

**SYMPOSIUM NN**  
**Materials for Space Applications**

November 29 - December 3, 2004

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\* Invited paper

## TUTORIAL

**FT NN: The Space Environment Effects on Materials, Components, and Devices**  
**Monday November 29, 2004**  
**9:00 AM - 12:00 PM**  
**Room 207 (Hynes)**

The tutorial will start with a description of the space environment. The nature of the radiation component of the space environment (main characteristics of electromagnetic radiations, particles type, energy, and flux) and the temperatures will be briefly discussed. The low Earth orbit and geostationary orbit environmental conditions will be explored in detail. Attention will be paid to the presence of atomic oxygen in low Earth orbits, to Van Allen radiation belts, to solar flares, and to cosmic radiation.

The second part of the tutorial will cover radiation-induced effects in materials. The main radiation-induced modifications in materials are critically reviewed. The focal issues of dosimetry, microdosimetry, and nanodosimetry will be briefly discussed. Synergetic effects resulting from the competition of different degradation processes (radiative, thermal, and oxidative degradation) will be analyzed in detail and exemplified with experimental data regarding the effect of the space environment on space materials. The limits of the on-Earth simulation of the space environment effects on materials will be critically reviewed. Particular attention will be paid to new emerging nanomaterials, with emphasis on polymer-based nanocomposites, multifunctional materials, gradient materials, and smart materials.

The last part of the tutorial will focus on the space environments effect on components and devices. Short-term and long-term modifications induced by ionizing radiation in different electronic components and devices will be briefly discussed.

### Instructors:

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SESSION NN1: Nanomaterials for Space Applications

Chair: Jeffrey Zaleski

Monday Afternoon, November 29, 2004

Room 207 (Hynes)

### 1:30 PM \*NN1.1

**Fabrication of Nanostructured Functional Materials for Space Applications.** Christopher R.M. Grovenor, John Topping, Zhibin Xie, Bernard Henry and Hazel Assender; Department of Materials, Oxford University, Oxford, United Kingdom.

We have been working in Oxford for some years on the design of processing strategies for thin film functional materials for photovoltaic, electrochromic and thermochromic devices, and for advanced permeation barriers. In each case, our key focus has been on how the processing conditions control film nanostructure and hence performance. As well as exploring the properties of oxide and oxynitride films deposited in conventional sputtering apparatus, we are also working on transferring this expertise to the operation of a commercial-scale 10m/sec web coater for large area processing of functional films on polymer substrates. This presentation will discuss some of the key achievements of this work, and comment on the critical technical issues that remain to be solved in this kind of materials fabrication programme.

### 2:00 PM NN1.2

**Polymer derived ceramic foams for light weight applications.**

Juergen Zeschky<sup>1</sup>, Michael Scheffler<sup>2</sup>, Thomas Hoefner<sup>1</sup>, Claudia Arnold<sup>1</sup>, Nahum Travitzky<sup>1</sup>, Peter Greil<sup>1</sup> and Rajendra K. Bordia<sup>2</sup>;  
<sup>1</sup>Department of Materials Science, Glass and Ceramics, University of Erlangen-Nuernberg, Erlangen, Germany; <sup>2</sup>Department of Materials Science and Engineering, University of Washington, Seattle, Washington.

Ceramic foams are a class of porous materials with unique physical, thermal, electrical and chemical properties. A novel foaming method demonstrated that the cell morphology and the microstructure of composite foams can easily be tailored by a molecular approach. Thermoset foams were manufactured from a specific

poly(silsesquioxane) as a preceramic polymer which was loaded with 30-70 wt. % of a blend of Si and SiC. The foaming process was carried out in the temperature range from 220 to 270 degree C. The driving force of the foam formation is a condensation reaction of hydroxy and alkoxy groups of the preceramic polymer activated at the foaming temperature. Simultaneously, a viscosity increase due to curing reactions stabilizes the foam structure. The morphology of the foam was controlled by the foaming temperature. Foaming at temperatures of 270-300 degree C resulted in isotropic foams with a narrow cell size distribution with average cell sizes ranging from 0.5 to 2.0 mm. Foaming temperatures of 220-240 degree C lead to a structure with gradient porosity with the porosity varying linearly from 90 % to 40 %. Concurrently, the structure changed from completely open celled to close celled. The transition is accompanied by a permeability drop from 300 D to 0 D, an exponential decay of the surface/volume ratio (16 mm<sup>-1</sup> to 2 mm<sup>-1</sup>) and the connectivity density (3.0 mm<sup>-3</sup> to 0.1 mm<sup>-3</sup>). The mean strut thickness rose from 0.2 to 1.0 mm, the cell diameter increased from 1.0 mm showing a spherical shape to 1.6 mm with a flattened shape. The thermal and mechanical properties were varied by changing the filler amount and the pyrolysis conditions. The electrical resistance was adjusted by pyrolysis temperature control between 1\*10<sup>14</sup> Wcm at 600 degree C and 1 Wcm at 1600 degree C. High strength ceramic foams with compression strength exceeding 4 MPa and exceptional reliability at a porosity of 75 % were obtained from foams with a filler load of 60 wt. %. The thermal conductivity ranged between 0.15 and 0.45 W/mK, the coefficient of thermal expansion was adjustable via filler load between 4 and 10 ppm/K. The thermal shock resistance was remarkably high reaching R' 1000 degree C.

### 2:15 PM \*NN1.3

**Materials Research for Space Exploration.** M. Wargo, Exploration Systems Mission Directorate, NASA Headquarters, Washington, District of Columbia.

A major challenge of NASA's exploration vision is the development of robust capabilities for long-term missions beyond low earth orbit. NASA's scientific advisory groups and internal mission studies have identified several enabling capabilities that require substantial advancements in materials science and materials technologies if these goals are to be accomplished in a safe, timely, and cost effective manner. For example: Crews must be protected from the severe radiation environment beyond the earth's atmosphere and magnetic field without an appreciable spacecraft mass penalty. Advances in chemical propulsion are predicated on materials improvements that provide greater efficiency and require less mass. The overall mass of the mission launch complement must be reduced by developing repair and fabrication techniques that use or recycle available materials. Significant materials related problems currently limit the development of nuclear systems for propulsion and other space applications. New materials research and development is recognized as being essential to success in these and other exploration related areas. NASA has engaged the US research community in developing the scientific foundations for many of these new technologies. Initial technical workshops held with representatives of the materials research community and with internal NASA customers have established the early roadmaps for this research and the first NASA Research Announcements have been issued. This is the beginning; the successful evolution of the program will depend on the scientific innovation of the materials research community.

### 2:45 PM NN1.4

**Irradiated Single Crystals for High Temperature**

**Measurements in Space Applications.** Alex A. Volinsky<sup>1</sup>, V.

Timoshenko<sup>2</sup>, V. Nikolaenko<sup>3</sup> and V. Morozov<sup>3</sup>; <sup>1</sup>Mechanical Engineering, University of South Florida, Tampa, Florida; <sup>2</sup>Russian Research Center, Kurchatov Institute, Moscow, Russian Federation; <sup>3</sup>Molnitya-T, Moscow, Russian Federation.

While space crafts experience temperatures from -120 to 110C on the orbit, their surface reaches extremely high temperatures, well above 1000C, during decent into the atmosphere due to aerodynamic heating. Sophisticated insulation systems are designed for thermal protection. One of the steps in designing a protection system is experimental temperature measurements. Neutron flux induces point defects formation and accumulation in diamond and SiC single crystals, which causes overall lattice expansion. During thermal annealing this process is reversed, so the annealing temperature and time result in the "reduced" lattice parameter (measured by X-Ray diffraction), which allows calculating the maximum temperature, if the exposure time is known, and visa versa. This paper describes the use of irradiated single crystal high temperature sensors for measuring temperatures in thermal protection systems during space craft descent, as well as other space applications. These additional applications include measuring the furnace temperature during single crystal growth in space at zero gravity, and measuring the rocket combustion chamber turbo pump temperature.

### 3:15 PM \*NN1.5

#### The Mechanical Properties of Thin Polymer Films.

F. R. Jones, Simon A. Hayes and Alexander A. Goruppa; Department of Engineering Materials, University of Sheffield, Sheffield, United Kingdom.

The degradation of thin polymer films is of major concern to designers of smart structures. However assessment of relevant properties is not so simple because the determination of mechanical properties is complicated by their time-dependent response. Dynamic analysis techniques, such as dynamic mechanical thermal analysis (DMTA) can be used to characterise the viscoelastic properties of bulk polymers but this is not applicable to thin films. Nanoindentation offers the possibility of determining the properties of thin films but has generally only been used to measure static properties. More recently dynamic nanoindentation with a heating stage has been developed. Most nanoindentation studies have concentrated on relatively hard materials because soft rubbery materials have a high specimen compliance and time-dependent characteristics, making the location of the surface with the indenter difficult. In this paper the performance of the dynamic nanoindenter technique has been validated using the principles of time-temperature superposition to generate mastercurves for a range of polymers. A Hysitron Triboscope nanoindenter, attached to a Digital Instruments Dimension 3100 AFM with depth sensing resolution of less than 0.1nm and load resolution of 10nN was used to apply an oscillatory load at a known frequency. A thermal stage developed jointly by Hysitron and Sheffield University which replaced the standard sample stage, provided thermal control in the range -15°C to +125°C. Thus forces of 150  $\mu$ N and 200  $\mu$ N static, with a 10  $\mu$ N oscillation at frequencies ranging from 10-100Hz were used at temperatures ranging from +10 to +60°C in 5 or 10°C intervals. A typical test would therefore be conducted isothermally at the selected temperature, while the frequency was ramped in steps of 2-5Hz from 10 to 100Hz. The data-sets were processed using the WLF equation to determine shift factors, and the curves overlaid to give a mastercurve. A range of standard polymers which could be examined as thin films or in bulk have been used to establish confidence in the technique. In addition, a number of polymers have been deposited from a range of monomers, using plasma polymerisation. Dynamic nanoindentation data in the form of a master curve was obtained for a polycyanurate using time-temperature superposition. The data was interpolated to provide a  $\tan\delta$  thermogram and a glass transition temperature. DMTA was used to record  $\tan\delta$  thermogram at a frequency of 1Hz. The DSC was also used to determine the glass transition temperature. It was shown that the maximum  $\tan\delta$  from dynamic nanoindentation and DMTA were very similar in intensity and breadth. However, the measured glass transition temperatures are slightly different, being 21°C from dynamic nanoindentation and 29°C from DMTA. These are compared with 19°C from DSC. A similar analysis for an epoxy resin achieved two identical  $\tan\delta$  curves. The relevance of this to the degradation of thin polymer films will be discussed.

### 3:45 PM NN1.6

#### Polymeric Materials for Space Sails: The Combined Effects of Polymer Thickness, Radiation, and Glass Transition.

Mircea Chipara<sup>1</sup>, David L. Edwards<sup>2</sup> and Mary Nehls<sup>2</sup>; <sup>1</sup>Indiana University Cyclotron Facility, Indiana University, Bloomington, Indiana; <sup>2</sup>NASA MSFC Marshall Space Flight Center, Huntsville, Alabama.

The solar sail project is an innovative low-thrust project that exploits the momentum transferred by solar photons to a large, highly reflective, low mass structure. This passive propulsion endeavor depends critically on the availability of lightweight materials with outstanding mechanic, thermal, electric, and optical capabilities. For such a drastic reduction of spacecraft weight, it is mandatory to focus on composites and nanocomposites based on polymeric matrices. Far and very far term missions are expected to last for several years, imposing the use of materials with a very long lifetime or of self-healing materials. These requirements are augmented by the harsh conditions of the space environment such as ionizing particles, extreme temperatures, IR, and UV radiations capable to trigger or to enhance degradation processes occurring in polymers and composites. To reduce the areal density of the solar sail is necessary to reduce the thickness of the polymeric film. If the thickness of the polymeric film is of the order of 100 nm or smaller, the glass transition temperature of the polymeric membrane starts to become a function of the polymeric film thickness, due to the confinement of the macromolecular chain (the radius of gyration of the polymer is comparable or larger than the film thickness). The polymeric materials considered for such applications have a high glass transition temperature. Under the effect of ionizing radiations (such as accelerated electrons, protons, and ions) these polymers are subjected to dominant degradation reactions that result in the decrease of the average molecular mass. This will decrease the glass transition

temperature of the polymeric film and will affect the radiation stability of the polymer. This decrease has to be added to the decrease in the glass transition temperature due to the confinement of the polymeric chain within the film. For particular distances and orientations of the solar sail relative to the Sun, the local temperature of the film may exceed the glass transition of the polymer resulting in the destruction of the polymer film. An analysis of the complex degradation of the ultra thin polymeric film subjected to the space environment is presented. The potential effects of the competition between polymeric chain confinement, radiation (ionizing) component of the space environment, film erosion, and temperature are tentatively analyzed within a unified description. The main limitations to the use of polymeric thin films (local temperature, radiation fluence) are discussed. Techniques to overcome these limitations are suggested and analyzed in detail.

### 4:00 PM \*NN1.7

#### Multiscale Phenomena in Bruggeman Composites.

Ralph Skomski, Jian Zhou and D. J. Sellmyer; Physics and Astronomy, University of Nebraska, Lincoln, Nebraska.

Many mechanical, electromagnetic, and other materials used in space applications are composites. Bruggeman's early work on dielectric composites [1] has led to the concept of an effective medium and been applied to a variety of systems. By analyzing the mathematical structure of the theory we obtain a generalized formulation that describes mechanical [2], transport, dielectric, and magnetic properties of composites. The theory can be used for a wide range of volume fractions and also yields percolation effects, although the long-range critical fluctuations near the percolation threshold are ignored and the critical exponents are mean-field like. An important point is that the theory is, essentially, a single-parameter approach. The parameter  $g$ , which is numerically equal to the percolation threshold, describes both the original materials parameter and the corresponding reciprocal (inverse) parameter, for example modulus and compliance (or resistivity and conductivity). Extraction from the literature and explicit calculation yields values of  $g$  for a variety of systems and geometries, such as dielectric and conductive materials, elastic and viscoelastic systems, heat conduction and diffusion, and demagnetizing fields. In general,  $g$  depends on the considered physical phenomenon and on the geometry and dimensionality of the composite. In cases such as diffusion, heat conduction, dielectric response and demagnetization,  $g = D$ , where  $D$  is the demagnetizing factor describing the considered geometry. However, the situation is slightly more complicated for mechanical properties, such as Young's modulus. A key limitation of the Bruggeman theory is that the underlying equations are macroscopic. For example, the elasticity of a fiber composite depends on the volume fraction of the fibers but is independent of the fiber radius. When specific length scales are involved, then there are nontrivial corrections to the Bruggeman behavior. Examples are Debye-Hückel corrections in liquids and exchange-length corrections in hard- and soft-magnetic materials. Focusing on the magnetic analogy [3], we analyze the structure of these corrections and show how they affect the materials properties of the composite. In the simplest case, the difference between the Bruggeman and non-Bruggeman regimes is analog to the difference between the Coulomb and Yukawa potentials in particle physics, and the corresponding Yukawa screening length limits the applicability of the Bruggeman theory. Finally, we briefly discuss some aspects of a Bruggeman theory of multifunctional materials with interactions involving two or more physical properties, such as magnetoelastic coupling. This work is supported by NSF-MRSEC, the W. M. Keck foundation, AFOSR and ARO. [1] D. A. G. Bruggeman, *Ann. Physik* **24**, 636 (1935). [2] R. M. Christensen, "Mechanics of Composite Materials", Wiley, New York 1979. [3] R. Skomski, *J. Magn. Magn. Mater.* **272-276**, 1476 (2004).

### 4:30 PM \*NN1.8

#### Materials Reactivity and Gas-Surface Collisional Energy Transfer in the Space Environment.

Steven J. Sibener, James Franck Institute and Department of Chemistry, University of Chicago, Chicago, Illinois.

The MURI Center for Materials Chemistry in the Space Environment seeks to elucidate the fundamental chemical and physical processes which are responsible for the degradation of materials in the space environment. This MURI also seeks to refine our understanding of hyperthermal gas-surface collisional energy and momentum transfer, supplying data needed to more accurately assess vehicle drag and orbital decay in Low-Earth-Orbit (LEO). This is a critical area of materials research under extreme conditions, where the physical and chemical environment aggressively induces materials oxidation and degradation due to the presence of, and synergisms between, highly-energetic atomic oxygen, a diversity of charged particles, and intense unfiltered, i.e., photochemistry inducing, solar illumination. Illustrative activities to date have: elucidated the mechanism by which graphite is eroded by high-kinetic energy atomic oxygen (this

work is presently being extended to include other carbon-based materials such as diamond); quantified the pathways by which organic molecules and films react with atomic oxygen in regimes not typically found in the terrestrial environment; discovered a new gas-surface collisional energy-transfer mechanism in which atoms become embedded and re-emitted from thin organic films with specific directionality and excess momentum; developed new theoretical methodologies for treating rare events which can dominate the morphological evolution of eroding interfaces; explored synergistic effects in polymer degradation involving atomic oxygen & radiation commensurate with solar illumination in LEO. Our ability to focus experimental scattering and scanning probe imaging measurements in concert with appropriate theory and simulation gives the necessary foundation to probe incisively the fundamental materials chemistry issues characteristic of this extreme environment. This talk will present research highlights in materials reactivity involving O(3P) atomic oxygen as well as recent discoveries in hyperthermal gas-surface collisional energy transfer. It is with great pleasure that I acknowledge my collaborators in these projects: Kenneth Nicholson, Kevin Gibson, Bill Isa, Amadou Cisse, Irene Popova, and Tim Minton. Theoretical calculations from Lance Kelly, John Tully, Tianying Yan and William Hase are also gratefully acknowledged.

SESSION NN2: Carbon Nanotubes and Composites  
Based on Carbon Nanotubes  
Chair: Kin Tak Lau  
Tuesday Morning, November 30, 2004  
Room 207 (Hynes)

#### 8:30 AM \*NN2.1

**Raman Spectroscopy for Carbon Based Amorphous Thin and Ultra-Thin Films.** Giuseppe Compagnini, dipartimento di chimica, catania, Italy.

Space science has always devoted interest into carbon materials for two main reasons. First of all DLC, diamond films, carbon fibers and, more recently fullerenes and nanotubes are considered materials appealing for aerospace applications. Secondly, since carbon materials are present in many astrophysical objects (comets, asteroids and interstellar dusts), it is interesting to study their formation and evolution under several outer space conditions through suitable experiments in the laboratories. In this frame vibrational spectroscopies and in particular Raman spectroscopy are known to be among the most useful tools to characterize and control the structural properties of the obtained carbon materials. The aim of this work is to present some experiments on the formation and evolution of carbon films obtained by energetic particle irradiation of solids targets and by deposition of unusual carbonaceous species onto suitable surfaces. Vibrational spectroscopy will be considered to give decisive contributions to the understanding of the structure of the obtained carbon materials such as the evaluation of defects states, the bonding formation and rearrangement and the role of foreign species. Some special topics related to the use of Raman spectroscopy for monolayer and sub-monolayer films will be given by discussing the so called Surface Enhanced Raman effect applied to amorphous carbon and organic molecules.

#### 9:00 AM NN2.2

**Vertically Aligned Carbon Nanotubes as the Sputter Resist in Space Propulsive Systems.** Yoke Khin Yap<sup>1</sup>, Vijaya Kayastha<sup>1</sup>, Jitendra Menda<sup>1</sup>, Lakshman Kumar Vanga<sup>1</sup>, Jiesheng Wang<sup>1</sup>, Alex Kieckhafer<sup>2</sup>, Dean Massey<sup>2</sup> and Lyon B. King<sup>2</sup>; <sup>1</sup>Physics, Michigan Technological University, Houghton, Michigan; <sup>2</sup>Mechanical Engineering-Engineering Mechanics, Michigan Technological University, Houghton, Michigan.

The space exploration program faces enormous challenges as it seeks to achieve dramatic improvements in safety, cost, and speed of missions to the frontiers of space. Plasma propulsion systems have been recognized as far more efficient than chemical thrusters. This recognition has led to the development of highly efficient electric propulsion (EP) thrusters that are currently the only feasible technology for many deep space missions. However, the lifetimes of these EP devices are limited by an electrode sputter erosion process. Inspired by their impressive stiffness, we have tested carbon nanotubes (CNTs) as the protective coating for space propulsive systems. We compared two types of CNTs to CVD diamond, amorphous carbon and boron nitride films as exposed to the exhaust beam of a flight-quality Hall-effect thruster. We found that only CVD diamond films and vertically aligned multiwall carbon nanotubes (VA-MWNTs) survived the erosion due to the 250 eV propellant ions. VA-MWNTs were shown to have higher resistance against ion erosion as compared to horizontally laid CNTs. This is consistent with the fact that CNTs are as stiff as diamond along the tubular axes. Detailed analysis by field emission scanning electron microscopy

(FESEM), backscattered electron (BSE) imaging, and Raman spectroscopy indicate that these VA-MWNTs were bundled at their tips before the erosion. An erosion mechanism was then formulated and verified by a series of experiments with Xe propellant. The ion current density on the samples was 5 mA/cm<sup>2</sup>. Two types of VA-MWNTs were evaluated concurrently: ultra high density (>10<sup>10</sup> cm<sup>-2</sup>) VA-MWNTs grown by thermal CVD and high density (>10<sup>8</sup> cm<sup>-2</sup>) VA-MWNTs grown by a dual-RF-plasma enhanced CVD techniques. Despite the density difference, all the VA-MWNTs bundled together, which is explained by the surface tension initiated by the vaporization of the catalyst particles at the tips of the nanotubes. The bundling of MWNTs alone does not lead to significant erosion in the first 20 minutes of ion irradiation. However, drastic erosion (0.8 μm/min) was detected within the next 10 minutes. During this time frame, these catalyst particles melted, flushed downward and coated the MWNTs and are responsible for the fast erosion rate. We think that this is a solvent-assisted erosion process. We propose that removal of catalytic nanoparticles from the tips of VA-MWNTs could enhance their resistance against ion erosion in advanced propulsive systems. As viewed from their fast growth rate at relatively low temperatures (as high as 300 μm / hour at 650 °C in our experiment), VA-MWNTs without catalyst particles are very prospective protective coatings for advanced space propulsive systems.

#### 9:15 AM \*NN2.3

**Spectroscopic Analysis of Structural Parameters of Nanocomposites and Porphyrine Liquid Crystals for Space Applications.** J. M. Zaleski<sup>1</sup>, David F. Dye<sup>1</sup>, Brian D. Pate<sup>1</sup>, Kristina Stephenson<sup>1</sup> and Malcolm H. Chisholm<sup>2</sup>; <sup>1</sup>Department of Chemistry, Indiana University, Bloomington, Indiana; <sup>2</sup>Dept. of Chemistry, The Ohio State University, Columbus, Ohio.

Space applications require lightweight materials possessing multifunctional capabilities such as magneto-optical switching, high mechanical strength, and thermal stability. Lightweight materials with specific physical properties are extremely important to reducing mission costs while maintaining effectiveness. Polymeric materials are ideal matrices for such materials because of their enhanced strength to weight ratios. However, they often lack thermal and mechanical stability due to the simplicity of their 1-dimensional architectures. Additionally, they are frequently susceptible to the harshness of the space environment, especially to radiation-induced damage. For magneto-optical switching applications, enhanced molecular flexibility and structural responsiveness are desirable physical properties. Due to their stunning physical characteristics (mechanical strength, anisotropic electrical and thermal conductivities, and high aspect ratio), carbon nanotubes (CNTs) are perceived as excellent fillers to enhance the properties of polymeric matrices. Above the percolation threshold (0.5 weight %), these composites exhibit conducting features. For CNTs, the details of electron confinement, spin-spin, and electron-lattice (adhesion) interactions can be obtained from electron spin resonance (ESR) lineshape analysis. The ESR line shape is sensitive to the length of the nanotube and concentration. For long CNTs, the increase in the ESR signal (i.e. number of detected spins) is non-linear, indicating that the entire conducting domain is not being effectively interrogated. Similarly, the mesoscopic structure of liquid crystals is also governed by subtle molecular interactions. Magnetic alignment and optical switching of the microscopic structure is directly related to the electronic and chemical properties of the individual molecular building blocks. Strongly absorbing, paramagnetic (Cu<sup>2+</sup>, Co<sup>2+</sup>) and diamagnetic (Ni<sup>2+</sup>, Zn<sup>2+</sup>) porphyrine liquid crystals exhibit broad mesophases that can be magnetically aligned upon warming to their clearing temperatures and cooling in a magnetic field. The threshold temperature is sensitive to the nature of the central metal ion, revealing a competition between the paramagnetic and diamagnetic contributions to the molecular magnetic moment. Correlated solid state and mesophase <sup>13</sup>C NMR studies on the Ni and Zn complexes indicate time-averaged disk tilt angles of 27 deg. with respect to the columnar directors in the liquid crystal phase. Finally, Raman spectra of these compounds reveals a dependence of linewidth and Raman shift on the phase of the material. A detailed temperature dependence of the vibrational signatures of the porphyrine backbone are used to report the phase of the material. Moreover, the strong extinction of the macrocycle allows laser-induced phase transitions to be initiated with very low excitation powers (< 2 mW at 785 nm), providing an avenue to potential magneto-optical switches.

#### 9:45 AM NN2.4

**Advanced Life Support for Space Exploration: Air Revitalization using Amine Coated Single Wall Carbon Nanotubes.** Padraig Moloney<sup>1</sup>, Chad Huffman<sup>2</sup>, Olga Gorelik<sup>2</sup>, Pasha Nikolaev<sup>2</sup>, Sivaram Arepalli<sup>2</sup>, Michael Waid<sup>1</sup> and Leonard Yowell<sup>1</sup>; <sup>1</sup>NASA Johnson Space Center, Houston, Texas; <sup>2</sup>GBTech Inc., Houston, Texas.

The challenges posed by long duration human space flight have made

regenerable air revitalization a critical technology. Current systems using disposable lithium hydroxide do not address the difficulties presented by long duration missions. Solid amine systems offer the capability to regeneratively absorb CO<sub>2</sub> using an amine-impregnated porous substrate. Desorption of CO<sub>2</sub> is then achieved by exposing the system to vacuum or by increasing temperature. However, thermal inefficiencies and system size constraints prevent adoption of regenerable systems on current and future space vehicles. A key challenge is the thermal management of the absorbing bed. The absorbing surface increases in temperature which reduces absorbing efficiency. The removal of CO<sub>2</sub> reduces temperature, which in turn produces a loss in regeneration efficiency. These thermal inefficiencies necessitate prohibitively large volumes of traditional solid-amine materials, which do not have optimized surface areas and pore distributions. Single-wall carbon nanotubes (SWCNTs) may provide a means to increase surface area of the amine support and thermal efficiency. Recent work by Cinke et al. provided a method of functionalizing SWCNTs and increasing the surface area to the order of 1500 m<sup>2</sup>/g [1]. We will report on the production of free standing, high surface area carbon nanotube structures currently being impregnated with amines. This novel SWCNT/amine approach will be compared with the current state of the art polymer structure-based system and characterized using SEM, TEM, surface area analysis through BET, and also thermogravimetric equilibrium absorption. Results of SWCNT material improvements from processing modifications, including freeze-drying and vacuum sublimation, will also be presented. [1]Martin Cinke et al, Chemical Physics Letters 365 (2002) 69-74

#### 10:00 AM NN2.5

**Nondestructive Damage Sensing and Load Transfer Mechanisms of Functionalized Carbon Nanotube and Nanofiber/Epoxy Composites Using Electro-Micromechanical Technique.** Joung-Man Park, Dae-Sik Kim, Jae-Rock Lee and Tae-Wook Kim; Dept of Polymer Science & Engineering, Gyeongsang National University, Jinju, Gyeongnam, South Korea.

Carbon nanotube (CNT) and nanofiber (CNF) reinforced polymeric matrix composites have been recently intensively investigated in the research and industrial field due to their unique mechanical and attractive electrical properties. Carbon nanocomposites have high stiffness, strength and good electrical conductivity at relatively low concentrations of reinforcing materials. Electrical and mechanical properties of carbon nanomaterials (CNM) reinforced polymer composites depend on many factors such as inherent properties of CNM, the degree of dispersion, orientation, interfacial adhesion, aspect ratio, fiber shape and content, etc. Especially, the degree of dispersion is well known as one of the most important factors in electrical and mechanical properties. In this work, electro-micromechanical techniques were applied using four-probe method for carbon fiber/epoxy-CNT or CNF composites with their content and functionalization condition. Carbon black (CB) was used to compare to CNT and CNF. To improve the degree of dispersion and interfacial adhesion, CNM were functionalized by dilute nitric acid. The fracture of carbon fiber was detected by nondestructive acoustic emission (AE) relating to electrical resistivity by double-matrix composites test. Sensing for fiber tension was performed by electro-pullout test under cyclic strain. The results obtained from fiber damage sensing were correlated with the morphological observation of nano-scale structure using FE-SEM. Homogeneous dispersion and interfacial interaction between CNM and epoxy matrix could be one of keying parameters for better damage monitoring as well as mechanical performance. Damage sensing and reinforcing effect of carbon nanocomposites could be evaluated well nondestructively by the electrical resistance measurement with AE. Acknowledgement: This work was financially supported from CNMT of the 21st C Frontier R&D program by Ministry of Science and Technology (MOST), through Engineering Research Institute (ERI), Gyeongsang National University, KOREA.

#### 10:30 AM \*NN2.6

**Super-Tough Performance of Modified Carbon Nanofiber (MCNF)/UHMWPE Nanocomposite Films.** Xuming Chen, Kyunghwan Yoon, Christian Burger, Igors Sics, Benjamin Chu and Benjamin S. Hsiao; Chemistry, SUNY-Stony Brook, Stony Brook, New York.

Improvements of mechanical toughness on high performance polymeric materials have been the focus of scientific and industrial research for the past several decades. In this study, we aimed to improve the toughness of ultrahigh molecular weight polyethylene (UHMWPE) films, which have already exhibited excellent mechanical properties, by incorporating modified carbon nanofibers (MCNFs) through sequential solution blending and melt pressing. The surface modification of CNF consisted of two synthetic steps: oxidation and chemical reaction with octadecylamine. The MCNF was characterized by using TGA and showed approximately 30% of conversion from

carboxylic acid groups to octadecylamide groups. The surface modification of carbon nanofiber with octadecyl groups (short hydrocarbon chains) significantly facilitates the dispersion of MCNF in UHMWPE by melt mixing. The optimal toughness improvement occurs at a low fraction of MCNF incorporation. The composite film with only a small amount of MCNF (e.g. 0.2 wt%) showed a significant improvement on the elongation to break ratio and thus the toughness. The plastic flow at the MCNF/UHMWPE interface may be induced by the attached oligomeric groups on the surface of carbon nanofiber, which served as a solvent to soften the UHMWPE chains and reduce the entanglement density at the fiber interface. In-situ synchrotron WAXD showed that the higher the MCNF content, the more plastic flow in the amorphous region, which is consistent with the mechanical properties.

#### 11:00 AM NN2.7

**Sensing and Actuating Characteristics of SWNT-Electroactive Polymer Nanocomposites for Space Applications.** Joycelyn S. Harrison<sup>1</sup>, Nancy M. Holloway<sup>1</sup>, Purnell Hopson Jr.<sup>1</sup>, Ralph M. Stephens<sup>1</sup>, Sharon E. Lowther<sup>1</sup>, Zoubeida Ounaies<sup>2</sup>, Jinho Kang<sup>3</sup>, Kristopher E. Wise<sup>3</sup> and Cheol Park<sup>3</sup>; <sup>1</sup>Advanced Materials and Processing Branch, NASA Langley Research Center, Hampton, Virginia; <sup>2</sup>Mechanical Engineering, Virginia Commonwealth University, Richmond, Virginia; <sup>3</sup>National Institution of Aerospace, Hampton, Virginia.

Structures and materials in space are exposed to a number of harsh environmental conditions including high-energy protons and electrons, ultraviolet radiation, atomic oxygen, high and low temperature extremes, hard vacuum, galactic cosmic radiation, micrometeoroids, and man-made debris. Because state-of-the-art terrestrial materials often fail to perform due to premature degradation of their functionalities after prolonged exposure in space, developing novel materials which can survive in a space environment while maintaining their functions is essential for successful long-term space missions. A series of high performance electroactive polymers (EAP) were developed for use as sensing/actuating components in future space vehicles and astronaut suits. Single wall carbon nanotubes (SWNTs) were incorporated to form a composite material in hopes of enhancing the multifunctionality of these polymers while maintaining or improving their already impressive mechanical, thermal, and electronic characteristics. In this presentation, sensing and actuating characteristics of SWNT-EAP nanocomposites are reported. Understanding the electrical and dielectric properties of these nanocomposites is critical to future efforts aimed at optimizing their capabilities for specific applications. Sensing properties of the nanocomposites were explored as a function of strain, stress, pressure, and temperature. Actuation was demonstrated by the large displacements observed in the nanocomposites at low applied electric fields relative to commercial actuating materials such as PZT and PVDF. Based on the results of analytical modeling and numerical simulation, the origin of sensing and actuation responses of the SWNT-EAP nanocomposites will be addressed. These SWNT-EAP nanocomposites are scheduled to be installed at the International Space Station (ISS) in the MISSE (Materials International Space Station Experiment) 3 and 5 experiments, which were designed to characterize the performance of new prospective spacecraft materials when subjected to the combined deleterious effects of the space environment. These nanocomposites will be retrieved for further evaluation after several years' exposure in space.

#### 11:15 AM NN2.8

**Synthesis, Characterization, and Modeling of Nanotube Materials with Variable Stiffness Linkers.** S. J. V. Frankland<sup>1</sup>, M. N. Herzog<sup>2</sup>, G. M. Odegard<sup>3,1</sup>, H. M. Herring<sup>4</sup>, T. S. Gates<sup>5</sup> and C. C. Fay<sup>5</sup>; <sup>1</sup>National Institute of Aerospace, Hampton, Virginia; <sup>2</sup>National Research Council, Hampton, Virginia; <sup>3</sup>Mechanical Engineering-Engineering Mechanics, Michigan Technological University, Houghton, Michigan; <sup>4</sup>Lockheed Martin Space Operations, Hampton, Virginia; <sup>5</sup>NASA Langley Research Center, Hampton, Virginia.

Novel single-walled carbon nanotube (SWNT) materials are being developed at NASA Langley Research Center as durable, light-weight materials for aerospace applications. In the present work, the SWNT were randomly tethered with a di-functional, variable stiffness linker using diazonium chemistry. This functionalization was confirmed using Raman spectroscopy, and the amount of SWNT incorporation was determined using thermogravimetric analysis in an argon atmosphere. After creating a thermal compression mold of the material, the room temperature storage modulus was determined as a function of depth and frequency via nanoindentation. These results were then compared with a detailed model of the constitutive properties of the material. The model development occurred in several stages. First, an atomistic model of the variable stiffness tether was developed using input from ab initio calculations to customize the force field. Then molecular dynamics simulation, equivalent

continuum modeling, and micromechanics were used to determine the constitutive properties of the material as a function of nanotube concentration and functionalization. The paper will present results from the synthesis and experimental characterization of the material along with model predictions for its mechanical behavior.

**11:30 AM \*NN2.9**

**Enhancement on the Mechanical Strength of Advanced Composites with Nano-structural Materials.** Kin-tak Lau, Mechanical Engineering, The Hong Kong Polytechnic University, Hong Kong SAR, Hong Kong.

Since the discovery of carbon nanotubes a decade ago, many substantial works related to these nano-structural materials in different scientific and engineering aspects have emerged dramatically. The extraordinary mechanical, electrical and thermal properties of the nanotubes are governed by their atomic architectures, commonly called chiral arrangement. Ideally, all carbon atoms in the nanotubes are covalently bonded and formed repeated close-packed hexagonal structures in each layer of shell(s). Due to these naturally chemically-formed atomic arrangements, the carbon nanotubes possess superior mechanical properties, which are stronger than any kind of known metallic materials in the World. Many critical results have been reported recently by using nanotubes as an atomic force microscope (AFM) probe, conductive devices in artificial muscles, nano-thermometer and fuel cells. Although all these works are still under research stage, it demonstrates that it is a high potential in the development of nanotube-related products and components in real-world applications. In the past few years, many researchers and engineers from the advanced composite community has attempted to use these nano-structural materials to enhance the properties of conventional advanced composite structures by altering their mechanical, thermal and electro-static behaviors for space and infrastructure applications. However, in order to achieve these goals, certain aspects such as (1) understanding the mechanical properties of both singlewalled and multiwalled types of carbon nanotubes; (2) investigating appropriate fabricating processes of nanotube/polymer composites; (3) clarifying the interfacial bonding properties between the nanotubes and surrounding matrix and (4) justifying the benefit on the strength of composites after mixing with the nanotubes. This paper provides a critical review on the above aspects basing on the works recently done by the authors and other researchers. All these aspects cannot be considered individually in the development of nanotube/polymer composites. A detailed discussion on each of these aspects and how to co-link to others is also addressed.

SESSION NN3: Dosimetry and Nanodosimetry  
Chair: Richard Wilkins  
Tuesday Afternoon, November 30, 2004  
Room 207 (Hynes)

**1:30 PM \*NN3.1**

**Space Radiation Effects on Electronics: Simple Concepts and New Challenges.** Kenneth A. LaBel, NASA/GSFC, Greenbelt, Maryland.

Research on Space Radiation Effects on Electronic Systems is applying science concepts (physics) to engineering applications. In this talk, I will present: - A brief review of the space radiation environment as it applies to electronics focusing on near-earth and free-space environments, - The effects of concern for electronic systems ranging from single energetic particle interactions to long-term degradation of device performance, - Examples of the environmental interaction with in-flight electronic systems including commercial electronics responses to solar events and variance with the solar cycle, - A NASA perspective on utilization of commercial electronics from mission requirements to proactive research efforts, and, - A first-hand prognostication of the future of electronics and radiation effects and tolerance including new efforts to utilize commercial semiconductor foundries to provide radiation tolerant products. A final portion of the talk will cover some of the recent issues plaguing the aerospace community with regards to radiation effects as well as discuss the role of space validation for radiation test protocols and predictive techniques. It should be pointed out that when discussing commercial electronics, only a small minority of semiconductor manufacturers design and develop products built for performance in the space radiation environment. The remainder of these companies is focused on the terrestrial markets dominated by handheld electronics, tabletop computing, and gaming. It is a law of economy of scale: sell a million devices to Sony or 100 to NASA. However, the electrical performance (think speed of a processor) of these commercial devices is two or more generations faster than the best device designed for the space radiation environment specifically. This is the daily challenge of designing electronics for space systems: higher performance using commercial components with uncertain

radiation response (the possibility of lost data or mission failure), the complexity of mitigating those radiation responses, or utilization of the lower electrically performing radiation hardened devices. This trade space will be interleaved throughout this talk.

**2:00 PM NN3.2**

**Working Group 4 of the International Standardization Organization - Experience of Developing Standards in the Space Environment Field.** Mikhail Igorevich Panasyuk, Skobeltsyn Institute of Nuclear Physics of Moscow State University, Moscow, Russian Federation.

During the past several years space environment standards are being developed in scope of ISO WG4 activities. This is the first attempt to develop international standards of physical phenomena of the natural environment such as: galactic cosmic rays, solar energetic particles, the magnetic field of the Earth, the ionosphere, the plasmasphere, solar irradiations, upper atmosphere etc. The major difficulties associated with the development of such standards is the absence of widely recognized for many natural phenomena. However, in the case of successful development, such standards can play an outstanding role in the engineering estimates, required for spacecraft designing. This report will contain an overview of the current status of the standards being developed in scope of the WG4 activity plan.

**2:15 PM NN3.3**

**Simulation of Absorbed Dose Distribution in Space Materials.** Boris Aronovich Briskman, Radiation Material Science, Karpov Institute of Physical Chemistry, Obninsk, Kaluga region, Russian Federation.

The problems of absorbed dose distribution simulation at on-ground space material tests are discussed. Several approaches to such simulation directed to increase of the test adequacy and economy are analyzed. Sometimes, it is possible to use quantitative criteria of absorbed dose distribution depending on the specific space vehicle orbit. The assessment of reliable simulation of the radiation spectrum may be made, for example, by introducing a special numerical characteristic of the depth dose profile in a material - depth dose criterion. For this purpose, it is recommended to use the ratio of the exponent index of the depth dose profile ( $m$ ) to the density of the material ( $r$ ). In the simplest form, the depth dose profile can be represented as a sum of two exponents. The first depth dose profile applies to a near-the-surface layer of 5 to 10 mcm in thickness, and the second to a layer of from 10 mcm up to, as a minimum, 100 mcm in thickness. The reference values of  $m/r$ , calculated for standard spectra of ionizing radiation, are calculated. For simulation, depth dose profile for the tested material should be calculated for both the space environment and test conditions. It is necessary to perform the calculations using the same mathematical code and the same geometry of particle incidence for both the space and simulated space radiation environments. The depth dose profiles shall be presented in the form of two exponents. The next step is to find the values of depth distribution criterion for both types of conditions and then to adjust the values of  $m/r$  varying the radiation source energy and the particle fluences. Permissible difference between the depth dose profile criteria for orbit flight and ground test is a complex function of material properties, values of absorbed dose, and dose rate. For optical properties, for example, in a majority of nonmetallic materials, a linear response results except at high doses, and this response in broad range of dose rates with irradiation in vacuum changes not more than two times. The recommended permissible difference between the  $m/r$  values for the adjustment process is about 30%.

**2:30 PM \*NN3.4**

**Moscow State University Activities on Research of the Space Environment Effect on Materials.** Lev Novikov, Nuclear and Space Research, Skobeltsyn Institute of Nuclear Physics, Moscow State University, Moscow, Russian Federation.

Main areas of spacecraft-space environment interaction research in Moscow State University are following: - effect of the space radiation on spacecraft materials and equipment; - spacecraft charging in the space plasma in various orbits; - influence of atomic oxygen flux on materials in LEO; - surface erosion and destruction under impact of micrometeoroids and small particles of the space debris. The brief description of accelerators of ions, electrons, hard microparticles and the oxygen plasma, used in Skobeltsyn Institute of Nuclear Physics Moscow State University (SINP MSU) for ground-based testing of the spacecraft materials and equipment elements is given. The accelerators used provide the energy range of electrons an ions 0.1-30 MeV. The electrostatic accelerator of the hard particles provides velocities for metal particles (Al, Cr, Fe, Ni, Cu, Mo, W etc.) with diameter 0.2-5.0 micrometers from 0.1 up to 30 km/s. Plasmadynamic accelerator gives the beam of the oxygen neutral atoms and ions with energy 5-50 eV. Mathematical models of the spacecraft charging in GEO and LEO, and models for 3D calculation of distribution of the

space radiation absorbed dose developed in SINP MSU are presented. Results of tests of various materials (thermal control coatings, optical glasses, polymers etc.) are discussed. Some experiments onboard the MIR station and ISS are described. Principle problems for contemporary and future research are proposed for discussion.

### 3:00 PM **NN3.5**

**Near-Earth Radiation Environment During Solar Extreme Events.** Mikhail Igorevich Panasyuk, Skobeltsyn Institute of Nuclear Physics of Moscow State University, Moscow, Russian Federation.

The main components of the radiation environment, surrounding the Earth are: galactic cosmic rays, solar energetic particles and the radiation belts. All these components are subject to studies from the point of view of radiation impact on spacecraft material and elements. Galactic cosmic rays which mainly consist of protons and nuclei and especially their highly ionizing component (heavy nuclei) are the predominant source of radiation damages in condensed matter at micro-volume level, causing such malfunctions in electronic devices such as latch-ups, single event upsets and etc. Energetic particles generated in solar flares and coronal mass ejections can also lead to serious radiation hazards. During periods of powerful solar events, lasting several days and accompanied by magnetic storms, the most highly energetic solar particles can overcome the barrier, induced by the magnetic field, reaching low altitudes. In this case, the radiation doses from solar particles can significantly exceed the corresponding doses from trapped particles. The solar energetic particles exhibit a long-term variation associated with the 11-year solar activity cycle. The physical interpretation and model representation of this variation is far from being complete. The fluxes of particles in the radiation belts are the highly dynamical. Temporal variations of the particle fluxes (mainly electrons and protons) undergo intensity variations on time scales from about 1 minute to several years. Rapid variations, generally, are associated, with the generation of electric fields and electromagnetic emissions during substorm activity, while slow variations are induced by variations of solar activity and, as a consequence, by variations of particle transport velocities and loss rates inside the radiation belts. No comprehensive engineering model of the radiation belts that would adequately describe their spatial and dynamic properties is yet available. Radiation hazard for space vehicles in near-Earth space is caused by a number of factors among which, besides the time of exposure to the radiation environment, the most significant are the orbital parameters of satellites, as well as the levels of solar and geomagnetic activities leading to radiation flux enhancement. This report devoted to near-Earth radiation environment effects during mainly solar extreme events.

### 3:30 PM **\*NN3.6**

**MeV Ion Beam Modification Polymers.** Daryush Ila<sup>1</sup>, Robert L. Zimmerman<sup>2</sup>, A. Leslie Evelyn<sup>1</sup>, Gwyn M. Jenkins<sup>1</sup>, David B. Paker<sup>3</sup>, Dale K. Hensley<sup>3</sup> and Cydale C. Smith<sup>4</sup>; <sup>1</sup>Physics, Alabama A&M University, Normal, Alabama; <sup>2</sup>University of Sao Paulo, Sao Paulo, Brazil; <sup>3</sup>Oak Ridge National Laboratory, Oak Ridge, Tennessee; <sup>4</sup>NASA - Marshall Space Flight Center, Huntsville, Alabama.

The effect of MeV ion beams in their passage through various polymers will be discussed and compared to similar effects produced by Pyrolysis. Certain chemical species such as sulfur, chlorine, and other pendant and ligand molecules contributes to the cross linking of chains. The effects of these cross-linking agents during Pyrolysis process have been studied by polymer scientists and by chemists for many years. The effects of these cross linking agents on the ion beam modification processes are of interest to us. We will highlight the structure of certain polymer chains, such as partially cured resin, Polyethylene (PE), polyethersulfone (PES), polyvinyl chloride (PVC) and polystyrene (PS), before and after bombardment by MeV ions and observe change in their electrical, optical, and chemical structure. A direct comparison of the properties of these polymers before and after exposure to MeV ion beam with the before and after thermal annealing (Pyrolysis) provides us with a conclusion that the energy transferred by MeV ions in their track, due to the electronic excitation (electronic stopping power), may be considered a soft effect and is similar to that of thermal annealing of polymers.

### 4:00 PM **NN3.7**

**Nanodosimetry: Present and perspectives.** Mircea Chipara, Indiana University Cyclotron Facility, Indiana University, Bloomington, Indiana.

More and more contributions concerning the synthesis and characterization of materials, confined, structured, or possessing interfaces at nanometer scale are reported. Some of these materials are changing already our life while many others are expected to be used in a wide variety of applications in the near future. Nanomaterials explosion is anticipated to change the fundamentals of radiation dosimetry. There is a general perception that the size reduction to nanometer scale will imply a significant increase of the

radiation sensitivity of the material. The mechanisms of radiation transfer from the incident radiation to the nanometer sized features and mainly the processes involved in the delocalization and confinement of the deposited energy within these features are not yet fully understood. The theoretical point of view supports the idea that the development of a new theoretical framework such as nanodosimetry is pointless as long as the models that describe the interaction of ionizing radiation with the matter are valid even at sub nanometer scale. These models represent the core of dosimetry. The extension of dosimetry to micron sized targets and the subsequent development of microdosimetry did not affect the basic principles of energy transfer from the incident radiation to the target or of energy dissipation within the target. Accordingly, a thorough investigation of radiation effects in nanomaterials is required to assess their radiation stability, to firmly establish the theoretical background, and to decide about the future of nanomaterials in harsh environments such as the space environment. An important step in the theoretical modeling of radiation-induced modifications in nanomaterials is an accurate and precise theory concerning the interaction of ionizing radiation with nanomaterials. Such a theory - named nanodosimetry - will allow us to predict and simulate the behavior of nanomaterials in extreme radiation environments. The very first step, representing the transition from macroscopic dosimetry to microdosimetry was successfully accomplished in the last two decades. The main theoretical ideas and experimental facts that would eventually trigger the development of nanodosimetry are still accumulating. This contribution will critically review the most important limits of dosimetry and microdosimetry generated by the nanometer size confinement of the target. Particular attention will be paid to the latest theoretical and experimental results that will be eventually embedded in nanodosimetry. The main limits in the development of a nanodosimetry theory are analyzed and the need for such a theoretical development of dosimetry is seriously reviewed.

### 4:15 PM **NN3.8**

**Total dose radiation effects in Si nanocrystal non-volatile memory transistors.** Mihail P. Petkov<sup>1</sup>, L. Douglas Bell<sup>1</sup>, Robert J. Walters<sup>2</sup> and Harry A. Atwater<sup>2</sup>; <sup>1</sup>Jet Propulsion Laboratory, Pasadena, California; <sup>2</sup>T.J.Watson Laboratory, California Institute of Technology, Pasadena, California.

We report results pertinent to the high tolerance of prototype Si nanocrystal (nc-Si) field effect transistors (FET) to ionizing radiation. Nc-Si crystals were formed in a thin SiO<sub>2</sub> blanket layer on a Si wafer by Si implantation and subsequent high-temperature anneal. This structure was patterned into ring-gate FETs with different width-to-length ratio of the gate. The active part of the gate contained large number of nc-Si domains, on each of which charge can be deposited by suitable electric fields. This technology acts as conventional flash non-volatile memory (NVM), with the added novelty of statistically assessing bit status (open/closed FET). It is believed that the radiation-induced loss of charge on a large fraction of the crystals may not necessarily lead to information loss (bit flip causing soft error). Yet, studies of the nc-Si radiation tolerance are scarce to-date. We monitored the FETs Ids-Vg characteristics, which exhibit a hysteresis that is the signature of a memory effect. An intense <sup>60</sup>Co gamma-source, whose Compton-scattered electrons created the ionizing dose, was used to emulate high radiation environments. Devices were irradiated to different doses of up to 15 Mrad(Si) with a dose rate of 25 rad/s. During the irradiation, the device terminals were either grounded, or a square wave 'write'/'erase' potential was applied to the gate. Changes in the hysteresis due to the cumulative dose were expressed in terms of changes in the gate threshold voltages for both the 'on' and 'off' states of the FETs. A comparison was made with conventional FETs with identical structure, shape and dimensions, but does not contain Si nanocrystals. While the conventional FETs exhibit shift in the threshold voltage for up to 2 Mrad(Si) the nc-Si FETs show no significant functional changes. Preliminary photoluminescence measurements have also confirmed that the nc-Si domains remain unaffected up to at least 1 Mrad(Si) cumulative dose. Radiation-induced defects, which retained charge for at least several hours, were also registered, although the 'write'/'erase' gate potential reinstated the initial pre-radiation state. The density of these charge traps appeared to saturate at 10 Mrad(Si).

### 4:30 PM **\*NN3.9**

**Radiation-Induced Modifications in Polymeric Materials Subjected to Ionizing Radiation.** D. Hui<sup>1</sup> and Magdalena D. Chipara<sup>2</sup>; <sup>1</sup>Department of Mechanical Engineering, University of New Orleans, New Orleans, Louisiana; <sup>2</sup>Indiana University, Bloomington, Indiana.

Polymeric materials present an particular importance for space applications due to their reduced weight and good mechanical properties. Nevertheless, the use of polymeric materials for space applications is restricted by the harsh conditions of the space

environment. The complex space environment implies at least extreme temperatures, ionizing radiation, low gravity, and micrometeorites. The present lecture is restricted to the effect of ionizing radiation on the physical properties of polymers. Whenever possible, the combined action of ionizing radiation and temperature is analyzed. The main long-term effect of the interaction of ionizing radiation with polymeric materials consists in the change of the average molecular mass and distribution. If cross-linking reactions are dominant, the average molecular mass is increased by irradiation while if chain scissions reactions are dominant the average molecular mass is decrease by irradiation. These changes shift the glass transition temperature of the polymer to a higher value (dominant cross-linking reactions) or to a lower value (dominant chain-scission reactions). The presence of gases may affect significantly the degradation of polymeric materials. The polymeric chain is sensitive to oxygen attack. In Low Earth Orbit and Geostationary orbits, the presence of atomic oxygen introduces a very fast degradation pathway that will compete and interact with the radiation-induced degradation pathway. As a result, a rapid failure of polymeric materials is expected. In most cases, the ionizing radiation component of the space environment will involve electromagnetic radiations and accelerated particles (starting from protons up to heavy nuclei). The equilibrium of scissions and cross-linking reaction is sensitive to the temperature of the sample. For low temperatures, the polymer is far below its glass transition temperature and the main degradation process is expected to be chain scission. This reflects the freezing of the segmental motions in polymers at low temperatures. The weight of chain scissions reactions relative to chain cross-linking is decreased as the temperature of the sample is increased up to a point at which eventually chain cross-linking reactions become dominant. The effect of ionizing radiation on the mechanical properties of some simple polymers are presented and analyzed. Additionally, spectrometric data that points to fundamental mechanism occurring in polymers during irradiation, are presented and discussed.

SESSION NN4: Multifunctional Materials  
Chair: Michael Wargo  
Tuesday Evening, November 30, 2004  
Room 208 (Hynes)

#### 7:00 PM \*NN4.1

**Manipulation of Nanoscopic Core-shell Materials Originating from Block Copolymers.** Karen Lynn Wooley, Haiyong Huang, K. S. Murthy, Kai Qi and Qi Zhang; Chemistry, Washington University, Saint Louis, Missouri.

This presentation will discuss the design of nanoscopic core-shell materials, the development of procedures for their preparation, the interesting physical properties that result, and the ability to manipulate such nanostructured materials physically and chemically. The covalent stabilization of supramolecular assemblies of block copolymers is a convenient methodology by which robust nanoscopic materials may be produced. When the assemblies are polymer micelles and the covalent stabilization is in the form of intramolecular crosslinking between the polymer chains that constitute the shell layer, an interesting morphology is produced, for which linear polymer chains are confined within a nanoscopic volume and also attached to a crosslinked surface. Emphasis will be placed upon the physical properties of these materials, in particular, the thermal properties of nanoconfined crystalline polymer chains, and upon the thermally-induced reshaping of these structures. Potential for these materials to be utilized as self-healing nanoparticles and as thermally-addressable memory storage bits will be highlighted.

#### 7:30 PM NN4.2

**Self-healing Polymer Composite via Polycondensation of Polydimethylsiloxane.** Soo Hyoun Cho<sup>1</sup>, Magnus H. Andersson<sup>2</sup>, Paul V. Braun<sup>1</sup>, Scott R. White<sup>2</sup> and Nancy R. Sottos<sup>3</sup>; <sup>1</sup>Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>2</sup>Aerospace Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>3</sup>Theoretical and Applied Mechanics, University of Illinois at Urbana-Champaign, Urbana, Illinois.

We demonstrate self-healing of a flexible polydimethylsiloxane based system through the polycondensation of hydroxyl terminated polydimethylsiloxane (HOPDMS) with polydiethoxysiloxane (PDES) in the presence of organotin catalyst. Importantly, the polycondensation reaction of polydimethylsiloxane is accomplished at room temperature using a catalyst which is highly stable against water and oxygen. This self-healing system is composed of a matrix, epoxy vinyl ester, in which HOPDMS and PDES are dispersed as mixed phase separated liquid droplets, and microencapsulated droplets of an organotin catalyst. The microcapsules of the organotin catalyst consist of a polyurethane shell material formed through the interfacial polymerization of urethane with a chain extender in organic solvent.

The average particle size of microcapsules can be controlled by mechanical stirring speed during the polymerization reaction. When a crack propagates into the matrix, the catalyst is released from the microcapsules driving the cross-linking of the HOPDMS and PDES forming PDMS in the crack plane, sealing it shut. The self-healing efficiency of the polymer composite is investigated by comparing the fracture toughness of virgin and self-healed samples. The addition of acryl ethoxy silane to the matrix as an adhesion promoter significantly improves the healing efficiency of the polymer composite. Due to the very low Tg and high char temperature of PDMS, this material system may be an excellent candidate for space applications.

#### 7:45 PM NN4.3

**Molecular Self-Healing Processes in Polymers.** Mircea Chipara<sup>1</sup> and Karen Wooley<sup>2</sup>; <sup>1</sup>Indiana University Cyclotron Facility, Indiana University, Bloomington, Indiana; <sup>2</sup>Chemistry Department, Washington University in St. Louis, Saint Louis, Missouri.

Self-healing materials are obtained by dispersing small bubbles containing catalysts and monomers within various polymeric materials. The wall of these polymeric vesicles has to be fragile in order to release the catalysts and the monomers within the polymer during its mechanical solicitation. The size of these polymeric vesicles or bubbles is of the order of microns. This "classical" self-healing process is triggered by mechanical stresses that move the polymeric chains relative each to other. The "classical" self-healing process occurs at micron or larger scales and lasts as long as a sizable concentration of unreacted monomers and catalysts is still present in the polymer. Diffusion processes may limit the efficiency of the self-healing process mainly if the density of catalysts and monomers within the polymer is low. A self-healing process, operating at molecular scale has been identified in a relatively narrow class of polymeric materials. In contrast with the "classical" self-healing process, this process may be triggered not only by mechanical stresses but virtually by a wide range of processes such as radiation, light, and/or temperature besides mechanical stress. These features make this class of polymeric materials extremely attractive for space applications and in particular for long-term mission. The theoretical mechanism involved by the molecular self-healing process, the main features of the molecular self-healing as well as the potential space applications of self-healing polymers are discussed in detail. Preliminary experimental data, as obtained by electron spin resonance spectroscopy are reported.

#### 8:00 PM NN4.4

**Multifunctional Carbon Nanotube/Polymer Composites.** Jian Chen<sup>1</sup>, Rajagopal Ramasubramaniam<sup>1</sup> and Haiying Liu<sup>2</sup>; <sup>1</sup>Zyvec Corporation, Richardson, Texas; <sup>2</sup>Department of Chemistry, Michigan Technological University, Houghton, Michigan.

Carbon nanotubes, due to their high-aspect ratio, small diameter, lightweight, high-mechanical strength, high-electrical and thermal conductivity, high-thermal and air stability, are recognized as the ultimate carbon fibers for high performance, multifunctional composites. However, smooth carbon nanotube surfaces (i.e., sidewalls) are incompatible with most solvents and polymers, which result in poor dispersion of nanotubes in the polymer matrix. We report here that homogeneous nanotube polymer composites can be fabricated using noncovalently functionalized, soluble single-walled carbon nanotubes (SWNTs)(Ref. 1). In contrast to pristine SWNTs, soluble SWNTs can be homogeneously dispersed in commercial polymers such as polycarbonate, polystyrene etc. These composites showed dramatic improvements in the electrical conductivity with extremely low percolation threshold 0.05-0.1 wt% of SWNT loading. By significantly improving the dispersion of SWNTs in commercial polymers, we show that only very low SWNT loading is needed to achieve the conductivity levels required for various electrical applications without compromising the host polymer's other preferred physical properties and processability. In contrast to previous techniques, our method is applicable to various host polymers and does not require lengthy sonication. The resulting polymer composites also show significant enhancement in mechanical strength. The mechanical measurement showed that 2 wt % of soluble SWNT filling resulted in a 79% increase in the tensile strength of polycarbonate. We also observed a stress-induced SWNT alignment in polycarbonate, which is very difficult to achieve with insoluble SWNTs. The SEM study of the fracture surface indicates excellent interfacial interaction/load transfer between SWNTs and polycarbonate, possibly due to the considerably increased roughness of carbon nanotube surfaces by PPE non-covalent functionalization. We will also report an isotropic nanotube-based conducting thermoplastic with the tensile strength higher than aluminum. Reference: 1. Chen, J. et al. J. Am. Chem. Soc. 2002, 124, 9034-9035.

#### 8:15 PM NN4.5

**Conducting Polyaniline Composites as Microwave Absorbers.** Honey John<sup>1</sup>, Rani Joseph<sup>1</sup> and K.T.Mathew Mathew<sup>2</sup>; <sup>1</sup>Dept. of



Polymer Science and Rubber Technology, Cochin University, Kerala, cochin, India; <sup>2</sup>Dept. of Electronics, Cochin University, Kerala, cochin, Kerala, India.

Conducting polymers are excellent microwave absorbers and they show technological advantage compared to inorganic electromagnetic absorbing materials, being light weight, easily processable, and the ability of changing the electromagnetic properties with nature and amount of dopants, synthesis conditions etc. Among the conducting polymers, polyaniline is superior mainly because of its ability to exhibit varied electromagnetic properties by reversible proton doping. In this paper we report the synthesis, dielectric properties and expected application of conducting composites based on polyaniline. Cyclohexanone soluble conducting polyaniline composites of microwave conductivity 12.5 S/m was synthesized by the insitu polymerization of aniline in the presence of emulsion grade polyvinyl chloride. The composites were characterized using TGA and DSC, which shows that the thermal stability of the composite was better compared to polyaniline. 1M HCl was optimized as the dopant for the composite with respect to its dielectric properties among the different dopants like sulfuric acid, nitric acid, perchloric acid, toluene sulfonic acid, camphor sulfonic acid etc. The dielectric properties of the composites, especially the dielectric loss, conductivity, dielectric heating coefficient was found superior to polyaniline. Moreover, the absorption coefficient of polyaniline was increased with polyvinyl chloride loading and it reaches a maximum at 1:1.5 Pani:PVC composite. The penetration depth was very low (below .004 m) for this composition. The microwave absorption of this particular composite was studied at different frequency bands i.e, S, C and X bands (2-12 GHz) and at varying thickness. The absorption coefficient was found to be higher than 200 m-1 and it can be used for making microwave absorbers in space applications. The composite also shows selectivity in microwave absorption at different frequency bands. The dielectric properties were studied using HP 8510 vector network analyzer and cavity perturbation technique.

#### 8:30 PM \*NN4.6

**Multifunctional Power-Generating and Energy-Storing Structural Composites for U.S. Army Applications.** Joseph T. South, James F. Snyder, Robert H. Carter, Jim Campbell, Eric D. Wetzel and Shawn M. Walsh; Weapons and Materials Research Directorate, U.S. Army Research Laboratory, Aberdeen Proving Ground, Maryland.

The performance of many U.S. Army systems depends on the efficient use of material mass and volume. In particular, many components on these systems are often dedicated to power generation and energy storage. Examples include next-generation ground vehicles, which utilize hybrid powertrains requiring large banks of batteries; unmanned aerial vehicles, whose range and speed are currently limited by battery life; and individual soldiers, whose sensing and communication equipment requires both continuous and burst power requirements. Many of these systems also include a significant amount of structural and/or armor materials. Therefore, important system-level reductions in mass and volume are possible by creating multifunctional materials that simultaneously offer both power generation or energy storage capabilities with structural or armor properties. Three candidate multifunctional materials are under investigation. First, structural lithium-ion batteries are being designed by formulating ion-conductive polymer electrolytes with continuous fiber reinforcement. The electrolytes are designed to balance structural and ion-conduction properties, through the use of functionalized structural polymers, block copolymers, or microscale mixtures of conductive and structural materials. Second, structural fuel cells have been fabricated by creating skin-core composite structures. In these fuel cells, the skins are composed of thin, glass fiber or carbon fiber-reinforced composite laminates. The core consists of layers of open-cell metal foam with a Nafion<sup>®</sup>-based membrane electrode assembly interlayer. This core generates power when hydrogen and oxygen sources are circulated through the porous foam layers. Simultaneously, the high shear and compressive stiffness of the core transfers loads efficiently to the skins, resulting in a structure with high specific stiffness and strength. Finally, electroded surfaces have been integrated into continuous fiber-reinforced polymeric composites, to create structural capacitors. Fabrication and design details for these multifunctional composites, as well as structural and power/energy performance results, will be reported.

#### 9:00 PM NN4.7

**Thermoplastic matrix composites for SPACE SOLAR POWER TRUSS (SSP).** Hao Zhang<sup>1</sup>, Koorosh Guidanean<sup>2</sup> and Steven Nutt<sup>1</sup>; <sup>1</sup>Materials Science, University of Southern California, Los Angeles, California; <sup>2</sup>L'Garde Inc., Tustin, California.

**ABSTRACT:** Rigidizable thermoplastic matrix composites can be repeatedly softened and folded by heat. By using low T<sub>g</sub> (Glass transition temperature) thermoplastic matrix, SPACE SOLAR

POWER TRUSS can be easily packaged at room temperature, and expand in the outer space. The present paper discusses RFI (resin film infusion) and hot pressing process that were used to fabricate laminate tubes. Polyurethane and carbon plain-weave fabrics are used. Sample tubes have the dimension of length 16.76cm and diameter 2.54cm, which is formed according to the proportion 1:5 to the real tubes. The performance of the bent tubes under compression and compared with that of the unbent tubes. Results show that modulus increases after bending. Fiber waviness also affects the final stiffness properties of composites. High waviness results in easy of losing stability, especially for thin laminates. The dimension of laminate appears to affect compressive properties due to buckling. In order to improve resistance to delamination, stitching technique has been used. Stitching parameters have been investigated. Keywords: Thermoplastic matrix composites, compressive properties, rigidizable, RFI, bent, stitching, fiber waviness

SESSION NN5: Poster Session  
Chair: Ramon Artiaga  
Tuesday Evening, November 30, 2004  
8:00 PM  
Exhibition Hall D (Hynes)

#### NN5.1

**Screening of the thermal endurance of space materials by kinetic modelling.** Stan Heltzel, Jason R. Williamson, Gustavo Garcia Martin, Martin Moser and Christopher O.A. Semprinoschnig; Materials Physics and Chemistry Section, ESA/ESTEC, Noordwijk, Netherlands.

The knowledge of the thermal endurance is an important aspect for ESAs upcoming missions to the inner part of the solar system. These missions require a precise understanding about the high temperature resistance of materials. In the optimum case, this requires to know the detailed space environmental behaviour at elevated temperature. That implies to know outgassing kinetics, decomposition kinetics, evolution of thermo-mechanical properties versus temperature and to know the general effect of high temperature ageing. To establish that knowledge is a very time consuming task and therefore methods are required to generate a faster screening methodology to down-select materials. We have worked to establish one way of methodology which allows us in a relatively short time to predict the thermal endurance of space materials. This is commonly known as kinetic modelling. Such models rely on test results obtained by TGA (Thermo-Gravimetric Analysis). By performing a minimum of three experiments such models enable lifetime predictions in a relatively short time. We applied two commonly used kinetic models. We have applied the model proposed in ASTM E 1641 that relies on a standard Arrhenius equation. We have also applied another model called model free kinetics proposed by Sergei Viazovkin. This one relies on a similar approach but it is able to establish the activation energy as a function of temperature. In this paper several space relevant materials were screened and test results are shown. The applicability and the limitations of the two models are discussed. It is shown that depending on what type of decomposition reaction occurs one model yields better results than the other. For that the results of the models are compared with a selected number of long term isothermal tests and additionally the impact of long duration ageing and the comparison with predictions are elaborated.

#### NN5.2

**Nanocrystalline Soft Magnetic Alloys for Space Applications.** Matthew A. Willard<sup>1</sup>, Thomas Francavilla<sup>1</sup> and Vincent G. Harris<sup>2</sup>; <sup>1</sup>Code 6324, U. S. Naval Research Laboratory, Washington, District of Columbia; <sup>2</sup>Electrical and Computer Engineering Department, Northeastern University, Boston, Massachusetts.

Flywheel and magnetic bearing systems require soft magnetic materials possessing high strength and low eddy current and hysteretic losses. Although amorphous alloy magnets exhibit good loss characteristics, their long-term use at elevated temperatures, near 300 to 400°C, degrades their properties. In recent years, nanocrystalline materials with exceptional soft magnetic properties have been developed with potential use for such applications. Nanocrystalline soft magnetic materials possess higher magnetization and operation temperatures than amorphous alloys with similar compositions. Recently, nanocrystalline alloys with the nominal composition (Fe,Co,Ni)<sub>89</sub>Zr<sub>7</sub>B<sub>4</sub> have been produced by a single wheel melt spinning technique, followed by an isothermal anneal above the primary crystallization temperature.<sup>1,2</sup> The resulting materials are multi-phase with residual amorphous phase between nanocrystallites. The high frequency response of these materials is aided by the multi-phase microstructure, primarily by the reduction of eddy current losses by the high resistivity of the residual amorphous phase. This study focuses on a Co-based nanocrystalline alloy (Co<sub>84.55</sub>Fe<sub>4.45</sub>Zr<sub>7</sub>B<sub>4</sub>) with potential for long-term high temperature

use. Differential thermal analysis and X-ray diffraction have been used to determine the primary crystallization temperature (e.g. 480°C at 20°C/min) and product phases (e.g. FCC and BCC grains when annealed at 550°C), respectively. Vibrating sample magnetometry provided the magnetization and hysteretic losses as a function of temperature. The resulting magnetization of 120 emu/g was reduced by less than 20% over the temperature range from room temperature to 400°C. As an indication of their performance, core losses have been measured on toroidal samples using a Walker AC permeameter over a frequency range of 0.1 to 500 kHz and at induction amplitudes of 0.1, 0.3, and 0.5 T. A sample annealed at 550°C for one hour had a core loss of 0.0025 W/g at an operation frequency of 1 kHz and applied magnetic induction of 100 mT. References 1.) M. A. Willard, J. H. Claassen, R. M. Stroud, and V. G. Harris, *Journal of Applied Physics* **91**(10) (2002), 8420-8422.

### **NN5.3**

**Multifunctional Metal/Polymer Hybrid Fiber for Space and Aerospace Applications.** Jar-Wha Lee<sup>1</sup>, Tom W. Omohundro<sup>2</sup>, David P. Zika<sup>2</sup>, Dale L. Hart<sup>3</sup>, Erik Schomburg<sup>3</sup>, Elizabeth T. Shinn<sup>4</sup>, Abigail J. Cooley<sup>4</sup>, Edward L. White<sup>4</sup>, Paul W. MacDowell<sup>4</sup>, George A. Slenski<sup>4</sup>, Anthony N. Watkins<sup>5</sup> and Robert L. Yang<sup>5</sup>; <sup>1</sup>Syscom Technology, Inc., Columbus, Ohio; <sup>2</sup>The Boeing Company, St. Louis, Missouri; <sup>3</sup>University of Dayton Research Institute, Dayton, Ohio; <sup>4</sup>Wright-Patterson AFB / AFRL, Dayton, Ohio; <sup>5</sup>Langley Research Center / NASA, Hampton, Virginia.

The stringent weight and space requirements of advanced space and aerospace systems have led to a need for stronger, lighter, smaller, and more flexible cable and wiring components. Syscom has fabricated a multifunctional metal/polymer hybrid fiber from a rigid-rod type of polymer, such as poly(p-phenylene benzobisoxazole) (PBO) fiber, for signal transfer and electromagnetic interference (EMI) shielding in wiring and cable applications. The test results indicated that the metal/polymer hybrid PBO fiber exhibited 67% the electrical conductivity, 73% the weight and 200% the breaking strength of a comparable size copper-beryllium CS95 wire. Additional experimental results of electric current carrying capability, cable shielding performance, atomic oxygen erosion protection and potential electronic textile applications will also be discussed.

### **NN5.4**

**Effect of Temperature on the Luminescence and Up-Conversion Dynamics of Praseodymium in LiLuF<sub>4</sub> Laser Crystal.** Gonul Ozen<sup>1,4</sup>, John M. Collins<sup>2</sup>, Ottavio Forte<sup>1</sup>, Misha Meylman<sup>1</sup>, R. Yu Abdulsabirov<sup>3</sup> and Baldassare DiBartolo<sup>1</sup>; <sup>1</sup>Department of Physics, Boston College, Chestnut Hill, Massachusetts; <sup>2</sup>Department of Physics and Astronomy, Wheaton College, Norton, Massachusetts; <sup>3</sup>Laboratory of Quantum Electronics and Radiospectroscopy, Kazan State University, Kazan, Russian Federation; <sup>4</sup>Department of Physics, Istanbul Technical University, Istanbul, Turkey.

Rare-earth doped crystalline solids are very attractive because of their important applications in lasers and non-linear optics. In particular, praseodymium (Pr<sup>3+</sup>) in various crystals has been widely studied because of its energy level structure and suitable lifetimes of the meta-stable levels in 4f<sup>2</sup> configuration. Praseodymium, in general, enters solid host lattices in its trivalent state. There are at least two potentially useful transitions associated with emissions in the 600nm region: the 1D<sub>2</sub> to 3H<sub>4</sub> transition and the 3P<sub>0</sub> to 3H<sub>6</sub> transition. Besides the practical applications of praseodymium doped crystals there are some interesting basic processes such as multi-phonon relaxations and up-conversion processes that need to be explored. Previous studies of Pr-doped LiLuF<sub>4</sub> crystals concentrated on the basic optical and spectroscopic properties such as the position of the energy levels of the Pr<sup>3+</sup> ions in 4f<sup>2</sup> configuration and 4f<sup>2</sup> to 4f<sup>5d</sup> excited state absorption [1]. This presentation reports on the investigation of the spectroscopic properties of Pr<sup>3+</sup> ions in LiLuF<sub>4</sub> laser host doped with 0.5% Pr<sup>3+</sup> ions as a function of temperature. Luminescence spectra were obtained by exciting selectively the sample at 439nm into the 3P<sub>2</sub> level and at 593nm into the 1D<sub>2</sub> level, resulting in direct excitation of the 3P<sub>0</sub> and 1D<sub>2</sub> levels, respectively. Also, a strong blue emission due to the 3P<sub>0</sub> to 3H<sub>4</sub> transitions upon excitation of 1D<sub>2</sub> level with 593 nm laser light between 24K and 300K has been investigated. The decay pattern inspection was used to assign spectral lines to specific radiative transitions and to study the dynamics of the excited ions in the crystal. Detailed measurements of the 3P<sub>0</sub> and 1D<sub>2</sub> emissions under pulsed excitation were made between the 24K and 300K. Under direct excitation, the 1D<sub>2</sub> decay curve is nonexponential at all temperatures, whereas the 3P<sub>0</sub> decay is exponential only at low temperatures. Under excitation into the 1D<sub>2</sub> level, the decay curve of the blue emission shows a rise, indicating that the 3P<sub>0</sub> level is populated via upconversion due to energy transfer. The temperature dependence of this emission and a mechanism for the upconversion will be presented. We gratefully acknowledge the support of this research under the NASA grant #

NNL04AA30G. [1] S. Nicolas, E. Descroix, Y. Guyot, M.-F. Joubert, R.Yu. Abdulsabirov, S.L. Korableva, A.K. Naumov, V.V. Semashko, *Optical Materials* **16** (2001) 233-242.

### **NN5.5**

**Thermal Conductivity Reduction in Nanocomposite for Thermoelectric Power Generation.** Hohyun Lee<sup>1</sup>, Shankar Kunwar<sup>2</sup>, Dezhi Wang<sup>2</sup>, Zhifeng Ren<sup>2</sup>, Mildred Dresselhaus<sup>3</sup> and Gang Chen<sup>1</sup>; <sup>1</sup>Department of Mechanical Engineering, MIT, Cambridge, Massachusetts; <sup>2</sup>Department of Physics, Boston College, Chestnut Hill, Massachusetts; <sup>3</sup>Department of Physics and Department of Electric Engineering and Computer Science, MIT, Cambridge, Massachusetts.

Radioisotope thermoelectric generators are used for deep space missions. Such applications call for materials with high thermoelectric figure of merit, which is proportional to the electric conductivity, the square of Seebeck coefficient, and inversely proportional to the thermal conductivity. Properly designed nanocomposites have the potential to improve the thermoelectric figure of merit over that of bulk materials. This paper reports experimental results on the thermal conductivity of Si/Ge nanocomposites. The experimental data shows that the thermal conductivity of Si/Ge nanocomposites is reduced below that of the homogeneous alloy.

### **NN5.6**

**Investigation upon Mechanical Properties of Thin Film Silicon Under Cryogenic Temperature.** Yi Zhao and Xin Zhang; Department of Manufacturing Engineering, Boston University, Brookline, Massachusetts.

With growing micro-satellite technologies, more and more instrumentations are being compacted into a limited space for scientific concept validation. These instruments, especially the infrared imaging components, need to be maintained within a narrow cryogenic temperature range for an acceptable signal-to-noise ratio. Most of the passive cooling approaches are not appropriate due to the extreme temperature condition in outer space, while it calls for an efficient cooling mode in which the cooling rate should be adjustable in response to surrounding environment. Micro pump array is a feasible option which transfers liquid cryogen through inlet and outlet flipper at a desired rate via the actuation of the thin silicon diaphragm. The mechanical properties of the diaphragm are critical for cooling rate estimation. However, little is known about that under the cryogenic temperature. In this paper, we present the preliminary work about the investigation upon the stiffness of thin film silicon under liquid nitrogen temperature. It is expected to shed some light on the further application of thin film silicon in extreme temperature environment. The thin film silicon (30 μm in thickness) was mounted in double layered dewar which was filled with liquid nitrogen. The film was actuated using compressive gas and its deflection was achieved using Michelson interferometer. Given the lateral dimension and the thickness, Young's modulus of the film was derived. It is found that the Young's modulus increases with decreasing temperature, which indicates a much stiffer film under cryogenic environment. The stiffness change is compared with semi-empirical equation and the discrepancy is explained mainly in two folds: the difference in mechanical properties (Young's modulus, CTE, and etc.) between the thin film and bulk material, and lattice distortion due to the mismatch between silicon and doped boron when temperature changes. Moreover, it is discussed that the direct contact of the silicon film with sample holder as in the experiment setup may also serve as a contribution. As the calibration, a skewed Michelson interferometer was built using a 532-nm Nd:YAG laser for surface wave generation and a 514.5 nm Ar/ion laser as the detection probe. A smaller propagating time was found associating with cryogenic temperature, indicating an increased elastic modulus. This increased Young's modulus under cryogenic temperature further validates the stiffness change with temperature.

### **NN5.7**

**InAs Quantum Dot Development for Enhanced InGaAs Space Solar Cells.** Ryne P. Raffaele<sup>1</sup>, Sam Sinharoy<sup>2</sup>, William King<sup>2</sup> and Sheila G. Bailey<sup>3</sup>; <sup>1</sup>NanoPower Research Labs, Rochester Institute of Technology, Rochester, New York; <sup>2</sup>Essential Research Incorporated, Cleveland, Ohio; <sup>3</sup>NASA Glenn Research Center, Cleveland, Ohio.

The majority of high-efficiency space solar cells being produced today are based on multi-junction devices of lattice-matched III-V materials. An alternative which has been receiving an increasing amount of attention is the a lattice mis-matched or metamorphic approach to multi-junction cell development. In the current state-of-the-art metamorphic triple junction cell, the InGaAs junction (bottom cell) of the three-cell stack is the current limiting entity, due to the current matching which must be maintained through the device. This limitation may be addressed through the incorporation of InAs quantum dot array into the depletion region of an InGaAs cell. The

InAs quantum dots in the InGaAs cell will provide sub-gap absorption and thus improve its short circuit current. This cell could then be integrated into the three-cell stack to achieve a space solar cell whose efficiency would dramatically exceed current state-of-the-art standards. A theoretical estimate predicts that a InGaAlP(1.95eV)/InGaAsP(1.35 eV)/InGaAs(1.2 eV) triple junction cell incorporating quantum dots to improve the bottom cell current would have an efficiency exceeding 40%. In addition, theoretical estimates have demonstrated that the use of quantum dot structures may also hold other cell benefits such as improved temperature coefficients and better radiation tolerance, which are especially important for utilization in space. As a first step towards achieving that goal, we have initiated the development of InAs quantum dots on lattice-mismatched InGaAs (1.2 eV bandgap) grown epitaxially on GaAs by metallorganic vapor phase epitaxy (MOVPE). These quantum dots have been characterized via photoluminescence (PL) and atomic force microscopy (AFM). The correlation between the quantum dot size and resulting optical band structure as a function of the synthesis parameters will be presented. The procedure for incorporating these quantum dots into prototype InGaAs devices will be reviewed. The resulting optoelectronic performance the prototype devices, including photovoltaic efficiency under simulated 1 sun intensity and air mass zero (AM0) illumination and spectral response, will be presented.

#### **NN5.8**

**In-situ Polymerization of Poly(methyl methacrylate)/clay Nanocomposites in Supercritical Carbon Dioxide.** Qian Zhao<sup>1</sup> and Edward T. Samulski<sup>1,2</sup>, <sup>1</sup>Curriculum in Applied Materials Sciences, University of North Carolina, Chapel Hill, North Carolina; <sup>2</sup>Chemistry, University of North Carolina, Chapel Hill, North Carolina.

A novel route to synthesize PMMA/clay nanocomposites were presented by a pseudo-dispersion polymerization of MMA in presence of fluorinated surfactant modified clay (10F-clay) in supercritical carbon dioxide. The nanocomposites were characterized by SEM, TEM, X-ray Diffraction (XRD), TGA, and DMA, and showed partially exfoliated/intercalated structures as well as enhanced thermal stability, glass transition temperature and mechanical properties. It was also found that 10F-clay served not only as inorganic filler, but also an effective stabilizer for PMMA growth in CO<sub>2</sub>. More stabilizing mechanisms were suggested by FTIR studies. This general route allows for clean synthesis of nanocomposites with high yields in supercritical CO<sub>2</sub>, without the need for adding extra surfactant to stabilize the polymerizing system.

#### **NN5.9**

**Materials for High-Energy Radiation Shielding on Extra-Terrestrial Surfaces.** Richard Kiefer<sup>1</sup>, Janet L. Chapman<sup>1</sup>, Amanda R. Boone<sup>1</sup> and Sheila A. Thibeault<sup>2</sup>, <sup>1</sup>Chemistry, College of William and Mary, Williamsburg, Virginia; <sup>2</sup>Advanced Materials and Processing Branch, NASA Langley Research Center, Hampton, Virginia.

Long-term missions to extra-terrestrial surfaces such as the Moon or Mars will require habitats to protect humans and electronic equipment from the effects of high-energy radiation from galactic cosmic rays (GCR) and solar particle events (SPE). Shielding from this radiation is best accomplished by using materials composed of elements with small atomic numbers. The best shield would be liquid hydrogen, though obviously that would not be practical. Polymeric materials made from carbon and hydrogen provide a reasonable alternative. Habitats constructed on the surface of the Moon could utilize surface material (regolith) along with polymeric material both for shielding and for structural integrity. We have used polyethylene and polypropylene with simulated lunar regolith to make micro-composites as possible materials for habitat construction. Polyethylene and polypropylene have the highest hydrogen content of any polymer. Polyethylene and polypropylene powders were used in various mass percents and thoroughly mixed with regolith powder before heating. A microwave oven was used to heat the polymer/regolith mixture and form the micro-composite. The resulting materials were characterized by thermal analysis. In the future, promising materials will be irradiated with high-energy iron ions to measure their shielding effectiveness.

#### **NN5.10**

**Electron Spin Resonance Studies on Electron Beam Irradiated Carbon Nanotubes Dispersed in Styrene-Isoprene-Styrene Block Copolymer.** Mircea Chipara<sup>1</sup>, Wendland Beezhold<sup>2</sup>, Jeffery Zaleski<sup>4</sup>, Kristina Stephenson<sup>4</sup>, David Dye<sup>4</sup> and Kin-tak Lau<sup>3</sup>, <sup>1</sup>Indiana University Cyclotron Facility, Indiana University, Bloomington, Indiana; <sup>2</sup>Physics Department, Idaho State University, Pocatello, Idaho; <sup>3</sup>Mechanical Engineering, The Hong Kong Polytechnic University, Hong Kong, Hong Kong; <sup>4</sup>Chemistry Department, Indiana University, Bloomington, Indiana.

The huge mechanical strength, high thermal and electrical conductivity, the strong anisotropy of the physical properties correlated with the capability to tailor the physical properties by controlling the chirality of nanotubes fuelled many research. While nanotubes have an impressive mechanical strength, the ropes of carbon nanotubes have rather modest mechanical strength. Much effort is directed now towards the preparation and characterization of composite materials based on polymeric matrices, in a tentative to further increase the mechanical strength of such matrices. This research has a particular relevance to space applications due to the reduced weight of these composites and to the capability to obtain multifunctional composites based on carbon nanotubes. Experimental data confirmed an increase in the mechanical properties of polymeric matrices due to the reinforcement with carbon nanotubes but the results are still below expectations. Most efforts are focused on the interface between nanotubes and polymeric matrix, which controls the adhesion of the polymeric chain to nanotubes and finally governs the mechanical properties of the composite. The stability of carbon nanotubes subjected to space environment has yet to be assessed. The sensitivity of carbon nanotubes to oxidative degradation will eventually prevent the use of carbon nanotubes in low orbits such as Low Earth Orbit, where the presence of atomic oxygen will open and destroy carbon nanotubes. The effect of the radiation component of the space environment is not yet fully understood. Recent studies revealed the fusion of carbon nanotubes in electronic microscopes. The absence of oxygen and a high stopping power are expected to be required for carbon nanotube fusion. The possibility to fuse carbon nanotube suggests that in polymer based composites, the irradiation may graft the nanotubes to the polymeric chain. Such a cross linked material would exhibit outstanding mechanical properties. We reported the electron spin resonance investigations on carbon nanotubes dispersed in styrene-isoprene-styrene block copolymer subjected to accelerated electron beams. Multiwalled carbon nanotubes were dispersed in a dilute solution of styrene-isoprene-styrene block copolymers and sonicated several hours. The solvent was removed by evaporation. The irradiation has been performed in air, at room temperature, at integral doses of 0.1 MRads, 1.0 MRads, and 10 MRads. The resonance spectra were recorded by using a JES-ME-3X spectrometer, operating in X band. The experimental data revealed the high resistance of carbon nanotubes dispersed in polymeric matrices to ionizing radiation, even in the presence of oxygen. No traces of free radicals generated by the electron irradiation of the polymeric matrix were noticed, within experimental errors.

#### **NN5.11**

**Preparation and Characterization of Electrospun poly(ethylene oxide) (PEO) Nanofibers-reinforced Epoxy Matrix Composites.** Jae-Rock Lee<sup>1</sup>, Soo-Jin Park<sup>1</sup>, Min-Kang Seo<sup>1</sup> and Jung-Man Park<sup>2</sup>, <sup>1</sup>Advanced Materials Division, Korea Research Institute of Chemical Technology, Yusong, Daejeon, South Korea; <sup>2</sup>Department of Polymer Science and Engineering, Engineering Research Institute, Gyeongsang National University, Chinju, South Korea.

In this work, electrospinning was carried out using 12 wt.% poly(ethylene oxide) (PEO) solution under fixed tip-to-collect distance (10 cm) and voltage (15 kV) in order to fabricate nanofibers-reinforced composites. The content of PEO nanofibers was varied from 0 to 10 wt.% in the epoxy (EP) matrix resins. And the PEO powders-impregnated composites were also prepared to compare with physicochemical properties of nanofibers-reinforced composites. Thermal and mechanical interfacial properties of EP/PEO nanocomposites were characterized by thermogravimetric analysis (TGA) and fracture toughness test, respectively. As a result, the PEO-based nanofibers-reinforced composites showed an improvement of thermal stability parameters (initial decomposed temperature (IDT) and integral procedural decomposition temperature (IPDT)) and fracture toughness factors (KIC and GIC), compared to the composites impregnated with PEO powders. And the thermal and mechanical interfacial properties of the composites were increased with increasing the PEO content, which could be probably attributed to the higher specific surface area and larger aspect ratio of PEO nanofibers, resulting in improving the demand performance of the nanocomposites.

#### **NN5.12**

**Computer Simulation of Displacement Damage in Silicon Carbide.** Ram Devanathan, Fei Gao and William J. Weber; Fundamental Science Directorate, Pacific Northwest National Laboratory, Richland, Washington.

Silicon carbide is a wide-bandgap semiconductor that is a promising candidate material for radiation-resistant electronics applications. In the space environment, irradiation by energetic protons and electrons can result in displacement damage leading to new energy levels in the

band gap. These defects can degrade the electrical as well as physical properties of the material. In an effort to understand the atomic-level details of the displacement process, we have performed molecular dynamics simulation of displacement events on Si and C sublattices for displacement doses ranging from 0.005 to 0.5 displacements per atom. Our results indicate that the displacement threshold energy is about 20 eV for C and 35 eV for Si, and amorphization can occur by accumulation of displacement damage regardless of whether Si or C is displaced. In addition, we have simulated defect production in high-energy cascades as a function of the primary knock-on atom energy and observed features that are different from the case of damage accumulation in Si. These systematic studies shed light on the phenomenon of non-ionizing energy loss that is relevant to understanding space radiation effects in semiconductor devices.

#### **NN5.13**

**Non-Equilibrium Ti-Fe Bulk Alloys with Ultra-High Strength and Enhanced Ductility.** Dmitri V. Louzguine, Hidemi Kato and Akihisa Inoue; Institute for Materials Research, Tohoku University, Sendai, Japan.

The relatively low density of Ti (4.5 Mg/m<sup>3</sup>) implies high strength/density ratio for Ti-based alloys which makes them suitable for aerospace applications. Typical commercial Ti-based alloys have an ultimate tensile strength slightly exceeding 1 GPa. At the same time, Ti alloys exhibit capability for further strengthening. For example, mould casting technique allows to produce Ti-based bulk glassy alloys having a high strength of about 2 GPa which, however, suffer from the lack of ductility. In the present work we report the formation of the high-strength and ductile hypo-, hyper- and eutectic Ti-Fe alloys in the form of the arc-melted ingots 20-30 mm diameter and 10-15 mm height. The structure of the samples studied by X-ray diffractometry and scanning electron microscopy is found to consist of cubic ordered Pm3m TiFe and disordered BCC Im3m  $\beta$ -Ti supersaturated solid solution phase. The Ti65Fe35 alloy having a dispersed hypereutectic structure consisting of the primary TiFe phase and submicron-size eutectic (TiFe +  $\beta$ -Ti) structure showed the best combination of the mechanical properties among the studied Ti-Fe alloys. A sample of the arc-melted Ti65Fe35 alloy cut from the central part of the ingot being tested by an Instron-type machine exhibits excellent mechanical properties: a Young's modulus of 149 GPa, a high mechanical fracture strength of 2.2 GPa, a 0.2 % yield strength of 1.8 GPa and 6.7 % ductility. The hard round-shaped intermetallic TiFe phase, the supersaturated  $\beta$ -Ti solid solution and the dispersed eutectic structure obtained result in a high strength of the Ti-Fe alloy which in addition to that has much higher ductility compared to that of bulk glassy alloys because the ductile  $\beta$ -Ti solid solution phase enables plastic deformation of the sample. The deformation behaviour and the fractography of the Ti-Fe alloys are studied in details. The reasons for the high strength and good ductility of the hypereutectic alloy are discussed. The Ti-Fe alloys are characterized by low cost of the alloying elements and do not require an additional injection mould casting procedure.

#### **NN5.14**

**Optical and Upconversion Studies on New Electro-Optic Ceramic Materials.** Xuesheng Chen<sup>1</sup>, Kewen Li<sup>2</sup>, Yanyun Wang<sup>2</sup>, Kevin Zou<sup>2</sup>, Gonul Ozen<sup>3</sup>, B. Di Bartolo<sup>3</sup> and Hua Jiang<sup>2</sup>; <sup>1</sup>Department of Physics & Astronomy, Wheaton College, Norton, Massachusetts; <sup>2</sup>Boston Applied Technologies, Inc., Woburn, Massachusetts; <sup>3</sup>Department of Physics, Boston College, Chestnut Hill, Massachusetts.

We report optical studies, including the non-linear effect of upconverting frequency of light, on newly developed transparent electro-optic ceramic materials, Er or Nd doped PLZT. These materials have many important applications such as high-efficiency high power lasers for remote sensing and space exploration. These ceramic materials have successfully been made highly transparent in the wavelength range 500nm-7000nm with intrinsic absorption bands due to the rare-earth doping ions, Er or Nd. Transparent ceramic laser materials have several critical advantages over popular single crystal laser materials - they are easy to accommodate high-concentration rare earth ions and to be fabricated in large size. The host ceramic material PLZT also has high electro-optic coefficient, which might have unique features in phase and mode self-modulation that would lead to an innovative laser system with higher efficiency, more compactness, and integrated multi-functions. In this presentation, we focus on luminescence and upconversion luminescence studies on this ceramic material family, rare-earth doped PLZT. This work provides crucial information for promising applications in new optical devices including high-efficiency high-power ceramic lasers. This work was supported in part by NSF and DOE.

#### **NN5.15 TRANSFERRED TO NN11.4**

#### **NN5.16**

**FIB-Based Cryogenic Characterization and Testing of Silicon Nitride Thin Film Materials and Devices for Space Applications.** Wen-Hsien Chuang<sup>1</sup>, Rainer Fettig<sup>2</sup> and Reza Ghodssi<sup>1</sup>; <sup>1</sup>Electrical and Computer Engineering, Institute for Systems Research, University of Maryland, College Park, Maryland; <sup>2</sup>NASA Goddard Space Flight Center, Greenbelt, Maryland.

Two-dimensional microshutter arrays are currently being developed at NASA Goddard Space Flight Center, to be used as programmable aperture masks for a Multi-Object Spectrometer on the James Webb Space Telescope (JWST). The microshutter arrays, made of silicon nitride thin films using microelectromechanical systems (MEMS) techniques, require cryogenic operation at 30 K to reduce thermal emission into the instrument. Since the JWST's second Lagrange point orbit will be inaccessible to space shuttle re-servicing missions, the microshutter arrays must operate in a cryogenic vacuum environment reliably over a 10 year mission lifetime without repair after launch. Therefore, a complete understanding of mechanical properties and reliability of silicon nitride thin films at cryogenic temperatures is critical for the development of the microshutter arrays. We have developed a cryogenic measurement setup installed inside a Focused-Ion-Beam (FIB) system to emulate the operating environment of the microshutter arrays. To obtain an accurate temperature measurement, a thin-film thermo-resistor with a sensitivity of 7.85 ohm/K was fabricated with test devices as an integrated temperature sensor. The device temperature down to 20 K with a FIB chamber pressure lower than 10<sup>-6</sup> torr has been successfully obtained using this setup. *In-situ* measurement techniques for Young's modulus and fracture strength characterization were also developed based on the designed setup. From the experiment, the average Young's modulus of silicon nitride thin films varies from 260.5 GPa at room temperature (298 K) to 266.6 GPa at 30 K, and the average fracture strength ranges from 6.9 GPa at room temperature to 7.9 GPa at 30 K. For reliability tests of the microshutter arrays, a novel MEMS test device with the same stress operating condition was designed. Unlike slow magnetic actuation utilized in the microshutter arrays, this device was actuated with electrostatic force to achieve accelerated lifetime tests. In addition, the disadvantage of high input voltages in most electrostatic MEMS devices was solved using a mechanism of vibration amplification. In this device design, two resonators were connected serially with a common torsion bar. Electrostatic energy was applied to the first resonator via a small fixed gap between electrodes and then transferred to the second resonator which can be moved freely with the common torsion bar. When the frequency of the applied energy matched the resonant frequency of the second resonator, large vibration movement of the second resonator can be obtained due to a high quality factor inside the vacuum chamber while keeping the vibration of the first resonator small. The stress amplitude of the second resonator can then be controlled by the frequency and amplitude of the input electrostatic energy. From the test results, no device failure has been found even up to 10<sup>9</sup> loading cycles when testing at the operating stress lower than 6.6 GPa.

#### **NN5.17**

**Whisker Formation in Sn Coatings on Cu.** Eric Chason, Lucine Kabakian and Sharvan Kumar; Division of Engineering, Brown University, Providence, Rhode Island.

In the electronics industry, Cu conductors are often coated with pure Sn and Pb-Sn alloys to enhance corrosion resistance and solderability. However, pure Sn layers have a tendency to develop whiskers that may cause component failures. Currently, Pb is alloyed with the Sn to suppress whisker formation, but the push towards Pb-free processing will make this unacceptable in the future. In space applications, the whiskers can become electron emitters and carry large amounts of current; Sn whiskers have been implicated in the loss of several satellites. We have been studying pure Sn coatings on Cu in order to understand the driving forces and mechanisms of whisker formation so that we can develop alternative processing approaches or materials systems that are reliable and environmentally acceptable. Using a real time, wafer-curvature based system we have measured the evolution of stress in vapor-deposited layers of Sn on Cu. We will present measurements of how stress develops in the Sn and Cu layers and describe a model for how interface reactions between the Sn and Cu leads to whisker formation.

#### **NN5.18**

**Energetics of Interstitial Oxygen in Cr and V.** Brian S. Good<sup>1</sup> and Evan Copland<sup>1,2</sup>; <sup>1</sup>Materials Division, NASA Glenn Research Center, Cleveland, Ohio; <sup>2</sup>Case Western Reserve University, Cleveland, Ohio.

The deleterious effects of dissolved oxygen in high-temperature materials are well known, yet the details of many aspects of the phenomenon remain poorly understood. In particular, the solubility of

oxygen in group IIIA-VA (Nb, Ti, Zr, Y) based alloys is a fundamental problem affecting both mechanical properties and oxidation resistance. In these alloys, at concentrations less than 5-10at%, oxygen is more stable dissolved in the metal than it is as an oxide-compound. Protective oxidation behavior is obtained when the alloy is saturated with oxygen and equilibrium is approached at the metal-scale interface, but this generally yields unacceptable mechanical properties. In contrast, alloys based on Ni, Fe, Al and Cr exhibit almost no oxygen solubility and readily form protective oxide layers. Therefore, it is important to understand the fundamental differences in the details of oxygen solubility in these two groups of metals. As a first step toward this understanding, we consider the energetics of interstitial oxygen in pure-V and pure-Cr. Both of these metals are BCC, yet their oxygen solubilities are very different, with that of V being much higher than that of Cr. We obtain total energies, densities of states, band structures and population analyses using the CASTEP plane-wave pseudopotential density functional computer code. The differences in the energetics and electronic structures of the two materials, particularly the partial densities of states associated with the individual metal ions and the interstitial oxygen, are discussed.

#### **NN5.19**

**Preliminary Assessment of Materials Returned by the Genesis Mission.** Karen M. McNamara, Johnson Space Center, NASA, Houston, Texas.

The Genesis Mission returns to Earth September 8, 2004 and represents a milestone in the US space program, comprising the first sample return since the Apollo Missions as well as the first return of materials exposed to the space environment outside of low Earth orbit and beyond the Earth's magnetosphere for an extended period (nearly three years). For material scientists, it represents an unprecedented opportunity to study the effects of the space environment of materials ranging from pure single crystals to composites to metals to plastics. We have no other comparable materials in all of our collections on Earth. The goal of the Genesis Mission is to collect a representative sample of the composition of the solar wind. This is done by allowing the naturally accelerated species to implant shallowly in the surfaces of ultra-pure, ultra-clean collector materials. These collectors include single crystal silicon (FZ & CZ), sapphire, and silicon carbide; those materials coated with aluminum, silicon, diamond like carbon, and gold; and polycrystalline diamond and amorphous carbon. Once returned, the materials are allocated by an independent committee to the science community on the basis of merit of their proposals. The collectors are reserved primarily for studies related to cosmochemistry and solar system formation. In addition to the science collector materials, the remainder of the spacecraft capsule is to be returned to the Johnson Space Center for curation and subsequent allocation for materials and engineering studies. Many of these materials were extremely well characterized pre-flight and their handling and processing thoroughly documented, making them outstanding specimens for study. In this presentation, I will give a brief overview of the mission and spacecraft. I will report primarily on the condition and assessment of the returned science payload and return capsule and its comparison to pre-flight. The techniques we will use to characterize the materials include visual inspection, photo-documentation, optical scanning, microscopy, and spectroscopic ellipsometry. All of these techniques are carried out non-destructively in a cleanroom environment to preserve the as-returned conditions of the specimen. At the time of the presentation, we expect to be well into this preliminary assessment but by no means finished with the work. The purpose of this presentation is to introduce the materials community to this unique suite of materials and the opportunities available for their study.

#### **NN5.20**

**The Effect of Simulated Hypervelocity Space Debris on Polymers.** E. Grossman<sup>1</sup>, R. Verker<sup>1</sup>, N. Eliaz<sup>2</sup>, I. Gouzman<sup>1</sup>, S. Eliezer<sup>3</sup>, M. Fraenkel<sup>3</sup>, S. Maman<sup>3</sup>, F. Beckmann<sup>4</sup> and K. Pranzas<sup>4</sup>; <sup>1</sup>Space Environment Division, Soreq NRC, Yavne, Israel; <sup>2</sup>Department of Solid Mechanics, Materials and Systems, Tel Aviv University, Ramat Aviv, Tel Aviv, Israel; <sup>3</sup>Plasma Physics Department, Soreq NRC, Yavne, Israel; <sup>4</sup>Neutron Scattering Department, GKSS, Geesthacht.

Space debris population in Low Earth Orbit has been increasing constantly with the increase in spacecraft missions. Hypervelocity space debris impacts limit the functionality of polymeric outer surfaces and, in extreme cases, might cause a total loss of a spacecraft. In this work, the fracture of various polymers (Kapton, Teflon and POSS-Polyimide) films by ultrahigh velocity impacts was studied. A laser-driven flyer ground simulation system was used to accelerate aluminum flyers to impact velocities as high as 2.9 km/s against polymer films with different thicknesses. Besides of impact effects, synergistic effects of combined ultrahigh velocity impacts and thermal cycling and ionizing radiation were also studied. Scanning electron microscopy was used to characterize the fracture morphology.

Impact effects on the internal structure of the polymer were studied by means of neutron and X-ray scattering techniques and X-ray microtomography. It was found that high-velocity impacts generate spalls in the Kapton film, with ultrahigh strain rate of about  $10^6 \text{ s}^{-1}$ . SAXS measurements reveal formation of new micro structural features in the Kapton and Teflon films. POSS-Polyimide film, although possess a high atomic oxygen durability, suffered from brittle fracture formation even for low velocity debris. Increase in the debris velocity resulted in a ductile-to-brittle transition. However, fractures created by impacts at velocities above 1.7 km/s showed central impacts regions, which experienced the highest strain rate and were of ductile-type fracture, while the outer regions, which experienced a lower strain rate, failed through brittle cracking. A model explaining this phenomenon is suggested based on the high impact temperature ( $T > T_g$ ) developed at the central impact region, and the low temperatures ( $T < T_g$ ) at remote regions.

#### SESSION NN6: Radiation Effects in Composite Materials

Chair: Mathias Celina  
Wednesday Morning, December 1, 2004  
Room 207 (Hynes)

#### **8:30 AM \*NN6.1**

**An Overview and Status Report of the Center for Integrated Space weather Modeling (CISM).** H. Spence, Department of Astronomy, Boston University, Boston, Massachusetts.

The Center for Integrated Space weather Modeling (CISM) is a new Science and Technology Center funded by the NSF. CISM is a ten-year, \$40M project, now in its second year, with a strong mandate from the National Space Weather Program. The overarching CISM vision is to understand our changing Sun and its effects on the solar system, life, and society. The main goal is to develop a series of ever-improving, comprehensive, physics-based numerical models describing the coupled Sun-to-Earth space environment. These models will be used as scientific tools for increased understanding of the complex space environment, as specification/forecast tools for space weather prediction, and as tools for teaching about the space environment. Headquartered at Boston University, CISM coordinates and integrates the scientific efforts of a diverse set of existing research groups from over 12 different institutions to form a single team of focused collaborators who are creating a product with multiple applications. In this talk, I will provide an overview of CISM and a status report on its activities, with an emphasis on those elements relevant to material science concerns.

#### **9:00 AM NN6.2**

**Processing and Characterization of Carbon Nanotube/Nanofiber Films and Nanocomposites.** Jihua Gou, Krishna Anumakonda and David Tacchi; Department of Mechanical Engineering, University of South Alabama, Mobile, Alabama.

Carbon nanotubes and carbon nanofibers have extraordinary mechanical, electrical, and thermal properties. To take the advantage of their unique properties, carbon nanotubes and carbon nanofibers were preformed as nano-films through a filtration process. A high quality of dispersion of carbon nanotubes and nanofibers in the solvent was achieved with the aid of surfactants under sonication. The nano-films consisted of densely packed, highly interconnected networks of carbon nanotubes and nanofibers and were further used as multi-functional reinforcements by integrating them into composite structures. The microstructures of the nano-films and nanocomposites were characterized with SEM and AFM. The properties of the nanocomposites were studied with tensile test and dynamic mechanical analysis.

#### **9:15 AM NN6.3**

**Nanostructured Carbon Thin Films as Space Radiation Shields.** Sanju Gupta, Gerardo Morell and Brad R. Weiner; Physics, NCSU, Raleigh, North Carolina.

Severe environmental tolerability is the prime factor in the development of novel space materials exhibiting excellent physical properties accompanied by lightweight, reusability, and multifunctional capabilities. Diamond is known for its reputation being radiation hard besides a range of other outstanding physical properties (electronic, optical, mechanical, and chemical) and hence it is preferable over the other existing semiconductors as detectors used in harsh environments. Thin films of micro- and nanocrystalline diamond were deposited by microwave plasma-assisted CVD technique. They were then submitted to gamma radiation doses of 1, 5, and 20 Mrads sequentially in order to study the radiation induced structural transformation and the corresponding changes in the electron field emission properties in order to establish

property-structure correlations [1]. Microstructural characterizations include Raman spectroscopy (RS), scanning electron microscopy (SEM), atomic force microscopy (AFM), and X-ray photoelectron spectroscopy (XPS). Microcrystalline diamond showed a dramatic improvement in the field emission properties, while nanocrystalline carbon showed a relatively small but systematic decrease in turn-on field from 9 to 4 Volt/micron with increasing radiation dose. The enhancement in emission characteristics associated to the radiation induced microstructural transformation shows the critical role of defects with their associated electronic defect states and of sp<sup>2</sup>-bonded carbon channels in the electron field emission mechanism of these so-called nanocomposite carbon materials. The results also indicate that nanocrystalline carbon tends to reach a state of damage saturation when subjected to 26 Mrad doses of gamma radiation, suggesting the possibility of fabricating radiation buffer/shields materials that would undergo internal sp<sup>3</sup> <-> sp<sup>2</sup> C inter-conversion while absorbing or scattering ionizing and/or electromagnetic radiation without changing their average microstructure, which can be employed for preventing radiation from reaching sensitive materials [1]. [1] S. Gupta, B. L. Weiss, B. R. Weiner, L. Pilione, A. Badzian, and G. Morell, J. Appl. Phys. 92, 3311 (2002) and references therein.

#### 9:30 AM NN6.4

**Proton Beam Induced Modifications in Multi-Functional Polyethylene-Based Carbon Nanotubes Composites.** Merlyn X. Pulikkathara<sup>4,3</sup>, Kristina Stephenson<sup>2</sup>, Laura Laura Pena-Paras<sup>4</sup>, Daneesh McIntosh<sup>4</sup>, Richard Wilkins<sup>3</sup>, David Dye<sup>2</sup>, Jeffrey Zaleski<sup>2</sup>, Enrique V. Barrera<sup>4</sup> and Mircea Chipara<sup>1</sup>; <sup>1</sup>Indiana University Cyclotron Facility, Indiana University, Bloomington, Indiana; <sup>2</sup>Chemistry Department, Indiana University, Bloomington, Indiana; <sup>3</sup>NASA Center for Advanced Radiation Research, Prairie-View A&M University, Prairie-View, Texas; <sup>4</sup>Department of Mechanical Engineering and Material Science, Rice University, Houston, Texas.

High-density polyethylene (HDPE) is currently considered the best material for space radiation shielding. In addition to its high concentration of hydrogen atoms, lightweight, and reduced radiation-induced activation, HDPE is a structural material with good mechanical properties and high radiation stability. During HDPE irradiation cross-linking reactions are dominant. The mechanical and thermal capabilities of HDPE are further boosted by filling the polymer with various materials. For certain applications, antistatic or even electrical conducting features are also required. These features can be achieved by using conducting fillers below and above the percolation threshold, respectively. Particular attention is paid to carbon-based fillers and in particular to carbon nanotubes. Minute amounts of nanotubes increase significantly the Young modulus of HDPE, and can add electric conductivity to the insulating polymeric matrix. In previous studies, percolation thresholds lower than 0.5% in weight were reported for long nanotubes dispersed within polymeric matrices. The radiation stability of nanotubes is still under debate. It has been reported that electron beam irradiation of nanotubes in vacuum resulted in nanotubes fusion and eventually in the grafting of HDPE chains on nanotubes. The irradiation of nanotubes in oxygen may degrade the nanotubes resulting in the formation of volatiles. These effects are amplified if oxygen is replaced by single oxygen (which is present in Low Earth Orbits). We report a comparative study on the radiation stability of HDPE doped with graphite and single wall nanotubes (SWNT). The composites were obtained by dispersing 5 % weight of SWNT fillers (including functionalized SWNT, fluorinated-SWNT, F-C11H23 doped-SWNT and graphite). The samples were irradiated in vacuum with protons accelerated at 40 MeV and 10 MeV at three fluences (3x10<sup>8</sup> protons cm<sup>-2</sup>, 3x10<sup>9</sup> protons cm<sup>-2</sup>, and 3x10<sup>10</sup> protons cm<sup>-2</sup>). Proton irradiation is important for space applications, as accelerated protons have one of the most important contributions to the radiation component of the space environment. After irradiation, the samples were stored in air, at room temperature. The radiation-induced modifications were investigated by electron spin resonance spectroscopy, using a Bruker ESP 300 spectrometer, operating in the X band, equipped with a variable temperature accessory. Additional measurements were performed by Raman spectroscopy and thermal analysis. Raman spectra revealed that the nanotube signature was unaltered by the irradiation, and that the functionalization of the nanotubes with peroxide (f-C11H23) was not affected by the irradiation. The analysis of ESR data revealed that the composites based on SWNT have a higher resistance to proton irradiation than the composite based on graphite. Due to the large thickness of samples, the effect of carbon nanotubes oxidation at the interface carbon nanotubes-air cannot be observed.

#### 9:45 AM \*NN6.5

**Ground-Based Radiation Studies on Single-Walled Carbon Nanotube Materials for Space Applications.** Richard Wilkins<sup>1</sup>, Merlyn X. Pulikkathara<sup>2,1</sup>, Enrique V. Barrera<sup>2</sup> and Ranji K. Vaidyanathan<sup>3</sup>; <sup>1</sup>Center for Applied Radiation Research, Prairie View A&M University, Prairie View, Texas; <sup>2</sup>Department of Mechanical

Engineering and Material Science, Rice University, Houston, Texas; <sup>3</sup>Advanced Ceramics Research, Tucson, Arizona.

Materials based on carbon nanotubes hold great promise for a variety of applications relevant to space exploration and the aerospace industry. Materials used for these applications will be subject to hostile environments including increased levels of high-energy particulate radiation. The type, energy range and fluence (and therefore the adsorbed dose) of the radiation will depend on the environment of the space mission. While it is not feasible to conduct an exhaustive study of the effects of space radiation on the earth's surface, experiments can be designed to simulate space exposure using sources which represent particular components of the relevant radiation environments. We will present results from irradiation experiments conducted on "buckypapers" of carbon single-walled nanotubes (SWNT) and composites of SWNT and polyethylene (currently, the principal material used for passive shielding in space). We have exposed these materials to a variety of radiation types relevant to the space environment: high-energy neutrons, protons, heavy ions and gamma rays. Experiments with buckypapers have focused on radiation damage mechanism to the nanotubes. Experiments with the composites have concentrated on radiation effects on the material and shielding properties of the composites. The nanotubes used in the studies of composite materials have been both functionalized and non-functionalized SWNT. We will discuss the implications of our experimental results on the material considerations for aerospace application of carbon nanotube materials.

#### 10:30 AM \*NN6.6

**Radiation and Temperature Effects on Carbon nanotube/polymer Composite Thin Films.** Kwanwoo Shin and Ebrahim Najafi; Materials Science and Engineering, Gwangju Institute of Science and Technology, Gwangju, South Korea.

Carbon nanotubes are one of the preferred fillers for polymer composites due to their exceptional electrical and mechanical properties. We have studied degradation of Poly (methyl methacrylate)-CNT nanocomposite thin films, using a UV-ozone and an e-beam radiation as a function of CNT concentration. We have shown that the addition of CNT fillers can have a dramatic reinforcement effect on the nature of degradation by both high-energy radiations, where polymer free radicals are mainly responsible for the proliferation of degradation. In addition, CNT networks can effectively disperse the radiation energy. The saturation in the reinforcement effect was observed when a concentration of CNT was approximately 0.5 wt %. This concentration was interpreted in terms of a critical concentration for percolation of the CNT network, and the result was consistent with the sheet resistivity measurement for which physical contact between CNT fibers was evident. The results are then correlated with systemic study of the rheological and chemical properties of polymer-CNT composites by thermal analysis (TGA and DSC), where the activation energies responsible for polymer thermal degradation are accurately measured. Part of this work was supported by the Ministry of Science and Technology of Korea through Proton Accelerator User Program (No. M102KS010001-02K1901-01810).

#### 11:00 AM NN6.7

**In Situ Formation of Carbon Nanotubes in Polymer Derived Ceramics.** Michael Scheffler, James D. Whitt and Rajendra K. Bordia; Department of Materials Science and Engineering, University of Washington, Seattle, Washington.

Due to their outstanding mechanical and physical properties Carbon Nanotubes (CNTs) are considered to be the ultimate reinforcement for composite materials. However, the processing of CNT reinforced ceramics is a challenge. CNTs are expensive, difficult to disperse and potentially a health hazard. Here we present results on a new approach of ceramic processing which combines preceramic polymers and in situ generation of CNTs. These materials can be processed at comparatively low temperatures. A special characteristic of the thermal transformation of the preceramic polymer to ceramic is the release of hydrocarbons in the temperature range from 400 to 900 degree C which overlaps with the temperature range of CNT formation by CVD processing. In this work preceramic polymers (polycarbosilanes, polysiloxanes) were doped with transition metal compounds (Fe, Co, Ni and mixtures thereof), crosslinked and pyrolyzed in argon atmosphere between 800 degree C and 1200 degree C. The pyrolysis products were investigated with respect to the formation of CNTs and their morphology. Characterization was carried out with thermal analysis, XRD, SEM and TEM analysis. The ceramic residue was in the range 70 to 80 %. The samples doped with transition metals showed the formation of CNTs after pyrolysis in argon atmosphere. The morphology of the CNTs was found to be influenced by the type and concentration of transition metal, and the pyrolysis parameters. The in situ formation of CNTs from decomposition gases of preceramic polymers offer potential for the in situ formation of carbon nanotubes in a (polymer derived) ceramic

matrix for high temperature light weight applications.

#### 11:15 AM NN6.8

**Investigation of Space Radiation Resiliency of Carbon Nanotube Based Nanocircuits.** B. W. Jacobs<sup>1</sup>, V. M. Ayres<sup>1</sup>, M. A. Crimp<sup>1</sup>, R. M. Ronningen<sup>1</sup>, A. F. Zeller<sup>1</sup>, H. C. Shaw<sup>2</sup>, J. B. Benavides<sup>2</sup>, A. J. Kogut<sup>2</sup>, M. P. Petkov<sup>3</sup> and J. B. Halpern<sup>4</sup>;  
<sup>1</sup>Michigan State University, East Lansing, Michigan; <sup>2</sup>NASA Goddard Space Flight Center, Greenbelt, Maryland; <sup>3</sup>NASA Jet Propulsion Laboratory, Pasadena, California; <sup>4</sup>Howard University, Washington, District of Columbia.

Space exploration is entering a new era, with the advent of widespread use of new micro space probes. However, the increased use of miniaturized equipment corresponds to a decreased availability to carry radiation shielding against the space radiation environment. It is well documented that silicon electronics based on diffusive transport through a channel are degraded or damaged by space radiation environments that alter the conduction path, by any of several mechanisms<sup>i</sup>. A switch to new nanoelectronics may obviate several of the problems encountered current electronics. In prototype carbon nanotube transistor circuits, there are no conventional pn junctions or isolated gates to damage, as transistor action occurs primarily by varying the contact resistance rather than the channel conductance<sup>ii</sup>. In previously reported research, single and multi-walled carbon nanotubes have exhibited enhanced heavy-ion radiation resilience<sup>iii</sup>. Enhanced proton radiation resilience has been observed in GaAs quantum dots<sup>iv</sup>. We will present results of an investigation into fundamental radiation issues in the new nanoscale materials and circuit architectures. We have fabricated simple nanocircuits to investigate the electrical characteristics of carbon nanotubes and gallium nitride nanowires so as to analyze the radiation resilience of these novel circuits, and specifically how robust the nanotube-metal contact is after significant doses of radiation. Total dose and heavy ion radiation experiments are ongoing at the NASA Jet Propulsion Laboratory, and at the National Superconducting Cyclotron Laboratory, Michigan State University. Electrical characterization is ongoing at the NASA Goddard Space Flight Center. Scanning probe microscopies, transmission and scanning electron microscopies and Raman and FTIR spectroscopies are used to analyze damage points on the samples. The research goal is to determine if the promise of ultra lightweight circuits with improved space radiation resilience can be realized. <sup>i</sup>P. Lenahan and J.F. Conley, Jr., "A comprehensive physically based predictive model for radiation damage in MOS systems", Appl. Phys. Lett., Vol. 71, pp.2413-2423 (1998) <sup>ii</sup>S. Heinze, J. Tersoff, R. Martel, V. Derycke, J. Appenzeller, and P. Avouris, "Carbon nanotubes as Schottky barrier transistors", Phys. Rev. Lett., Vol. 89 (2002) <sup>iii</sup>B. W. Jacobs, V. M. Ayres, M.A. Crimp, and H. C. Shaw, "Nanotube and nanowire devices in the space radiation environment", Bull. Am. Phys. Soc., Vol. 49, No. 1, p. 249 (2004). <sup>iv</sup>R. Leon, S. Marcinkevicius, J. Siegert, B. Cechavicius, B. Magness, W. Taylor and C.Lobo, "Effects of proton irradiation on luminescence emission and carrier dynamics of self-assembled III-V quantum dots", IEEE Trans. Nucl. Sci., Vol. 49, pp.2844-2851 (2002)

#### 11:30 AM \*NN6.9

**Polyimides Containing Pendent Perfluoromethyl Groups: Synthesis, Properties, and Space Applications.**

Stephen Z. D. Cheng and Frank W. Harris; The Maurice Morton Institute of Polymer Science, The University of Akron, Akron, Ohio.

The overall goal of this ongoing research is to modify the structure of aromatic polyimides to attain solubility in common organic solvents without substantially decreasing the rigidity of their backbones. The primary approach of this research has involved the introduction of trifluoromethyl-substituted, twisted-biphenyl structures in polyimide backbones. The steric repulsion of the substituents in the 2- and 2'-positions of the biphenyl moieties in such monomers twist the rings dramatically out of plane. The resulting twisted conformation inhibits chain packing and crystallization. The severe twist also breaks up the conjugation along the backbone and reduces color. Hence, it was initially postulated that polymers obtained from these monomers would be soluble in common organic solvents and would form optically-clear films that would be transparent in the visible light region. A secondary approach has involved the synthesis and polymerization of trifluoromethyl-substituted 3,6-diarylpromellitic dianhydrides. The pendent substituents also break up the chain packing resulting in solubility in organic solvents. Space applications of the polyimides will be discussed. **General Properties.** The polymers had intrinsic viscosities as high as 10 dl/g (m-cresol at 30 °C) and glass transition temperatures ( $T_g$ s) as high as 370 °C. Many of the polymers, particularly those based on the substituted dianhydrides and 6FDA, were soluble in acetone and tetrahydrofuran. **Thin Film Properties.** Depending on their structure, the polymers could be cast from phenolic or ketone solvents into 10-20  $\mu$ m-thick, colorless films. WAXD and FTIR analysis revealed that the chains aligned parallel to the substrate surface during the casting process. The extent of

in-plane orientation was found to depend on the polymer chain rigidity and linearity and on the polymer molecular weight. The orientation resulted in anisotropic thermal, mechanical, optical and dielectric properties. The  $T_g$  along the out-of-plane direction was lower than the  $T_g$  determined along the in-plane direction. The coefficient of thermal expansion (CTE) was approximately one order of magnitude higher out-of-plane than that in-plane, which was as low as  $6.98 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  for BPDA-PFMB films. The refractive index in-plane was 0.02 to 0.2 higher than the refractive index out-of-plane, which varied from 1.500 to 1.710. This anisotropy in refractive index was utilized in the development of retardation layers for liquid crystal displays. The out-of-plane dielectric constant, which was determined according to ASTM-150-181, ranged from 2.25 to 2.75 at 1 MHz. **Fiber Properties.** Fibers were prepared from BPDA-PFMB from m-cresol and p-chlorophenol solutions using a dry-jet wet spinning method. The fibers were elongated and annealed above 400 °C to achieve tensile strengths of 3.3 GPa and tensile moduli over 130 GPa. The fibers were more thermally stable than any other reported high performance fiber.

SESSION NN7: Irradiation Effects in Materials for Space Applications

Chair: Zelina Iskanderova  
Wednesday Afternoon, December 1, 2004  
Room 207 (Hynes)

#### 1:30 PM \*NN7.1

**Nanocomposites made out of nanoclusters as building blocks for space applications.** You Qiang, Jiji Antony, Amit Sharma,

Sweta Pendyala, Joe Nutting, Daniel Meyer and Daniel Sikes; Department of Physics, University of Idaho, Moscow, Idaho.

The development on new advanced nanocomposites that possess unique mechanical, thermal, magnetic, electrical and chemical properties with sustainability in different harsh space environments will be a future challenge in materials science and engineering industry. To successfully make these nanocomposites into real-life applications, we have developed new types of nanocomposites assembled by monodispersed nanoclusters as building blocks from our newly developed ultrahigh intensity nanocluster source. Different nanostructured composites are synthesized by controlling independently the incident nanocluster size, concentration, and impact energy to have a wide variety of controlled electronic, magnetic, mechanical and chemical properties for many possible space applications. Nanocluster size, size distribution and nanocrystalline structures have been studied by TOF, AFM, TEM and HRTEM. Superhard nanocomposites (e.g. TiN and TiAlN coatings) are deposited at room temperature by energetic nanocluster impact on the surface of a substrate (HSS steel, glass even Teflon and plastic). These coatings can enhance greatly the lifetime of high-speed bearings in satellite gyroscope and other moveable parts in space. Magnetic properties of soft nanocomposites made out of Fe or Co nanoclusters have been investigated by SQUID, MFM and magnetic x-ray circular dichroism (MXCD). An interesting dependence of magnetization was found on the cluster size and the concentration in a matrix. Ultrahigh magnetic moment has been found due to more enhanced orbital moment of nanoclusters in the nanocomposites. We also found the photoluminescence of ZnO nanoclusters at room temperature is size dependent. There is a blue shift from 3.13 eV of bulk ZnO to 3.45 eV of ZnO nanocluster films that is important for space applications of photoelectronic devices. \* Research supported by Idaho NSF-EPSCoR, Battelle-PNNL, ONR and DOE-EPSCoR. Contact information: youqiang@uidaho.edu

#### 2:00 PM NN7.2

**Simulation of InAsSb/InGaAs Quantum Dots for Optical Device Applications.** Paul von Allmen, Seungwon Lee and Fabiano Oyafuso; Jet Propulsion Laboratory, Pasadena, California.

Self-assembled InAsSb/InGaAs quantum dots are candidates for optical detectors and emitters in the 2-5 micron band with a wide range of applications for atmospheric chemistry studies. While photoluminescence peaks at wavelengths as high as 2.2  $\mu$ m have been measured in InAsSb dots [1], the present study aims at determining the maximum wavelength theoretically achievable. The energy band gap of unstrained bulk InAs(1-x)Sb(x) is smallest for x=0.62 but biaxial strain for bulk InAs(1-x)Sb(x) grown on In(0.53)Ga(0.47)As shifts the energy gap to higher energies and the smallest band gap is reached for x=0.51, which seems therefore to be the preferred concentration for long wavelength optical devices. We next examine how the electronic confinement in the quantum dots modifies these simple considerations. We have calculated the electronic structure of lens shaped InAs(1-x)Sb(x) quantum dots with diameter 37 nm and height 4 nm embedded in a In(0.53)Ga(0.47)As matrix of thickness 7 nm and lattice matched to an InP buffer. The relaxed atomic

positions were determined by minimizing the elastic energy obtained from a valence force field description of the inter-atomic interaction. The electronic structure was calculated with an empirical tight binding approach with the parameters obtained from Ref. [2]. We find that for Sb concentrations larger than  $x=0.5$ , the electrons are not confined in the dot, which results from the conduction band edge in the  $\text{InAs}(1-x)\text{Sb}(x)$  being at higher energy than in the  $\text{In}(0.53)\text{Ga}(0.47)\text{As}$  matrix. The respective positions of the bulk conduction and valence band edges clearly illustrate that the  $\text{InSb}/\text{In}(0.53)\text{Ga}(0.47)\text{As}$  heterostructure is of type II. We will further show the variation of the exciton energy and oscillator strength as a function of Sb concentration throughout the region where the electron is confined in the  $\text{In}(0.53)\text{Ga}(0.47)\text{As}$  buffer material. [1] Y. Qiu and D. Uhl, Appl. Phys. Lett. 84, 1510 (2004). [2] J.M. Jancu, R. Scholz, F. Beltram and F. Bassani, Phys. Rev. B 57, 6493 (1998).

**2:15 PM NN7.3**  
**Correlation of Optical Luminescence with Radiation Hardness in Doped  $\text{LiNbO}_3$  Crystals.** Kelly Simmons-Potter<sup>1</sup>, William J. Thomes<sup>2</sup>, Barrett G. Potter, Jr.<sup>3</sup> and Louis Weichman<sup>2</sup>; <sup>1</sup>Electrical and Computer Engineering, University of Arizona, Tucson, Arizona; <sup>2</sup>Sandia National Laboratories, Albuquerque, New Mexico; <sup>3</sup>Materials Science and Engineering, University of Arizona, Tucson, Arizona.

Transient ionizing radiation fields have been observed to cause substantial optical loss in undoped  $\text{LiNbO}_3$  crystals operating at 1.06 microns. This loss is slow to recover and makes the selection of this material for Q-switch applications in radiation environments unfeasible. We have studied the effects of Mg doping on the radiation response of  $\text{LiNbO}_3$  crystals, and have investigated the optical luminescence of doped and undoped samples. Our results indicate a strong correlation between crystal defects, mainly formed during crystal growth, and the radiation response of the materials. These findings have enabled us to produce rad-hard  $\text{LiNbO}_3$  crystals for use in high gamma-field environments. This work was partially supported by Sandia National Laboratories. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

**2:30 PM \*NN7.4**  
**Nanosensors for Rapid Hydrogen Detections.** Zhili Xiao<sup>1,2</sup>, Michael P. Zach<sup>1</sup>, Tao Xu<sup>1</sup>, Ulrich Welp<sup>1</sup>, Wai-Kwong Kwok<sup>1</sup> and George W. Crabtree<sup>1</sup>; <sup>1</sup>Materials Science Division, Argonne National Laboratory, Argonne, Illinois; <sup>2</sup>Department of Physics, Northern Illinois University, DeKalb, Illinois.

The successful transition to a hydrogen based economy depends on the ability to fully control and monitor hydrogen. Hydrogen sensors will play an important role in this endeavor. Currently, commercial sensors have slower response times (8s or longer) than is needed for most hydrogen economy applications, e.g., 0.1 s for vehicle passenger compartments/trucks, and 0.1-0.6s for personnel monitoring. In space application, hydrogen sensors with short response time and miniature size are highly desired for real time hydrogen leak detection. We fabricated hydrogen sensors with response times down to a few tens of milliseconds by utilizing nanomaterials. The hydrogen sensors that will be elaborated in this talk are based on two-dimensional (2D) arrays of palladium nanobeads on porous anodized aluminum oxide (AAO) substrates. Highly ordered nanopore arrays were achieved in aluminum oxide membranes through an electrochemical anodization approach. The process converts aluminum foils with various thicknesses into aluminum oxide by placing the foils in an acid solution under a positive electric field. The aluminum oxide contains an array of self-organized pores with diameters and pore-pore distances that can be adjusted by changing the anodization voltage and the acid concentration. Porous AAO membranes with pore diameters ranging from 8 nm to 400 nm have been successfully fabricated. By controlling the pore-to-wall ratio of the AAO membrane and adjusting the film thickness, we could control the morphology of the film to form a 2D palladium nanobead array on top of the membrane. We found that the rapid change of the resistance in a palladium nanobead array can be as fast as a few tens of milliseconds. The mechanism and the influence of doping the nanobeads by either using an alloy target or by multiple target co-sputtering was explored. We will also present results on hydrogen sensors based on other mechanisms, for example, nanoscale Schottky barriers between palladium and semiconductors.

**3:00 PM NN7.5**  
**Composite Satellite Housings Materials Containing Nickel Nanostrands for Elimination of Metal Sheet Electromagnetic Interference (EMI) Shielding Materials.** George Hansen<sup>2</sup>, Max D. Alexander<sup>1</sup>, Matt Pettit<sup>2</sup> and Heather J. Dowty<sup>1</sup>; <sup>1</sup>Polymer Branch, Air Force Research Laboratory, Dayton, Ohio; <sup>2</sup>Metal Matrix Composites, Heber City, Utah.

EMI shielding of electrical boxes and electrostatic discharge are both of key concern for satellites and other space systems. Without the continuous conductive coatings electrical charge can build up on large structures and then destructively discharge, damaging sensitive electrical systems. Likewise, the electrical components require EMI shielding to prevent system interference from adjacent or external sources. Traditional solutions have involved the use of Aluminum or other metal sheets bonded to the increasingly more popular light-weight composite electronic housings. However, this can present a myriad of integration and bonding issues. Here we present data on the use of a novel form of nano-size high aspect ratio nickel (Nickel Nanostrands) which when directly incorporated into the composite structure provides shielding equivalent to metal bonded structures. These systems have been tested in both the DC (static dissipation) and high frequency (Shielding Effectiveness) regimes at a variety of low loading resulting in the determination of the corresponding structure-property relationships. The durability of these materials will also be reported under a variety of testing conditions.

**3:30 PM \*NN7.6**  
**Radiation Effects and Defects in Cubic Boron Nitride. A Promising Multifunctional Material for Severe Environment Conditions.** Vasile Sergiu Nistor, National Institute for Materials Physics, Magurele-Bucuresti, Romania.

Cubic boron nitride (cBN) crystals, the second hardest known material, with sphalerite structure exhibits properties which are superior to diamond in what concerns its lower chemical reactivity, higher thermal stability and semiconducting properties. The energy band-gap of boron nitride, varying between 5.4 and 7.0 eV at room temperature, is the highest among all covalent-bonded materials known so far. This band-gap is suitable for ultraviolet (UV) detectors and UV light emitting diodes operable at wavelengths in the deep UV regime. Moreover, it has been shown [1] that p-n junctions and UV-light emitting diodes could be operated at temperatures as high as 900K without significant parameters changes. It is also highly resistant to radiation damage [2]. It all shows that cBN is a very promising material for semiconductor and optical devices in extreme conditions, as found in the space environment. In spite of the increasing technological interest in cBN, its growth and characterization are in a relatively early state of development. In particular, very little progress has been made to understand the defect structure and associated energy states, essential in controlling the majority of most important materials properties. We shall present the recent results of X(9.4GHz)- and W(95GHz)-band multifrequency Electron Spin Resonance (ESR) studies, in the 2.5-295K temperature range, on nominally pure (dark and amber) and beryllium doped cBN crystalline powders and single crystals, respectively. Such correlated multifrequency ESR studies in a broad temperature range have resulted in the clear identification of several paramagnetic species with axial  $\langle 100 \rangle$  and  $\langle 111 \rangle$  type symmetry and different g-tensor components, such as the D-type centers [3]. It has been also found that some of these point defects are photosensitive under in-situ illumination with Ar<sup>+</sup> and Kr<sup>+</sup> lasers at various wavelengths. New types of paramagnetic defects have been also produced following such low temperature illumination as well as irradiation with energetic particles. Based on available experimental data, as well as on previous theoretical estimations of stability and properties of the intrinsic point defects in cBN, possible structural models shall be discussed. The results of ESR measurements shall be also compared with results of TL (Thermo Luminescence), micro-PIXE (Particle Induced X-ray Emission) and micro-IL (micro-Ion Luminescence) measurements. This last technique in particular can be used as a sensitive method to map radiative recombination centres due to crystal imperfections or doping levels. References [1]. T. Taniguchi et al., Diamond and Related Materials 12, 1098 (2003) and references cited therein [2] C. Manfredotti et al., Diamond and Related Materials 10, 568 (2001) [3] S. V. Nistor et al., Diam. Rel. Mater. 10, 1408-1411 (2001).

**4:00 PM NN7.7**  
**Magnesium Implantation by Plasma Immersion in Kapton for Oxidation Protection in Low Earth Orbit.** Mario Ueda<sup>1</sup>, Ing Hwie Tan<sup>1</sup>, Renato S. Dallaqua<sup>1</sup>, Jose O. Rossi<sup>1</sup>, Nicole Demarquette<sup>3</sup> and Antonio F. Beloto<sup>2</sup>; <sup>1</sup>Associated Laboratory of Plasma, National Institute for Space Research, Sao Jose dos Campos, S.Paulo, Brazil; <sup>2</sup>Associated Laboratory of Materials and Sensors, National Institute for Space Research, Sao Jose dos Campos, S.Paulo, Brazil; <sup>3</sup>Departamento de Engenharia Metalurgica e Materiais, Escola Politecnica da USP, Sao Paulo, S.Paulo, Brazil.

Low Earth Orbit environment is rich in atomic oxygen, degrading polymer based materials and limiting their lifetime to a few years. A thin metal oxide layer on the surface of these polymers is a promising protection against oxidation. Implantation of metal ions and subsequent oxidation is an ideal mean of obtaining these protective coatings since a mixing layer is created providing superior adhesion to the substrate. We will report on the implantation of magnesium ions



into Kapton films to create a protective MgO layer. Direct implantation is accomplished in a vacuum arc system with a straight magnetic duct, with samples oriented parallel to the plasma stream to prevent contamination by macroparticles and minimize deposition. Implantation with and without magnetic field will be compared since charging effects are expected to be severe in the much denser magnetically confined plasmas. A third implantation approach is described which consists of depositing a thin Mg coating on the samples and subsequently biasing it to high voltages in a nitrogen plasma for recoil implantation. All samples will be tested for oxygen degradation resistance by exposure to oxygen plasmas generated in an RF plasma reactor. Thermal cycling test is accomplished through successive dips in liquid nitrogen and in a pre-heated oven. Adhesion is qualitatively checked by peeling an adhesive tape and inspecting the surface with a Scanning Electron Microscope.

#### 4:15 PM NN7.8

##### **Modeling the photovoltaic response of p+n GaAs solar cells using Silvaco following 53 MeV proton irradiation.**

Jeffrey Hamilton Warner<sup>1</sup>, Aaron L. Crespin<sup>2</sup> and Robert J. Walters<sup>1</sup>;  
<sup>1</sup>US Naval Research Laboratory, Washington, District of Columbia;  
<sup>2</sup>Naval Postgraduate School, Monterey, California; <sup>3</sup>US Naval Research Laboratory, Washington, District of Columbia.

In this paper, we report the results of modeling the photovoltaic response of p+n GaAs solar cells following 53 MeV proton irradiation using Silvaco. The devices were irradiated with seven fluence values ranging from  $1 \times 10^{12} \text{ cm}^{-2}$  up to  $7 \times 10^{13} \text{ cm}^{-2}$ . Deep Level Transient Spectroscopy measurements were performed to determine the defect trapping centers introduced after irradiation. The activation energy, capture cross-section, and defect concentrations for all the defects are calculated and used for inputs in the Silvaco model. Illuminated current-voltage measurements were performed under 1 sun, AM0 conditions, to determine the degradation of the photovoltaic parameters as a function of proton fluence. The theoretical calculations of the degradation of the photovoltaic parameters are compared with the experimental data to verify the model used.

#### 4:30 PM NN7.9

##### **Glassy Polymeric Carbon Heat Management Structures for Space Applications.** Claudiu I. Muntele, Lawrence R. Holland, Iulia C. Muntele, Robert L. Zimmerman and Daryush Ila; Physics, Alabama A&M University, Normal, Alabama.

We developed an innovative composite material based on the decades-old polymeric carbon technology altered at nanoscale level by selective incorporation of metallic nanoclusters in order to obtain enhanced in-plane thermal and electrical conductivity. The concept takes advantage of the already good thermal conductivity of glassy polymeric carbon (GPC) and the ability of this form of carbon of alloying easily to most of the common metals. These properties along with a very good oxidation resistance at high temperatures and extremely low permeability for any chemical for a large temperature interval make GPC composites an almost ideal candidate primarily for heat management structures, but also for radioactive fuel containment aboard a nuclear-powered space system. We tested several Metal-GPC combinations for thermal and electrical conductivity, as well as thermal and electrical anisotropy of in-plane vs. transversal conductivity and compared to the current materials used for space applications. Variations of these properties with the type and amount of metal incorporated in the GPC structure will be presented during the meeting.

#### 4:45 PM NN7.10

##### **Beam Properties of the New Radiation Effects Research Stations at Indiana University Cyclotron Facility.**

Barbara vonPrzewoski<sup>1</sup>, Thomas Rinckel<sup>1</sup>, William Manwaring<sup>1</sup>, Charles C. Foster<sup>2</sup>, Edmund R. Hall<sup>1</sup>, Tony Kinser<sup>1</sup> and Kenneth M. Murray<sup>3</sup>, <sup>1</sup>Indiana University Cyclotron Facility, Bloomington, Indiana; <sup>2</sup>Foster Consulting Services, LLC, Tacoma, Washington; <sup>3</sup>KM sciences, Bloomington, Indiana.

Two new beamlines for radiation effects studies at the Indiana University Cyclotron Facility will be described. The new, state-of-the-art radiation effects research stations (RERS1 and RERS2) were completed in fall of 2003. Their applicability to radiation hardness testing of materials for space applications will be discussed. Beam characteristics such as transmission, energy, energy spread and lateral profile are described and compared to calculations. Beam is extracted from the cyclotron at 205 MeV and delivered by a fast kicker system to each of the new irradiation stations. One of the beamlines (RERS2) has Beryllium degraders well upstream of the radiation effects station which may be used to degrade the beam energy to 52, 72, 102, or 149 MeV. Both beamlines have the option to degrade the beam energy immediately upstream of the device under test (DUT). Momentum analysis downstream of the degrader guarantees nearly mono-energetic beams at the location of the DUT. Available fluxes

are between  $10^2$  and  $10^{11}$  protons/second/cm<sup>2</sup> (at 200 MeV). During irradiations, the dose is automatically measured with a calibrated secondary electron monitor and logged.

SESSION NN8: Polymers in Space  
Chair: Bruce Banks  
Thursday Morning, December 2, 2004  
Room 207 (Hynes)

#### 8:00 AM \*NN8.1

##### **Low Earth Orbital Atomic Oxygen Interactions with**

**Spacecraft Materials.** Bruce A. Banks, Sharon K. Miller and Kim K. de Groh; Electro-Physics Branch, NASA Glenn Research Center, Cleveland, Ohio.

Atomic oxygen, formed in Earth's thermosphere, interacts readily with many materials on spacecraft flying in low Earth orbit (LEO). All hydrocarbon based polymers, graphite-epoxy composites and silicones are easily oxidized by the impact of 4.5 eV atomic oxygen as the spacecraft rams into the residual atmosphere. The resulting interactions can change the chemistry, morphology, optical and emissive properties as well as the thickness of these materials. As a result of the erosive properties of atomic oxygen on polymers and composites, protective coatings have been developed and are used to increase the functional life of polymer films and composites that are exposed to the LEO environment. The results of in-space and ground laboratory exposure of materials to atomic oxygen will be presented for hydrocarbon based polymers, graphite, graphite-epoxy composites and silicones. The merits, risks and issues associated with the use of atomic oxygen protective coatings over hydrocarbon based polymers will be presented to show recommended practices and explain observed in-space failures.

#### 8:30 AM NN8.2

##### **Radiation Testing of POSS-Kapton Films for Space**

**Applications.** Jenn-Ming Yang, <sup>1</sup>UCLA, Los Angeles, California;

<sup>2</sup>Aerospac Corporation, El Segundo, California; <sup>3</sup>Air Force Research Laboratory, EdwardsAFB, California.

Radiation Testing of POSS-Kapton Films for Space Applications H. Katzman, M. Meshishnek, D. Coleman, and G. Steckel Space Materials Laboratory The Aerospace Corporation El Segundo, CA 90245 S. Tomczak ARFL/PD Edwards AFB, CA 93524 J.-M. Yang Department of Materials Science & Engineering University of California Los Angeles, CA 90095 Polyimide films such as Kapton are used extensively in space applications. Therefore, understanding the behavior of these materials when exposed to the earth-orbit environment is important in predicting performance characteristics such as in-space durability. In the low earth orbit (LEO) environment, the presence of high atomic oxygen (AO) flux and solar radiation can rapidly degrade the polymers. Recent studies conducted at the AFRL have conclusively demonstrated that the incorporation of POSS (polyhedral oligomeric silsesquioxanes) into organic polymers results in extended lifetimes in low earth orbit. When exposed to atomic oxygen, POSS-polymers form a passivating ceramic layer on the surface that protects the virgin polymer underneath. The objective of this work is to investigate the behavior and degradation mechanisms of POSS-Kapton films subjected to radiation simulating that found in geosynchronous (GEO) orbit. The radiation exposure includes ultraviolet radiation, as well as high fluxes of electrons and protons of widely varying energies. POSS-Kapton films have been exposed to the equivalent of 5-year GEO radiation environments in the Aerospace Space Simulation Chamber. Their chemical, mechanical and physical degradation as a result of this exposure will be discussed.

#### 8:45 AM \*NN8.3

##### **Investigation of Oxidative Degradation of Bisphenol A Polycarbonate under Swift Heavy Ion Irradiation.**

Frederique Dehaye<sup>1</sup>, Emmanuel Balanzat<sup>2</sup>, Etienne Ferain<sup>1</sup> and Roger Legras<sup>1</sup>; <sup>1</sup>High Polymer Physics and Chemistry Lab, Catholic University of Louvain, Louvain-la-Neuve, Belgium; <sup>2</sup>CIRIL, CEA/CNRS/ENSICAEN, Caen, France.

Bisphenol A polycarbonates (BPA-PC) are widely used for applications related with ionizing radiation. These applications include the particle track detectors, the spacesuit helmets, the medical devices, the micro and nanoporous particle track etched membranes (nano-PTM), ect. Involved in the nano-PTM development for supplying optimized templates for the synthesis of metallic nanowires and polymeric nanotubules with diameters going from 10 to 100 nm<sup>a</sup>, we are studying the effects of the electronic stopping power  $(dE/dx)_e$  and the irradiation atmosphere (vacuum and oxygen) on the chemical modifications in BPA-PC films by performing swift heavy ion (SHI) irradiations with ion beams at energies of a few MeV/amu. Therefore Fourier transform infrared (FTIR) spectroscopy has been used to

monitor *insitu* as well the BPA-PC films degradation as the emitted gases under SHI irradiation. The results related to the irradiations under vacuum have already been presented (films<sup>b</sup> and gas<sup>c</sup> analysis). We present here an investigation of oxidative degradation of BPA-PC under SHI irradiations and post-irradiation short-term storage. SHI irradiations were performed at the GANIL accelerator using medium energy facilities. The covered (dE/dx)<sub>e</sub> range goes from 1.6 to 50 MeV/mg/cm<sup>2</sup>. To distinguish the contribution to the gaseous carbon oxides production of the BPA-PC oxygen from the atmosphere oxygen, the SHI irradiations were performed under <sup>16</sup>O<sub>2</sub> and <sup>18</sup>O<sub>2</sub>. The FTIR irradiated films analysis shows that the radiochemical yields of the isopropyl groups destruction and the hydroxyl groups creation are increased by the presence of oxygen. The FTIR emitted gases analysis enhances the formation of C<sup>16</sup>O and C<sup>18</sup>O as well as C<sup>16</sup>O<sup>16</sup>O, C<sup>16</sup>O<sup>18</sup>O and C<sup>18</sup>O<sup>18</sup>O. Methane and ethylene are produced when BPA-PC is irradiated. Above a (dE/dx)<sub>e</sub> threshold value, the formation of acetylene is observed whatever the irradiation atmosphere. <sup>a</sup>:<http://www.it4ip.be> <sup>b</sup>:F. Dehaye, E. Balanzat, E. Ferain, R. Legras, Nucl. Instr. and Meth. B, 209 (2003), 103-112. <sup>c</sup>:F. Dehaye, E. Balanzat, E. Ferain, R. Legras, IRAP 2004 conference.

#### 9:15 AM NNS.4

##### **Enhancement of Surface Durability and Stability of Conductive and Non-conductive Paints in Space Environment.**

Yuri Gudimenko<sup>1</sup>, Richard Ng<sup>1</sup>, Jacob Kleiman<sup>1</sup>, Zelina Iskanderova<sup>1</sup>, Anatoli V. Grigorievskiy<sup>2</sup> and David L. Edwards<sup>3</sup>, <sup>1</sup>Space Materials, ITL Inc, Toronto, Ontario, Canada; <sup>2</sup>Institute Kompozit-Test, Korolev, Moscow Region, Russian Federation; <sup>3</sup>NASA Marshall Space Flight Center, Huntsville, Alabama.

Enhancement of Surface Durability and Stability of Conductive and Non-conductive Paints in Space Environment. Y. Gudimenko-1, R. Ng-1, J. Kleiman-1, Z. Iskanderova-1, A. Grigorievskiy-2, L. Kiseleva-2, M. Shuiskiy-2, D. Edwards-3, M. Finckenor-3 Various space environmental factors have shown to cause damage and degradation to thermal control components and materials on orbiting spacecrafts, producing changes in optical, mechanical, and chemical properties. Prolonged exposure of these sensitive surface spacecraft materials to the space environment can result in degraded system performance. Due to the importance of thermal control material durability and performance in various space missions, a surface modification program was continued at ITL Inc. to evaluate its patented surface modification technology. Known as Photosil, this surface modification was used to evaluate a variety of space-related thermal control coatings, and assess its impact on the stability of coatings functional properties and space durability. Significant enhancement of AO erosion resistance was achieved on organic-based conductive and non-conductive paints, treated by various versions of the developed technology. For some of the paints, identical samples are still being exposed to the LEO space environment as part of the Material on International Space Station Experiment (MISSE). This protective technology has shown, as per ground-based accelerated testing, to significantly improve the AO resistance of polyurethane-based paints (Aeroglaze), Russian acrylic-based conductive advanced EKOM and other types of paints, while leaving their functional properties almost unchanged. Various examples of these applications are presented. Recent results are also presented to further improve the AO durability of a wide range of thermal control coatings, never previously treated by Photosil(TM). The thermal control coatings evaluated in this program represent the materials from various international development and manufacturing sources. They include conductive and/or non-conductive white, black, gray, and silver-gray space paints. These paints vary in the types and concentrations of binders (acrylic, silicone, or silicate) and pigments (inorganic and/or organic). The paper provides a more detailed description on the various types of thermal control coatings used and as well as some examples of data that have been generated. Functional properties and characteristics, such as durability in an imitated LEO environment, thermal-optical characteristics - solar absorbance and thermal emittance, electrical surface resistivity, adhesion, stability at thermal cycling, and outgassing characteristics of the Photosil(TM)-treated coatings are being verified. Pristine (untreated) and Photosil(TM)-modified samples are tested in an AO ground-based accelerated simulator and evaluated for changes in mass loss, thermo-optical properties, surface morphology, and surface chemistry changes.

#### 9:30 AM NNS.5

##### **Preventive Surface Treatment of Silicone Materials for Outgassing and Contamination Reduction in Space Applications.**

Zelina Iskanderova<sup>1</sup>, Jacob Kleiman<sup>1</sup>, Yuri Gudimenko<sup>1</sup>, Richard Ng<sup>1</sup> and Daniel Kaute<sup>2</sup>; <sup>1</sup>Space Materials, ITL Inc, Toronto, Ontario, Canada; <sup>2</sup>PlasmaTreat, Mississauga, Ontario, Canada.

Preventive Surface Treatment of Silicone Materials for Outgassing and Contamination Reduction in Space Applications. Z. Iskanderova\*, J.

Kleiman\*, Y. Gudimenko\*, R. Ng\*, D. Kaute\*\* \*Integrity Testing Laboratory Inc.; Markham ON, Canada \*\*PlasmaTreat; Mississauga ON, Canada Application of organosilicone polymers as protective coatings has a long history and tradition. These polymers are clearly advantageous over their organic analogs in high thermal stability and resistance to environmental factors such as ultraviolet and atomic oxygen in space. At the same time, it has been found [1] that contaminating films on spacecraft sensitive materials surfaces in many cases consisted primarily of products resulting from the interaction of atomic oxygen with silicones. The instability of silicone in space systems has been a barrier to their effective use as coatings, despite many useful and sophisticated applications. Necessity of a drastic reduction of the outgassing of volatiles and the following contamination has thus become a challenging problem. Undoubtedly, this required a revision of the existing approaches to synthesis, production, and application of organosilicone coatings. However, the general tendency for development of the organosilicone polymer coatings provides little ground for optimism to prevent the outgassing of volatile contaminating moieties in oxidative space environment. Development of a concept of ground-based preventive surface treatment for surface stabilization and space durability enhancement of silicone materials is presented in this paper, launching with formulation of the idea and concluding with efficiency demonstration by the examples of silicone materials treatment by special low temperature plasma technologies. Few advanced aerospace and space-related organosilicone polymers have been used to demonstrate the promising potential application of this approach. It was shown, that the application of FLUME? plasma systems, introduced by PlasmaTreat? [2], not only expands opportunities to modify the surface structure, but also essentially simplifies the technological development to reduce or prevent contaminations caused by silicone-coated space materials and structures. References [1] E.M. Silverman. Space Environment Effects on Spacecraft: LEO Material Selection Guide, NASA CR-4661, Aug. 1995. [2] [www.PlasmaTreat.com](http://www.PlasmaTreat.com) Contact Information \* Integrity Testing Laboratory Inc. 80 Esan Park Drive Markham ON, Canada L3R 2R7 (905) 415-2207 \*\* PlasmaTreat North America Inc. 2810-1 Argentinia Road, Mississauga ON, Canada, L5N 8L2 (905) 816-2350

#### 10:00 AM \*NNS.6

##### **Surface Modification of Polymers, Paints and Composite Materials Used in the Low Earth Orbit Space Environment.** Jacob I. Kleiman, Integrity Testing Laboratory Inc., Markham, Ontario, Canada.

Polymers and composites, exposed to low Earth orbit (LEO) environment undergo dramatic changes and irreversible degradation of physical characteristics. While many protective schemes are used to reduce the effects of LEO environment, protection of polymer materials in LEO still remains a major challenge, especially for future long duration missions or space stations. Surface modification processes proposed [1-4] as an approach to protect polymer materials from LEO environment will be discussed. Among them the PHOTOSIL<sup>TM</sup> [1-3], a surface modification process with a new approach to silicon functionalization of high-performance polymers that incorporates up to 36 at.% of Si into the polymer surface that attains new properties and is able to withstand AO erosion. The conducted tests in a fast atomic oxygen (FAO) beam facility demonstrated erosion yields lower than  $10^{-26}$  cm<sup>3</sup>/at. The treated layers exhibited excellent thermal match with the bulk of the treated material. The IMPLANTOX<sup>TM</sup> [1,2,4] is a modified ion implantation process that allows to form oxide based highly protective surface layers on polymers. The high dose ion implantation with selected elements has been performed on a range of polymers in the energy range 10 - 100 keV, using different sources. The treated materials were tested using FAO facility, with fluencies (1- 2)x10<sup>20</sup> at.O/cm<sup>2</sup> that are comparable to fluencies in LEO. After FAO testing, the samples were clear and transparent, with a glassy-like shiny surface with no signs of any surface erosion. Examples of use of the surface modification processes on various parts and systems will be presented. 1. J.I. Kleiman et al. "Polymers and Composites in the Low Earth Orbit Space Environment: Interaction and Protection", Canadian Aeronautic and Space Journal, v.45, No.2, (June 1999), 148-160. 2. J. Kleiman, "Surface Modification of Polymers Used in the Low Earth Orbit Space Environment", Metallized Plastics 5/6, Fundamental and Applied Science, ed. K. Mittal, (1998) 331-351 3. Yu. Gudimenko, et al. "Modification of Subsurface Region of Polymers and Carbon-Based Materials". U.S. Patent #5,948,484, issued September 7, 1999. 4. Z.A. Iskanderova, et al. "Surface Modification of Polymers and Carbon-Based Materials by Ion Implantation and Oxidative Conversion". U.S. Patent #5683757, issued 11 April, 1997; J.I. Kleiman et al. "Ion Implantation Protects Surfaces", Advanced Materials and Processes, (April 1998), No.4, 26-3.

#### 10:30 AM NNS.7

##### **Highly Stable Polymers Based upon Poly(m-Carborane-Siloxane) Elastomers.** Julian James Murphy, Organic Materials,

Many commonly used polymer materials contain significant amounts of inorganic fillers. These are incorporated to decrease the cost of polymer feed stocks but also to modify and improve the materials properties. Many ageing effects seen in polymers are caused by subtle modification of the polymer/filler interface. As this can be extremely complex developing a predictive ageing model is exceedingly difficult. Poly(dimethyl siloxane) is a commonly used elastomeric material, which in general is stable. Important properties of the polymer are, however, dependent upon the incorporation of particulate fillers. The interaction of such fillers with the polymer phase is complex, in addition it is intimately dependent upon a layer of water situated at the interface. When in the service environment that such materials experience is harsh and strongly desiccating certifying the performance of components is exceedingly difficult. Poly(siloxane) polymers incorporating small closed polyhedral C<sub>2</sub>B<sub>10</sub>H<sub>12</sub> species, commonly referred to as a carborane, have been produced. The carborane cage is incorporated into the polymer chain and acts essentially as a micro filler. This overcomes some of the difficulty associated with changes in the filler/polymer interface upon ageing. In addition, incorporation of the carborane is shown to yield a significant increase in the thermal stability of the material.

#### 10:45 AM NNS.8

**Polyhedral Oligomeric Silsesquioxane (POSS)-Polymers: Engineering Multi-Functional Benefits with Nanomaterials.** Shawn H. Phillips, Material Applications Branch, Edwards AFB, Edwards AFB, California.

Twelve years of research on POSS-polymers has resulted in numerous publications on both the nanoscale mechanical reinforcement and physical property improvements of incorporating truly compatible silicon oxide frameworks within polymer matrices. However, it has only been within the last few years that we have attempted to control and predict property improvements while taking advantage of their inherent multi-functional benefits. This talk will center around the engineering of 2-D and 3-D POSS networks within polymer systems, maximizing improvements through crystal/aggregate optimization, and multi-functionality with respect to space-survivable, self-healing polymers. The future need for studying the processing of POSS-polymers will also be discussed.

#### 11:00 AM NNS.9

**Volatile Evolution from Polymer Materials Induced by Irradiation with Accelerated He<sup>++</sup> Ions.** Julian James Murphy, Organic Materials, AWE, Reading, United Kingdom.

Experimentally investigating ageing caused by irradiation with energetic particles is very difficult. Firstly, performing the irradiation itself poses a number of problems. Radioactive sources can be employed but these are difficult to handle and contaminate the material precluding subsequent chemical and physical characterisation. Accelerated ion beams can also be employed to simulate particle irradiation. The high dose rates employed in such procedures means the irradiation must be performed in a vastly reduced timescale. This raises questions about the applicability of such procedure to accelerate material ageing. Secondly, difficulties arise because of the small amount of material irradiated when a sample is exposed to particle radiation. The penetration of energetic particles tends to be small so any change is localised in the near surface region. Analysing changes in such thin layers causes a number of additional problems. To simulate ageing induced by particle radiation polymer samples have been exposed to fast He<sup>++</sup> ions in an accelerated ion beam. The ions pass through a 10micron thick window of Havar foil before impacting upon the sample. Volatile species evolved from the materials upon bombardment are contained within the irradiation chamber by the foil window. Analysis of such species is shown to be a highly sensitive probe for investigating chemical changes in the exposed materials. A number of important chemical changes induced in polymer materials have been identified. Volatile products have been shown to be very different to those arising from thermal degradation. Trends in the relative rates of volatile evolution have been identified which correlate with chemical changes identified in other radiation experiments. As these experiments are performed at far slower irradiation rates the implications for using accelerated ion beams to simulate particle irradiation induced ageing will be discussed.

#### 11:15 AM NNS.10

**Assessment of Shielding Material Performance for Deep Space Missions.** Louis K. Mansur<sup>1</sup>, Barbara J. Frame<sup>1</sup>, Nidia C. Gallego<sup>1</sup>, Chris J. Janke<sup>1</sup>, Jeffrey O. Johnson<sup>2</sup> and Lawrence W. Townsend<sup>3</sup>; <sup>1</sup>Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; <sup>2</sup>Nuclear Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; <sup>3</sup>Nuclear Engineering Department, University of Tennessee, Knoxville,

In deep space, radiation doses to personnel from galactic cosmic rays (GCR) become a significant issue that largely constrains potential missions. In the present work a range of possible materials is being evaluated for personnel shielding against GCR in spacecraft. Conventional spacecraft structural materials, such as aluminum or higher atomic number structural alloys, provide little shielding and can under certain conditions substantially increase radiation doses to personnel. Materials containing high proportions of hydrogen and other low atomic mass number nuclei, which also minimize higher atomic mass components, provide improved GCR shielding. Polyethylene is generally considered as a good performance benchmark shield material. In this work we are investigating both shielding materials that are superior to polyethylene in shielding performance, and spacecraft materials that incorporate desirable shielding properties and are multifunctional in ways that polyethylene is not. Candidate materials that we are investigating can be categorized as carbon forms with potential for hydrogen storage, polymers and polymer composites, and novel materials such as hydrides. Work is in progress to identify the most promising materials and to assess their key properties with respect to space radiation shielding and suitability for other spacecraft applications. Assessments of materials are being carried out with respect to physical, mechanical and shielding properties. Desirable attributes such as 1.) potential performance in structural applications, 2.) hydrogen storage, 3.) thermal management, and 4.) electrical performance will be assessed on a material specific basis. Calculations of shielding effectiveness for realistic GCR spectra are underway. These will be compared with experiments utilizing heavy ion beams with energies of order GeV per nucleon at the NASA Space Radiation Laboratory located at the Brookhaven National Laboratory AGS booster accelerator.

#### 11:30 AM NNS.11

**Conducting and Antistatic Composites for Space Applications.** Mircea Chipara<sup>1</sup>, Jagannathan Sankar<sup>2</sup>, Petre

Notinger<sup>5</sup>, Denis Panaitescu<sup>6</sup>, David Hui<sup>3</sup>, Gheorghe V. Aldica<sup>6</sup>, Magdalena D. Chipara<sup>1</sup> and Kin-tak Lau<sup>4</sup>; <sup>1</sup>Indiana University Cyclotron Facility, Indiana University, Bloomington, Indiana; <sup>2</sup>Department of Mechanical Engineering, North Carolina A&T State University, Greensboro, North Carolina; <sup>3</sup>Department of Mechanical Engineering, University of New Orleans, New Orleans, Louisiana; <sup>4</sup>Department of Mechanical Engineering, The Hong Kong Polytechnic University, Hong Kong, Hong Kong; <sup>5</sup>Politehnica University, Bucharest, Romania; <sup>6</sup>National Institute for Materials Physics, Bucharest, Romania.

Conducting polymers (CP) are of particular interest for space applications due to their high conductivity (in highly doped forms) and lightweight. Among potential space applications of CP are EMI shielding, antistatic coating, rechargeable batteries, gas separation membranes, and actuators (artificial muscles). CP exhibit either good mechanical properties and poor thermal/thermo-oxidative stability (such as polyacetylenes) or good thermal/thermo-oxidative capabilities and poor mechanical features (such as polypyrrole and polyaniline). To overcome these drawbacks several research directions were considered. Most of these studies were focused on the dispersion of polymers with high thermal and thermo-oxidative stability in polymeric matrices with outstanding mechanical properties. For space applications, CP should present a high stability. The thermo-oxidative stability is of particular importance for polymers designed to be used in Low Earth Orbit, where the presence of atomic oxygen (more reactive than molecular oxygen) triggers a fast degradation. We report on the physical properties of composites based on polyaniline (PANI) and polypyrrole (PPY) dispersed in various conducting matrices such as polyethylene and polyvinyl chloride. Various experimental data were used to analyze conducting polymers (PANI and PPY) and their composites. Electron spin resonance (ESR) was used to monitor the concentration and the temperature dependence of conducting electrons. The resonance line shape was used to analyze the mesoscopic nature of the conduction. The resonance line position and amplitude revealed the absence of high spin bipolarons and indicated that the charge transport is dominated by polarons. The temperature dependence of the resonance line width was correlated with polarons mobility. DC electrical conductivity measurements, at low temperatures, revealed a dominating one-dimensional variable range hopping in both CP and composites above the percolation threshold. The dependence of the DC conductivity on the amount of filler dispersed within the polymeric matrix showed that the conductivity percolation threshold is at relatively large fillings. Mechanical testing (elongation at break and tensile strength) on polymeric matrices loaded with various fractions of conducting polymers indicated a close connection between electrical and mechanical properties. The data are compared with the results obtained by dispersing metallic fibers within polymeric matrices. The radiation stability of CP and of composites based on conducting polymers is tentatively discussed. The tuning of mechanical and electrical properties of these composites

is realized by adjusting the amount of conducting filler introduced in system and by controlling the conductivity of CP through the doping level. The effect of acidic doping on polyaniline, as revealed by ESR spectroscopy is discussed.

#### 11:45 AM NN8.12

**Raman Spectroscopy of Graphitic Foams.** Eduardo Bede Barros<sup>2,1</sup>, N. S. Demir<sup>1</sup>, G. Dresselhaus<sup>1</sup> and M. S. Dresselhaus<sup>1</sup>;  
<sup>1</sup>Physics, Massachusetts Institute of technology, Cambridge, Massachusetts; <sup>2</sup>Departamento de Física, Universidade Federal do Ceara, Fortaleza, Ceara, Brazil.

The pitch based graphitic foams recently developed by J. Klett of the Oak Ridge National Laboratory (ORNL) have been found to have a very high thermal conductivity to weight ratio. Several thermal management applications to this novel material are expected to be found in the aeronautics and aerospace industries. The heat transfer in solids is mainly governed by the phonon propagation properties. Therefore, Raman spectroscopy should prove to be a key technique to probe the thermal properties of graphitic foams. The pores in graphitic foams are distributed on a tetragonal pattern and the graphitic planes are found to have different orientations throughout the structure surrounding the pores. Raman spectroscopy studies were performed on the different regions of the foam. The D-band intensity, which indicates the presence of defects in the graphitic structure, is observed to change relative to the G-band intensity for regions of the foam with different types of microstructures. Also, the two peak structure related to the stacking of graphene layers of the double resonance feature known as G' band was studied yielding interesting correlations between structural features and characteristics of the Raman feature. The authors thank Dr. J. Klett of the ORNL for providing the samples and the Intel Corporation for their support.

SESSION NN9: Space-Induced Degradation of Materials  
Chair: Kenneth Label  
Thursday Afternoon, December 2, 2004  
Room 207 (Hynes)

#### 1:30 PM \*NN9.1

**Properties and Improved Space Survivability of POSS Polyimides.** Sandra J. Tomczak<sup>1</sup>, Darrell Marchant<sup>2</sup>, Rusty Blanski<sup>1</sup>, Timothy K. Minton<sup>3</sup> and Amy Brunsvold<sup>3</sup>.<sup>1</sup>AFRL/PRSM, Edwards AFB, Edwards Air Force Base, California; <sup>2</sup>ERC Inc., Edwards AFB, Edwards Air Force Base, California; <sup>3</sup>Chemistry and Biochemistry, Montana State University, Bozeman, Montana.

Polyimides such as Kapton are used extensively in spacecraft thermal blankets, solar concentrators, and space inflatable structures. Atomic oxygen (AO) in lower earth orbit (LEO) causes severe degradation in Kapton resulting in reduced spacecraft lifetimes. One solution is that SiO<sub>2</sub> coatings impart remarkable oxidation resistance and have been widely used to protect Kapton. Imperfections in the SiO<sub>2</sub> application process and micrometeoroid / debris impact in orbit damage the SiO<sub>2</sub> coating leading to erosion of Kapton. A self passivating, self healing silica layer protecting underlying Kapton upon exposure to AO may result from the nanodispersion of silicon and oxygen within the polymer matrix. Polyhedral oligomeric silsesquioxane (POSS) composed of an inorganic cage structure with a 2:3 Si:O ratio surrounded by tailorably organic groups is a possible delivery system for nanodispersed silica. A POSS diamine was copolymerized with pyromellitic dianhydride and 4,4'-oxydianiline resulting in POSS Kapton Polyimide. The glass transition temperature (T<sub>g</sub>) of 5 to 20 weight % POSS Polyimide was determined to be 5 – 10 % lower than that of unmodified polyimides (414 °C). Furthermore the room temperature modulus of polyimide is unaffected by POSS, and the modulus at temperatures greater than the T<sub>g</sub> of the polyimide is doubled by the incorporation of 20 wt % POSS. To simulate LEO conditions, POSS Polyimide films were exposed to a hyperthermal O-atom beam. Surface analysis of exposed and unexposed films conducted with X-ray photoelectron spectroscopy, atomic force microscopy, and surface profilometry support the formation of a SiO<sub>2</sub> self healing passivation layer upon AO exposure. This is exemplified by erosion yields of 10 and 20 weight % POSS Polyimide samples which were 3.7 and 0.98 percent, respectively, of the erosion yield for Kapton H at a fluence of 8.5 x 10<sup>20</sup> O atoms cm<sup>-2</sup>.

#### 2:00 PM NN9.2

**Adhesion of Copper to Teflon® PFA Surfaces Modified by Vacuum UV Photo-oxidation Downstream from Ar Microwave Plasma.** W. Dasilva<sup>1</sup>, A. Entenberg<sup>2</sup>, Bruce Kahn<sup>3</sup>, T. Debies<sup>4</sup> and G. A. Takacs<sup>1</sup>; <sup>1</sup>Chemistry, Center for Materials Science and Engineering, RIT, Rochester, New York; <sup>2</sup>Physics, RIT, Rochester, New York; <sup>3</sup>Imaging and Photographic Technology, RIT, Rochester, New York; <sup>4</sup>Xerox Corporation, Rochester, New York.

Fluoropolymers, like Teflon®PTFE (polytetrafluoroethylene), FEP (fluorinated ethylene-propylene co-polymer) and PFA (polytetrafluoroethylene-co-perfluoroalkoxy vinyl ether) have been extensively used in space applications, protective coatings, microelectronic packaging and biotechnology. However, their low surface energy properties present significant challenges for adhesion and wettability when bonding to other materials, such as, the conductor copper. Therefore, surface modification processes, that affect wettability or enhance the adhesion of copper to Teflon, are of considerable interest. PFA was modified with vacuum UV (VUV) radiation downstream from an argon microwave plasma. During most of the experiments, oxygen flowed over the VUV exposed samples. Surface modification was investigated by water contact angle, X-ray photoelectron spectroscopy (XPS), and scanning electron microscopy (SEM). Copper was sputter coated onto the modified surfaces. The adhesion of copper to the modified surfaces was monitored by use of a tape test. The results for PFA will be compared to the earlier reports on adhesion of copper to VUV-modified PTFE [1,2] and FEP [3]. \*To whom correspondence should be addressed. Phone: 585-475-2047, Fax: 585-475-7800. E-mail address: gatsch@rit.edu. ®Teflon is a registered trademark of E. I. duPont de Nemours & Co., Wilmington, DE. [1] S. Zheng, A. Entenberg, G. A. Takacs, F. D. Egitto and L. J. Matienzo, J. Adhesion Sci. Technol., 17, 1801 (2003). [2] H. Desai, L. Xiaolu, A. Entenberg, B. Kahn, F. D. Egitto, L. J. Matienzo, T. Debies and G. A. Takacs, in: Polymer Surface Modification: Relevance to Adhesion, K. L. Mittal (Ed.), Vol. 3, in press, VSP, Utrecht (2004). [3] W. Dasilva, A. Entenberg, B. Kahn, T. Debies and G. A. Takacs, Polymer Materials Science and Engineering, 90, 833 (2004).

#### 2:15 PM NN9.3

**Degradation and Oxidation of Silver due to Hyperthermal Atomic Oxygen.** Long Li<sup>1</sup>, Liang Wang<sup>1</sup>, Timothy K. Minton<sup>2</sup> and Judith C. Yang<sup>1</sup>; <sup>1</sup>Materials Science and Engineering Department, University of Pittsburgh, Pittsburgh, Pennsylvania; <sup>2</sup>Department of Chemistry and Biochemistry, Montana State University, Bozeman, Montana.

Atomic oxygen (AO) formed by the photodissociation of molecular oxygen is the primary species of oxygen in the low-Earth orbit (LEO), ranging from approximately 200 to 700 km above the earth. The high relative velocity between space vehicles and ambient atomic oxygen leads to hyperthermal collisions of O-atoms with spacecraft materials. These collisions, which are equivalent to O atoms with 5 eV of translational energy striking a surface, may cause rapid degradation and/or oxidation of materials. Silver is used as a mirror material on spacecraft and as a sensor material to monitor atomic oxygen fluence. We have chosen silver as a typical model metal to gain insights into degradation and passivation mechanisms of metals exposed to atomic oxygen. In this work single crystal silver bulk crystals, held at a temperature of 220°C, were exposed to a 5 eV atomic oxygen beam created by laser detonation of O<sub>2</sub>. The O-atom exposure fluence was 8x10<sup>19</sup> atoms/cm<sup>2</sup>. We have characterized the resulting oxide and oxide-metal structures by cross-sectional (scanning) transmission electron microscopy ((S)TEM) and high-resolution TEM. Our results show that a more than 10 micron oxide scale formed on each silver substrate and the oxide scales were predominately composed of polycrystalline silver grains with only a small amount of silver oxide in the scale. This result differs markedly from a silver surface that is oxidized by O<sub>2</sub>, where a silver oxide layer of few nanometers formed on the surface prevents the further oxidation of silver from O<sub>2</sub> at temperatures below 350°C. The oxide scale/substrate interface is very rough and detached in some areas. Many defects and large lattice deformations inside the single crystal substrate are observed near the interface. The results suggest a bulk oxidation of silver single crystals with atomic oxygen and a high permeability of atomic oxygen in the silver lattice. These results suggest that atomic oxygen beam with a 5 eV energy enhanced the diffusion of O-atoms into the substrate. For further investigation, a unique Physical Sciences Inc. FAST<sup>TM</sup> AO laser detonation atomic oxygen source in a UHV chamber is employed for *ex/situ* oxidation studies. The system is equipped with a Maxtek Inc.RQCM system, a quartz crystal microbalance with dual-sensor, to dynamically measure the mass change of metals coated onto the sensor crystals when exposing to atomic oxygen, thereby allowing the *in situ* measurement of mass gain and atomic oxygen flux. Thin films of single crystalline silver will be exposed inside the atomic oxygen source for incremental time periods and then characterized *ex/situ* by transmission electron microscopes in order to observe the evolution of the oxide.

#### 2:30 PM NN9.4

**Structural Characterization of Oxide layers on Aluminum Formed by Exposure to Hyperthermal Atomic Oxygen.** Long Li<sup>1</sup>, Liang Wang<sup>1</sup>, Timothy K. Minton<sup>2</sup> and Judith C. Yang<sup>1</sup>;  
<sup>1</sup>Materials Science and Engineering Department, University of Pittsburgh, Pittsburgh, Pennsylvania; <sup>2</sup>Department of Chemistry and

Space vehicles traveling in the Low Earth Orbit ( 200 to 700 km above the earth) are subject to a strong interaction with atomic oxygen (AO) with a 5 eV relative energy. The violent oxidation of materials by atomic oxygen causes rapid degradation and premature failure of materials in this harsh environment. Aluminum and its alloys, are used as structural components on space vehicles and as a coating material to enhance reflectivity. In this work single crystalline aluminum bulk crystals were exposed to a 5 eV atomic oxygen beam created by laser detonation. The samples were maintained at a temperature of 220°C and were exposed to an O-atom fluence of  $8 \times 10^{19}$  atoms/cm<sup>2</sup>. We have characterized the resulting oxide and oxide-metal structures by cross-sectional (scanning) transmission electron microscopy ((S)TEM) and high-resolution TEM. Our results on aluminum single crystals show that an amorphous aluminum oxide layer with 5 nm thickness formed on each of the aluminum crystals, and this oxide layer had a rough interface at the atomic level. The plane-view TEM results of the amorphous oxide and the area near the interface between amorphous and crystalline material suggest a bulk oxidation of Al single crystals with atomic oxygen. For further investigation, a unique Physical Sciences, Inc. FAST<sup>TM</sup> AO laser detonation atomic oxygen source in a UHV chamber is employed for *ex/in situ* oxidation studies. The system is equipped with a Maxtek Inc. RQCM system, quartz crystal microbalance with a dual-sensor head, to dynamically measure the mass change of metals coated onto the sensor crystals during exposure to atomic oxygen. Thin films of single crystalline aluminum will be exposed inside the atomic oxygen source for incremental time periods and then characterized *ex situ* by transmission electron microscopes to observe the evolution of the oxide.

#### 2:45 PM NN9.5

##### Increased Ordering in the Amorphous SiO<sub>x</sub> due to Hyperthermal Atomic Oxygen. Maja Kisa<sup>1</sup>, William G.

Stratton<sup>2</sup>, Ross Harder<sup>3</sup>, Timothy K. Minton<sup>4</sup>, Ian K. Robinson<sup>3</sup>, Paul M. Voyles<sup>2</sup> and Judith C. Yang<sup>1</sup>; <sup>1</sup>Materials Science and Engineering Department, University of Pittsburgh, Pittsburgh, Pennsylvania; <sup>2</sup>Materials Science and Engineering Department, University of Wisconsin-Madison, Madison, Wisconsin; <sup>3</sup>Department of Physics, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>4</sup>Department of Chemistry and Biochemistry, Montana State University, Bozeman, Montana.

Atomic oxygen (AO) is considered to be a hazardous factor causing rapid degradation of the spacecraft materials exposed to a harsh environment present in the low earth orbit (LEO). Protection of spacecraft material from AO attack can be achieved by overlaying thin-film metal or semiconductor oxides. The thickness, uniformity and structural characteristics of protective coatings, as well as their durability and resistance to reactive AO are important factors in predicting AO diffusion through the coating. Therefore, the effects of hyperthermal (5.1eV) AO on the structural characteristics of the silica layer and Si/SiO<sub>x</sub> interface formed by the oxidation of Si -single crystal were studied by a variety of microcharacterization techniques. A laser detonation source (LDS), based on the breakdown of oxygen gas by a CO<sub>2</sub> TEA laser, was used to produce AO with 5.1eV kinetic energy. The O/O<sub>2</sub> ratio in the beam was 2.3 and the duration of the exposure was 7 hours, with the surface held at a temperature of 493 K. The average AO flux was  $3.2 \times 10^{15}$  atoms cm<sup>-2</sup> sec<sup>-1</sup>. The oxidation of Si(100) by molecular oxygen (MO) was done in a conventional furnace and the temperature was ramped to 493K and P<sub>O<sub>2</sub></sub>=1atm for 48 hours, to create a completely passivating silica layer. The silica layer formed on Si(100) by AO was found to be thicker, more homogeneous, and less amorphous, as compared to the oxide layer created by molecular oxygen, as evidenced by High Resolution Transmission Electron Microscopy (HRTEM) and Selected Area Electron Diffraction (SAED). Electron Energy Loss Spectroscopy (EELS) results revealed that the Si/SiO<sub>x</sub> interface created by AO is abrupt containing no suboxides as opposed to the broad interface with transitional states formed by MO. SAED technique was used to observe sharper diffraction rings present in the diffraction pattern of Si(100) oxidized by reactive AO as opposed to the diffused haloes present in the diffraction pattern of Si(100) oxidized by MO. Initial Fluctuation Electron Microscopy (FEM) results confirmed increased ordering in SiO<sub>x</sub> formed by AO when compared to the non-regular arrangement present in the amorphous oxide formed by MO oxidation of Si(100). The increased ordering of SiO<sub>x</sub> due to AO was expected, since the high energy of the oxidizing species can contribute to the formation of more thermodynamically favorable silica structure. The increase in ordering will be further investigated and quantified by synchrotron X-ray diffraction (XRD) and Radial Distribution Function (RDF) analyses of SAED patterns. The determination of the degree of ordering, periodicity, as well as the interatomic distances and bond angles will provide more detailed insight in the oxide structure formed by reactive AO species.

#### 3:15 PM NN9.6

##### Spectroscopic Investigations on the Effect of Proton

**Bombardment of Polyimide.** David L. Edwards<sup>3</sup>, Kim K. de Groh<sup>2</sup>, Mary Nehls<sup>3</sup>, Sharon K. Miller<sup>2</sup>, Bruce Banks<sup>2</sup>, Chris Stephens<sup>1</sup>, Ramon Artiaga<sup>5</sup>, Roberto Benson<sup>4</sup> and Mircea Chipara<sup>1</sup>; <sup>1</sup>Indiana University Cyclotron Facility, Indiana University, Bloomington, Indiana; <sup>2</sup>NASA Glenn, Cleveland, Ohio; <sup>3</sup>NASA MSFC Marshall Space Flight Center, Huntsville, Alabama; <sup>4</sup>University of Tennessee, Knoxville, Tennessee; <sup>5</sup>University of Coruna, Ferol, Spain.

Polyimide is a high glass transition temperature with excellent mechanical properties, high temperature stability even in the presence of oxygen, and high dielectric strength. The polymer is important for space applications mainly due to its reduced weight, low electrical conductivity, and mechanical strength. The high dielectric strength of polyimide (2500 V/mil for Kapton) is preserved up to 4000C. Polyimide has been already used in space for inflatable structures, multi layer-insulation, flexible circuit boards, thermal blanket, and acoustical fuselage blanket. The transparent polyimide (LaRC) has a high transparency (90%), and a decomposition temperature of about 5500C. The polymer exhibits liquid crystal features and is characterized by a stiff molecular chain. Recent studies revealed that the use of polyimide as insulator has to be considered with caution. High fluence heavy ions as well as short circuit arcs are capable of producing conducting channels in polyimide. The pyrolysis like nature of these modifications that result in the production of graphite like conducting nanostructures, is responsible for the failure of polyimide-based insulators. A spectroscopic research on the effect of proton irradiation on polyimide aiming to a better understanding of the molecular basis of polyimide failures is reported. The final goal of the research is to assess the limits of in space applications of polyimide. Thin films of polyimide where subjected to irradiation with protons irradiated at 200 MeV, at different fluences. The irradiation has been done in air, at room temperature. Electron spin resonance spectroscopy was used to identify the nature of free radicals and to study the effect of temperature on the recombination of free radicals. Additional studies were done by using FTIR and UV-VIS measurements on the same samples. Dynamical mechanical analysis tests revealed the decrease of the glass transition temperature of polyimide upon irradiation. This indicates that chain scissions is the main degradation process of proton irradiated polyimide.

#### 3:30 PM NN9.7

##### Au Ion Induced Modification of C<sub>60</sub> Thin Film Samples.

Navdeep Bajwa<sup>1,3</sup>, Alka Ingale<sup>2,4</sup>, D. K. Avasthi<sup>3</sup>, Ravi Kumar<sup>3</sup>, A. Tripathi<sup>3</sup>, K. Dharamvir<sup>1</sup>, V. K. Jindal<sup>1</sup>, Michael Schmitt<sup>4</sup> and W. Kiefer<sup>4</sup>; <sup>1</sup>Department of Physics, Panjab University, Chandigarh-160014, India; <sup>2</sup>Laser Physics Division, Center For Advanced Technology, Indore 452013, India; <sup>3</sup>Material Science Division, Nuclear Science Center, New Delhi 110067, India; <sup>4</sup>Institut fuer Physikalische Chemie, Universitaet Wuerzburg, D-97074 Wuerzburg, Germany.

The present work reports phase transformations of thin films of C<sub>60</sub> irradiated with 100 MeV <sup>197</sup>Au<sup>8+</sup> ions. This work is in continuation with our earlier work using <sup>58</sup>Ni<sup>10+</sup> and <sup>16</sup>O<sup>6+</sup> ions<sup>1,2</sup>, to study the modification in C<sub>60</sub> thin films. The study of C<sub>60</sub> thin films using <sup>197</sup>Au<sup>8+</sup> ions, provides us enough additional data to investigate thoroughly the role of S<sub>e</sub> in causing phase transformations under different ion fluences. Thin films of C<sub>60</sub> ( 230 nm) were deposited on Si(100) and quartz substrates and then irradiated using <sup>197</sup>Au<sup>8+</sup> ions of 100 MeV energy. The S<sub>e</sub> and S<sub>n</sub> values for these ions incident on C<sub>60</sub> solid were obtained to be 1.292 X 10<sup>3</sup> eV/Å and 1.895 X 10<sup>1</sup> eV/Å, respectively. The S<sub>e</sub> value being about 2 orders of magnitude more than the S<sub>n</sub> value, shows that the electronic energy loss is dominant for the irradiation of <sup>197</sup>Au<sup>8+</sup> ion at 100 MeV. The irradiated films of C<sub>60</sub> were characterized using Raman spectroscopy measurements and optical absorption measurements. However, only results of Raman measurements have been presented here. Raman spectra of the films were measured using LABRAM Raman spectrometer. Micro-Raman data was recorded at room temperature with Ar ion laser excitation 514.5nm (10X objective) at Universitaet Wuerzburg, in a range of 750 – 2150 cm<sup>-1</sup>. Power and exposure time was so adjusted as to avoid any phototransformed polymer peak. The Raman spectra indicate that swift heavy ion (SHI) irradiation results in transformation of crystalline C<sub>60</sub>. At low fluences along with the fragmentation of C<sub>60</sub> there is dimer/polymer formation. As fluence increases the dimer/polymer content first rises, optimizes, decreases and finally vanishes at very high fluences. At high fluences, all the C<sub>60</sub> molecules as well as the polymer C<sub>60</sub> break up resulting in nano-crystalline graphite<sup>1,2</sup> embedded in amorphous carbon (a-C). A comparison of Raman spectra of Au, Ni and O ion irradiated C<sub>60</sub> thin films indicate that the decrease of all the modes (A<sub>g</sub> and H<sub>g</sub> modes) of C<sub>60</sub> is fastest for the Au ion with increasing fluence. We observe that damage of C<sub>60</sub> is maximum by the Au ion followed by the Ni ion (S<sub>e</sub> = 7.3 x 10<sup>2</sup> eV/Å) and then the O ion (S<sub>e</sub> = 81 eV/Å). We conclude that the phase transformations taking place at different

fluence values for different ions used are strongly correlated with the  $S_e$  value of the ion. Thus lower the  $S_e$ , the larger the range of fluence for which  $C_{60}$  persists. The details will be published separately. It seems that the transformation of  $C_{60}$  to polymerized  $C_{60}$  as well as amorphous carbon is dependent mainly on the total energy deposited in the film. A critical value of energy is responsible for onset and the vanishing of polymerized  $C_{60}$ . REFERENCES 1. Navdeep Bajwa, Alka Ingale, D.K.Avasthi, Ravi Kumar, A.Tripathi, K.Dharamvir and V.K.Jindal J. Appl. Phys. **94**, 326 (2003). 2 Navdeep Bajwa, Alka Ingale, D.K.Avasthi, Ravi Kumar, K.Dharamvir and V.K.Jindal, Radiation measurements **36**, 737 (2003).

### 3:45 PM NN9.8

#### TEM Studies of the Protective Al Coatings on Kapton.

Judith C. Yang<sup>1</sup>, Huiping Xu<sup>1</sup>, Long Li<sup>1</sup> and Bruce Banks<sup>2</sup>;

<sup>1</sup>Materials Science & Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania; <sup>2</sup>John H. Glenn Research Center at Lewis Field, NASA, Cleveland, Ohio.

Polymeric materials undergo rapid erosion when exposed to the harsh low-earth-orbit (LEO) environment. Coatings can reduce the erosion rate of polyimide Kapton from atomic oxygen (AO) attack. Specifically, we are investigating how thin Al coatings can protect Kapton. Protective Al layers with variations in layer thickness and growth conditions were deposited on circular plates of Kapton with 1" diameter and 0.005" thickness. The quality of these protective coatings are evaluated by mass loss measurement as a function of AO fluence within an oxygen plasma asher and compared to Kapton alone, where dramatically decreased erosion rate has been noted. To understand how these coatings protect Kapton as well as how the AO interacts with the coatings, we are investigating the microstructure of these coated materials by plane view and cross-sectional transmission electron microscopy (TEM) methods. TEM samples were prepared by Ultramicrotomy. Special attention is focused on defects, cavities and grain size of the protective layer as well as interfaces between this layer and Kapton.

### 4:00 PM NN9.9

#### Materials Testing Activities within ESA in Support of Future Inner Solar System Missions.

Christopher O.A. Semprinoschnig, Marc R.F. Van Eesbeek and Stan Heltzel; Materials Physics and Chemistry Section, ESA/ESTEC, Noordwijk, Netherlands.

ESA (European Space Agency) is currently planning spacecraft to the inner solar system such as Venus Express to Venus, BepiColombo to Mercury and Solar Orbiter that all reach the inner part of the solar system. The closer vicinity to the sun (compared to standard earth orbit missions) is posing several challenges to the selection of materials used on these spacecraft. In some cases it will require design solutions that are different to commonly used solutions for earth orbit applications, in other cases standard materials can be used on the limit of their capability. Temperature and radiation are the two main drivers and need to be thoroughly considered regarding the selection, testing and qualification of materials for the various functional applications. In this paper ESA internal work regarding the assessment of materials for inner solar system will be presented. A main part of the work is devoted to thermal control materials and space environmental testing at elevated temperature. As these materials are the most exposed it is important to understand how they will interact with the relevant space environment at elevated temperature. This interaction requires the assessment in a multi and an interdisciplinary manner. Common materials properties that need to be considered are: Mass stability and outgassing, contamination aspects, thermal endurance, ageing behaviour, embrittlement issues, thermo-mechanical stability and others. An important input parameter for thermal models is the knowledge of the thermo-optical properties as these determine the equilibrium temperatures. This requires a good understanding on the stability of these properties as function of the mission and also as function of temperature. For the materials engineer it is paramount to evaluate whether the so called end of life values are within the functional limit of the material. For the latter it is important to understand what drives materials degradation mechanisms. We will give an overview and will present detailed results on thermal control materials obtained and tested specifically for a Venus as well as for a Mercury orbit. This implies data on the thermal endurance of materials and includes a ranking developed at ESTEC to quickly compare materials. Furthermore results of low (Venus) and high intensity (Mercury perihelion >10 Solar constant) UV testing are shown. The impact that this test environment is posing on thermal control materials is shown, estimates for the current understanding of end of life values are shown and critical issues for future activities are highlighted. KEY WORDS: Thermal endurance, thermal control materials, space environmental testing

### 4:15 PM NN9.10

#### He Ion and Electron Irradiation Effects on Compositionally

**Tuned MgZnO Based UV Detectors.** Shiva Hullavarad<sup>1</sup>, R. D. Vispute<sup>1,2</sup>, S. Dhar<sup>1</sup>, T. Venkatesan<sup>1</sup> and I. Takeuchi<sup>1</sup>; <sup>1</sup>Center for Superconductivity Research, University of Maryland, College Park, Maryland; <sup>2</sup>Bluewave Semiconductors, Baltimore, Maryland.

MgZnO is a novel oxide based UV sensitive material. The band gap of  $Mg_xZn_{1-x}O$  can be tuned by varying the composition of Mg to achieve band gaps corresponding to UV-A, UV-B, UV-C regions of UV spectrum. This material is of significant importance for various applications in flame sensors, UV index monitors and missile plume detection. The interesting property that makes this material unique is its existence in multiple phases for different Mg compositions. This allows picking up desired Mg composition corresponding to suitable UV sensitive window and growing on lattice matched substrate. In the present work we have studied the effect of 1.5 MeV He ion and 200 keV electron irradiation on M-S-M Photoconductors fabricated on MgZnO. The MgZnO films are synthesized by Pulse Electron Deposition. X-Ray Diffraction and Rutherford Back Scattering-channeling techniques are used to characterize the crystalline quality and composition of the samples respectively. We also measure the change in optical transmission of the absorption edge at the band gap as a function of irradiation dose. The leakage current, sharpness of photoresponse under proper UV light illumination are also studied as a function of ion dose.

### 4:30 PM \*NN9.11

#### Piezoelectric PVDF Materials Performance and Operation

Limits in Space Environments. Mathew C. Celina, Timothy R. Dargaville, Roger A. Assink and Roger L. Clough; Dept. 1811, Sandia National Laboratories, Albuquerque, New Mexico.

Polymer based adaptive optic mirrors have been identified as an alternative approach to overcome weight limitations in large aperture spaced-based telescopes. Dimensional adjustments using electron beams have been successfully demonstrated but are dependent on reliable piezoelectric properties and responses of thin films. While polyvinylidene fluoride (PVDF) as a generic polymer type has been identified as a suitable piezoelectric material for control purposes, it is also well known that fluorinated polymers are highly radiation-sensitive materials. Mechanical properties will change under various types of radiation ( $\gamma$ -, x-ray, e-beam, ion-beam, strong vacuum UV). Extreme temperature fluctuations with annealing effects and cyclic stresses are of similar concern, as is atomic oxygen exposure. While the radiation degradation of fluoro polymers has been discussed in the literature, there is little knowledge on the behavior of the piezoelectric and critical mechanical properties of PVDF in these environments. We have studied the degradation of PVDF and related copolymers under a range of stress environments and specifically investigated the impact of these environments on the piezoelectric properties necessary for reliable operation of thin film mirrors in space. We have established fundamental correlations between chemical and physical features of various PVDF copolymers and their piezoelectric properties. Among the techniques used were x-ray diffraction, 19F NMR with high speed spinning, DSC, tensile strength, measurement of d33 coefficients and characterization of D-E hysteresis loops. Material performance was found most importantly to depend on crystallinity, processing history, moduli, and net polarization. For example, high temperature functionality was severely impacted by mechanical relaxation phenomena in highly oriented materials. Conversely, at low temperature materials with limited Tg-moduli transitions showed more consistent responses. Therefore, physical property changes appear to be the dominant factor controlling performance rather than radiation induced chemical changes. A framework for material qualification issues and overall system survivability predictions in low earth orbit conditions has been developed. It will allow for improved material selection approaches, feedback for manufacturing and processing technologies, avenues for material optimization/stabilization strategies and provide the necessary guidance on alternative materials. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

SESSION NN10: Magnetic Materials for Space

Applications

Chair: Ralph Skomski

Thursday Evening, December 2, 2004

Room 206 (Hynes)

### 7:00 PM \*NN10.1

**Laser and Particle Beam Irradiation Effects in Amorphous Metals.** Monica Sorescu, Physics, Duquesne University, Pittsburgh, Pennsylvania.

This work presents a comparison between laser and particle beam

induced effects in amorphous alloys. Excimer laser irradiation effects in FeBSiC, FeNiMoB and FeCoBSi metallic glasses produced two types of kinetics for the transverse induced magnetic anisotropy: out-of-plane magnetic texture for the higher magnetostriction samples and random orientation of magnetic moment directions for the lower magnetostrictive systems. Contrary to the pronounced out-of-plane reorientation of the magnetic moments, the surface magnetic texture was found to remain in-plane using conversion electron Mossbauer spectroscopy. This correlates with the formation of laser-induced closure domain structures in the irradiated samples. Thin films of FeBSiC were obtained by laser ablation deposition on Si substrates and magnetic properties were found to be correlated with the values of the laser fluence employed: the magnetic moments were oriented in plane for low laser fluences, at random for intermediate laser fluences and out-of-plane for high values of the laser fluence. High energy electron beams ( $W=7$  MeV) and their interaction with amorphous alloys were studied by Mossbauer spectroscopy and scanning electron microscopy. Changes in chemical short-range order were found to occur and a spherical mode of growth was identified for the crystalline precipitates in FeBSiC. When low energy electron beams were used ( $W=30$  and  $50$  keV) the dominant mechanism was found to be radiation-enhanced diffusion. Several crystalline phases were found to precipitate out and their relative abundance was found to depend on the fluence employed. Alpha particle beams with  $W=2.8$  MeV were directed towards the surface of FeBSiC metallic glass at two different radiation doses. Transmission and conversion electron Mossbauer spectroscopy, hysteresis loop and magnetic susceptibility measurements were used to elucidate the fundamental effects underlying the interaction of alpha particles with the amorphous structures. The system was found to undergo the onset of surface and bulk crystallization, at stages depending on the dose employed. The magnetic moment values were tailored by the values of the irradiation parameters. Moreover, the real part of the susceptibility component was modulated by the radiation dose employed.

#### 7:30 PM NN10.2

**Smart Radiation Device Using a Metal-Insulator Transition of (La,Ca,Sr)MnO<sub>3</sub>.** Yuichi Shimakawa<sup>1,2</sup>, Akira Okamoto<sup>3</sup>, Yasuyuki Nakamura<sup>3</sup>, Atsushi Ochi<sup>2</sup>, Sumitaka Tachikawa<sup>4</sup> and Akira Ohnishi<sup>4</sup>, <sup>1</sup>Institute for Chemical Research, Kyoto University, Uji, Kyoto, Japan; <sup>2</sup>NEC Corporation, Tokyo, Japan; <sup>3</sup>NEC TOSHIBA Space Systems, Ltd., Yokohama, Japan; <sup>4</sup>Japanese Aerospace Exploration Agency, Sagami, Japan.

A variable emittance radiator for a spacecraft, named SRD (Smart Radiation Device), which uses a metal-insulator transition of (La,Ca,Sr)MnO<sub>3</sub>, was developed. The emittance property of this material significantly changes at the metal-insulator transition temperature (close to room temperature); that is, (La,Ca,Sr)MnO<sub>3</sub> is a highly emissive insulator above room temperature and a less emissive metal below room temperature. Therefore, the (La,Ca,Sr)MnO<sub>3</sub> material installed on spacecraft surfaces can automatically control the radiation heat transfer from the spacecraft to space without consuming any electrical power. The developed radiators also greatly improve the weight and the production cost over the conventional thermal control louvers. Several forms of the device, which include ceramic tiles and thin films, have been developed. After undergoing ground tests against simulated space environment, the developed device is indeed employed on the JAXA's asteroid sample return spacecraft MUSES-C (Hayabusa), which was launched on May 9, 2003. The emittance property of the material will be discussed from a physical point of view. The outstanding device performance will also be reported.

#### 7:45 PM \*NN10.3

**Fabrication of LBMO/YBCO Double Layers for Tunable Microwave Filters.** Tamio Endo<sup>1,2</sup>, Michi Ogata<sup>1</sup>, Takahisa Sakurada<sup>1</sup>, Hidetaka Nakashima<sup>1</sup>, Hirofumi Yamasaki<sup>3</sup> and Kazuhiro Endo<sup>2</sup>, <sup>1</sup>Faculty of Engineering, Mie University, Tsu, Mie, Japan; <sup>2</sup>Electronics Division, Advanced Industrial Science and Technology, Tsukuba, Ibaraki, Japan; <sup>3</sup>Energy Division, Advanced Industrial Science and Technology, Tsukuba, Ibaraki, Japan.

Double Layers of ferromagnetic La(Ba)MnO<sub>3</sub> (LBMO) and superconducting YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> (YBCO) are promising for tunable microwave filter devices used at base stations of mobile and satellite communication systems. In order to complete excellent double layers, we must first obtain techniques to fabricate perfect a/c-phase of YBCO and excellent crystalline LBMO single layers on substrate at low substrate temperatures (Ts), and then fabricate their double layers. At low Ts, a-phase growth of YBCO is promoted while c-phase is promoted at high Ts in ion beam sputtering (IBS) deposition. The c-phase is promoted at low oxygen pressures (Po), while the a-phase is promoted with increasing Po. The a-phase growth is enhanced by oxygen plasma supply. Thus we can obtain the perfect a-c orientation growths of YBCO with in-plane aligned grains. The minimum surface roughness is 1 nm for the c-phase and 0.3 nm for the a-phase,

corresponding to one unit cells. Excellent crystalline thin films of LBMO can be grown by IBS with controlling Ts, Po and oxygen molecule or plasma on MgO and LAO. It can be grown down to 480 deg C on LAO. The minimum rocking half-width is 0.01°. The minimum surface roughness is 0.8 nm. As-grown LBMO film shows metal-insulator transition temperature at 84 K and Currier temperature at 174 K, while annealed sample shows at 31 K and 180 K, respectively. The samples show a magnetoresistance (MR). They show negative MR at high temperatures (T) while positive MR at lower T. Ferromagnetic resonance of the samples shows extremely small half-width of 100 G indicating superior spin homogeneity. They show a doublet peak at lower T. The results are interpreted by a phase separation. Thus LBMO films can be used as the ferromagnetic layer of the tunable filters. The double layers of LBMO/a,c-YBCO, LSMO/YBCO and a,c-YBCO/LBMO can be fabricated by IBS. The overlying LBMO layer can be easily grown on the underlying YBCO layer. XRD patterns of LBMO/YBCO show clear separated peaks from YBCO and LBMO. Then we can estimate the crystallinities of YBCO before the double layer deposition, and of underlying YBCO and overlying LBMO after the double layer deposition. The crystallinity of overlying LBMO is excellent, and that of underlying YBCO is degraded. The better crystalline LBMO grows on the better crystalline YBCO. The double layer surface is smooth. LSMO can also be grown on YBCO layer but their peaks are located closely. The overlying YBCO of YBCO/LBMO showing the separated peaks has good crystallinity, furthermore the underlying LBMO crystallinity is improved during the double layer deposition. YBCO mosaicity is almost the same with that of single YBCO layer on substrate, or better. The surface, however, is roughened.

#### 8:15 PM NN10.4

**Magnetic Nano Particles for Space Applications.** S. K. Sharma<sup>1,2</sup>, Singh Mahavir<sup>1</sup>, Ravi Kumar<sup>2</sup>, V. V. Shiva Kumar<sup>2</sup> and S. N. Dolia<sup>3</sup>, <sup>1</sup>Physics Dept., H.P. University, Shimla, Himachal Pradesh, India; <sup>2</sup>Material Science Division, Nuclear Science Centre, New Delhi, Delhi, India; <sup>3</sup>Physics Dept., University of Rajasthan, Jaipur, Rajasthan, India.

Radiation resistant ferrite materials have potential applications in space stations. Mg-Mn spinel was chosen for this study because of its radiation resistance and potential for its use as an insulator in radiation environments. The radiation damage expected in these environments can be quickly and conveniently simulated using ion irradiation. The results of swift heavy ion irradiation induced modifications in the magnetization behavior of the Mg-Mn ferrite nano particles have been investigated using 100 MeV Ni<sup>+8</sup> ion irradiation. The size of the synthesized particles is 4.7 nm. To ensure the single-phase spinel structure of the system powder x-ray diffraction has been performed. The powder samples were irradiated at three different fluences in the range  $1 \times 10^{12}$ – $1 \times 10^{13}$ . Isothermal dc magnetization studies have been performed using vibration sample magnetometer on the pristine as well as on the irradiated samples at 20 and 300 K. With irradiation we observe that saturation magnetization remains almost constant for all the fluences. However, magnetic hardness of the materials increased about 5% with fluence  $1 \times 10^{13}$  ions per cm<sup>2</sup> as compare to pristine nanoparticles. The magnetization studies is further supported by Mossbauer studies of these nano particles. These results have been explained on the basis of the existence of surface defects produced by swift heavy ions which generate orientational disorder of surface spins. The behavior of saturation magnetization with irradiation make these nanoparticles suitable for memory devices in space research.

#### 8:30 PM \*NN10.5

**Developing Glassy Magnets from Simulated Composition of Martian Soil for Exploration Applications.** Narayanan Ramachandran<sup>1,2</sup>, Chandra Ray<sup>2</sup> and Jan Rogers<sup>2</sup>, <sup>1</sup>BAE Systems Analytical Solutions Inc., Huntsville, Alabama; <sup>2</sup>NASA Marshall Space Flight Center, Huntsville, Alabama.

The long-term exploration goals of NASA include developing human habitation on Mars and conducting scientific investigations on Mars and other planetary bodies. In situ resource processing is a key objective in this area. We focus on the possibility of making magnetic glasses in situ for potential applications development. The talk will focus on ongoing work at NASA Marshall Space Flight Center on making magnetic glass from Mars soil simulants and its characterization. Analysis of the glass morphology, strength, chemistry and resulting magnetic properties will provide a fundamental understanding of the synthesized material that can be used for potential applications development. In an effort to characterize the magnetic properties of the Mars glasses, a series of tests were performed at NASA MSFC. Preliminary tests indicated that the glasses were attracted to a magnet and also had a small amount of residual magnetism. They were opaque (almost black in color). As the first step, a sample of Mars 1 glass (1 mm x 1 mm x 5 mm length) was machined, weighed and its hysteresis curve was

measured using a Vibration Sample Magnetometer (VSM). Next, a small furnace was designed and built and the sample was baked in a graphite (reducing agent) crucible at 800 C in an Argon atmosphere for 3 hours in the presence of a uniform, transverse (transverse to the 5mm length of the sample) magnetic field of 0.37 Tesla. The treated sample showed reddening on the outside and showed substantially increased residual magnetism. This sample was again analyzed in the VSM. The data clearly showed that some chemical change occurred during the heat treatment (color change) and that both the glasses have useful magnetic properties. Although no orientation effects of the magnetic field were considered, the data showed the following: 1. Both glass samples are primarily soft magnets and display ferromagnetic behavior (hysteresis, saturation, etc.) 2. The treated glass has improved saturation magnetism (order of magnitude increase), retentivity (factor of 6 increase) and susceptibility (order of magnitude increase) compared to the untreated glass 3. The untreated sample has higher coercivity (50% that of Nickel) than the treated sample 4. Both samples have similar energy density. Results from a systematic study to quantify the effects of processing conditions such as heat treatment, atmosphere, containerless processing (by electrostatic levitation), and applications of external magnetic fields of different strengths will be discussed. Efforts on optimizing the magnetic properties of the product and the feasibility of using it for a couple of specific magnetic applications such as heat generation using an ac field and for electro forming will also be covered. The latter is an in situ manufacturing technique being studied for in-space fabrication applications at MSFC.

#### 9:00 PM \*NN10.6

**Magnetization Dynamics in Nanostructured Materials for Nonvolatile Memory Applications.** Leonard Spinu<sup>1</sup>, Huy Pham<sup>2</sup> and Alexandru Stancu<sup>3</sup>; <sup>1</sup>Advanced Materials Institute, University of New Orleans, New Orleans, Louisiana; <sup>2</sup>Department of Physics, University of New Orleans, New Orleans, Louisiana; <sup>3</sup>Faculty of Physics, Al. I. Cuza University, Iasi, Romania.

As the conventional magnetic recording technology enabled recording densities unforeseeable only a couple of years ago, there is a lot of effort put in developing new types of memory that could combine the nonvolatility, high speed and high density. Nonvolatility, the ability to function in an environment where the power cannot be guaranteed, along with capacity to withstand radiation does would destroy conventional memory are relevant properties for military and space applications. Magnetic random memory (MRAM) is the exponent of such efforts and is essentially a hybrid between an electronic RAM chip and a hard disk drive that uses magnetism to store the information but is a solid-state device. This talk summarizes our recent experimental and theoretical results obtained on the study of magnetization dynamics in magnetic nanostructured materials for information storage applications. Magnetization dynamics is one of the key issues of magnetic materials that are part of new data storage devices. For two-dimensional (2D) devices used in MRAM, the magnetization dynamics is determined by the 2D magnetization switching properties as the MRAM cells require that the magnetic field be applied in two dimensions in the plane of the device. Moreover, study of two-dimensional magnetic switching enables us to determine the critical curve which provides information about micromagnetic and structural properties of magnetic systems. A new sensitive method for critical curve determination of 2D magnetic systems was proposed. It was shown that this method, based on reversible susceptibility's singularities detection, is general and can be applied independent of the expression free energy describing the magnetic system under study. Experimentally, the method has been used to investigate the magnetization reversal of a wide range of systems that include magnetic nanostructured materials with different dimensionalities as nanoparticles arrays, thin films, and magnetic multilayers. The study of magnetization reversal is very important in designing high density and high data transfer rate recording media. This determined the seeking of new strategies for magnetization reversal. Thus, recently it was observed that for Stoner-like magnetic particles without interactions the switching field and switching time can be decreased using short magnetic field pulses. Since in real magnetic systems the effect of interaction can not be avoided in this study we present a analysis of magnetization reversal in interacting Stoner-Wohlfarth particles. A network of interacting spins is considered and the switching diagrams for the reversing particle and its first neighbors are determined. The final state of magnetization and the switching time are found to be dependent on initial state of magnetization, pulse parameters, and the spatial arrangement of the particles.

SESSION NN11: Materials in Extreme Environments  
Chair: Narayanan Ramachandran  
Friday Morning, December 3, 2004  
Room 207 (Hynes)

#### 8:30 AM \*NN11.1

**Polymer Degradation From The Thermal Analysis Point Of View.** Ramon Artiaga<sup>1</sup>, Ricardo Cao<sup>2</sup>, Salvador Naya<sup>2</sup> and Ana Garcia<sup>1</sup>; <sup>1</sup>Dep. Industrial Engineering II, E.P.S. University of Coruna, Ferrol, Spain; <sup>2</sup>Dep. Mathematics, Fac. Computer Sci. University of Coruna, Coruna, Spain.

In this work the application of different thermal analysis methods to polymer based materials degradation is considered in two ways: the study of the degradation process itself and the evaluation of the degree of damage of a material as consequence of chemical degradation by thermal or radiation effects. The thermal degradation in different atmospheres is basically studied by TGA in dynamic experiments. The authors have found that the evolution of the sample mass follows a mixture of logistics model, so an overall TGA curve can be fitted by this model. The fitting parameters have important physical meaning related to the kinetics of the different processes involved and to the relative amount of each component in the sample. The method itself implies the separation of overlapping processes. Other improvements made by the authors are related to the noise reduction and smoothing of TGA and DSC data, particularly, the estimation of DTG derivatives using logistic regression for pilot bandwidth estimation. The analysis by TGA of many materials results in more or less complex traces that do not allow a simple parametric fit like the one previously described, since although it reproduces the asymptoticity at the beginning and end of the reaction, sometimes many processes are strongly overlapping giving a complex trace that would need an important number of logistic components to be adequately fitted. However, it is possible to use a local polynomial regression model instead, been also applicable to DSC traces, whose shapes are totally different from TGA. The authors propose a model based in a nonparametric estimation, where the goodness of the fit depends very much on the bandwidth selection, especially when the derivatives are concerned. The proposed model gave a satisfactory fitting. This way of fitting smoothes the noise and gives always reliable values, different than the obtained by other methods that strongly depend on the user choice. Concerning the evaluation of the degree of damage by thermal analysis methods, dynamic mechanical analysis (DMA) was applied to polyimides. The glass transition temperature was measured before and after the exposure to different doses of proton radiation, that emulates the space environment. Other examples show how the exposure for long times at moderately elevated temperatures results in reduction of some mechanical properties. Additionally, the effect of different nanofillers on styrene-isoprene-styrene block copolymers was evaluated by DMA. A shift of the glass transition temperature was observed to be dependent on the nano-filler content. The degradation of some materials of interest for space applications will be shortly reviewed. Polyethylene and polyimide are such two typical materials.

#### 9:00 AM NN11.2

**Mechanical and Thermal Properties of Particulate-Reinforced Composites.** Sea-Hoon Lee<sup>1,2</sup>, Fritz Aldinger<sup>1</sup>, Sung-Churl Choi<sup>2</sup> and Keun-Ho Auh<sup>2</sup>; <sup>1</sup>Max-Planck-Institut für Metallforschung, Stuttgart, Germany; <sup>2</sup>Ceramic processing research center, Hanyang university, Seoul, South Korea.

Effects of heat treatment of Si<sub>3</sub>N<sub>4</sub> filler on the mechanical and thermal properties of particulate-reinforced composites (PRC) made by precursor-impregnation and pyrolysis (PIP) method were investigated. The PRC are expected to have enhanced thermal shock resistance due to the similarity of coefficient of thermal expansion (CTE) between filler and Si-C-N matrix. The chemical composition of the filler was controlled by changing the heating conditions and/or using the Si<sub>3</sub>N<sub>4</sub> powder bed. Oxygen content of the filler always decreased after the heat treatment above 1300 degree celsius. Carbon and nitrogen contents of the filler were also affected by the heating condition and powder bed. Mechanical properties such as cutting resistance, strength, Young's modulus and hardness as well as high temperature mass stability of the PRC were improved by the heat treatment of the filler before the impregnation and pyrolysis of the liquid Si-C-N precursor.

#### 9:15 AM NN11.3

**Developing New Refractory Open Cell Metal Foams for Aerospace Applications.** Wassim E. Azzzi, William Roberts and Afsaneh Rabiei; Dept of Mechanical, Aerospace Engineering, North Carolina State University, Raleigh, North Carolina.

The thermodynamic efficiency of the Brayton cycle, upon which all gas turbines (aeropropulsion and power generation) are based on scales with the peak operating temperature. However, the peak temperature is limited by the turbine blades and the temperature they can withstand. The highest temperatures in the gas turbine obviously occur in the combustor region but these temperatures are often too high and the combustion products must be diluted with relatively cooler air from the compressor to reduce the temperature to



tolerable levels for the turbine blades. Thus, to maximize the thermodynamic efficiency, the amount of dilution air should be minimized. The best performance occurs when the temperature entering the nozzle guide vanes is perfectly uniform and equal to the maximum allowable temperature. If there are non-uniformities in temperature, the mean temperature must be decreased so that the local (in time or space) maximum temperature does not exceed the maximum allowable values. It is also desirable to have a uniform velocity profile. Considerable effort has been expended in reducing the pattern factor and moving the profile factor closer to unity. This will not only increase the efficiency of the engine but will also increase the lifetime of the turbine blades that constantly suffer from thermal fatigue cycling. However, current practices yield results that are far from optimal. In our study, an open cell porous material is being placed between the combustor section and the turbine section of the engine. This provides a better temperature and velocity profiles and allows operation at the maximum allowable temperatures. The open cell porous material with its increased surface area mixes the hot and cold air entering the turbine section and eventually provides a profile that is closer to unity. The main issues in this case will be to control the pressure drop at an acceptable level and finding a porous material with sufficiently low stagnation pressure losses yet able to withstand the thermal and mechanical stresses as well as corrosion and oxidation. This research focuses on studying the thermal and physical properties of various open cell porous materials with different cell sizes, materials, and thicknesses and their behavior at elevated temperatures and pressures. The temperature profile versus pressure drop for various porous media will be experimentally tested and analyzed and the effect of each parameter on the pressure drop and temperature profile is being studied.

#### 9:30 AM NN11.4

**Processing and Development of an Ultra-Light High Strength Material Through Powder Metallurgy.** Brian P. Neville and Afsaneh Rabiei; Department of Mechanical and Aerospace Engineering, North Carolina State University, Raleigh, North Carolina.

A new closed cell metallic foam that is produced by powder metallurgy is being developed. It is processed by mixing metallic hollow spheres with metal powder and sintering them into a solid structure. Although denser than similar foams made entirely of sintered hollow spheres, this new foam will have the advantage of greater strength due to the solid matrix around the spheres, while still maintaining a lower density than that of a bulk metal. The solid matrix can be tailored for different applications through the addition of alloying elements. Additionally, wire reinforcements can be added for even greater strength. This process also has the advantage of being able to control the size and distribution of the cells by controlling the size of the spheres. Both similar and dissimilar materials can be used for the hollow spheres and the matrix, whereas a casting technique only allows a matrix material of a lower melting point than that of the spheres. In this study, both similar and dissimilar materials have been used for the hollow spheres and the matrix to produce new metal foams. The material has been reinforced with fiber-reinforced wires to increase the strength of the material. Microstructural, mechanical and physical properties of all reinforced and unreinforced materials have been studied using various techniques, including optical microscopy, SEM, EDX, compression and fatigue tests. The results have been compared with the properties of various metal foams processed using different techniques including those processed using hollow spheres and produced by casting technique.

#### 9:45 AM NN11.5

**Aerothermal Analysis of an Advanced Hot Structure for Hypersonic Flight Tests.** Raffaele Savino<sup>1</sup>, Mario De Stefano Fumo<sup>1</sup>, Giuliano Marino<sup>2</sup> and Mario Tului<sup>3</sup>; <sup>1</sup>Space Science & Engineering "Luigi G. Napolitano", University of Naples "Federico II", Naples, Italy; <sup>2</sup>Centro Italiano Ricerche Aerospaziali(CIRA), Capua, Italy; <sup>3</sup>Centro Sviluppo Materiali(CSM), Roma, Italy.

The European Space Agency (ESA) is pursuing the Expert hypersonic flight test programme with the objective to improve knowledge of challenging problems of hypersonic flight. The configuration chosen for the Expert vehicle is basically a blunt cone with four flaps and flat surfaces ahead of them. The vehicle will take advantage of the Russian Volna launcher capabilities to make controlled ballistic suborbital flights to study all of the most critical aerothermodynamic phenomena. Among other scientific payloads, the vehicle will carry an advanced concept of hot structure based on the development of ultra high temperature ceramics (UHTC). Specific goals of this payload are: 1) to test UHTC (e.g. ZrB<sub>2</sub>/SiC and/or HfB<sub>2</sub>/SiC and/or ZrB<sub>2</sub>/SiC/C) for future spacecrafts at hypersonic re-entry conditions (total enthalpy > 10 MJ/Kg, stagnation point heat flux > 5MW/m<sup>2</sup>, surface temperature > 2300 K); 2) to assess potential advantages related to the relatively high emissivity and high thermal conductivity that make UHTCs particularly well suited for use as massive leading

edges of sharp fuselages and wings (boundary layer thermal protection); 3) to investigate the behaviour of UHTCs at overall radiative equilibrium conditions (radiative cooling); 4) to investigate surface catalytic properties of UHTCs with respect to recombination reactions in presence of a highly dissociated oxygen mixture; 5) to compare flight results with Plasma Wind Tunnel (PWT) results and with numerical results for numerical codes validation and verification. This paper provides a description of the structure architecture with its potential instrumentation. The attention is focused on the aerothermodynamic analysis performed to design the winglet shape, consisting essentially of a relatively sharp UHTC leading edge, able to withstand very high temperatures, and a metallic support of Oxide Dispersion Strengthened (ODS) alloy. Three-dimensional fluid dynamic computations have been carried out at the flight conditions of the reference Expert trajectory to evaluate the aerothermodynamic field and the thermal loads on the winglet. The physical model includes viscous effects, real gas properties, nonequilibrium chemical reactions and surface catalytic effects. The unsteady aerothermal analysis has been carried out considering different thermal boundary conditions (adiabatic wall or thermal coupling with the capsule metallic skin). The results are discussed with particular attention to the temperature distributions in the UHTC and in the structural support.

#### 10:15 AM NN11.6

**Solid State NMR Measurements For Preliminary Lifetime Assessments in Gamma-Irradiated and Thermally Aged Siloxane Elastomers.** Sarah C. Chinn, Robert S. Maxwell, Long Dinh, Bryan Balazs and Richard Gee; Lawrence Livermore National Laboratory, Livermore, California.

Siloxanes have a wide variety of applications throughout the aerospace industry which take advantage of their exceptional insulating and adhesive properties and general resilience. They are, however, subject to degradation in radiatively harsh environments. We are using solid state nuclear magnetic resonance techniques to investigate aging mechanisms in siloxane elastomers and correlations to changes in performance for a variety of polymeric materials. NMR parameters such as transverse (T<sub>2</sub>) relaxation times, cross relaxation rates, and residual dipolar coupling constants provide excellent probes of changes crosslink density and motional dynamics of the polymers caused by ionizing radiation and thermal degradation. The results of NMR studies on aged siloxanes are being used in conjunction with other mechanical tests to provide insight into component failure and degradation kinetics necessary for preliminary lifetime assessments of these materials. Results obtained both from high resolution NMR spectrometers as well as low resolution benchtop NMR screening tools will be presented.

#### 10:30 AM NN11.7

**Syntactic Closed-cell Foams Based on Silicon Carbide.** Engin Ozcivici and Raman P. Singh; Department of Mechanical Engineering, Stony Brook University, Stony Brook, New York.

Closed cell ceramic foams have been fabricated by incorporating hollow ceramic spheres into a silicon carbide matrix using the polymer infiltration and pyrolysis (PIP) technique. The hollow ceramic spheres used in this investigation were aluminosilicate cenospheres, which are produced as a by-product of coal fly ash, while the silicon carbide matrix was formed by the pyrolysis of a commercially available liquid pre-ceramic polymer. The fabrication process involved physical mixing of cenospheres with the liquid pre-ceramic polymer in measured quantities. The resulting slurry was compacted into high temperature molds and subsequently subjected to high temperature pyrolysis in an inert atmosphere. In this manner, various closed cell composite foams were fabricated using different grades of cenospheres, and different levels of infiltration with the preceramic polymer. Foam characteristics that were investigated include density, packing factor, porosity, mechanical properties (flexure strength, modulus and compressive strength), and thermal properties (conductivity and CTE). Various randomly graded, binary and ternary mixtures of cenosphere sizes were used in order to optimize packing density and the resulting materials properties. The pyrolysis procedure allows for low-cost and net shape fabrication of complex components with less fabrication time as compared to chemical vapor deposition. It has been demonstrated that these foams can deliver unique mechanical, physical and thermal properties that offer potential for various thermo-mechanical aerospace applications.

#### 10:45 AM NN11.8

**Investigation on Stress-Induced Martensite in a NiTi Shape Memory Alloy with Synchrotron Radiation.** Jafar Khalil-Allaf<sup>1,2</sup>, Wolfgang Wilhelm Schmahl<sup>1</sup>, Bernd Hasse<sup>3</sup> and Thomas Reinecke<sup>1</sup>; <sup>1</sup>Institute for Mineralogy, Ruhr-University Bochum, Bochum, NRW, Germany; <sup>2</sup>Faculty of Materials Engineering, Sahand University of Technology, Tabriz, West Azarbaijan, Iran; <sup>3</sup>HASYLAB, DESY, Hamburg, Niedersachsen, Germany.

Ni-rich NiTi shape memory alloys are the most promising materials for superelastic applications due to their high strength and recoverable strains up to 8%. Superelasticity is based on the stress-induced phase transition from cubic B2 austenite to monoclinic B19' martensite and recovery of the B2 phase upon release of stress. In flat specimens of superelastic NiTi under uniaxial tensile stress the austenite - martensite transformation is localised in shear transformation bands. We investigated the phase state in flat Ni50.7Ti49.3 tensile specimens by diffraction methods in a space-resolved mode using synchrotron radiation. The specimens were solution annealed at 850°C for 15 min and water quenched; subsequently the specimens were cold rolled at -150°C with a thickness reduction of 10%. Finally, the samples were aged at 300°C for 6 minutes. Inside the shear band, the transformation to martensite was incomplete even at 8% macroscopic tensile strain. The residual non martensitic grains deform elastically by 1% in the transverse direction as they have to bear an increased load when the neighbouring grains yield forming martensite. The matrix outside the shear band does not show enough martensite to be detectable by diffraction. Between the shear band and the adjacent matrix we observe a boundary region with transitional states of strain. The observed large lattice strain of the residual non martensitic grains can be attributed to a formation of the rhombohedral R-phase state or to stressed austenite. However, a very sharp preferred orientation of R-phase twin domains has to be postulated to get a precise fit of the full diffraction profile.

#### 11:00 AM NN11.9

##### **Template Synthesis of Boron Nitride Nanotubes for Space Applications.**

Jose Eneider Nocua<sup>2</sup>, Arturo Hidalgo Cordova<sup>2</sup>, Joel De Jesus<sup>2</sup>, Rafael Velazquez<sup>2</sup>, Fabrice Piazza<sup>2</sup> and Gerardo Morell<sup>1</sup>; <sup>1</sup>Dept of Physical Sciences, University of Puerto Rico, San Juan, PR, Puerto Rico; <sup>2</sup>Dept of Physics, University of Puerto Rico, San Juan, PR, Puerto Rico.

Boron nitride nanotubes (BNNTs) have received increasing attention due to their unique properties that exhibit advantages over carbon nanotubes (CNTs). BNNTs are semiconductors with a nearly constant wide band gap of 5.5 eV, independent of their chirality and size. They have a high elastic modulus of 1.2 TPa, and are resistant to oxidation at temperatures up to 900 C in air. Therefore, BNNTs are extremely promising building blocks for emerging technologies and novel systems concepts with high potential payoff. The growth, structure, properties, and purification of BNNTs is not yet well documented due to significant technological difficulties in making them. BNNT-based electronics represent a new generation of mission enabling technologies capable of performing under harsh conditions such as high temperature and ionizing radiation. BNNT nanocircuitry will feature low-power consumption, compactness, and robustness, thus enabling a high degree of redundancy for highly interconnected communication networks, wherein data from multiple modules are shared. Besides their electronic applications, BNNTs are promising materials for reinforcing metals, polymers and ceramics. Once BNNTs are assembled in ropes or bundles, they can be incorporated into structural materials in order to improve their performance. Such materials will show good performance when exposed to shock waves and similar situations. Hence, BNNTs will also help fulfill the goals of lighter, stronger, safer, and more reliable space ships and airplanes. We report on the growth, structure, and purification of BNNTs grown by arc discharge using CNTs as templates. The arcing conditions are maintained at 20-40 V DC and 50-100 A with a gap spacing nearly constant of around one mm. Scanning electron microscopy, transmission electron microscopy, and electron energy loss spectroscopy were used to investigate the structure of the tubes and the effect of purification treatments.

#### 11:15 AM NN11.10

##### **Modelling of Hydrogen Embrittlement: Combined Continuum Cohesive and Quantum Mechanics Calculations.**

Santiago A. Serebrinsky<sup>1</sup>, Emily A. Carter<sup>2</sup> and Michael Ortiz<sup>1</sup>;

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Metallic materials are often susceptible to hydrogen embrittlement (HE) when subject to adequate conditions. In particular, there were several cases along the history of space applications. Despite the long-standing nature of the problem, the mechanism responsible for HE was not elucidated as yet. Such knowledge is central for the prediction and prevention of HE. In this work, we present a model of HE based on a combination of quantum mechanical calculations and cohesive theories of fracture. The latter were successfully applied many times as an engineering tool to analyze crack propagation under various conditions. Its application depended on some calibration procedure to fit experimental data for specific samples. Here, we point to obtain a more fundamentally based construction to determine fracture properties. We present a model based upon: i) a cohesive law

dependent on impurity surface coverage that is calculated from first principles; ii) a stress-assisted diffusion equation for the impurity; iii) a static continuum analysis of crack growth; and iv) the Langmuir equilibrium relation determining the impurity coverage from its bulk concentration. An appealing feature of the model is that it provides quantities readily measured as an output, as opposed to many other models that give only a qualitative picture. We determine crack evolution as affected by several system variables, as yield strength, temperature, etc. Remarkably, crack advancement can be intermittent or smooth, depending on various quantities. We compare calculations for the specific cases of high strength steels and high strength aluminum alloys with experiments. The comparison shows that the proposed model of hydrogen-induced reduction in cohesion is likely the operative mechanism for HE in the former, while for the latter some additional embrittlement factor is required.

#### 11:30 AM NN11.11

##### **Microstructure Development in High-Temperature Mo-Si-B Alloys.**

Ridwan Sakidja and John H. Perepezko; MS&E, UW-Madison, Madison, Wisconsin.

Mo-Si-B alloys have been considered as potential high temperature structural materials due to their high melting points (above 2000oC) and excellent oxidation resistance caused by their self-healing characteristics over an extended temperature range (up to 1400oC). In the current study, the effect of alloying additions to achieve a lower weight density and microstructural stability has been examined. The critical factor to the alloying additions appears to be the stability of the high melting ternary-based T2 borosilicide phase. In addition, the application of a variety of processing coating methods to develop an integrated coating structure that provides the thermal and oxidation protection to the Mo-Si-B substrate at high temperatures has been evaluated. The support of AFOSR (for the microstructure development) and ONR (for the coating design) are gratefully acknowledged.

#### 11:45 AM \*NN11.12

##### **An Experimental Study of the High Strain Rate Behavior of Ice.**

P. K. Dutta, <sup>1</sup>Cold Regions Research and Engineering Laboratory, U.S. Army Research and Development Center, Hanover, New Hampshire; <sup>2</sup>Applied Research, Army-ERDC-CRREL, Hanover, New Hampshire.

High strain rate compression tests were conducted with columnar ice grown in laboratory. Besides measuring stress and strain to failure we analyzed the size, shape and nature of the fragments, fracture patterns, and effects of end conditions on the failure process. The tests were performed in a split Hopkinson pressure bar at two temperatures, 283 K and 233 K, and a strain rate in the range of 10 to 15 strains/sec. Additional tests were performed on the similarly grown ice at a low rate of 0.001 strain/sec for comparison. The results were further analyzed in terms of the energy absorption and the progressive failure process of the cylindrical specimens. The general observation is that the most failures occurred by splitting, and the final failure by the collapse of the columns, which become unstable under the sustained load during the passage of the stress wave through the material. Most failures occurred around a value of 1000 microstrain. The evidence of progressive failure was clear from the multiple peaks of the stress-strain curve in the loading phase. The collapse too was not instantaneous, but took a finite time of 100 to 120 microseconds, possibly for the time required for the ejection of the fragments. The temperature effect was evident in the small increase of peak stress when the test temperature was lowered from 260 K to 230 K. Comments are made on some aspects of the microstructural changes typical of high and low rate deformations.