ON A FAMILY OF BIS-UREAS. Rosa E. Melendez, Andrew J. Carr, Kazuki Sada and Andrew D. Hamilton, Dept of Chemistry, Yale University, New Haven, CT.

The use of organic molecules as gelators in certain organic solvents has been the target of recent research in materials science. The types of structures formed in the gel matrix have potential applications as porous solids that can be used as absorbents or in catalysis. We will present and discuss the organogelation properties of a family of bis-ureas. studies presented will include a molecule structure-activity relationship, thermodynamic properties, comparison to x-ray crystallographic data and potential functionalization of the gels formed by this class of compounds

4:15 PM LL7.10
PROCESSING AND CHARACTERIZATION OF COFIRED CAPACITOR AND VARISTOR CERAMICS. Barbara Malič, Marija Kosec, Jožef Stefan Institute, Ljubljana, SLOVENIA, Jošt Razinger, Zoran Živič, KEKO Varicon, Žužemberk, SLOVENIA.

The integration of passive ceramic electronic components consisting of various materials is a current topic in device development. One example is the integration of varistor and capacitor materials into monolithic elements offering high frequency and high amplitude transient voltage protection of electronic circuits. Cofiring two ceramic materials needs to consider different sintering intervals, shrinkages, thermal expansion coefficients and possible chemical reactions. The aim of the present work was to prepare a monolithic varistor-capacitor element. The starting powders were commercial zinc oxide based varistor and lead magnesium niobate based material. The respective powder compacts were prepared by uniaxial dry-pressing. The lateral shrinkages of the pellets were determined as functions of the compaction pressure, and of the sintering temperatures. Sandwich structured composite pellets were dry-pressed and sintered at the conditions that lead to shrinkages observed on the pellets of separately sintered materials. The microstructural analysis of the composite-pellets sintered at 950°C, 1 hour revealed that the microstructure of the capacitor ceramics remained essentially the same as in the separately sintered pellet of the capacitor ceramics. The microstructure of the varistor ceramics in the composite-pellet revealed increased porosity in the interface region as compared to the bulk of the same pellet or to separately sintered varistor ceramics. To obtain the electrical characteristics the composite-pellets were separated into their functional parts. The measured typical characteristics for the varistor and the capacitor are reduced as compared to the separate pellets, but are still adequate.

4:30 PM LL7.11
MAGNETOIMPEDANCE: A CHOICE PROPERTY OF SMART MATERIALS. K.L. Garcìa and R. Valenzuela, Institute for Materials Research, National University of Mexico, MEXICO.

When a metallic ferromagnet is submitted to a high-frequency electric current (typically in the 100kHz-10MHz range), its impedance response shows large variations as a function of the magnetic field applied on the material. This behavior is known as magneto-impedance(MI), and it has been shown (1) that (unlike magneto-resistance) it can be explained by classic electromagnetism, on the basis of the interaction between the magnetic field generated by the ac current and the domain structure of the ferromagnet. In this paper, we present a detailed study of magnetization processes that occur during MI in as-cast CoFeBSi amorphous wires obtained by the in-water-rotation technique. We show that magnetization processes such as domain wall bulging, domain wall displacements, and spin rotation can be distinguished by playing with the amplitud and frequency of the ac current. The variations in impedance response under a dc magnetic field are therefore associated with effects of such a field on the domain structure of the wire. The general behavior of magnetoimpedance can be synthesized in a simple equivalent circuit. Finally, some applications are describe.

4:45 PM LL7.12
STRUCTURAL CHANGES ON PHOTOCHROMISM OF ORGANIC-INORGANIC HYBRID MATERIALS. Keiichi Kuboyama, Kyoto Univ, Venture Business Laboratory, Kyoto, JAPAN; Kenji Ishida, Kazumi Matsushige, Kyoto Univ, Dept of Electronic Science and Engineering, Graduate School of Engineering, Kyoto, JAPAN.

Some transition metal oxides are known to exhibit the reversible coloration phenomena. Tungstic acid is one of such materials and exhibits the photochromism and the electrochromism, which can be applied to some devices such as a smart window, a memory and a display device. It is known that the coloration phenomena in the tungstic acid are caused by the redox reaction. We can obtain the tungstic acid by sol-gel process and it hardly exhibits the photochromism. However, we found that the photochromic efficiency became extremely higher by addition of some organic materials to the tungstic acid and we have studied the mechanism of such a remarkable photochromic enhancement. In some spectroscopic measurement as FTIR (Fourier transform infrared spectroscopy) and XPS (X-ray photoelectron spectroscopy), we obtained interesting features as follows. The addition of an organic material leads to reducing the tungstic acid to smaller pieces, that is, the surface area of the pieces that can react with the additive increases. Moreover, it was found that specific sites in the additive are oxidized when the sample colors. The fact suggests that the additives having such specific sites can enhance the photochromism of the tungstic acid.