# SYMPOSIUM OO

# Growth, Modification, and Analysis by Ion Beams at the Nanoscale

November 28 - December 1, 2005

# Chairs

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Proceedings to be published online (see ONLINE PUBLICATIONS at www.mrs.org) as volume 908E of the Materials Research Society Symposium Proceedings Series.

<sup>\*</sup> Invited paper

SESSION OO1: Swift Heavy and Light Ions: Fundamentals and Applications Chair: Marcel Toulemonde Monday Morning, November 28, 2005 Commonwealth (Sheraton)

9:00 AM \*OO1.1

Recent Developments in Ion Track Technology. Christina Trautmann, Gesellschaft fuer Schwerionenforschung, Darmstadt, Germany.

In many materials swift heavy ions of MeV to GeV energy produce tracks consisting of amorphous or otherwise transformed matter [1]. Given by the small track diameter of a few nanometers in combination with the large range of up to 100  $\mu$ m (and more), energetic ion beams offer unique possibilities of modifying materials properties and producing micro- and nano-structures [2]. A short glimpse on the present activities in the field of ion track technology is provided including single-pore membranes as sensors for biomolecules [3], ion-track perforated micromoulds and microfluidic systems [4], and templates for electro-deposition of nanowires [5]. [1] e.g. proceedings of SHIM (Swift Heavy Ions in Matter) conferences, Nucl. Instr. and Meth. B 209 (2003). [2] M.Toulemonde, C.Trautmann, E.Balanzat, K.Hjort, A.Weidinger, Nucl. Instr. Meth. B 216 (2004) 1. [3] S. Metz, C. Trautmann, A. Bertsch, and Ph. Renaud, J. Micromech. Microeng. 14 (2004) 324. [4] C.R. Martin, Z.S. Siwy, E. Heins, C. Harrell, C. Trautmann, Biophysical Journal 86 (2004) [5] M.E. Toimil-Molares, A.G. Balogh, T.W. Cornelius, R. Neumann, C. Trautmann, Applied Physics Letters 85 (2004) 5337.

9:30 AM \*OO1.2

Simulations of Swift Heavy Ion Processes. Eduardo M. Bringa, Computational Material Sciences, Lawrence Livermore National Laboratory, Livermore, California.

There are numerous atomistic simulation studies in the area of keV ion bombardment, but relatively few studies on MeV-GeV ion bombardment, where energy is deposited mostly into electronic excitations. I will focus on three different models to include electronic effects into classical molecular dynamics simulations: the Coulomb explosion model, the thermal spike  $\bmod el$ , and a thermal spike including a two-temperature model (TTM). In the thermal spike model the electronic energy transferred to the ions is given as an initial high temperature for ions inside the track. When a TTM is included, the energy of the incident ion is deposited into the "fluid" of electrons, allowing for electronic heat conduction and using an electron-phonon coupling term to transfer energy to the ions. Using these various models, current parallel computers allow for simulations with empirical potentials of systems with up to several million atoms. Therefore, damage features of several nanometers can be simulated, including amorphous tracks, surface craters, etc. While these schemes are relatively simple, several examples from materials science and astrophysics will show that they do provide a reasonable description of several experimental results where electronic stopping power dominates. Few possible directions to improve the description of electronic excitations will be discussed. This presentation contains contributions from a number of people, including R.E. Johnson, D. Ivanov, O. Tucker, L. Zhigilei, R. Papaleo, A. Caro, L. Davila, B. Doyle, P. Rossi, P. Durham, W. Weber, R. Devanathan, and R. Corrales. The work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under contract of No.W-7405-Eng-48.

SESSION OO2: Sputtering, Surface Topography, Ripples & Dots Chair: George Vizkelethy Monday Morning, November 28, 2005 Commonwealth (Sheraton)

10:30 AM \*OO2.1

Shocks in Ion Sputtering. H. Henry Chen, Omar A. Urquidez, Stefan Ichim, Humberto Rodriguez, Michael P. Brenner and Michael J. Aziz; Div. Engrg. & Appl. Sci., Harvard University, Cambridge, Massachusetts.

Through theory, experiments and computer simulations, we report a new regime of ion beam sputtering, occurring for sufficiently steep slopes. High slopes propagate over large distances without dissipating the steepest features. Both the propagation velocity and the dynamically selected slope are universal, independent of the details of the initial shape of the surface. Our theoretical results follow from the classical theory of sputtering and develop a small-curvature approximation that is valid for any slope. We show that the resulting behavior can be understood as the propagation of a shock that self-selects a stable slope; the mathematical structure of the solutions

is the same as that previously observed in thin film fluid flows. Experiments show striking experimental confirmation of the predictions of the theory. An important implication of the propagative behavior at high surface slopes is that a pattern can be fabricated at a large length scale and, through uniform ion irradiation, reduced to a smaller length scale while preserving the sharpest features.

### $11:00 \text{ AM } \underline{\text{OO}2.2}$

Nanoscale Mass Redistribution Mechanisms and Morphology Evolution due to Ion Sputtering. N. Kalyanasundaram<sup>1</sup>, B. Davidovitch<sup>2</sup>, Michael J. Aziz<sup>2</sup>, Michael P. Brenner<sup>2</sup>, Jonathan B. Freund<sup>3</sup> and Harley T. Johnson<sup>1</sup>; <sup>1</sup>Department of Mechanical and Industrial Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>2</sup>Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts; <sup>3</sup>Department of Theoretical and Applied Mechanics, University of Illinois at Urbana-Champaign, Urbana, Illinois.

We study nanoscale mechanisms of mass redistribution and dynamics near a surface due to ion bombardment. We analyze the impact of ion-stimulated surface mass redistribution, in addition to sputter erosion, on morphology changes such as ripple and dot formation. We extract from molecular dynamics simulations a response function. defining the change in elevation at any point on a surface due to an impact at an arbitrary point. The spatial dependence of this function reflects the combined effects of mass redistribution and sputter removal. We use our new response function to derive a linear continuum equation for surface morphology, finding new terms that can imply qualitative and quantitative variations from Bradley-Harper theory in the linear instability of an initially flat surface. Furthermore, we use our formalism to find the leading non-linear terms in the continuum equation, and relate the results to pattern formation.

### 11:15 AM OO2.3

Formation of Nanodots on Si (100) Surfaces During Low Energy Ion Bombardment in the Presence of Mo Seed Atoms. Gozde Ozaydin<sup>1</sup>, Ahmet S Ozcan<sup>2</sup>, Yiyi Wang<sup>2</sup>, Karl F Ludwig<sup>2</sup>, Hua Zhou<sup>3</sup> and Randall L Headrick<sup>3</sup>; <sup>1</sup>Aerospace and Mechanical Engineering, Boston University, Boston, Massachusetts; <sup>2</sup>Physics, Boston University, Boston, Massachusetts; <sup>3</sup>Physics, University of Vermont, Burlington, Vermont.

Real time x-ray studies of the formation of nanodots during Ar<sup>+</sup> ion bombardment of Si (100) surfaces in the presence of Mo seed atoms are presented. Silicon (100) surfaces are bombarded with low energy (100-1000eV) Ar<sup>+</sup> ions at room temperature. Without Mo seeding, Si surfaces develop only power-law roughness. However, a small amount of Mo seeds supplied to the Si surfaces during ion bombardment initiates the formation of highly correlated nanodots that are typically 3 nm high with a spatial wavelength of approximately 30 nm. As ion bombardment continues these nanodots saturate and the overall roughness is dominated by the larger length scale roughness. Formation of GaSb nanodots during low energy Ar<sup>+</sup> ion bombardment of GaSb surfaces at normal incidence is also studied in real time.

11:30 AM  $\underline{OO2.4}$  Quantifying the Order of Spontaneous Ripple Patterns on Ion-Irradiated Si(111). H. Bola George<sup>1</sup>, Ari-David Brown<sup>2</sup>, Jonah Erlebacher<sup>2</sup> and Michael J. Aziz<sup>1</sup>; <sup>1</sup>Division of Engineering & Applied Sciences, Harvard University, Cambridge, Massachusetts; <sup>2</sup>Department of Materials Science & Engineering, Johns Hopkins University, Baltimore, Maryland.

Uniform keV ion irradiation causes a morphological instability known to result in the spontaneous formation of topographic ripple and dot patterns. The degree of order of these patterns, which has important implications for non-lithographic patterning applications, varies markedly with fabrication conditions. We investigate the influence of systematic variations of fabrication conditions, including ion energy, current density, ion fluence, and substrate temperature upon the degree of order of argon ion bombarded Si(111) surfaces. For quantifying order in sputter rippled topographic images, we develop an algorithm that evaluates the density of topological defects, such as ripple bifurcations and terminations, and suitably normalizes the result in order to present a scalar figure of merit: the normalized defect density. We discuss fabrication conditions that lead to extremely well ordered ripple patterns upon irradiation.

11:45 AM OO2.5

Morphological Evolution and Non-Equilibrium Relaxation Kinetics during Sputter Ripple Formation. Wai Lun Chan and Eric Chason; Division of Engineering, Brown University, Providence, Rhode Island.

Depending on the processing conditions, a large variety of morphologies can be formed on a surface during low energy ion bombardment, e.g., nano-ripples, nano-dots, smoothening, and kinetic roughening. The resulting morphology is due to a complex interaction between ion-surface interactions and defect mediated surface transport. The surface relaxation mechanism, often simplified in current instability models, plays a very important role in determining the correlation length and the roughening rate of the resultant morphology. Recent results from kinetic Monte-Carlo simulation and experiments on Cu(001) will be used to show how the relaxation kinetics on a stepped surface during ion bombardment not only differ from classical Mullin's type diffusion but also from the relaxation of a stepped surface under thermal equilibrium condition. The scaling behavior of the relaxation rate with the average defects concentration and wavelength of the ripples morphology is reported. The results are important in determining the wavelength of the sputter ripples and its flux and temperature dependence during ion bombardment. The authors gratefully acknowledge the support of the U.S. Department of Energy under contract DE-FG02-01ER45913.

> SESSION OO3: Focused Ion Beams Chair: Anny Michel Monday Afternoon, November 28, 2005 Commonwealth (Sheraton)

### 1:30 PM \*OO3.1

A Novel, Focused Ion Beam Directed Route for the Local Synthesis of Nanowires at Room Temperature. Alois Lugstein and Emmerich Bertagnolli; Institute for Solid State Electronics, Technical University of Vienna, Vienna, Vienna, Austria.

The exciting discovery of nanowires has been sparked by a desire to tune the fundamental optical, electronical, mechanical and magnetic properties of materials through rational control of their physical size. The broad field of possible applications ranges from new-generation nanoelectronics to catalysis. Several, chemical and material techniques for the production of various types of nanowires have been reported, whereby most of them are based on the vapor-liquid-solid mechanism. However, there is still an on-going effort in developing new synthesis methods with the main goal to grow nanowires at moderate temperatures not to damage preexisting modules and to grow them at a prespecified location while eliminating the requirement of a later assembly process. We developed a novel focused ion beam based technique for the local synthesis of nanowires without the requirement of any additional materials source. Nanowire growth took place in room temperature ambient without any sample heating thereby opening the door to cheaper and faster commercialization, and being compatible with on-chip microelectronics. Gallium antimonide, antimony and germanium nanowires were grown with diameters of about 20 nm and lengths of a few micrometers. The focused 50 keV Ga+ ion beam with a beam current density of 0.8 A/cm2 was used to form catalytic nanoparticles eliminating the usual requirement of pre-patterning quantum sized droplets. The morphological evolution of the sample surface was investigated by in-situ FIB-SEM, SEM, EDX, HRTEM, and the chemical composition of the pattern was analyzed using high resolution AES. We completed this many-facetted experimental study with Raman and XRD measurements. In contrast to the Ge and Sb nanowires, which appear to be fully amorphous, HRTEM investigations revealed that for thin GaSb nanowires, crystallites are embedded in the amorphous matrix. The average size of the nanocrystallites is in the range of 3 to 15 nm. Some thicker GaSb nanowires appear to be polycrystalline with no obvious preferred growth direction and with an amorphous sheet on the outer surface. The elemental composition of the nanowires investigated using EDX revealed an almost ideal 1:1 stoichiometry of Ga and Sb. Studies carried out with different substrate materials and beam energies confirmed the central details of the growth mechanism and suggest that our approach can be used, in principle for synthesis of other material nanowires.

### 2:00 PM OO3.2

Focused Ion Beam-induced Ripple Structure and Phase Decomposition in  $Cd_2Nb_2O_7$ . Jie Lian<sup>1</sup>, Wei Zhou<sup>2</sup>, Lumin Wang<sup>1</sup>, Lynn A. Boatner<sup>3</sup> and Rodney C. Ewing<sup>1</sup>; <sup>1</sup>University of Michigan, Ann Arbor, Michigan; <sup>2</sup>Nanyang Technological University, Singapore, Singapore; <sup>3</sup>Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Ion sputtering-induced ripple structures have been of particular interest for the fabrication of nanoscale-textured materials via self-organization processes or as templates for the growth of nanowires, nanorods and nanodots. Here, we report the first experimental result of the simultaneous formation of a ripple structure with the characteristic wavelength varying from nm to sub- $\mu m$  range induced by ion sputtering and the formation of uniformly-distributed metallic nanoparticles with the size of  $3{\sim}10$  nm caused by ion irradiation-induced phase decomposition. Ripple structure perpendicular to the ion projection direction in  $Cd_2Nb_2O_7$ 

pyrochlore was created by focused ion beam (FIB) irradiation with 30 keV Ga<sup>+</sup>, at off-normal incident angles varying from 37° to 60°. The surface morphologies upon ion beam sputtering were investigated by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The characteristic wavelength of the ripple structure can be controlled from ~130 nm to 550 nm, varying with the incident angles and irradiation dose. The corresponding surface roughness also varies as a function of irradiation dose and incident angles. Ripples were patterned at different areas of a Cd<sub>2</sub>Nb<sub>2</sub>O<sub>7</sub> thin foil for TEM observation in order to characterize the ripple microstructure in detail. The ripple structure was clearly evident by high-angle annual dark field (HAADF) image and EDS mapping. A phase decomposition occurred in the ion sputtered pyrochlore, and Cd metallic nanoparticles with the size of 3~10 nm were observed. These results demonstrate that focused ion beam patterning combining with the FIB micro-milling has significant impacts in understanding the self-assembled processes and the microstructure of the ripple structure in a wide range of materials.

## 2:15 PM <u>OO3.3</u>

Local Charging Induced Chemical Modification of Focussed Ion Beam Processed Simox. Marion A. Stevens-Kalceff and Thomas L. Sobey; School of Physics, University of New South Wales, Sydney, New South Wales, Australia.

Processing of materials used in semiconductor technology by focussed (keV) ion beam irradiation is an expanding and important area of research and technique development. For example, ion beam assisted deposition and sputtering processes enable the maskless modification of materials at the nanoscale. When semiconductors and dielectrics are modified by a focussed ion beam, localized charging may occur and the assessment of the consequences of charging and associated physical processes must be assessed to enable optimal processing. SIMOX (Separation by IMplantation of Oxygen) Silicon-On-Insulator (SOI) wafers are widely used in applications including high-performance/ low power CMOS and MEMS devices. The influence of incidental charging of SIMOX resulting from focussed ion beam processing has been assessed using Kelvin Probe Microscopy (KPM). KPM is a specialized Atomic Force Microscopy technique in which long-range Coulomb forces between a conductive atomic force probe and a specimen enable the electrical potential at the specimen surface to be characterized. The surface potential may be associated with a localized charge distribution within the specimen, and information about the spatial extent and relative magnitude of this charge distribution can be deduced from the surface potential data. Significant focussed ion beam irradiation induced surface potentials are measured and modelled using Finite Element Analysis and are also compared with spatially resolved Electron Microprobe data of the relative Ga, O and Si concentrations. The observations are consistent with a number of complex competing physical processes including ion neutralization, positive/secondary ion emission/ sputtering, ion implantation and charge trapping resulting in local electromigration of oxygen. This investigation shows that ion beam processing of SIMOX can result in local modification of the chemical composition of the specimen via implantation and electromigration. This has implications for the nanoscale processing of semiconductor materials in a Focussed Ion Beam system.

> SESSION OO4: Structural Modifications I: Defect Accumulation, Amorphization, Strain Engineering, Grain Orientation Control Chair: Daryush Ila Monday Afternoon, November 28, 2005 Commonwealth (Sheraton)

# 3:30 PM <u>\*OO4.1</u>

Glass Transition in Silicon Induced by Swift Heavy Ions.

Andre Hedler<sup>1</sup>, Siegfried Klaumuenzer<sup>2</sup> and Werner Wesch<sup>1</sup>; <sup>1</sup>Institut fuer Festkoerperphysik, Friedrich-Schiller-Universitaet Jena, Jena, Germany; <sup>2</sup>Hahn-Meitner-Institut Berlin, Berlin, Germany.

Amorphous silicon (a-Si) is a tetrahedrally coordinated semiconductor with a density of  $2.29~\mathrm{g/cm^3}$  at ambient temperatures, whereas liquid silicon (l-Si) is metallic, has an average coordination number of about 6 and a density of  $2.55~\mathrm{g/cm^3}$  at the equilibrium melting point of crystalline silicon at 1685 K. Due to these structural differences a-Si has not been considered to be a glass. In fact, numerous experiments and simulations have been interpreted as evidence for a first order phase transition from a-Si to l-Si, denoted as melting of a-Si. According to an extrapolation of the Gibbs free energies the melting point of a-Si is expected to be at around 1450 K. In contrast to this, recent computer simulations on supercooled liquid silicon have revived the idea of a liquid-liquid phase transition between the high-density metallic liquid and a low-density liquid followed by a glass transition to solid a-Si. However, due to the crystallization of a-Si above 1000 K

on the nanosecond time-scale conventional analyzing methods fail and the nature of the phase transition has never been fully clarified in experiments. In this work the effects of swift heavy ion irradiation of a-Si have been studied as a function of electronic energy deposition, sample temperature, ion fluence and ion incident angle. It will be shown that a-Si flows plastically in the same way as conventional glasses. The positive sign of the deformation yield provides experimental evidence for the existence of the low-density liquid. A consistent quantitative description for the dependence of the deformation yield on the electronic energy deposition for low temperatures will be shown by following closely the viscoelastic model for ion hammering. In this way, the glass transition temperature for a time-scale of 10 ps is estimated to be about 1000 K independent of the precise details of the liquid-liquid phase transition. Our results support the idea of liquid polymorphism as a general phenomenon in tetrahedral networks.

### 4:00 PM <u>OO4.2</u>

Ion-Beam-Induced Amorphization in Gallium Nitride and Silicon Carbide. Weilin Jiang<sup>1</sup>, Jie Lian<sup>2</sup>, Yanwen Zhang<sup>1</sup>, William J. Weber<sup>1</sup> and Rodney C. Ewing<sup>2</sup>; <sup>1</sup>Pacific Northwest National Laboratory, Richland, Washington; <sup>2</sup>The University of Michigan, Ann Arbor, Michigan.

Both gallium nitride (GaN) and silicon carbide (SiC) are wide bandgap semiconductor materials that have great potential for a wide range of electronic and optoelectronic applications. SiC also has significant potential for use in future nuclear power applications. A fundamental understanding of the amorphization processes in these materials is needed to assess or predict device performance or nuclear operations. In this study, room-temperature irradiation with 1 MeV Au<sup>+</sup> ions has been used to produce amorphized layers on the 6H-SiC and GaN single crystal surfaces. Thin samples for transmission electron microscopy (TEM) were prepared and energy dispersive x-ray (EDX) analysis in a line-scan mode was performed to profile host elements as a function of depth across the amorphized regions. The GaN results suggest that there is a volume expansion in the buried disorder saturation regime, which is less significant than in the amorphization regime. The nitrogen atoms in GaN are mobile during the irradiation at room temperature, and a significant amount of N atoms diffused from the disorder saturation regime to the amorphized surface. N loss near the surface region is also observed. In the case of SiC, a gradual increase of C and decrease of Si concentrations with the decreasing depth are observed in the amorphized regime. Since C atoms are more mobile than Si in SiC, the behavior is likely due to C diffusion from a greater depth to the surface during the ion irradiation at room temperature. In addition, microstructural evolutions from perfect crystal to complete amorphization in the irradiated GaN and SiC, along with some striking features (such as bubble/cavity distributions) will also be presented and discussed.

# 4:15 PM OO4.3

Kinetics of Damage Accumulation and Annealing in Au-irradiated SrTiO<sub>3</sub>. Yanwen Zhang<sup>1</sup>, Chongmin Wang<sup>1</sup>, Weilin Jiang<sup>1</sup>, Fei Gao<sup>1</sup>, Mark H. Engelhard<sup>1</sup>, Thevuthasan Suntharampillai<sup>1</sup>, William J. Weber<sup>1</sup>, Jie Lian<sup>2</sup> and Rodney Ewing<sup>2</sup>; Pacific Northwest National Laboratory, Richland, Washington; <sup>2</sup>Department of Geological Sciences, University of Michigan, Ann Arbor, Michigan.

Single crystal strontium titanate ( $SrTiO_3$ ) is of technological interest in microelectronics industries due to its high dielectric constant, good insulating properties, outstanding wear resistance, high resistance against oxidation, and chemical and thermal stability. Strontium titanate and other titanate ceramics have also been proposed as phases for immobilization of nuclear waste. In many of these applications, knowledge of dynamic accumulation, recovery and nanostructure evolution is critical. In the current work, damage accumulation in strontium titanate (SrTiO<sub>3</sub>) irradiated with 1.0 MeV Au ions has been investigated at temperatures from 150 to 400 K. The relative disorder on the Sr and Ti sublattices at the damage peak has been determined as a function of local dose and temperature. A disorder accumulation model, with contributions from the amorphous fraction and the crystalline disorder, has been fit to data, and the results indicate that defect-stimulated amorphization is the primary amorphization mechanism below the critical temperature. The response of ion-beam induced amorphous layers in SrTiO<sub>3</sub> to electron beam (e-beam) irradiation was also studied. Recrystallization of the amorphous layer was measured in situ under 200 keV electron-beam (e-beam) irradiation over temperatures from 300 to 400 K. The e-beam enhanced recrystallization rates are orders of magnitude higher than those expected from thermal epitaxial recrystallization. A sub-linear-like regrowth dependence on exposure time may be used as a fingerprint for the e-beam enhanced recrystallization, as opposed to the super-linear-like behavior normally observed in thermal recrystallization. Analyses of damage accumulation and recrystallization data indicate that the irradiation-enhanced and

thermal recovery processes have activation energies of  $0.1\mathrm{eV}$  and 0.7eV, respectively.

## 4:30 PM <u>OO4.4</u>

Irradiation-Enhanced Second-Phase Precipitation in Zr-Fe Nanocrystalline Thin Films. Djamel Kaoumi<sup>1</sup>, Arthur Motta<sup>1</sup> and Robert Birtcher<sup>2</sup>; <sup>1</sup>Penn State University, University Park, Pennsylvania; <sup>2</sup>Argonne National Laboratory, Argonne, Illinois.

In situ observations in a transmission electron microscope (TEM) were used to study ion-beam enhancement of second-phase precipitation in Zr-Fe nanocrystalline thin films. The free-standing films were prepared by co-sputter deposition with Fe contents ranging from 0 to 4.5 at%. TEM diffraction analysis showed that only the hcp Zr crystal structure was present in the as-deposited films. No second phases were detected, although Rutherford Backscattering Spectroscopy (RBS) confirmed a Fe content well beyond the solubility limit of Fe in Zr (on the order of ppm), which means the thin films were supersaturated solid solutions of Fe in Zr. Heat treatment in the absence of irradiation was observed to cause precipitation of the Zr2Fe intermetallic phase, but only above 673 K. The same second-phase precipitation occurs at lower temperatures in the presence of ion irradiation. Samples were irradiated in-situ at the Intermediate Voltage Electron Microscope (IVEM) at Argonne National Laboratory with Ar and Kr ions to fluences in excess of 1016 ion/cm2, at temperatures ranging from 20 to 573 K. Second phase precipitation was detected by electron diffraction patterns and by dark-field imaging comparing regions exposed to the beam and regions protected from the beam by the TEM support grid. At all irradiation temperatures nanometer-sized Zr2Fe intermetallic precipitates formed, similarly to the thermal runs. In the bulk, this phase is thermodynamically metastable in the range of temperatures investigated (relative to the orthorhombic Zr3Fe intermetallic phase). The effect of irradiation temperature and the influence of the ion type on the kinetics of the reaction were studied. The kinetics of the irradiation-enhanced second-phase precipitation was followed by recording the diffraction patterns at regular intervals. The dose to a given level of precipitation was found to decrease with irradiation temperature and with increasing Fe content. The dose in dpa to a given level of second phase precipitation was comparable for the two types of ions used. The results are discussed in terms of existing models of precipitation under irradiation.

 $4{:}45~\mathrm{PM}~\underline{\mathrm{OO4.5}}$  Nano-Sized Titanium and Zirconium Carbides: Synthesis, Characterisation and Irradiation. Mickael Dolle<sup>1</sup>, Dominique Gosset<sup>1</sup>, Christine Bogicevic<sup>2</sup>, Fabienne Karolak<sup>2</sup>, Gianguido Baldinozzi<sup>2</sup> and David Simeone<sup>1</sup>; <sup>1</sup>DEN-DANS-DMN-SRMA, Gif sur Yvette, France; <sup>2</sup>SPMS, ECP, Chatenay Malabry, France.

Nano-sized grain materials have shown recently an increasing interest explained by the new properties, which arise for example from the absence of extended defects in the particles [1]. In fact, unusual mechanical properties are expected for such materials in which the usual characteristic distance between defects (e.g. dislocations) is higher than the grain size and then the proportion of atoms involved in grain boundaries is no longer negligible as compared to the bulk material ones. Controlling the properties of the grain boundaries then leads to new properties such as superplasticity [2] or toughening even in the case of brittle matrix materials. The development of a new generation of nuclear reactors (Gen-IV project [3]), with improved thermodynamic yield and a drastical reduction of waste production, makes it necessary to consider new materials able to withstand very high temperatures (1000-1200°C in normal conditions, up to 1500°C in incidental ones). Moreover, in the case of fast-neutron reactors to be used for nuclear waste burning, low-Z materials can no longer be used due to too high neutron slowing-down efficiency. Compounds, among which the transition metal carbides ZrC and TiC, are then to be considered. Those materials are highly refractory, have good thermal conductivity [4], low neutron absorption or scattering cross sections, low damage under irradiation [5]. Unfortunately, they have a brittle mechanical behaviour. We have then undertaken the elaboration of nano-sized carbide powders in order to test the potentialities of those materials in two different directions. improvement of the mechanical properties (toughness, yield) and behaviour under irradiation (defect clustering, swelling).Our presentation will then focus on the preparation of nanotextured ceramics. We will first present the synthesis of nano-sized powders by different ways in order to obtain materials with a good homogeneity and few impurities (oxygen and free carbon). For example, we succeeded, using the sol-gel method, in preparing nano-sized powders by carbothermal reduction. The particles size is around 30-40 nm for TiC and 60-100 nm for ZrC and the samples have a low free carbon content. These materials were irradiated by 4 MeV Au ions. The structural stability of these phases under irradiation were then followed by X ray grazing diffraction. Results are discussed. [1] R.W. Siegel, Mat. Sci. Forum, **235-238**, 851-860 (1997). [2] Yulin Lu, P.K. Liaw, JOM **53(3)**, 31-35 (2001). [3] J. Bouchard, IAEA

International conference on fifty years of nuclear power - The next fifty years. Book of extended synopses **234**, 22-23 (2004). [4] E.K. Storms, P. Wagner, High temp. Sci. **5**, 454-462 (1973). [5] R.A. Andrievskii et al., Inorg. Mat. 14-4, 530-533 (1977).

> SESSION OO5: Poster Session I Chair: Yanwen Zhang Monday Evening, November 28, 2005 8:00 PM Exhibition Hall D (Hynes)

005.1

The influence of surface charging on the modification of surface properties by means of low energy ion implantation. Mourad Yedji and Guy G. Ross; INRS-Energie, Materiaux et Telecommunications, Universite du Quebec, Varennes, Quebec,

Low energy ion implantation produces an accumulation of electric charge on the surface of insulators. In previous works [1,2], we studied the effect of charge accumulation on the implanted ion profile and investigated an original technique for charge neutralization. In the present work, we use these mechanisms as a tool to effectively modify the surface properties of polymers. Samples of polyethylene (PE) and polystyrene (PS) have been implanted with 2.2 keV D2+ ions to a fluence of 3.9 x 1016 D+/ cm2 with and without the use of the neutralization system. The physicochemical properties of the surface have been characterized by elastic recoil detection (ERD), Rutherford backscattered spectroscopy (RBS), photoelectron spectroscopy (XPS) and atomic force microscopy (AFM). For both samples, AFM characterization shows that the roughness of their surfaces decreases by a factor of 10 after ion beam bombardment with neutralization. However, the roughness increases during the implantation with charge accumulation. The wettability of the treated surface is increased with both methods of implantation. We note that the effect of charge accumulation enhances the decrease of the contact angles of water on both PE and PS. RBS analysis shows a physical trapping of oxygen with both treatments. The samples acquire more oxygen in the case of implantation without neutralization. XPS results confirm this result and reveal the formation of C-O, C=O, and O-C=O molecular bonds. This behavior increases with the degree of the charge accumulation. Our results show that the level of surface charging affects the morphology, the kind of damage at the surface, and gives a complementary tool to adjust the wettability of polymer surfaces. [1] G.G. Ross and C. Sevigny, Nucl. Instrum. Meth. B 211 (2003) 351. [2]
 M. Yedji and G.G. Ross, Nucl. Instrum. Meth. B 230 (2005) 386.

OO5.2
DLC Formation in C Implanted and Deposited PET. Wu Yuguang<sup>1</sup>, Zhang Tonghe<sup>1</sup> and Wu Zhenglong<sup>2</sup>; <sup>1</sup>Key Laboratory for Radiation Beam Technology and Material Modification, Institute of Low Energy Nuclear Physics, Beijing Normal University, Beijing, China; <sup>2</sup>Analytical and Tester Center, Beijing Normal University, Beijing, China.

Polyethylene terephthalane (PET) has been implanted and deposited with C ions attracted from a metal vapor arc source (MEVVA). The thinckness of C deposited on PET is 400nm. After implantation, sheet resistance decreased. When the dose increases from 1x10<sup>16</sup>/cm<sup>2</sup> to 2x10<sup>17</sup>/cm<sup>2</sup>, the resistivity decreases 7-8 orders of magnitude. The surface hardness (H) and modulus (E) measured by nano-indenter tester increase obviously. The surface hardness of 6.28 GPa of implanted PET is 15 times greater than that of PET, 2 times greater than stainless steel hardness (3GPa), the Modulus increases 2.6 times comparing with PET. The hardness of C deposited PET is 8-9.6GPa. It is 15-18 times greater than that of PET. TEM photo shows that the nano-particles were formed. It can be seen that the surface structure has greatly changed. The PET has been strengthened by these dispersed nano-particles. The surface wear resistance improved extremely. XPS analysis shows that sp<sup>3</sup> content is 27.7%, 32.7% and 60% for C implanted PET with dose of  $1x10^{17}/cm^2$ ,  $2x10^{17}/cm^2$  and C deposited PET respectively. It indicated that the content of sp<sup>3</sup> increased with increasing of C ion dose. The diamand-like film is formed by C ion deposition. The stability of electrical properties is very good. The resistance of implanted layer increases by a factor of 1.5 within 730 days only. The wear times of C deposited polymer increases by a factor 30 to 50 comparing with undeposited molymer.

Low Energy High Current Ion Beam modifications on Polyimide Substrates by Vacuum Web Sputtering System. Jung Cho<sup>1</sup>, Byung Jae Kim<sup>1</sup>, Young Seop Kim<sup>1</sup> and Won Kook Choi<sup>2</sup>; <sup>1</sup>TL business team, Toraysaehan, Seoul, South Korea; <sup>2</sup>Thin Film Technology Research Center, Korea Institute of Science and

The modification mechanism of C implanted PET was discussed.

Technology, Seoul, South Korea.

Surface modification on polyimide films have been widely adopted in information technology such as production of flexible electronics, drive IC substrates for liquid crystal display and optical pickup for hard disk suspensions. For these applications, two kinds of flexible copper clad laminates (F-CCL) have been developed. One is casting type two layer F-CCL which polyimide varnish is coated on copper foil and cured on copper foil surfaces. The other is oppositely making of copper foil on polyimide films by using both vacuum sputtering and electroplating. In this paper, we deposit a thin tie layer  $(10\sim15~\mathrm{nm})$ and Cu seed layer (200 nm) on polyimide film (Kapton-EN and Upilex-S) after very low energy ion beam irradiation with various gases for the improvement of adhesion and thermal stability. The surface energy of unmodified and ion beam modified polyimide film were measured 38 and 81 erg/cm, respectively. After electroplating  $9\mu$ m thick Cu foils on polyimide, the thickness uniformity, adhesion strength and a thermal stability of the FCCL are investigated by a x-ray thickness measurement, a 900 peel strength tester and thermal curing treatment. Also an interfacial reaction between Cu and polyimide with the irradiation of the reactive gas ion species are examined by x-ray photoelectron spectroscopy and oxidation state of the deposited Cu influencing the adhesion are also analyzed. As the results, the peel strength of the as-received FCCL is higher than 0.8 kgf/cm and kept 0.65 kgf/cm even after thermal treatment 7 days at 150 o<sup>C</sup> and a 9  $\mu$ m thick FCCL shows a very good thickness uniformity with only  $\pm 2.8\%$  standard deviation over 520 mm in width.

Analytical TEM Investigations of Gold Nanowires Formed Along Swift Heavy Ion Tracks in Nickel Oxide. Ch. Dais $^1$ , Joerg K. N. Lindner $^2$ , B. Stritzker $^2$  and Wolfgang Bolse;  $^1$ Institut fuer Strahlenphysik, Universitat Stuttgart, Stuttgart, Germany; <sup>2</sup>Insitutut fuer Physik, Universitaet Augsburg, Augsburg, Germany.

Structure and phase formation occuring within the limited volume of an ion track are an analytical challenge even for highly resolving TEM techniques. In the present case we have investigated the formation of metallic nanowires by gettering of Au atoms in the tracks of swift heavy ions in Au/NiO multilayer sandwiches using analytical TEM techniques. The thickness of the individual Au-layers was 2 - 10 nm, those of the NiO were ranging from 20 - 50 nm. The samples were irradiated at 80 K with Ar, Kr, Xe and Au ions of energies ranging between 90 and 600 MeV. After irradiation TEM shows that the Au is not anymore located in the initial marker planes, but has been relocated into cylindrical regions of about 5 nm in diameter at typical distances of 10 - 20 nm, which are oriented along the ion beam direction. In case of Au and Xe irradiation these cylinders often extend from the NiO/Si interface up to the surface, where part of the Au was found to segregate in small nano-sized particles. In case of Kr ions only short cylinders are formed on both sides of the initial Au marker planes. Ar irradiation did not affect the initial Au distribution. The formation of these nano-wires can be understood taking into account a transient melting and rapid  $(10^{-11}s)$  resolidification process in a cylindrical region of about 10 nm in diameter along the ion trajectories. The insoluble Au, which is located in this molten region will be transported into center of the track by Au segregation at the solid-liquid front moving from the outer parts of the track towards its center. Because of volume conservation the Au will at the same time be transported along the track direction. Subsequent ion impacts in the vicinity of these Au-segregates will result in further relocation of the Au until the nano-wires of some nm in diameter and typical distances of the molten track radius are formed.

Porous SiO2/Si layers produced by ion bombardment: dependence on the ion energy. Aricia Dallanora<sup>1</sup>, Gerardo G. Bermudez<sup>2</sup>, Christina Trautmann<sup>3</sup> and Ricardo M. Papaleo<sup>1</sup>; <sup>1</sup>Faculty of Physics, Catholic University of Rio Grande do Sul, Porto Alegre - RS, RS, Brazil; <sup>2</sup>Laboratorio Tandar, Comision Nacional de Energia Atomica, Buenos Aires, Argentina; <sup>3</sup>Gesellschaft fur Schwerionenforschung, Darmstadt, Germany.

Ion bombardment and chemical etching were employed to produce porous SiO2 thin films with a narrow size distribution and pore sizes from few tens to hundreds of nanometers. The size of the conical  $\,$ holes, the cone angle, and the size dispersion were determined as a function of ion energy and the energy loss dE/dx for a fixed etching condition. Gold ions with energies from 0.03 to  $2200~\mathrm{MeV}$  from three different accelerators (at Porto Alegre, Brazil, Buenos Aires Argentina, and Darmstadt, Germany) were used to bombard SiO2 films grown onto Si at low fluences (1E8-1E9 ions/cm2). As expected, no distinguishable holes are formed at the lowest energies where the nuclear stopping dominates (0.03-2 MeV). This occurs even when the total stopping power is higher than the threshold electronic stopping power for track etching, which was found to be around 300 eV/Å.

Apparently no synergistic effects between the nuclear and electronic energy deposition processes take part, at least in what concerns track etching. A linear increase in the mean pore diameter was observed in the energy range of 5 to 85 MeV. For higher energies, the hole size increase very little because of the significant increase in the velocity of the ions and the consequent decrease of the energy density in the tracks. The dispersion of the hole size distribution is around 15% close to the threshold, but decrease to less than 5% for stopping powers higher than 1000 eV/Å.

### 005.6

Wafer Bonding Method for MEMS Fabrication and Packaging Using Proton Beams. E. H. Kim<sup>1</sup>, H. S Kim<sup>2</sup>, Y. J Kong<sup>1</sup>, H. K Jang<sup>3</sup>, J. W Hyun<sup>1</sup>, Yongmin Kim<sup>1</sup> and S. J Noh<sup>1</sup>; <sup>1</sup>Applied Physics, Dankook University, Seoul, South Korea; <sup>2</sup>PTL Lab., KAPRA, Cheorwon, South Korea; <sup>3</sup>Mecharonics Co., Ltd, Pyeongtaek, Gyeonggi-Do, South Korea.

Wafer-bonding techniques are key issues for the commercialization of MEMS(Microelectromechanical Systems) devices. The anodic bonding method and the wafer direct-bonding method are well-known major techniques for wafer bonding. However, the anodic bonding method includes high voltage processes above 1.5 kV and the wafer direct-bonding method includes high temperature processes above 1,000 °C. Thus, the MEMS devices can be damaged, degraded, malfunctioned etc. During the past few years, diverse effort has been undertaken to find reliable bonding processes that can be conducted at low temperature. Unfortunately, these new bonding processes are highly dependent upon the bonding material, surface treatment and surface flatness. Now, we propose a new bonding method which is based on the localized heating at a bonding interface by energy transport at the Bragg-peak of proton beams. 13-MeV proton beams of diverse currents from 1  $\mu A$  to 10  $\mu A$  were used for the irradiation of pyrex glass and silicon wafer of 1 cm x 1cm. Bonding between the pyrex glass and silicon wafer was successfully achieved without extra heating or electric fields. Detailed results and the applications for the MEMS fabrication and packaging will be presented.

Acknowledgement: This work is supported by the Ministry of Science and Technology of Korea through Proton Accelerator User Program of Proton Engineering R&D Project from the Nuclear R&D Program and the 21th Century Frontier R&D Program.

### OO5.7

Molecular dynamics simulations of cluster-size effect on sputtering process with reactive gas cluster ions. <u>Takaaki Aoki</u> and Jiro Matsuo; Quamtum Science and Engineering Center, Kyoto University, Uji, Japan.

For the last decade, surface modification processes utilizing gas cluster ion beam (GCIB) have been proposed. Especially, clusters generated from reactive gas source such like O2, \$F6, CF4, etc. have been studied for high-speed nano-scale etching processes. The characteristics of GCIB process is that, when a cluster, which consists of several tens to thousands atoms, impacts on a target surface, large number of collisions occurs simultaneously, which result in local heating, large motion and chemical excitation of surface atoms. The enhancement of sputtering yield by reactive cluster impact has been demonstrated by experiments but the mechanism is still unknown. In this study, molecular dynamics simulations of reactive cluster ions with various sizes impacting on solid targets were performed to investigate size-effect of reactive clusters on sputtering processes. Various sizes of fluorine clusters,  $(F_2)_{30}$ ,  $(F_2)_{300}$  and  $(F_2)_{3000}$ , were irradiated on Si(100) target at same total incident energy of 6keV. These clusters were irradiated on same target one after another in order to reproduce real experimental condition, such as accumulation of fluorine atoms in the target. The MD simulations of sequential cluster impacts allowed various statistical analyses about sputtered particles and they showed cluster size dependence obviously. For example, the major sputtered particles were deferent with each other; Si for  $(F_2)_{300}$  (100eV/atom), SiF<sub>2</sub> for  $(F_2)_{300}$  (10eV/atom), and SiF<sub>3</sub> for  $(F_2)_{3000}$  (1eV/atom). At the impact of large size cluster with low incident energy, large number of Si-F bonding were generated at the interface between cluster and target surface, which enhances formation of volatile SiFx compounds with much fluorine atoms. On the contrary, small cluster with high kinetic energy-per-atom could cause much energetic surface atoms at near surface region, which could be sputtered without well fluoridized. Fo all irradiation cases, the kinetic energy distributions of sputtered particles obey high-temperature Boltzmann distributions. This result indicates that each sputtering process is induced under hyper-thermal equilibrium condition due to multiple collision process of cluster impact. Through these time evolutional or statistical analyses about sputtered particles, formation and desorption process of precursors by reactive cluster impact will be discussed.

### 005.8

Sputter Yield Variations and Their Impact on Pattern

Formation during Ion Bombardment. N. Kalyanasundaram<sup>1</sup>, Jonathan B. Freund<sup>2</sup> and Harley T. Johnson<sup>1</sup>; <sup>1</sup>Department of Mechanical and Industrial Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>2</sup>Department of Theoretical and Applied Mechanics, University of Illinois at Urbana-Champaign, Urbana, Illinois.

Using molecular dynamics simulations, silicon sputter yield is studied as a function of conditions such as ion beam incidence angle, ion beam divergence and pre-stress in the target. The influence of sputter yield variations on ion-induced pattern formation is then considered. Statistically converged descriptions of sputter yields are obtained by performing multiple randomized simulations of argon ions incident on silicon (001) and (111) surfaces at 500eV beam energy. Silicon sputter yield exhibits a maximum yield at large but non-grazing incidence angle. The maximum yield in an initially stress free silicon target bombarded on the (001) surface is 2.5 silicon atoms per incident argon atom at an incident beam angle of 60 degrees away from the surface normal. Changes in sputter yields under varying input conditions like pre-stress in the sample and beam divergence affect morphology evolution. The effect of externally applied compressive and tensile stresses on sputter yields and on pattern formation is explained. The relation between sputter yield and spatial energy density deposited by ion beam in the target is investigated in detail. Momentum transfer considerations are used to explain the variations in sputter yield.

# 005.9

Models of Amorphous Hydrogenated Carbon Targets for Fusion-Reactor Sputtering Calculations. <u>Jaime Marian</u>, George H. Gilmer, Luis A. Zepeda-Ruiz, Eduardo Bringa and Thomas Rognlien; Lawrence Livermore National Laboratory, Livermore, California.

Due to their high temperature mechanical stability, graphite composites are being intensely examined as candidates for first-wall materials in magnetic fusion reactors. However, prolonged exposure of the graphite tiles to the plasma in the near-surface region is expected to produce two undesirable effects. The first one is carbon sputtering back into the edge plasma region, which entails heat energy losses and magnetic disturbances. Carbon sputtered in this fashion may appear as isolated atoms or in any light hydrocarbon (CxHy) form and sputtering is expected to be more pronounced at the divertor plates, where the plasma ion flux is thought to be maximum. Secondly, continued carbon erosion in the first wall causes thinning and may compromise its structural integrity. Over time, the graphite tiles develop a thin layer of amorphous hydrogenated carbon with hydrogen contents that typically range between 30 and 40%. It is believed that the dynamics of carbon sputtering from the first wall are governed by the physical characteristics of this amorphous layer, including its thickness and its associated surface roughness. Because of the atomic nature of these processes, molecular dynamics (MD) is an ideal tool to study them. In this paper we present results of MD simulations of graphite amorphization by low-energy deuterium. The original graphite target is bombarded by deuterium atoms at high dose rates under conditions representative of those found at the divertor plates. Results showing the time evolution of amorphization will be presented, along with a chemical composition analysis of the material. The samples produced in this manner will be used as targets for subsequent carbon sputtering calculations and the results will be compared with data obtained using targets obtained from graphite/deuterium atomic systems processed by simulated melting, quenching, and annealing.

# OO5.10

Abstract Withdrawn

### 005.1

Holistic Analysis of Successive FIB Slices by SEM Imaging and 3D Reconstruction. Terence S. Yeoh<sup>1</sup>, Neil A. Ives<sup>1</sup>, Nathan Presser<sup>1</sup>, Martin S. Leung<sup>1</sup>, Gary W. Stupian<sup>1</sup>, John L. McCollum<sup>2</sup> and Frank Hawley<sup>2</sup>; <sup>1</sup>The Aerospace Corporation, Los Angeles, California; <sup>2</sup>Actel Corporation, Mountain View, California.

The conductive region of an antifuse structure was analyzed using a method of scanning electron microscopy image slicing and three-dimensional reconstruction. Due to the nanometer scale feature sizes, the sample could not be easily imaged using a single focused ion beam cross-section. A method of voxel imaging and interpretive 3-D reconstruction with nanometer feature sizes was developed in order to resolve volumes as small as 10 nm<sup>3</sup>. From this analysis it was determined that the fusing region for an antifuse is comprised of an elliptical volume approximately 75 nm in diameter. The antifuse was found to be an inhomogeneous mass with voids as small as 10 nm<sup>3</sup>.

### 005.12

Heavily As-doped crystalline silicon: Rutherford back-scattering spectra interpreted by atomistic models.

Alessandra Satta<sup>1</sup>, Eros Albertazzi<sup>2</sup>, Simone Balboni<sup>3</sup>, Marco Bianconi<sup>2</sup>, Luciano Colombo<sup>1</sup> and Giorgio Lulli<sup>2</sup>; <sup>1</sup>Physics, SLACS-INFM, Monserrato (Ca), Italy; <sup>2</sup>CNR-IMM Sezione di Bologna, Bologna, Italy; <sup>3</sup>CeSIA - Settore Reti e Comunicazioni, Universita' di Bologna, Bologna, Italy.

Structures of vacancy-arsenic complexes are determined with ab initio method and used for atomistic simulation of Rutherford back-scattering channeling (RBS-C) spectra in heavily As-doped crystalline silicon. The purpose is to investigate whether the relaxation patterns of clusters containing different numbers (from 1 to 4) of As atoms, can be used as a fingerprint in structural analysis by conventional RBS-C. Simulation of RBS-C spectra in large (millions of atoms) model supercells populated with the relaxed configurations of  $As_mV$ , show that the feature which can distinguish between complexes is the off-lattice displacement of the Si atoms nearest neighbors of the vacancy. This is relatively large in the case of  $\mathrm{As}_1\mathrm{V}$ and As<sub>2</sub>V, negligible in the case of As<sub>4</sub>V and absent in the case of As<sub>4</sub>V. From these results it is deduced that in the case of samples equilibrated at high temperature, the lack of any significant disorder of Si atoms is consistent with the hypothesis of electrically inactive As being in the form of either  $\rm As_3V$  or  $\rm As_4V$  complexes. The effects of strain due to the doped-silicon lattice compression are taken into account. Such hypothesis is consistent with experimental measurements. The comparison of simulated spectra with experimental angular-scans observations is reported.

005.13

Surface and Thin Layer Analysis on the Nanometer Scale using TOF-SIMS with Bi and C60 Cluster Ion Beams.

Nathan Havercroft<sup>1</sup>, Albert Schnieders<sup>1</sup>, Felix Kollmer<sup>2</sup>, Rudolf Moellers<sup>2</sup>, Derk Rading<sup>2</sup> and Ewald Niehuis<sup>2</sup>; <sup>1</sup>ION-TOF USA, Inc., Chestnut Ridge, New York; <sup>2</sup>ION-TOF GmbH, 48149 Muenster, Germany.

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is a very sensitive surface analytical technique, covering a wide range of organic and inorganic applications. It provides detailed elemental and molecular information about surfaces, thin layers, interfaces, and full three-dimensional analysis of the sample. In recent years cluster primary ions have been successfully applied for the analysis of organic surfaces by TOF-SIMS. Compared to monoatomic primary ion bombardment the use of clusters leads to a considerable enhancement of the secondary ion efficiency up to several orders of magnitude. This enhancement not only increases the sensitivity for molecular species up to several orders of magnitude, but also improves the useful lateral resolution in organic imaging down to the sub-micrometer range. In this contribution, fundamental as well as practical aspects of a newly developed Bi cluster liquid metal ion source (cluster LMIS) will be addressed and compared with the more established Ga LMIS and Au cluster LMIS. The Bi source offers excellent prospects for organic and inorganic surface and thin layer analysis. Imaging with lateral resolution down to 100 nm as well as inorganic depth profiling with high depth resolution in the sub-nm range can be performed. On the other hand, C<sub>60</sub> is currently discussed to be a possible projectile for organic depth profiling and first results have been published. We therefore have integrated a newly developed C<sub>60</sub> ion gun into a TOF-SIMS instrument. The new setup allows beside a direct comparison between different primary ions such as  $C_{60}$ ,  $Au_n$ ,  $Bi_n$  the combination of metal cluster ions (e.g.  $Bi_n$ ) and  $C_{60}$  in dual beam depth profiling. Here the C<sub>60</sub> beam is used to erode the surface of organic samples while the Bi source is used to analyse the centre of the sputtered crater. The potential of the new cluster ion sources will be demonstrated along examples ranging from inorganic materials like metals, semiconductors, glass, etc to organic materials like polymers, cells and biological tissues.

### 005.14

Holmium silicide on Si(100): a detailed study of the c(2x2) structure by medium-energy ion scattering and scanning tunnelling microscopy. Steven Tear<sup>1</sup>, Tim Wood<sup>1</sup>, Chris Bonet<sup>1</sup>, Tim Noakes<sup>2</sup> and Paul Bailey<sup>2</sup>; <sup>1</sup>Dept of Physics, University of York, York, United Kingdom; <sup>2</sup>CCLRC Daresbury Laboratory, Warrington, United Kingdom.

There is considerable interest in the growth and structure of rare earth (RE) metals deposited onto  $\mathrm{Si}(100)$  because of the observation of the formation of near one-dimensional (1D) structures which can be several microns in length while typically less than 10 nm wide and 1 nm high [e.g. 1,2]. These nanowires of rare earth silicide are formed because of the anisotropy in the strain that arises from the different lattice mismatch between the hexagonal rare earth silicide a-axis and c-axis, with that of the underlying  $\mathrm{Si}(100)$  unit cell. The a-axis is quite closely matched whereas the c-axis is not. At higher coverages of the RE, there is some uncertainty about the exact nature of the structure of the silicide that is present, whether it is hexagonal or tetragonal. We present here a detailed structural study using

medium-energy ion scattering (MEIS) and scanning tunnelling microscopy (STM) of the c(2x2) structure that is formed when six monolayers of RE metal is deposited onto Si(100) and annealed to  $500\,^{\circ}$ C. MEIS is particularly well suited to this as it is possible to select, by energy analysis of the scattered ions, only those ions which have been scattered by the heavier RE atoms in the surface. This means that only areas of the surface which are occupied by rare earth atoms will comprise the MEIS data for analysis. We have used both  $100 \, \text{keV}$  H ions and  $50 \, \text{keV}$  He ions as the primary beam, the latter case to gain greater surface sensitivity. The MEIS analysis presented will distinguish between the two proposed structures for the RE silicide on Si(100), and provide details of the atomic arrangement in the silicide layer. [1] C Preinesberger, S Vandre, T Kalka, M Dahne-Prietsch, J. Phys. D 31 (1998) L43 [2] J. Nogami, B. Z. Liu, M. V. Katkov, C. Ohbuchi, N. O. Birge. Phys. Rev. B. 63 (2001) 233305

005.15

XμPIXE a Non-Invasive, Rapid Method for Elemental Analysis. Paula P. Provencio and Barney L. Doyle; Beam Solid Interactions 01111, Sandia National Laboratory, Albuquerque, New Mexico

Conventional approaches to elemental analysis typically require radiochemical and/or mass spectrometric analysis and often yield only qualitative results. We report an external microbeam particle induced X-ray emission (X $\mu$ PIXE) system that uses accelerator-based ion beam analysis (IBA) techniques and x,y position sensitive spatial acquisition to nondestructively detect, characterize, and interpret elemental composition. In contrast to conventional X-ray analysis,  $X\mu PIXE$  produces little Bremsstrahlung background radiation and is therefore more sensitive than standard electron-induced x-rays to detect trace elements. Quantitative measurements can be made of complex samples quickly. A sub-millimeter particle beam is quickly scanned over a sample in air with little or no sample preparation. A desired region is chosen and a more quantitative acquisition and analysis performed. Using the capabilities of  $X\mu PIXE$  with quantitative computer codes, such as GeoPIXE, the utility of performing in-air analyses of any sample can be easily accomplished.

### 005.16

Development of a low energy Ne atom scattering system for insulator surface strctural analysis. Kenji Umezawa<sup>1</sup>, Shigemitsu Nakanishi<sup>1</sup>, Eisuke Narihiro<sup>1</sup>, Kazufumi Oda<sup>1</sup> and Walter M Gibson<sup>2</sup>; <sup>1</sup>Dept. of Physics, Osaka Prefecture University, Sakai, Osaka, Japan; <sup>2</sup>Physics, Osaka Prefecture University, Sakai, Osaka, Japan.

We have been developing a low energy Ne atom scattering systems combined with a time-of-flight ion scattering spectrometer for insulator surface structural analysis. Insulator surface structure is difficult to study because of charging effects during electron or ion beam bombardment. Structural analysis of insulartor surfaces are very important in fundamental research as well as teh cnological fields. In our system, charged ion beams of  $2\rm{keV-Ne+}$  , are converted into neutral beams by charge exchange with the same element gas or a glass capillary in a small cell, after the primary beam passes through a beam chopper. Beam neturalization was around 35 %. This is in good agreement with the calculated value using electron transfer cross section, particle density and collision length. Other features of this system are pulsed beams, time-of-flight measurements, and a microchannle plate (MCP) detector is coaxially mounted along the primary beam. For interactions of keV atom beams with crystals, the distances of closest approach are < 0.1 A, collisions are between atomic cores. The atomic positions and composition are able to be determined at an outermost layer as well as a few surface layers. This is a home made equipment. We will show the detection systems with electric circuits, as well.

005.17

Withdraw

005.18

In Situ Growth Analysis of Epitaxial Ultrathin Films by Low Energy ISS. Wakana Hara, Masashi Kitamura, Sei Otaka, Takashi Okada and Mamoru Yoshimoto; Materials & Structures Laboratory, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan.

Coaxial impact-collision ion scattering spectroscopy (CAICISS) utilizing low-energy ion scattering has emerged as useful tool to identify both the topmost surface atomic species and epitaxial films. So far, we have reported on analysis of the surface structure of ZnO [1] and GaN [2] thin films by CAICISS. Wurtzite-type crystals such as GaN, AlN, and ZnO have been attracted much attention due to their superior physical properties as materials for the next generation electro-optic devices. The wurtzite-type structure crystals have two polar directions of [0001] (+c) and [000-1] (-c). The polarities of wurtzite-type thin films are known to affect on a light emitting property and surface chemical property. On the other hand, we have

investigated about low temperature (near room temperature) epitaxial growth of functional ceramic thin films by laser MBE (i.e. pulsed laser deposition in ultrahigh vacuum), e.g. CeO<sub>2</sub> thin films on Si (111) substrates [3]. We have recently achieved the room-temperature epitaxial growth of ZnO and AlN thin films on sapphire (0001) substrates with epitaxial NiO or TiN ultrathin buffer layers by laser MBE, respectively. In this work, the surface structures and the polarities of room-temperature epitaxially grown wurtzite-type films were examined by in situ CAICISS. The buffer-enhanced epitaxial ZnO thin films grown at room temperature had +c (Zn-surface) polarity, while the polarity of high-temperature grown ZnO thin films on the sapphire were -c (O-surface). Furthermore, we have successfully formed NiO nanowires along atomic step edges on the ultrasmooth sapphire (0001) substrates by laser MBE. The nanowires were found to be about 20 nm in width and about 0.5 nm in height along the straight, 0.2-high step edges of the substrate. From the viewpoint of nanoscale growth control of films, the crystal structure of NiO nanowires was also examined by CAICISS. [1] T. Ohnishi et al., Appl. Phys. Lett. 72 (1998) 824. [2] M. Sumiya et al., Appl. Phys. Lett. 75 (1999) 674. [3] M. Yoshimoto et al., Jpn. J. Appl. Phys. 32 (1995) Ĺ688.

### 005.19

Growth Modes and Surface Structural Analysis of Pd/Ni(111) using Low Energy Ion Beam Scattering Spectroscopy.

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Bimetallic catalysts are often used in industrial reactions. After a 0.5 ML Pd deposit (at 300 K) onto Ni(111) crystal a strong increase in activity was previously observed. The Pd/Ni(111) phase is a large lattice mismatched combination (10%). Impact-collision ion scattering spectroscopy and low energy electron diffraction (LEED) have been used to determine the surface strcture in the initial stage of Pd adsorption on Ni(111). Pd of 99.99% purity was evaporated at a rate of about 0.1 ML/min. onto a Ni(111) crystal to a coverage of 3ML at room temperature. The azimuthal angle scans clearly show that the drops in the magnitude of the Pd intensity are observed at every 60 ° for a polar angle of 78°. A periodicity of 120° for the azimuthal angle could be seen. This suggests that Pd(111) planes are grown on the Ni(111) substrate. Moreover, the polar angle scans show that the mixed domains of about 50% of Pd[11-2]//Ni[11-2] and Pd[11-2]//Ni[11-2] and Pd[11-2]//Ni[11-2] and Pd[11-2]//Ni[11-2] and Pd[11-2]//Ni[11-2] and Pd[11-2]//Ni[11-2] and Pd[11-2]//Ni[11-2]/ Pd[-1-12]//Ni[11-2] exist at the substrate temperature of 300 K during Pd deposition. First layer of Pd atoms with an outward displacement of 0.3 A for Pd deposition less than a coverage of 1/3 ML. The Pd-Pd distance of 2.80 A obtained along a [11-2] azimuth is slightly larger than the Pd-Pd distance (2.75A) in a bulk.

## 005.20

Ion beam analysis of single crystal CVD diamond.
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Croatia; <sup>3</sup>Mechanical Engineering, University of Roma II, Roma, Italy.

Frontal IBIC ( Ion Beam Induced Current ) measurements have been carried out on a new single crystal epitaxial CVD diamond deposited over a HPHT diamond substrate and equipped with circular Al electrode, 2 mm diameter, deposited on both sample surfaces in a sandwich geometry without removing the HPHT substrate. Crystalline and structural quality was characterized by X-ray diffraction, Raman spectroscopy, cathodoluminescence and scanning electron microscopy. Both proton and alpha microbeams of energies 3 and 4.5 MeV were used, with a beam diameter spot of about 1.2 mm for scanned areas from 450x450 um down to 150x150 um and below. The homogeneity of charge collection efficiency (cce) was suitably monitored over these surface area and turned out to be much better than for previous CVD diamond samples. Since cce non-homogeneity is strongly affecting energy resolution in diamond and similar semiconductor materials, for these single crystal samples energy resolutions were very good and ranged from 3.5 down to 1.3 %FWHM depending on the scanned surface area and on the beam type. At voltage bias of 80V, the average cce, as measured across the whole sample thickness (substrate + CVD layer ), almost reached 50 % The values of cce together with homogeneity and energy resolution, which include a also a not negligible electrical noise due to experimental conditions, compare quite well not only with the best values obtained till now on single crystal CVD diamond, but even with Si. In fact, in the same conditions, Si detector reached 0.85% FWHM. The stability and reproducibility of the detector was very good without any preliminary priming. The event rate, which was generally kept at 50 cps or below, was pushed in some cases up to 700 cps by using a 1.2 um spot size of the beam with apparently no cce losses and with only a slight worsening of energy resolution. By the ion microprobe it was also possible to investigate surface defects and the behaviour of

electrodes. Detailed results and indications to further improve CVD diamond performances will be presented and discussed in the paper.

### 005.21

Īon Beam Induced Interface Modification for Relaxed SiGe on SiO<sub>2</sub>. Masanori Ikishima<sup>1</sup>, Ryo Matsuura<sup>1</sup>, Taizoh Sadoh<sup>1</sup>, Masaharu Ninomiya<sup>2</sup>, Masahiko Nakamae<sup>3</sup>, Toyotsugu Enokida<sup>4</sup> and Masanobu Miyao<sup>1</sup>; <sup>1</sup>Electronics, Kyushu University, Fukuoka, Fukuoka, Japan; <sup>2</sup>SUMCO, Noda, Japan; <sup>3</sup>SUMCO, Saga, Japan; <sup>4</sup>Fukuryo Semicon Engineering, Fukuoka, Japan.

The high carrier mobility of strained Si channels in the strained-Si/relaxed-SiGe-on-insulator structures is very useful for the high-performance MOSFET. To realize the next-generation fully depleted devices, the thickness of SiGe-on-insulator (SGOI) should be decreased less than 30nm. To fabricate such an ultrathin SGOI, the Ge condensation method by oxidation of SiGe/Si-on-insulator (SOI) structures has been intensively studied. However, high temperature (1200-1300°C) processing is required to obtain highly stress relaxed SGOI because of stiff bonding at SiGe/buried SiO<sub>2</sub> (BOX) interfaces. The processing temperature should be decreased (<1100°C) to prevent wafer warping. In order to solve these problems, we propose a new method of ion-beam induced interface modification, which is expected to weaken the bonding at SiGe/BOX interface. The quantitative relations between H<sup>+</sup> implantation to top-Si/BOX interfaces and stress relaxation during oxidation are presented in this paper. In the experiment, Si (thickness: 30nm)/Si<sub>0.85</sub>Ge<sub>0.15</sub> (55nm) layers were epitaxially grown on SOI wafers (top Si thickness: 55nm) by CVD. These samples were implanted with H<sup>+</sup> ions (energy: 8.1keV, dose: 0-5x10<sup>16</sup>cm<sup>-2</sup>) and subsequently oxidized at 1100°C. The projected range of H<sup>+</sup> ions matched to the top Si/BOX interfaces. Ge fractions and stress relaxation rates of the formed SGOI were evaluated by Raman and Auger electron spectroscopy. By analyzing the samples after oxidation (120 min), it was indicated that high dose implantation ( $>5x10^{15} \text{ cm}^{-2}$ ) increased the stress relaxation rates from 20% to 50%. However, Ge fractions in the SGOI were also increased by the enhanced oxidation by implantation, i.e., the final Ge fractions deviated from the expectation by the enhanced oxidation, which would make difficult to precise control of the lattice constant of SGOI. In order to eliminate the enhanced oxidation, two-step annealing (500°C for 30min, 850°C for 60min) was performed before oxidation. From analysis of the samples, it was shown that the relaxation rates abruptly increased for doses above  $1 \times 10^{15} \mathrm{cm}^{-2}$ , e.g., from 65% to 90% for samples oxidized for 120min. On the other hand, the Ge fractions were almost a constant value (53%). In order to investigate the mechanism for the stress relaxation by H<sup>+</sup> implantation, cross sectional TEM measurements were performed. No additional defects were observed for the implanted samples after oxidation. Moreover, the SiGe/BOX interfaces were very smooth for the implanted samples, though zigzag for the no-implanted samples. These results suggest that H<sup>+</sup> implantation realizes uniform gliding of SiGe layers on BOX during oxidation. We speculate that the implanted H atoms weaken the Si-O bonds at SiGe/BOX interfaces, and thus, induce the gliding of SiGe layers. These results clearly demonstrate the usefulness of the interface modulation by H<sup>+</sup> implantation for realization of highly stress relaxed ultrathin SGOI at a low temperature ( $\sim 1100^{\circ}$ C).

### OO5.22

Nucleation and Growth of Defects in He irradiated Metals using Rate Theory and Kinetic Monte Carlo Models. Christophe Juan Ortiz<sup>1</sup>, Maria Jose Caturla<sup>1</sup>, Chu Chun Fu<sup>2</sup> and Francois Willaime<sup>2</sup>; <sup>1</sup>Fisica Aplicada, University of Alicante, Alicante, Spain; <sup>2</sup>Service de Recherches de Metallurgie Physique, CEA/Saclay, Gif-sur-Yvette, France.

Presence of impurities such as He in metals under irradiation can significantly affect their mechanical properties. The macroscopic changes observed are ultimately related to the formation of nano-defects, in particular voids, that nucleate and grow during irradiation. Therefore, complex atomistic scenarios must be taken into considerations to understand and predict macroscopic transformations of materials. As the basic atomistic processes involved, i.e. diffusion and agglomeration of atoms, take place over very different scales of time and space, a multi-scale simulation approach is required. In order to achieve such a simulation tool, kinetic Monte Carlo and Rate Theory models can be used as two complementary simulation techniques. The former method accounts for the spatial correlation between defects and can be used to simulate the initial stages of nucleation at an atomic level. The latter method, based on coupled diffusion equations and on a mean-field approximation, is able to follow the evolution of the defect population at a macroscopic scale. In the present investigation, we studied by means of the two simulation methods described above, the nucleation of He-Vacancy clusters in He irradiated metals and their evolution for different temperatures, initial He concentration and sample thickness. The input parameters for the

migration energies and binding energies of different cluster types and sizes used in both models were obtained by ab initio calculations. For each experimental condition considered, we determined the preferential pathway in the formation of HeV clusters, which is essential for the prediction of void swelling and hardening phenomena in materials present in nuclear reactors. In order to validate the models as well as the set of basic input parameters, outdiffusion of He from implanted Fe was simulated and the results were compared to available desorption measurement data found in the literature. Finally, we compare the results of these two simulation approaches and extract some conclusions regarding the limitations of each model.

### 005.23

Fine Patterning of Oriented Nickel NanoCrystals using a Focused Ion Beam. Chiaki Nakajima, Timothy P. Halford and Yakichi Higo; Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Japan.

Oriented nickel Nanocrystals (NCs) have been successfully fabricated from a Ni-11.3wt%P amorphous alloy using Focused Ion Beam (FIB) irradiation at room temperature. Transmission Electron Microscopy (TEM) examination of the irradiated plane has revealed the formation of NCs throughout the irradiation area. Changing the alloy irradiation angle can enable or prevent the precipitation of oriented NCs. A two stage method has therefore been developed for forming patterned NCs. In this case the material is irradiated with gallium ions at an angle of 00 to the plane surface to reduce the specimen thickness to less than 100nm, whilst evenly forming NCs on the remaining exposed surface. The second stage of this process is then to change the irradiation angle to 900 from the plane surface and partially irradiate the desired areas in order to erase NCs from those selected areas. TEM shows that this technique successfully generates patterned Face Centered Cubic (FCC) Nickel NCs of diameter 5-10nm, with {111 parallel to the irradiated plane and [110] parallel to the projected ion beam direction. This is however an indirect fabrication method, requiring a significant time and ion irradiation. For these reasons development has begun upon a single stage method of NC patterning. This single stage method, although requiring an initial materials thickness of less than 100nm as a result of electro polishing, allows the patterning of NCs in only the selected areas. Specimens were pre-annealed at a temperature below that required for crystallization (180oC/30mins) in order to aid NC formation. The NCs were then formed in the irradiation area using FIB at a potential of 40kV and an ion beam incident angle of 350. NCs formed in this method have a diameter of about 5nm and the same orientation as that produced by the two stage method. Fine NCs have been produced by the two stage method, although it seems inefficient. In comparison, the single stage method is more efficient although the NCs produced are larger in size, with the larger irradiation angle making then less easy to crystallize. At an irradiation angle of more than 600, NCs have not been formed. The best method for oriented NC patterning is therefore to irradiate selected areas of annealed specimens at low angles using FIB (single stage method).

### 005.24

Nanostructure, Tribological Performance and Wear Resistance for Ti+C Dual Implanted Steel. Zhang Tonghe, Wu Yuguang and Liu Andong; Key Laboratory for Radiation Beam Technology and Material Modification, Institute of Low Energy Nuclear Physics, Beijing Normal University, Beijing, China.

Very good wear protective layer of H13 steel have been obtained using Ti+C dual implantation. The improvement wear protective properties of steel is explored. Several factors of influence on the steel strengths for Ti+C dual implantation are studied. The properties of wear resistance of the dual implanted steel are improved noticeably. The friction coefficent is reduced from 0.85 to 0.22; The surface hardness increased by 70%; the wear resistance increased by 2.5 times. Nanostructure is observed by TEM, The microstructure change is very important for wear resistance improvemet. Before ion implantation, long grains with narrow boundaries in martensitic steel were identified by TEM. The grains were refined, and dispersed phases with nano-meter size were distributed in the Ti-implanted layer. Electron diffraction analysis shows that the new phases are FeTi compounds. It is found that the nano-phases are formed and dispersed in the implanted layer. The size of the nano-phases ranges is from 10 to 30nm. If a large ion flux of  $50\mu\text{A/cm}^2$  was used for the implantation, the amount and size of the nano-phases are increased. More new nano-phases appear, such as Fe<sub>2</sub>Ti, TiC. The microstructure of Ti+C implanted H13 steel is very complex. The TEM images of cross section of samples shown that many fine circular columnar structures with diameter of 10-30nm are distributed in the dual-implanted layer. The new phases were identified by electron diffraction. The new phases are Fe<sub>2</sub>Ti, FeTi, TiC, FeC and Fe<sub>3</sub>C. The modified thickness of the implanted sample is obviously greater than the corresponding ion range. The formation of the new phases, with the appearance of compact structure, refined grains, low friction

coefficient are greatly improved the wear resistance properties.

### OO5.25

Monte Carlo simulation on the production and recombination of lattice defect in MgB2 by proton beam irradiation damage. Nianhua Peng, Christopher Jeynes and Roger Webb; University of Surrey, Guildford, United Kingdom.

Localised irradiation damage in YBCO thin films has been used successfully in Josephson junction fabrications using masked proton beam at 50 keV. Similar technology has been applied to Josephson junction fabrications in MgB2 superconducting thin films using proton beams with energies between 30 to 50 keV, though the experiments reveal a strong dependence of the defect production upon detailed irradiation conditions. An interesting time effect has also been observed in MgB2 crystals irradiated with a 1 MeV proton beam. We simulate the creation and recombination of lattice defects in MgB2 by proton beam irradiation process using CASCADE code, and compare them with experimental data.

### 005.26

High performance ultraviolet-detecting ZnO thin-film transistors isolated using energetic B ions. Kimoon Lee, Hee Sun Bae, Jae Hoon Kim, Seongil Im and Minsuk Oh; Institute of Physics and Applied Physics, Yonsei University, Seoul, South Korea.

Until recent years, ZnO-based thin-film transistors (TFTs) have been quite extensively studied, drawing much attention from researchers. It is because these new TFTs may achieve the following important goals: obtaining a high mobility channel layer on a flexible substrate through low temperature processes, realizing transparent TFTs, and achieving extra-functions such as photo-detection using the ZnO channel. Moreover, since ZnO deposited even at room temperature usually maintains a crystalline phase, ZnO-based TFTs often exceed a conventional amorphous Si TFT in terms of field effect mobility. For compound semiconductor devices, ion implantation for electrical isolation may be an effective and reliable technology because a conductive region can be made highly resistive through the trapping of free carriers by ion-beam-induced (IBI) deep level defects. Although device isolation has been conventionally achieved by oxidation or etching, the IBI isolation technique is sometimes preferred due to its simplicity, precise depth control, and compatibility with planar technology. It is an efficient and practical way to isolate the region between neighboring devices using energetic ions. The implantation for isolation has currently been applied to II-VI compound semiconductors including ZnO, but an isolation study on ZnO-based devices is still very rare. In the present study, we report on the fabrication of ultra-violet (UV) detecting ZnO-based thin-film transistors (TFT) with the active ZnO layer isolated by energetic B ions. After deposition on a SiO2/p<sup>+</sup>-Si substrate at 300 °C by rf sputtering, the ZnO layer was patterned with Al source/drain (S/D) contacts and a SiOx window. Then energetic B ions with 30 and 55keV were implanted onto the deposited structures for device isolation. Among the three samples of unimplanted,  $30~\rm keV$ , and  $55~\rm keV$ -implanted devices, the  $55~\rm keV$ -implanted one displayed the least gate current leakage ( $\sim$ 40 pA). The ZnO-TFT isolated with 55 keV B also showed a high field mobility of 1.34 cm<sup>2</sup>/Vs and decent on/off current ratio of more than  $\sim$ 10<sup>4</sup>, respectively. Under 364 nm UV light of 0.2 mW/cm2 and at zero volt of gate bias, the device exhibited a high photo-to-dark current ratio of  $\sim 10^3$  with a temporal response of

## 005.27

Ion-Induced Surface Modification in Synergy with Heat and Light of EUV and VUV Plasma-Facing Collector Mirrors.

Jean Paul Allain<sup>1</sup>, Ahmed Hassanein<sup>1</sup>, Martin Nieto<sup>1</sup>, Vladimir Titov<sup>1</sup>, Matthew Hendricks<sup>1</sup>, Perry Plotkin<sup>1</sup>, William Klimowych<sup>1</sup>, Chris Chrobak<sup>2,1</sup>, Edward Hinson<sup>3,1</sup>, Brent Heuser<sup>4</sup>, Robert Bristol<sup>5</sup> and Bryan Rice<sup>5</sup>; <sup>1</sup>Energy Technology Division, Argonne National Laboratory, Argonne, Illinois; <sup>2</sup>University of Wisconsin, Madison, Wisconsin; <sup>3</sup>Middlebury College, Middlebury, Vermont; <sup>4</sup>University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>5</sup>Intel Corporation, Hillsboro, Oregon.

Plasma-facing mirrors used to collect light in the EUV and VUV spectral bands must contend with an environment that can be detrimental to their performance and lifetime. Main mechanisms leading to their failure rate are ion-induced structural modification, sputtering, and impurity implantation. In extreme ultraviolet lithography (EUVL) environments plasma/surface interactions are important due to applications of plasmas to generate EUV light. Both laser produced plasma (LPP) and gas discharge produced plasma (GDPP) configurations face serious issues of components lifetime and performance under particle bombardment. For both configurations debris material, fast ions and neutrals, and condensable alternate EUV radiator fuels (Li, Sn) can irradiate collector optical surfaces. One critical challenge is to assess ion-induced damage in synergy with

off-band radiation heating, EUV radiator (Li, Sn) contamination, and background Ar plasma interaction on morphological and phase formation on surface evolution of the thin-film collector mirrors. In GDPP devices single thin-film (Ru or Pd) layers of 10-50 nm are used at glancing photon angles, whilst for LPP devices, multi-layer thin-film (Mo/Si) mirrors with d-spacing of 7.3-nm and capped with assorted 1-3-nm transition metal thin-films. Ion-induced damage and surface modification is also critical for multi-layer thin-film mirrors used in space telescope applications. Debris and radiation exposure also limit the lifetime and performance of these mirrors. The IMPACT (Interaction of Materials with charged Particles and Components Testing) experiment at Argonne National Laboratory studies ion-induced mechanisms that modify the behavior of optical mirror heterogeneous surfaces. IMPACT has up to five different ion sources at one time with assorted in-situ metrology. Irradiation conditions include: incident particle energies between 100 eV to 5 keV for He, Li, Xe or Sn singly-charged ions at oblique and normal incidence with varying sample temperatures up to 500 C. Ion bombardment is done in synergy with EUV or VUV light exposure. Low-energy (10 to 100 eV) Ar-ion bombardment is conducted simultaneously with Sn-ion irradiation on both single and multi-layer mirrors. Incident fluxes range from 1011-1014 ions/cm2/s and sample temperature ranges from 25 to 400 C. Measurement's conducted include: in-situ quartz crystal microbalance, in-situ surface analysis: auger spectroscopy and low-energy ion scattering spectroscopy, X-ray reflectivity, X-ray diffraction and atomic force microscopy.

> SESSION OO6: Swift Heavy and Light Ions II: Fundamentals and Applications Chair: Eduardo Bringa Tuesday Morning, November 29, 2005 Commonwealth (Sheraton)

### 8:30 AM \*OO6.1

High Energy Focused Ion Beam Nanoprobes: Design and Applications. Gary A. Glass, Bibhudutta Rout, Alexander D. Dymnikov, Richard R. Greco and Daniel P. Zachry; Louisiana Accerlerator Center, University of Louisiana, Lafayette, Louisiana.

An overview of the present state of high energy focused ion beam (HEFIB) system technology, nanoprobe system design and specific ion beam writing applications will be presented. In particular, the combination of P-beam, heavy-ion writing and ion implantation to produce microstructures in resists and silicon will be demonstrated. Heretofore, the development of HEFIB technology worldwide has progressed through a series of developments at independent research facilities, each having relatively narrow and mostly isolated, research purposes. However, a complete, versatile HEFIB nanoprobe system capable of both analysis and modification will require the combination of several component systems, each with specialized technology, and significant advances in the design of a complete system can only be expected from an effort that includes a coordinated development of the component parts.

# $9:00 \text{ AM } \underline{\text{OO6.2}}$

Relaxation of deformations produced by single ion impacts on glassy polymer surfaces. Ricardo M. Papaleo, Rafael Leal and Willyan Hasenkamp; Faculty of Physics, Catholic University of Rio Grande do Sul, Porto Alegre - RS, RS, Brazil.

Energetic heavy ions impacting a surface often produce at the site of penetration a crater and/or a raised region few nanometers in size, the so-called surface tracks. Our group has been systematically investigating surface tracks on polymers, aiming to establish a connection between the morphology of the impact features and materials properties, such as molecular weight, viscosity or the glass transition temperature. In this work, we present recent data on relaxation phenomena of single ion impacts on glassy PMMA surfaces. The viscoelastic nature of the mechanical behavior of such materials allows one to follow the relaxation dynamics of the nanometer-sized deformations induced by the ions. The experiments consist of  $\,$ bombarding the surfaces at a given temperature (from 55 to 100 C), which are allowed to relax in situ for a predetermined time, before quenching to room temperature, freezing the relaxation processes. For each temperature a set of samples are produced encompassing a suitable range of annealing times. The size of the surface tracks are then measured ex-situ by SFM, in order to obtain the temporal evolution of the (average) dimensions of the deformations or craters. The relaxation behaviour of the average height, length or volume of the deformations is well described by a stretched exponential function, with relaxation times  $\tau(T)$  several orders of magnitude smaller than typical values found for bulk PMMA at the same T. Extracted values of  $\tau$  varied five orders of magnitude (from few seconds to several hours) in the narrow temperature range of the experiments. Whilst at T close to or higher than the local Tg the average relaxation times

follows the Vogel-Fulcher-Tammann type of temperature dependence, at the low T regime a transition towards an Arrhenius behavior is seen. The crater walls relax much more slowly, because of the severe chemical modification of the chains in the core of the impact. Moreover, different relaxation rates were found for distinct parts of the hillocks, demonstrating the spatially inhomogeneous chemical nature of the protrusions induced by the ion impacts on the polymer. To our knowledge, these are the first measurements of relaxation rates of a surface track on a solid.

### 9:15 AM OO6.3

Quasi One-Dimensional Electrical Conductors Created by Swift Heavy Ion Tracks in Tetrahedral Amorphous Carbon Films. Hans C. Hofsaess<sup>1</sup>, Anne Katrin Nix<sup>1</sup>, Daniel Schwen<sup>1</sup>, Henrdik Zollondz<sup>1</sup>, Carsten Ronning<sup>1</sup>, Johann Krauser<sup>2</sup> and Christina Trautmann<sup>3</sup>; <sup>1</sup>2nd Institute of Physics, University Goettingen, Goettingen, Germany; <sup>2</sup>Department of Automatisation and Informatics, Hochschule Harz, Werningerode, Germany; <sup>3</sup>Gesellschaft fuer Schwerionenforschung mbH, Darmstadt, Germany.

Thin films of tetrahedral amorphous carbon (ta-C) were grown on low resistivity n-type Si substrates by ion beam deposition of 100 eV  $^{12}\mathrm{C}$ ions at room temperature. The films with several hundred nm thickness exhibit an sp<sup>3</sup>-content in excess of 80 % a density of about 3 g/cm<sup>3</sup> and a high room temperature resistivity up to  $10^{10}~\Omega cm$ . The films were irradiated with swift heavy ions (e.g. 350 MeV Au or 1  $\,$ GeV U ions) under normal incidence with fluences between  $10^9$  and  $10^{11}$  ion/cm<sup>2</sup>. The enormous uniform electronic energy loss along the ion track of about 28 keV/nm causes a conversion of the dense and high resistivity  ${\rm sp^3}$ -phase into a conducting graphite-like  ${\rm sp^2}$ -phase within a cylinder of about 10 nm diameter, thus creating nanometer-sized conducting filaments within the ta-C matrix. For individual conducting channels the room temperature I-V curves were measured by contact-mode atomic force microscopy using conducting metal-coated tips. Temperature dependent I-V measurements down to 10 K were done at ensembles of ion tracks connected through evaporated Au contacts. Frenkel-Poole conduction is the dominating transport mechanism at room temperature. With decreasing temperature, a further weakly temperature dependent transport process becomes apparent, most probably explained by variable range hopping. In addition, below about 100 K the Si/ta-C interface forms a weak Schottky-type barrier. We will discuss the temperature dependence of the I-V curves in order to identify possible one-dimensional transport processes. We also present first results on temperature dependent I-V curves at individual conducting ion tracks, contacted by electro-deposition of Cu through aligned pores in a polycarbonate film, which was spin-coated prior to ion irradiation.

### 9:30 AM <u>OO6.4</u>

Characterization of Swift Heavy Ion Tracks in CaF2 by Scanning Force. Marcel Toulemonde<sup>1</sup>, Nassima Khalfaoui<sup>1</sup>, Cristina Rotaru<sup>1</sup>, Jean Paul Stoquert<sup>2</sup>, Serge Bouffard<sup>1</sup>, Florent Haas<sup>3</sup> and Christina Trautmann<sup>4</sup>; <sup>1</sup>CIRIL, Caen, France; <sup>2</sup>INESS, Strasbourg, France; <sup>3</sup>IRES, Strasbourg, France; <sup>4</sup>GSI/MF, Darmstadt, Germany.

Single crystals of CaF2 were exposed to various high energy heavy ions, from Ca to U of energy 1 - 11.1 MeV per nucleon covering a large range of electronic stopping powers Se between 4.6 and  $35.5\,$ keV/nm. The irradiated surface was investigated by means of scanning force microscopy in the tapping mode. Nanometric hillocks produced by the ion projectiles were analyzed in terms of creation efficiency, diameter and height values, and diameter-height correlation. Hillock formation appears with a low efficiency above a stopping power threshold of  $\sim$ 5 keV/nm, with a constant height of  $\sim$ 1 nm for Se between 5 and 10 keV/nm. Above 10 keV/nm, the mean height increases linearly with Se, reaching 12.5 nm at Se= 35.5keV/nm. Similarly the efficiency becomes larger, reaching 100% for Se >15 keV/nm, i.e. each projectile produced an individual hillock. In this last regime a strong correlation appears between the height and diameter, allowing an internal determination of the tip size curvature. The diameter of the hillocks was deduced by graphical deconvolution of the scanning-tip curvature. In the entire Se regime, the mean diameter exhibits a constant value of  $\sim 13$  nm. Comparison with others experiments and with theory will be presented.

SESSION OO7: Metallic Nanoparticles in SiO<sub>2</sub> and Other Insulators
Chair: Joerg K.N. Lindner
Tuesday Morning, November 29, 2005
Commonwealth (Sheraton)

### 10:15 AM \*OO7.1

Ion Beam Shaping of Nanometals. Arjen M. Vredenberg<sup>1</sup>, A. Polman<sup>2</sup>, B. J. Kooi<sup>3</sup>, T. Van Dillen<sup>3</sup>, K.-H. Heinig<sup>4</sup> and M. Toulemonde<sup>5</sup>; <sup>1</sup>Utrecht University, Utrecht, Netherlands; <sup>2</sup>FOM-AMOLF, Amsterdam, Netherlands; <sup>3</sup>Groningen University,

Groningen, Netherlands;  $^4{\rm Forschungszentrum}$ Rossendorf, Rossendorf, Germany;  $^5{\rm CIRIL},$  Caen, France.

We present a novel type of ion-beam induced deformation of metal nano-objects. Under heavy ion irradiation Au nanosheres in a silica matrix first elongate, and at higher doses combine into nanowires that continue to grow under the ion beam. Such anisotropically shaped metal nanoparticles may have great potential in a wide range of fields. For example, nanorods exhibit a split plasmon resonance, with one of the bands shifting as far as the infrared. Arrays of such particles have great potential as nanophotonic guides in the (infra)red, an important telecom wavelength regime, but outside the range of plasmon resonances of spherical particles. Our samples consist of Au spheres (15 nm) in a single plane 150 nm below the surface of a SiO<sub>2</sub> matrix. At low dose  $(2x10^{14}/\text{cm}^2)$  the nanospheres elongate into nanorods, (/cm<sup>2</sup>) the nanospheres elongate into nanorods, with their long axis oriented in the ion beam direction (also verified by changing the ion incidence angle). At higher doses, nanowires form, still parallel with the ion path. This intriguing effect (the wires must have formed from many primary nanopsheres) will be discussed in detail, along with the elongation mechanism, based on Monte Carlo computer experiments. We also observe a clear threshold in the electronic energy loss. This threshold can be explained, assuming that the ion track has to be continuous for elongation to occur.

### 10:45 AM <u>OO7.2</u>

Ion-Beam-Synthesised Ag-SiO<sub>2</sub> Nanocomposite Layers for Electron Field Emission Devices. W.M. Tsang<sup>1</sup>, V. Stolojan<sup>1</sup>, A. A. D. T. Adikaari<sup>1</sup>, S. R. P. Silva<sup>1</sup> and S. P. Wong<sup>2</sup>; <sup>1</sup>Advanced Technology Institute, University of Surrey, Guildford,GU2 7XH, United Kingdom; <sup>2</sup>Department of Electronic Engineering & Materials Science and Technology Research Centre, The Chinese University of Hong Kong, Hong Kong, China.

The operational speed of solid-state electronic devices is limited by the saturation velocity of electrons ( $\sim 10^5~{\rm ms}^{-1}$ ) in solid due to the lattice scattering. The electron velocity in vacuum can approach the speed of light,  $3 \times 10^8$  ms<sup>-1</sup> [1], therefore, vacuum microelectronic devices are attractive for high-speed and high frequency applications. Using thin film cold electron field emission (FE) cathodes instead of hot filaments, as used in conventional vacuum electronic devices can reduce the size and improve the power efficiency of the device. In this work, Ag-SiO<sub>2</sub> nanocomposite layers were synthesised by Ag implantation on thermally oxidised SiO<sub>2</sub> layers and demonstrated to have excellent FE properties. These nanocomposite layers can give an emission current of 1 nA at electric fields less than 20 V/ $\mu$ m compared to several thousands volts per micrometre of pure metal surfaces. Their fabrication processes are fully compatible with existing integrated circuit technology. By correlating the FE results with other characterisation techniques including atomic force microscopy, Rutherford Backscattering Spectroscopy, transmission electron microscope and X-ray diffraction, it is clearly demonstrated there are two types of field enhancement mechanisms responsible for the excellent FE properties of these layers. The electrically conductive Ag nano-clusters embedded in the insulating  $\mathrm{SiO}_2$  matrix give rise to a local electric field enhancement due to an electrical inhomogeneity effect [2] and the dense surface protrusions provide a geometric local electric enhancement. The FE properties of these layers are critically dependent on the size and distribution of the Ag clusters, which could be controlled by the Ag dose and modified by the post-implanted pulse annealing with a high-power KrF excimer laser. The details of the structure and FE properties of these Ag-SiO<sub>2</sub> nanocomposite layers will be presented and their local field enhancement mechanisms will be discussed. [1] W. Zhu, Vacuum microelectronics, John Wiley & Sons, Inc (2001) [2] W.M. Tsang, S.P. Wong and J.K.N. Lindner, Appl. Phys. Lett. 81, 3942 (2002)

## 11:00 AM <u>\*OO7.3</u>

Controlling the size of nanocavities and nanoparticles in Si and SiO2 using ion irradiation. Jim S. Williams and Jenny Wong-Leung; RSPhysSE, ANU, Canberra, Australian Capital Territory, Australia.

When nanoparticles are generated in a solid by ion beam synthesis and annealing, the size distribution of the nanoparticles is controlled by the thermodynamics of the system. As a result, processes such as diffusion and Ostwald ripening can lead to unacceptably large particles and broad size distributions. In this study, we have employed a different approach whereby we first form nanocavities of controlled size in Si by ion irradiation and then use low temperature processing to fill such voids with high diffusivity metals. In such a manner, the system does not achieve thermodynamic equilibrium and it is possible to control the precipitate size distribution. We use two methods for forming nanocavities in Si. The first involves implanting Si into Si at elevated temperature to form a band of voids (up to 5nm in diameter) at depths up to half of the Si ion range. The second method involves H implantation to high dose followed by annealing to form cavities up to 30nm in diameter at depths centered around the H-ion range. Such

voids can then be shrunk to a desired size by subsequent Si ion irradiation under appropriate conditions that can either involve the formation of amorphous layers or the injection of Si interstitials into the cavity band. Nanoparticles are formed in Si by implanting a high concentration of a fast diffusing metallic species into Si (eg Au, Cu, Pt, Zn) and annealing at low temperatures to diffuse the metals to the cavities, where they precipitate to form either a metallic nanoparticle or metal silicide whose size is determined by the original nanocavity size. Such nanoparticles can be formed in SiO2 by initially starting with a buried oxide layer in Si and subsequently oxidizing the surface after the nanoparticles have been formed. We employ an ion-beam-induced process for oxidation so that low process temperatures can be used. Rutherford backscattering and transmission electron microscopy have been used to analyse the size, composition, depth distribution and structure of these nanostructures in Si and SiO2. Our results show that we can obtain a narrow band of nanoparticles with a narrow size distribution at a precise depth in SiO2 by the methods described above.

### 11:30 AM OO7.4

Magnetic anisotropy of strained cobalt nanoparticles.
Jung-Kun Lee, Michael Hundley, Joe Thompson and Michael Nastasi;
Materials Science and Technology, Los Alamos National Laboratory,
Los Alamos, New Mexico.

Recently, increasing interest has been placed on magnetism of nanoparticles because of their technological applications for future ultrahigh density magnetic data storage media. Ion implantation is a suitable method for synthesizing nanocrystals embedded in the matrix. Implantation of Co ions into dielectric matrix followed by thermal annealing leads to the composite material having uniformly dispersed magnetic nanocrystals. Up to now, most work to synthesize Co nanocrystals embedded matrix has used the amorphous SiO2 as a matrix material and nanocrystals do not have the crystallographic correlation with the matrix. Hence, the effect of epitaxial growth related strain between nanocrystals and matrix has been ignored. In this study, a combination of ion implantation and subsequent annealing is employed to synthesize cobalt based nanoparticles which are embedded in crystalline Al2O3 matrix. Magnetic characterizations are carried out by using a vibrating sample magnetometer (VSM) and a superconducting quantum interference device (SQUID) magnetometer. Compared to nanoparticles in amorphous SiO2 substrate, those in crystalline Al2O3 substrate show the stronger anisotropy of Curie constant and susceptibility. With the aid of transmission electron microscopy (TEM) and x-ray photoemission spectroscopy (XPS), cobalt based nanoparticles are found to grow semi-epitaxially in crystalline Al2O3 matrix. The semi-epitaxial growth of nanoparticles through lattice mismatch between nanoparticles and matrix is suggested to produce unique magnetic properties of cobalt based nanoparticles in Al2O3 matrix.

### 11:45 AM <u>OO7.5</u>

Mechanisms of Ion Beam Shaping of Metal Nanoparticles. Karl-Heinz Heinig, Institute of Ion Beam Physics and Materials Research, Forschungszentrum Rossendorf eV, Dresden, Germany.

The functionality of nanoparticles can be extended further by shape anisotropy. Thus, for future hard disks, rod-like nanomagnets are more resistant against thermally activated spin flipping than spheres, and, for photonics, light is guided as surface plasmon-polariton along a chain of rods with less damping than along a chain of spheres Recently it has been shown [1] that Au nanospheres embedded in SiO<sub>2</sub> can be shaped into rods (and even wires) by swift heavy ion irradiation. The underlying mechanisms are largely unknown. Van Dillen has proven [2] that the Trinkaus model [3], which describes successfully the ion beam shaping of dielectrics/semiconductors, can not be applied to ion beam shaping of metal nanoparticles. Here, a consistent mechanism of ion beam shaping and nanowire ripening will be presented. Using the temperature-time profiles of ion tracks in SiO<sub>2</sub> as delivered by Toulemonde [4], atomistic computer experiments performed with kinetic Monte-Carlo and Molecular Dynamics codes reproduce the experimental results [5]. Our comprehensive numerical studies facilitate a further optimisation of ion beam shaping. [1] A. Vredenberg et al., Int. Conf. "Ion Beam Modification of Materials" Monterey (USA), Sept. 5-10, 2004. [2] T. van Dillen, Int. Workshop on "Ion Beam Shaping", Amsterdam (Netherlands), Dec. 17, 2004. [3] H. Trinkaus, J. Nucl. Mater. 223, 196 (1995). [4] M. Toulemonde, Nucl. Instr. and Methods B66/67, 903 (2000), and private comm. [5] K.-H. Heinig, Int. Workshop on "Ion Beam Shaping", Amsterdam (Netherlands), Dec. 17, 2004.

SESSION OO8: Semiconducting Nanoparticles in SiO<sub>2</sub> and Other Insulators
Chair: James S. Williams
Tuesday Afternoon, November 29, 2005
Commonwealth (Sheraton)

### 1:30 PM OO8.1

ZnO Nanoparticles on SiO<sub>2</sub> Fabricated by Ion Implantation Combined with Thermal Oxidation. Hiroshi Amekura<sup>1</sup>, Oleg A. Plaksin<sup>1</sup>, Naoki Umeda<sup>1</sup>, Yoshihiko Takeda<sup>1</sup>, Naoki Kishimoto<sup>1</sup> and Christoph Buchal<sup>2</sup>; <sup>1</sup>Nanomaterials Lab., National Institute for Materials Science, Tsukuba, Japan; <sup>2</sup>Institut fuer Schichten und Grenzflaechen (ISG1-IT), Forschungszentrum Juelich GmbH, Juelich, Germany.

Metal-oxide nanoparticles (NPs) draw much attention because of their novel optical, magnetic and catalytic properties. Up to now, we have succeeded in fabrication of metal-oxide NPs, such as NiO, CuO and Cu<sub>2</sub>O in silica glass (SiO<sub>2</sub>) using metal-ion implantation and following thermal oxidation. In this paper, the method is applied to the fabrication of ZnO NPs in SiO<sub>2</sub>. First, Zn-metal NPs were formed in SiO<sub>2</sub> by implantation of Zn<sup>+</sup> ions of 60 keV to  $1.0 \times 10^{17}$  ions/cm<sup>2</sup>. Subsequently the samples were annealed in oxygen gas to oxidize the Zn NPs to ZnO NPs in SiO<sub>2</sub>. While the samples show brownish color due to Zn NPs in as-implanted state, the color disappears and a distinct absorption edge appears at  $\sim 3.3$  eV after the oxidation at 700°C for 1 hour. Grazing incident X-ray diffraction (GXRD) also confirms the formation of Zn-metal NPs in as-implanted state and the transformation to ZnO around 700°C. Cross-sectional transmission electron microscopy (XTEM) shows smaller ZnO NPs (diameter  $\sim 10$ nm) around the projectile range and larger NPs (diameter of  ${\sim}20~\text{nm}$ or larger) on the surface. Formation of NPs on the sample surface was also confirmed by atomic force microscopy (AFM). To understand the formation processes of this novel arrangement of ZnO NPs, the transformation processes from Zn NPs to ZnO NPs due to oxygen annealing were studied by optical absorption spectroscopy, Rutherford backscattering spectrometry (RBS), sputter depth profiling by X-ray photoelectron spectroscopy (XPS) and XTEM. To separate pure thermal annealing effects, some samples were annealed in vacuum under the same conditions except annealing atmosphere, and are compared with the samples annealed in oxygen gas. It should be noted that our samples fabricated in an optimum condition show defect-free exciton photoluminescence under He-Cd laser excitation at room temperature.

### 1:45 PM OO8.2

Photoluminescence Spectrum of Si-Nc Produced by Si Ion Implantation to Very High Concentration: Influence of Grain Size and Defects Inside the Nanocrystals. Guy G. Ross<sup>1</sup>, David Barba<sup>1</sup>, Martin Chicoine<sup>2</sup>, Chabha Dahmoune<sup>1</sup>, Viara Levitcharsky<sup>1</sup>, Francois Martin<sup>1</sup>, Robert G. Saint-Jacques<sup>1</sup>, Francois Schiettekatte<sup>2</sup>, Riadh Smirani<sup>1</sup> and Yiqian Q. Wang<sup>1</sup>; <sup>1</sup>INRS-EMT, Varennes, Quebec, Canada; <sup>2</sup>Laboratoire Rene J.A. Levesque, Universite de Montreal, Montreal, Quebec, Canada.

Silicon nanocrystals (Si-nc) were produced by the implantation of Si+ in excess into amorphous fused silica and SiO2 film thermally grown to different thicknesses on an Si substrate. The Si+ implantation was followed by annealing (1100 C) and passivation (500 C) in a forming gas (95% N2 and 5% H2). The intensity of the photoluminescence (PL) signal after excitation by means of an Ar laser beam increases with the Si concentration until an optimum concentration, CSiopt is reached (Si/SiO2  $\sim 15\%$ ). Further increase of the Si concentration leads to a strong decrease of the luminescent signal. In order to clarify the cause of this lower signal, we have investigated samples with a Si concentration three times higher than CSiopt using high-resolution transmission electron microscopy (HRTEM), x-ray photoelectron spectroscopy (XPS), and ellipsometry. It was found by TEM that the average diameter of the Si nc ranges from 2 to 22 nm in the sample with high Si concentration. The size of the Si nc in the middle region of the implanted layer are bigger than those near the surface or the bottom of the layer. For Si-nc with diameters larger than 6 nm, twinning structures have been observed in  $\sim 90\%$  of them as well as stacking faults and faceting in some nanoparticles. Most of the Si-nc (90%) larger than 10 nm are formed by the coalescence of smaller ones. Two kinds of coalescence, one being the preferential attachments of small particles to the {111} facets of a seed nanoparticle, and the other being an ordered combination of two or more small nanocrystals, have been observed. Characterization by TEM shows that a large quantity of oxygen was depleted from the first  $\sim 25$  nm in this sample and most of the SiO2 bonds have been replaced by Si-O bonds. All these microstructural defects have a great influence on the optical properties and play an important role in the light emission from the Si-nc. In samples with SiO2 films, the PL spectra are modulated by Fabry-Perot type interference of the emitted light reflected at the Si/SiO2 interface with that propagating directly towards the surface. We have investigated the PL spectral modulation as a function of the incidence angle of the pump laser and found that they are much less modulated by the interference effect than samples implanted to lower Si concentration. The measurement by ellipsometry of the depth distribution of the refractive index (n, k) has revealed a large increase of both at the depth of the Si-nc layer that would act as a light barrier blocking both the incoming pump laser and the Si-nc emitted

light. Both, the defect observed in the Si-nc and the effect of Si ions on the SiO2 layer surrounding the Si-nc can promote non radiative recombination and cause the strong decrease of the PL signal.

## $2:00 \text{ PM } \underline{\text{OO}8.3}$

Kinetic Monte Carlo and Rate Equations: A Comparison in a System of Nucleation and Growth of Nanocrystals.

Diana O. Yi<sup>1,2</sup>, Ian D. Sharp<sup>2,3</sup>, Qing Xu<sup>2,3</sup>, Liao Y. Chris<sup>2,3</sup>, Joel W. Ager<sup>2</sup>, Jeffrey W. Beeman<sup>2</sup>, Kin M. Yu<sup>2</sup>, Eugene E. Haller<sup>2,3</sup> and Daryl D. Chrzan<sup>2,3</sup>; Applied Science & Technology, University of California, Berkeley, Berkeley, California; Materials Science Division, Lawerence Berkeley National Lab, Berkeley, California; Materials Science & Engineering, University of California, Berkeley, Berkeley, California.

Implantation of atomic species into a bulk substrate and subsequent thermal annealing leads to nucleation and growth of nanometer sized crystals. The standard method to model this system uses the Kinetic Monte Carlo (KMC) algorithm that describes the time-dependent growth of particles as well as their size distribution. KMC is a stochastic method that atomistically simulates experiment. Each KMC event (diffusion, nucleation, or dissolution) occurs according to its rate of transition that, in turn, evolves the system in time. Alternatively, one can also describe this system using a coupled set of differential equations (rate equations) that describe the growth of cluster densities by monomer attachment and the coarsening of clusters via detachment kinetics. This approach has been used in 2-D and has been shown to describe well two dimensional epitaxial growth systems under constant deposition flux (Bales & Chrzan, PRB 50 6057 (1994), Bales & Zangwill, PRB 55 R1973 (1997)). Here, we show that rate theory can describe a three dimensional system by comparing rate theory results with those of KMC. Comparing the rate equation model to KMC simulations for the nucleation, growth and coarsening stages we find good agreement in the average cluster size, cluster and monomer densities and size distributions. However, the specific advantage of the rate equation approach is the ability to describe our system with a set of analytic expressions that encompass all the physics in a continuum environment and are easily solvable with a numerical integrator. KMC, on the other hand, relies on events occurring at the atomic level, is constrained to a finite size volume with a finite number of atoms and, to gain statistical accuracy, needs to be run many times. We used both the rate equation model and KMC simulations to study a system of ion beam synthesized Ge nanocrystals embedded in amorphous silica, thermally processed for one hour under 900øC. This work is supported in part by the Department of Energy under contract No. DE-AC02-05CH11231.

### 2:15 PM OO8.4

The Microstructure and Morphology of Ge Nanoparticles Fabricated Directly by Ion Implantation. Tiecheng  $\mathrm{Lu}^{1,3},$ 

Shaobo Dun<sup>1</sup>, Qiang Hu<sup>1</sup>, Sha Zhu<sup>2</sup> and Lumin Wang<sup>2</sup>; <sup>1</sup>Department of Physics, Sichuan University, Chengdu, China; <sup>2</sup>Department of Nuclear Engineering and Radiological Sciences, University of Michigan, Ann Arbor, Michigan; <sup>3</sup>International Center for Material Physics, Chinese Academy of Sciences, Shenyang, China.

It was reported in the MRS-2003 Fall Meeting that the Ge nanoparticles could be prepared directly by ion implantation at the room temperature. Results of a detailed study on the structure and morphology of these Ge nanoparticles are reported in this paper. The Ge nanoparticles embedded in a SiO<sub>2</sub> film on the top of a Si substrate was prepared by ion implantation with an energy of 40 keV and with the fluences of  $1 \times 10^{16}$ ,  $1 \times 10^{17}$ ,  $5 \times 10^{17}$  and  $1 \times 10^{18}$  cm<sup>-2</sup>, respectively. The microstructure and morphology of Ge nanoparticles were studied by cross-sectional TEM and SEM. The samples were etched in HF for suitable period of time to remove the surface SiO<sub>2</sub> layer above nano-particles in advance for SEM observation. The results show that, as for SEM images, the nano-particles have narrow size-distribution. As observed from TEM images, the depth of the layer that contains nanoparticles is about 4 nm nm below the surface, and the thickness of nanoparticle layer is about 20-30 nm. In sample implanted with the ion fluence of  $1\times10^{17}~{\rm cm}^{-2}$ , the nano-particles are quite uniform in size, about 5 nm. When the fluence increased to  $5\times1017~{\rm cm}^{-2}$ , the size of nanoparticles show a gradient-distribution, i.e. the size of nanoparticles near surface is bigger than that distant from surface. But when the fluence increasing to  $1\times10^{18}~{\rm cm}^{-2}$ , the nano-particles become to uniform in size (about 10 nm) again. Moreover, while some of Ge nano-particles are crystalline, some are amorphous. The ratio of number of nanocrystlline Ge particles versus that of amorphous ones increased with the increasing the implantation fluence, just as the size of the nano-particles. In addition, it seems that there are two kinds of different crystalline phase in nc-Ge. The mechanism responsible for the amorphous to crystalline transition, as well as the morphological control of the Ge nano-particles are discussed based on the experimental results.

SESSION OO9: Structural Modifications II: Defect Accumulation, Amorphization, Strain Engineering, Grain Orientation Control Chair: S.P. Wong Tuesday Afternoon, November 29, 2005 Commonwealth (Sheraton)

### 3:30 PM \*OO9.1

The use of ion implantation for the fabrication of strained silicon on SiO2 for nanoelectronic devices. Siegfried  $\mathrm{Mantl}^1$ ,

Dan Buca<sup>1</sup>, Bernd Hollaender<sup>1</sup>, Helmut Trinkaus<sup>1</sup>, Norbert Hueging<sup>2</sup>, Martina Luysberg<sup>2</sup>, Steffi Lenk<sup>1</sup>, Roger Loo<sup>3</sup>, Caymax Matty<sup>3</sup>, Herbert Schaefer<sup>4</sup>, Manfred Reiche<sup>5</sup> and Ionut Radu<sup>5</sup>; <sup>1</sup>Institut fuer Schichten und Grenzflaechen, Forschungszentrum Juelich and cni-Center of Nanoelectronic Systems for Information Technology, Juelich, Germany; <sup>2</sup>Institut fuer Festkoerperforschung, Forschungszentrum Juelich and cni-Center of Nanoelectronic Systems for Information Technology, 52425 Juelich, Germany; <sup>3</sup>IMEC, B-3001 Leuven, Belgium; <sup>4</sup>Infineon Technologies AG, 81739 Munich, Germany; <sup>5</sup>Max-Planck-Institut fuer Mikrostrukturforschung, 06210 Halle, Germany.

Strained silicon (sSi) is a new high mobility semiconductor material for nanoscaled electronic devices. The performance of Si-MOSFETs can be improved substantially by the implementation of sSi into the channel without further down-scaling the device dimensions. For electrons the mobility can be increased by 100%. In this contribution we will present an overview of the use of ion implantation for the fabrication of sSi on relaxed SiGe buffer layers and thin sSi directly on SiO2 (sSOI). sSOI fabrication involves wafer splitting by hydrogen implantation and wafer bonding. The epitaxial Si/SiGe heterostructures and various thicknesses (100 - 200 nm) and Ge concentrations in the range of 16 to 30 at% Ge were grown by CVD on 150 and 200 mm wafers. We have developed a special process to relax pseudomorphic SiGe layers by the implantation of light ions (e.g. He+) or heavy ions (Si+) and subsequent thermal annealing. Typically, a dose of 7xE15 He+/cm2 is implanted to a depth of about 200 nm below the substrate/SiGe interface. For Si+ implants the dose can be reduced by one order of magnitude. The implanted He forms high pressure bubbles in the Si substrate during annealing, and as consequence, dislocations are generated. The mechanism of the strain relaxation will be revealed by in in-situ TEM observations showing the nucleation and propagation of dislocations. We could show that for ultrathin sSi layers on SiGe (< 8nm) only one growth step is needed [1]. The thin Si cap layer becomes strained during the strain relaxation of the underlying SiGe layer after ion implantation and annealing at 850C. The elastic strain in the layers was measured by Raman spectroscopy and He ion channelling using angular scans to amount to 0.8% corresponding to an effective Ge concentration of 17at%. Using atomic force microscopy (AFM) the rms surface roughness was determined to be 0.5 nm. Furthermore, epitaxial overgrowth of the smooth Si/SiGe buffers at various temperatures with strain adjusted SiGe layers and sSi is investigated to further reduce defect density in the sSi and to obtain sSi layers with thicknesses well above 20 nm. Very thin sSi layers (10 nm) on SOI are ideal for fully depleted nanotransistors but larger thichknesses are needed for partially depleted MOSFETs. The density of threading dislocations was determined by TEM and defect etching and optical interference microscopy to be 1xE6 /cm2. For wafer splitting by H+ implantation the complex Si/SiGe heterostructure requires an adjustment of ion dose and energy. These H+ implantation results will be compared with plasma hydrogenation, a possible new alternative method for wafer splitting [2]. [1] D. Buca et al. Appl.Phys.Lett. 85 (2004) 2499 [2] Peng Chen et al. Appl. Phys. Lett. 85 (2004) 4944

# 4:00 PM <u>OO9.2</u>

Ion irradiation induced stress generation and stress relaxation in thin films. Aurelien Debelle, Anny Michel, Gregory Abadias, Christiane Jaouen, Philippe Guerin, Marc Marteau and Michel Drouet; Laboratoire de Metallurgie Physique, Universite de Poitiers, Futuroscope-Chasseneuil, France.

The physical and mechanical properties of thin films depend strongly on their intrinsic state of stress and recent developments have shown that the stress level influences specific properties, thus understanding the mechanisms of stress generation becomes of prime interest. Developing *insitu* or *exsitu* methods to adjust the stress level of thin films opens new opportunities for particular applications, and ion irradiation, offering a wide range of available energies, appears very promising. Thin films obtained by ion beam sputtering have large in-plane compressive stress, whereas thermal evaporation usually results in moderate tensile stress. As highly energetic particles are involved in sputtering, the origin of the growth stress is linked to the generation of point defects, and the increase in volume due to interstitial defects results in an in-plane compressive stress component. Since point defects are highly metastable, post-deposition ion irradiation at energies of a few hundreds of keV allows swift

relaxation of the growth stress, without significant modification of the microstructure of the thin films. Mo thin films and Mo/Ni multilayers were grown on Si and  $Al_2O_3$  [11-20] substrates at different temperatures. Analysis of X-ray diffraction data allows a full determination of the stress/strain state of the films. The evolution of the stress state in the Mo layers under  $\sim\!300$  keV Ar+ ion irradiation at low fluence ( $<5\mathrm{x}10^{14}~\mathrm{ions/cm}^2$ ) was studied. It was found that the intrinsic growth stress is triaxial, resulting from the superposition of a hydrostatic component linked to point defect-induced volume distortion, and a biaxial component due to constrains imposed by the substrate. Furthermore, when the state of stress is complex becaus epitaxial and/or thermal stresses have been superimposed on the growth stress, a separation of the different types of stress occurs naturally under ion irradiation since relaxation mechanisms and kinetics are very different; the growth stress is relaxed at low fluence, while releasing other stress components requires higher doses. In the case of thermal evaporated multilayers, where the growth stress is tensile, ion irradiation results in a mixing at the Mo/Ni interfaces even at low fluence. For a system where the epitaxial biaxial stress components are not equal, this mixing can be identified as a stress relaxation process, since the elastic energy corresponding to the difference in atomic volume of the two elements can be considerably reduced when a solid solution is introduced at the interfaces. In all cases, ion irradiation appears as a powerful tool not simply inducing stress relaxation, but also providing information on the mechanisms concerning stress generation and stress relaxation in thin films.

### 4:15 PM OO9.3

Focused  $\overline{\text{Ion Beam}}$  Induced Stresses and Deflections of Free-standing  $\operatorname{SiN}_x$  Membranes. Young-Rok  $\operatorname{Kim}^2$ , Daniel Branton<sup>2</sup>, Michael J. Aziz<sup>1</sup> and  $\overline{\text{Joost J. Vlassak}}^1$ ; <sup>1</sup>DEAS, Harvard University, Cambridge, Massachusetts; <sup>2</sup>Department of Molecular and Cellular Biology, Harvard University, Cambridge, Massachusetts.

Ion beams are often used for the micromachining, imaging, or surface modification of materials. Along with the desired effect, they sometimes generate large stresses in the irradiated material. Because micromachined systems often require nanometer-scale control of thin-film structures, deformation due to ion beam-induced stresses needs to be well characterized. We report the results of a study in which a focused Ga beam was used to induce stresses in free-standing silicon nitride membranes. Simple structures such as circles, squares and lines were written on the membranes with total irradiation doses varying from  $10^{14}$  to  $10^{17}$  ions/cm<sup>2</sup>. The deflections of the membranes caused by the ion beam were measured as a function of total dose and size of the irradiated area using atomic force microscopy. Both membranes with low and high residual stress were investigated. The deflections of the high-stress membranes are characterized by a flat profile and are relatively independent of the irradiation dose or size of the irradiated area; low-stress membranes, on the other hand, result in gradual profiles that depend sensitively on both dose and area. We propose a simple mechanical model to describe the deflection of the membranes. The model takes into account the membrane stress as well as bending stiffness and successfully predicts both modes of deflection observed in the experiments. Analysis of the experimental deflection profiles makes it possible to determine the stress induced by the ion beam provided the membrane stiffness, residual stress and thickness are known.

### 4:30 PM <u>OO9.4</u>

Local Molecular Structure of Erbium Implanted Silica Glasses. Jincheng Du and L. Rene Corrales; Pacific Northwest National Laboratory, Richland, Washington.

Erbium doped silica and silicate glasses are widely used in photonic devices such as optical amplifiers and lasers. Ion implantation is a very promising method for erbium doping in the fabrication of waveguide amplifiers or lasers because it offers a means to dope erbium ions into glasses in a controlled way. It is well known that the spectral features of erbium (and other rare earth) ions depend on their local coordination environments. Detailed information of how the structure changes during erbium implantation in glasses is still lacking. The experimental structure characterization in glasses is hampered by their inherent disorder. Molecular dynamics (MD) simulation can provide insight of the atomic level structure of glasses. Recently, MD has also been used in the displacement cascade simulations. In this work, we simulate the erbium cascade events up to k-eV range corresponding to the end of implantation process using molecular dynamics method. The changes of the local environment around rare earth ions and the corresponding relaxation of the silicon-oxygen network structure of the matrix glasses are determined. The molecular structures of implanted glasses are compared to the structures of glasses formed by normal melt-quench process, which are used to understand the experimentally observed spectral differences.

SESSION OO10: Ion Beam Analysis I Chair: Christina Trautmann Wednesday Morning, November 30, 2005 Commonwealth (Sheraton)

8:30 AM \*OO10.1 Abstract Withdrawn

9:00 AM OO10.2

Combined RBS/Channeling and HRXRD Study to Access Structural Properties and Lattice Strain of AllnN Epitaxial Layers Grown on GaN. <u>Katharina Lorenz</u><sup>1,2</sup>, N. Franco<sup>1,2</sup>, E. Alves<sup>1,2</sup>, I. M. Watson<sup>3</sup>, E. Nogales<sup>4</sup>, R. W. Martin<sup>4</sup>, K. P. O'Donnell<sup>4</sup> and P. J. M. Smulders<sup>5</sup>; <sup>1</sup>Departamento de Fisica, Instituto Tecnologico e Nuclear, Sacavem, Portugal; <sup>2</sup>CFNUL, Lisbon, Portugal; <sup>3</sup>Institute of Photonics, University of Strathclyde, Glasgow, United Kingdom; <sup>4</sup>Department of Physics, University of Strathclyde, Glasgow, United Kingdom; <sup>5</sup>Materials Science Centre, University of Groningen, Groningen, Netherlands.

During the last decade the group III nitride semiconductors GaN, AlN and InN as well as their ternary alloys InGaN and AlGaN with their wide and direct band gaps have attracted much attention in research and industry due to the wide variety of applications in (opto-) electronic devices, such as light emitters, transistors and sensors Work on the ternary AlInN is still scarce although several possible applications have been proposed. The fact that for a particular InN contents it is possible to grow AlInN lattice matched on GaN, thus reducing the defect production due to lattice strain, makes this material an interesting alternative for applications in cladding layers or Bragg mirrors in GaN-based devices. AlInN layers were grown by MOCVD on GaN buffer layers at different setpoint growth temperatures between 760 and 840 °C. The growth temperature influences the amount of InN introduced into the layer as well as its crystalline quality. Rutherford backscattering spectroscopy in the channeling mode (RBS/C) was used to study the crystalline quality, thickness and In concentration in the layers. Performing angular scans in a non-growth direction also allows the determination of lattice strain by determining the change of the tilt angle  $\Delta\Theta$  in the interface between the AlInN layer and the GaN buffer layer. However, in the case of high quality layers and  $\Delta\Theta$  comparable to or smaller than the critical angle, strong steering effects occur in the interface changing the angular yield in the substrate and making the analysis of angular scans difficult. Monte Carlo simulations were performed to analyze these unusual effects. The comparison with high resolution X-ray diffraction (HRXRD) shows that RBS/C in combination with Monte Carlo simulations gives a good picture of the strain in the layers. Additionally, in contrast to HRXRD, RBS/C gives unambiguous information about the InN content and its spatial distribution. In our samples we could evidence a change from tensile to compressive strain when increasing the InN content by lowering the growth temperature. Although the crystal quality deteriorates for lower growth temperatures, lattice matched AlInN material with InN concentrations around 17 % could be grown with good crystalline quality in a temperature range of 800 - 820 °C.

### 9:15 AM OO10.3

ERDA Characterization of Hydrogen Incorporation and Release in CVD  $Hf_xSi_{1-x}O_2/Si$  Structures.

Mohamed El Bouanani, Vaishali Ukirde, Lim ChangDuck, Jennifer Feng and Manuel Quevedo-Lopez; Materials Science and Engineering, University of North Texas, Denton, Texas.

Hydrogen is of paramount importance in semiconductor technology due to its prevalence in various deposition processes and post processing of electronic structures. Understanding the effects of hydrogen in semiconductors and its behavior such as incorporation, diffusion, trapping and release is of great importance in view of its role in defects passivation as well as degradation of electronic structures. Trap transformations under annealing treatments in hydrogen ambient are known to involve passivation of traps at thermal  $\mathrm{SiO}_2/\mathrm{Si}$  interfaces. Similar behavior is observed for high permitivity dielectrics based structures and is indirectly confirmed by low interfacial state density traps from electrical characterization of Metal Oxide Semiconductor (MOS) structures. However, little or no studies giving direct information on the location and concentration of hydrogen in high permitivity based MOS interface regions and how it is affected by hydrogen annealing treatments is available. Elastic Recoil Detection Analysis data are used to characterize the evolution of hydrogen in the bulk and the interface of  $\mathrm{Hf_xSi_{1-x}O_2/Si}$  structures during annealing in hydrogen ambient in combination with preprocessing in oxygen. Strong correlation between processing conditions and hydrogen trapping are demonstrated. This work was supported by the Texas Advanced Technology Program. Corresponding Author: Bouanani@unt.edu

### 9:30 AM OO10.4

ERD Analysis of T and <sup>3</sup>He in Aging ErT<sub>2</sub> Thin Films. James A. Knapp, James F. Browning, James C. Banks and William R. Wampler; Sandia National Labs, Albuquerque, New Mexico.

The measurement of  ${}^{3}\mathrm{He}$  in thin films containing tritium and other hydrogen isotopes is an important problem, especially for applications involving metal tritides where the buildup of  $^3{\rm He}$  from tritium decay may compromise performance. We have developed a new analysis system for performing measurements of such films with elastic recoil detection (ERD), using a  $\triangle$ E-E detection scheme to separately profile <sup>3</sup>He, tritium, deuterium, and protium as well as O and C. The analysis uses a 36 MeV Si ion beam incident at 10° and two pairs of  $\triangle$ E-E detectors to profile the elements of interest, one pair at 20° for <sup>3</sup>He, T, D, and H, and a second pair at 10° to profile O and C. Measurements are underway of films consisting of 100% T-loaded ErT<sub>2</sub> layers on Si(100) substrates with a thin Mo diffusion barrier, tracking the layer contents as a function of age past loading date. We will present the results of this ongoing study, as well as correlations with parallel measurements of the layer mechanical properties obtained using nanoindentation and finite-element modeling. 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-AC04-94A185000.

> SESSION OO11: Nanomasks & Nanopatterning, Ion Beam Mediated Selforganisation Chair: Barney Doyle Wednesday Morning, November 30, 2005 Commonwealth (Sheraton)

 $\begin{array}{c} 10:15 \ \mathrm{AM} \ \underline{\mathrm{OO11.1}} \\ \mathrm{Ion \ Beam} \ \overline{\mathrm{Induced}} \ \mathrm{Sintering} \ \mathrm{of} \ \mathrm{Colloidal} \ \mathrm{Nanoparticles}. \end{array}$ Joerg K. N. Lindner and Bernd Stritzker; Institute fuer Physik, Universitaet Augsburg, Augsburg, Germany.

Controlled drying of a colloidal suspension of 100 nm diameter silica spheres dispersed in water was used to create densely packed monolayers nanospheres on silicon substrates. Such self-organized monolayers can be used as stencil-type nanomasks, in which the gaps in between three neighbouring nanospheres are employed as mask openings e.g. for the deposition of metal dots on the substrate. In this paper it is shown that irradiation of such masks with light keV ions leads to significant material transport at the contact points of silica spheres, resulting in a sintering-type coalescence of neighbouring spheres. This sintering depends little on the implantation temperature and takes place at temperatures substantially smaller than typical sintering temperatures of SiO2. Evidence will be given that the effect is due to the large surface tensions involved with the small radii of curvature at the contact points of nanospheres. Investigations are in progress to show if the effect can be observed in other nanostructured materials systems as well.

# 10:30 AM <u>OO11.2</u>

Phase Separation and Dynamic Patterning in Cu-Co Films under Ion Irradiation. Pavel Krasnochtchekov, R. S. Averback and P. Bellon; Materials Science and Eng., UIUC, Urbana, Illinois.

It has been shown that ion beam irradiation can strongly affect the microstructure of an alloy system that is immiscible under normal conditions. For this study, Cu-Co alloy was chosen as the model system. Ion irradiations were carried out with Kr+ ions at 1.8 MeV energy. Thin ( $\sim 250$  nm) film samples were prepared by co-sputtering from Co and Cu targets in inert gas atmosphere. Co atomic content in thus prepared films varied from 10 to 20 %. The films were irradiated at different temperatures Ti up to the dose of  $2 \times 10^{16} \text{ cm}^{-2}$ . Exploiting the fact that the minority Co atoms become magnetic upon clustering while the host matrix atoms (Cu) remain non-magnetic, the development of phase separation in irradiated films (estimates of the average size of Co precipitates and concentration of Co in solution) was followed with the use of magnetic measurements. The measurements of magnetization curves were interpreted in the framework of the superparamagnetic theory. The analysis of magnetization data strongly indicates that ion beam irradiations at different temperatures result in three possible outcomes for the microstructure of the irradiated samples: randomizing mixing, macroscopic phase separation (coarsening), and, most interestingly, dynamic patterning, i.e. stabilization of phase separation at some temperature-dependent steady states. The mixing takes place at low temperatures (RT and lower). A nearly complete mixing is revealed from the FC/ZFC (field cooled / zero field cooled) measurements of samples irradiated at room temperature. At higher irradiation temperatures Ti, on the other hand, signs of thermodynamic-like coarsening are observed. Measurements of Cu-Co samples with both

10 and 15 % Co content irradiated at different temperatures consistently place the threshold value for the transition to the coarsening regime at Ti  $\sim$  330-350 C. The novel feature of the impact of ion irradiation on the alloy microstructure, composition patterning, is observed to occur at intermediate temperatures, i.e. between room temperature and Ti = 330 C. This regime is characterized by stabilization of phase separation by the dose of about 5 x  $10^{15}$  cm<sup>-2</sup>. The length scale of phase separation induced by ion beam irradiation is of the order of a few nanometers. Estimates from magnetic measurements for Cu90Co10 alloy yield the size of Co precipitates in steady state at Ti = 270 C of about 9,000 atoms (dia 5.7 nm), and a slightly larger size of 10,000 atoms (dia 5.9 nm) for Cu85Co15 alloy in the same conditions. The experimental results of this study are in qualitative agreement with Monte-Carlo simulations of phase separation in a model immiscible A90B10 alloy system with the heat of mixing similar to that of Cu-Co. The current experimental and simulation work lends support to the theory of driven systems under ion beam irradiation first put forward in the model of Enrique and Bellon (R. A. Enrique and P. Bellon, Phys. Rev. B 60, 14649 (1999)).

### 10:45 AM <u>OO11.3</u>

Size-Dependent Anisotropic Deformation, Capillary Forces and Newtonian Flow during Ion Irradiation. Teun van Dillen 1,2, Patrick Onck<sup>2</sup>, Alfons van Blaaderen<sup>3</sup>, Erik van der Giessen<sup>2</sup> and Albert Polman<sup>1</sup>; <sup>1</sup>Center for Nanophotonics, FOM-Institute AMOLF, Amsterdam, Netherlands; <sup>2</sup>Department of Applied Physics, Groningen University, Groningen, Netherlands; <sup>3</sup>Debye Institute, Utrecht University, Utrecht, Netherlands.

Ion irradiation of free-standing amorphous materials leads to anisotropic plastic deformation at constant volume at a well-defined rate. Previously, we have studied this process in detail for colloidal particles with diameters around 1 micron using ion energies between 300 keV and 30 MeV. Spherical silica colloids expand perpendicular to the ion beam and contract parallel to the ion beam, changing their shape to oblate ellipsoidal, the final anisotropy determined by the electronic energy loss and the ion fluence. In this new work, we study the deformation process in the colloid size regime where capillary forces play a role. Using colloidal silica particles with diameters in the range 38-1000 nm we find that the particle anisotropy after irradiation decreases with decreasing size. For example, for a 4 MeV Xe fluence of  $1 \times 10^{15}~{\rm cm}^{-2}$  the particle size anisotropy (major-over-minor axis) decreases from 1.70 for a 1020-nm diameter colloid to 1.30 for a 92-nm diameter colloid. The size dependence becomes even more pronounced at higher ion fluences. We model this behaviour with a visco-elastic continuum model that takes into account the anisotropic strain generation rate, capillary forces due to the (induced) surface curvature, and radiation-induced Newtonian viscous flow that serves to relax the surface stresses. The latter is induced by atomic displacements. Taking known values for the surface energy, and a measured value for the anisotropic deformation strain rate, we derive from our data a radiation-induced Newtonian viscosity of  $9x10^{21}$  Pa·ion/cm<sup>2</sup>, a typical value for 4 MeV Xe irradiation. These data show that the interplay between anisotropic deformation (an electronic stopping effect) and Newtonian viscous flow (a nuclear stopping effect) leads to size dependent effects at the nanoscale that may have implications for the irradiation of any material with a nanoscale geometry or topology.

### 11:00 AM OO11.4 Formation and characterization of swelled nano-porous

structures of ion-irradiated Ge surfaces. Junichi Yanagisawa<sup>1,2</sup>, Keiji Ogushi<sup>1</sup>, Kentaro Takarabe<sup>1</sup>, Kenji Gamo<sup>1,3</sup> and Yoichi Akasaka<sup>1,2</sup>; <sup>1</sup>Graduate School of Engineering Science, Osaka University, Toyonaka, Osaka, Japan; <sup>2</sup>Research Center for Materials Science at Extreme Conditions, Osaka University, Toyonaka, Osaka,

Japan; <sup>3</sup>Kansai Advanced Research Center, National Institute of Information and Communications Technology, Kobe, Hyogo, Japan.

Although there are several reports on the formation of swelled porous structures with nano-meter size on germanium (Ge) surfaces by high-energy (100 keV - 1.5 MeV) heavy (such as Gé, Kr) ion irradiations, characteristics of such swelled structures, as well as their formation mechanisms, have not been clarified so far. We have also reported that the swelled nano-porous structures on Ge surfaces were formed by 100-200 keV Si, Ga, and Au ion irradiations, but not formed by 15-30 keV Si and Au ion irradiations. In the present paper, composition of the 100 keV Ga irradiated Ge surfaces and the solubility of such nano-porous structures to water were investigated to obtain some information about the formation mechanisms and the characteristics of such structures, respectively. Ga ions were irradiated on (100) or (110) surfaces of single crystal of Ge wafers using a focused Ga ion beam (Ga FIB) system at an energy of 100 keV with doses of  $3x10\hat{1}5 - 3x1017$  /cm2. The atomic composition of Ga-irradiated Ge surfaces was investigated by Auger electron spectroscopy (AES). To investigate the stability of the nano-porous structure against water, the Ga-irradiated Ge samples were dipped in

ultra-pure water up to 10 hours, and the surfaces were observed by a scanning electron microscope (SEM). The swelling was not observed and the surface was etched for Ga irradiations at doses larger than 2x1017 /cm2, and the porous structures were squashed and changed to solid structures. However, atomic composition was not changed, that is to say, no Ga signal was observed in AES spectra for heavily irradiated samples. This indicates that the precipitation of irradiated Ga is not caused the crash of the porous structures. After dipping the samples in ultra-pure water, the nano-porous structures formed by Ga irradiation at doses lower than 2x1017 /cm2 were remained, but for the Ga-irradiated samples at doses larger than 2x1017 /cm2, the surface was partially peeled off and the Ga-irradiated region was partially etched ten times deeper than the projected range of Ga ions in Ge. These results indicate that the formation of both swelled nano-porous structures and deeply etched regions on Ge surfaces is possible by 100 keV Ga FIB irradiation controlling the ion dose, and formed nano-porous structures can be used as a functional surface even in water, such as a nano-filter in micro flow channels on a bio chip and highly contacting surfaces for bio-related materials.

### 11:15 AM OO11.5

Fabrication of pattered domains with graphitic clusters in amorphous carbon using a combination of ion implantation and electron irradiation techniques. Eiji Iwamura<sup>1,3</sup> and

Tatsuhiko Aizawa<sup>2</sup>; <sup>1</sup>New Business Creation Department, R&D Center, Arakawa Chemical Industries, Ltd., Osaka, Japan; <sup>2</sup>Japan office, University of Toronto, Tokyo, Japan; <sup>3</sup>PRESTO, Japan Science and Technology Agency, Kyoto, Japan.

A new form of carbon incorporating graphitic structures in amorphous carbon networks has attracted extensive interests since the hybridized structures are expected to lead to high performances by combining diverse physical properties, which arise from carbon structures. It is reported that carbon hybrid structures were synthesized at relatively low temperature using low-energy electron irradiation. Onion-like and nano-crystalline graphitic structures in amorphous carbon matrix can be fabricated via dynamic structural modification converting from amorphous to ordered structures with a help of catalytic metal dopants. This technique implies that it is possible to make arranged domains consisting of ordered/disordered carbon by controlling the region where catalytic elements are doped. In this study, fabrication of carbon composite structures, which contain patterned domains with graphitic structures in amorphous matrix, was demonstrated using a combination of an iron ion implantation and the low-energy electron irradiation techniques. Amorphous carbon thin films with 200-nm thickness were deposited on Si substrates by ion-beam sputtering. Iron atoms in a range from the order of 1013 to 1016 cm-2 were doped in selected regions of the a-C films using an ion implantation technique with a metal mask as a template. After removing the metal mask, the partially iron-containing a-C films were exposed to an electron shower. It was found that iron atoms were crystallized and preferably diffused toward the film surface leaving graphitic structures in the internal regions where iron atoms were implanted. On the other hand, the masked regions remained amorphous and at most a slight clustering with graphitic structures up to 1.5 nm was observed. The details of microstructural evolution and dependence of graphitization on iron content will be presented.

### 11:30 AM <u>OO11.6</u>

Self-assembled nanotiles of heteroepitaxial SiC on Si. <u>Takashi Matsumoto</u><sup>1</sup>, Masato Kiuchi<sup>1</sup>, Satoshi Sugimoto<sup>2</sup> and Seiichi Goto<sup>2</sup>; <sup>1</sup>Research Institute for Innovation in Sustainable Chemistry, National Institute of Advanced Industrial Science and Technology, Ikeda, Osaka, Japan;  $^2{\rm Graduate}$  School of Engineering, Osaka University, Suita, Osaka, Japan.

Using the organometallic ion beam technique, the self-assembled SiC nanotiles were fabricated on Si wafers. Nanosized, semiconducting tiles are important in electronics and photonics technologies, and will be applicable for single-electron and micro light-emitting devices. S is a wide bandgap semiconductor used for UV light emitters and power devices. We have fabricated the self-assembled SiC nanotiles using a very low-energy mass-selected ion beam deposition system. The precursor of methylsilicenium ion (SiCH<sub>3</sub><sup>+</sup>) was generated from dimethylsilane (SiH<sub>2</sub>(CH<sub>3</sub>)<sub>2</sub>). The ion beam was accelerated to 25 keV, and mass-selected by a sector magnet. In front of the substrate, the ion beam was decelerated to 100 eV and deposited on Si wafer. The ion beam current density was 0.1-0.2  $\mu$ A/cm<sup>2</sup> at the substrate. The pressure in the deposition chamber was kept below  $4x10^{-7}$  Pa. The growth temperatures of the self-assembled SiC nanotiles were 500-800 °C. The self-assembled SiC nanotiles were characterized by Raman spectroscopy, reflection high-energy electron diffraction (RHEED) and atomic force microscope (AFM). The crystal structure of the self-assembled SiC nanotiles was zinc-blende SiC (3C-SiC), and they were heteroepitaxy on Si substrates. The self-assembled SiC nanotiles had the length of 150-200 nm and the height of 15-30 nm. Using the organometallic ion beam, despite the low-temperature of

 $500~^{\circ}\mathrm{C}$  as SiC crystallization, the heteroepitaxial SiC nanotiles were fabricated on Si wafers.

> SESSION OO12: Ion Beam Analysis II Chair: Arjen Vredenberg Wednesday Afternoon, November 30, 2005 Commonwealth (Sheraton)

 $1:30~PM~*\underline{OO12.1}\\ Overview~of~Single~Ion~Effects~and~Analysis.~\underline{George~Vizkelethy}$ and Barney L. Doyle; Sandia National Labs, Albuquerque, New

Traditional Ion Beam Analysis (IBA) uses nanoampere currents of MeV ion beams to induce scattering or radiation producing events in the sample that is characteristic of its composition. All of these IBA techniques exploit very well understood ion-induced atomic and nuclear physics processes. Over the past decade, new techniques have evolved that exploit other well understood aspects of ion-material interactions: the deposition of charge including the generation of electron-hole pairs in semiconductors. The use of single MeV-energy microfocused single ions can therefore be used to position charges at specific locations with respect to electrodes or other semiconductor structures such as pn junctions for very basic studies of charge transport and induction in these materials. The effects of these ions on microelectronic devices is also identical to that caused by galactic and intergalactic cosmic rays in space. This new area of research is called Radiation Effects Microscopy, and this paper will review many of the developments that have occurred in this field over the past 5 years, both experimental and theoretical. Sandia is a multi-program 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.

# 2:00 PM OO12.2

SIMS Depth Profiling Free of Martix Effect. Peter Huber, <u>Helmut Karl</u> and Bernd Stritzker; University of Augsburg, Augsburg, Germany.

For samples incorporating high impurity concentrations (or concentration profiles across an interface) the SIMS count rate is no longer expected to be proportional to the concentration. We present a method to circumvent this problem and to retrieve a matrix free concentration profile by the use of implanted isotope profiles. The method described enables us to obtain SIMS (Secondary Ion Mass Spectroscopy) profiles corrected by all non-linearities between SIMS count rate and concentration (e.g. chemical matrix effect), resulting in an absolute concentration profile. The key is a self consistent evaluation technique allowing the determination of the relative sensitivity factors by comparing the numerically simulated implanted isotope depth profile with the measured SIMS-depth profile. The performance of the technique will be demonstrated for SiO2 implanted with Cd and Se at high doses.

### 2:15 PM OO12.3

Trends in the Structure and Strain of Two-Dimensional Rare Earth Silicides on Si(111) Investigated by Medium-Energy Ion Scattering. Steven Tear<sup>1</sup>, Tim J. Wood<sup>1</sup>, Ian M. Scott<sup>1</sup>, Dave J. Spence<sup>1</sup>, Chris Bonet<sup>1</sup>, Tim C. Q. Noakes<sup>2</sup> and Paul Bailey<sup>2</sup>; <sup>1</sup>Dept of Physics, University of York, York, United Kingdom; <sup>2</sup>CCLRC Daresbury Laboratory, Warrington, United Kingdom.

The epitaxial growth of the trivalent rare earth (RE) silicides on Si(111) has attracted considerable interest in recent years. This has arisen as a result of the potentially useful properties of low Schottky barrier heights of these interfaces with n-type silicon, and generally good lattice match with the silicon (111) surface. Such rare earth silicides have been suggested in alternative designs for source/drain regions in n-channel MOSFETs. The detailed surface structure of a series of two-dimensional (2D) RE silicides, formed when one monolayer of trivalent RE is deposited onto clean  $\mathrm{Si}(111)$  and annealed to  $\sim 500$  °C, have been analyzed using quantitative medium-energy ion scattering (MEIS) [e.g. 1, 2]. MEIS is an ideal technique to use for these materials as the RE atoms form an ordered subsurface layer, and hence energy and angular analyses of the scattered ions can be used to select those ions scattered from the subsurface RE's to determine the positions of the silicon atoms above the RE's. In a series of studies, the results of which are to be presented here, we examine a number of 2D RE silicides (Y, Gd, Dy, Ho, Er, and Tm) which exploit the sensitivity of MEIS to very small changes in the atomic positions above the RE layer and hence identify trends in the inter-atomic distance found in these systems. These trends will be discussed and compared to the small lattice mismatch, which varies by  $\pm$  a few percent, of the RE silicide with Si(111), and

to the RE atomic diameters. The results show a clear trend in the strain with lattice mismatch. [1] DJ Spence, SP Tear, TCQ Noakes, and P Bailey. Phys. Rev. B 61 (2000) 5707. [2] DJ Spence, TCQ Noakes, P Bailey, and SP Tear. Surf. Sci. 512 (2002) 61.

> SESSION OO13: Structural Modifications IV: Defect  ${\bf Accumulation,\,Amorphization,\,Strain\,\,Engineering,}$ Grain Orientation Control Chair: Bhupendra Dev Wednesday Afternoon, November 30, 2005 Commonwealth (Sheraton)

# 3:30 PM <u>\*OO13.1</u>

The Computer Simulation of Energetic Cluster Surface Interactions. Roger Webb, Advanced Technology Institute, University of Surrey, Surrey, United Kingdom.

The interaction of energetic clusters with solid surfaces has been a subject under investigation both experimentally and computationally for more than 30 years. Here we will concentrate on the simulation and modelling aspects of this work. In particular examples of cluster surface interactions will be drawn from molecular and cluster implantation for doping and materials modification, cluster erosion processes for surface erosion and deposition applications; and cluster and molecule scattering from surfaces. The behaviour of the cluster and target surface during and after impact can often be better understood with the help of computer simulation. Simulations will often give insight into what at first sight appear to be confusing experimental results. Examples of this will also be given.

### 4:00 PM \*OO13.2

Epitaxial Recrystallization of Nano-sized Amorphous Layers, Phase Nucleation and Growth in 4H-SiC. Fei Gao<sup>1</sup>, Yanwen Zhang<sup>1</sup>, Matthias Posselt<sup>2</sup>, Ram Devanathan<sup>1</sup> and William J Weber<sup>1</sup>; <sup>1</sup>MS K8-93, Pacific Northwest National Laboratory, Richland, Washington; <sup>2</sup>Forschungszentrum Rossendorf, Institute of Ion Beam Physics and Materials Research, Dresden, Germany.

Two nano-sized amorphous layers embedded in perfect crystals have been modeled to study the amorphous-to-crystalline (a-c) transition in the temperature range from 1000 to 2000 K in 4H-SiC by means of classical molecular dynamics methods. The results show that the epitaxial recrystallization of amorphous layer with a-c interface along the c axis is much faster than that along the basal plane, which suggests the anisotropies in the different activation energies for recrystallization. The recovery of bond defects and the rearrangement of atoms at the interfaces are important processes driving the epitaxial recrystallization of the amorphous layers. The nano-sized amorphous layer with the a-c interface oriented along the c axis can be fully recrystallized at all the temperatures considered. However, it is observed that second ordered phases, crystalline 3C-SiC, nucleate and grow during the recrystallization process inside the amorphous layer with the a-c interface along the basal plane, and these new phases are stable for long simulation times. Based on a model developed in the previous annealing simulations of 3C-SiC, the range of activation energies are determined to be from about 0.35 eV to 2.4 eV, which suggests that the recrystallization consists of multiple recovery processes, rather than a single process proposed previously. The present results are discussed and compared with the annealing simulations of 3C-SiC and experimental observations. The simulation results are in good agreement with previous experimental results in SiC, and thus, provide atomic-level insights into the interpretation of experimentally observed phenomena.

### 4:30 PM OO13.3

Ion irradiation induced densification of porous cluster-assembled thin films. <u>Kristoffer Meinander</u>, Kai Nordlund and Juhani Keinonen; Accelerator Laboratory, University of Helsinki, Helsinki, Finland.

Ionized cluster beam deposition has recently been introduced as a novel technique for the production of nanostructured thin films. Nanocrystalline thin films, grown by low-energy nanocluster deposition are, however, generally porous and extremely under-dense. Benefits of high deposition rates and nano-scale grain size, associated with cluster deposition, are outweighed by poor mechanical quality of the resultant films. Ion irradiation of crystalline materials is often associated with a volume expansion, and hence a decrease in density. In the case of initially under-dense materials, e.g. cluster-assembled  $\,$ thin films, irradiation causes local melting and subsequently results in a filling of voids. Irradiation induced processes will therefore facilitate an increase in density, simultaneously improving mechanical properties of the material. Heavy ion irradiation of porous thin films may thereby result in good mechanical qualities without greatly increasing grain sizes in the final films. Using molecular dynamics

simulations we have investigated the effects of heavy ion irradiation on cluster-assembled copper films. Porous Cu films with initial thicknesses of 15 - 20 nm, were produced by sequential thermal deposition of approximately 50 Cu nanoclusters, each containing 711 atoms, at 150 ps intervals between each cluster, on a smooth (100) Cu substrate. Irradiation with Xe and Au ions was thereafter carried out, during which the change in average density of the Cu films was calculated after each ion impact. This procedure was repeated for several ion energies, ranging from 1 keV to 30 keV. Results from these simulations will be compared with experiments. Initial simulation results indicate a fairly rapid increase in the density of the thin films, with irradiated film densities almost doubling those of as-deposited films after only a few dozen ion impacts. The final film densities converged at a level slightly below that of bulk copper. Optimal ion energies for the densification process were found to be dependent on the initial density and thickness of the thin films.

> SESSION OO14: Poster Session II Chair: Weilin Jiang Wednesday Evening, November 30, 2005 8:00 PM Exhibition Hall D (Hynes)

### 0014.1

Characterization of PIH&D deposited multilayer coatings by ion beam techniques combined with EFTEM. Florian Schwarz<sup>1,2</sup>, Joerg K. N. Lindner<sup>1</sup>, Maik Haeberlen<sup>1</sup>, Goetz Thorwarth<sup>1,2</sup>, Claus Hammerl<sup>2</sup>, Walter Assmann<sup>3</sup> and Bernd Stritzker<sup>1</sup>; <sup>1</sup>Institut fuer Physik, Universitaet Augsburg, Augsburg, Germany; <sup>2</sup>AnynTeC Duennschichttechnik GmbH, Augsburg, Germany; <sup>3</sup>Sektion Physik der LMU Muenchen, Garching, Germany.

Multilayered and nanostructured coatings offer a variety of advantages, for tribology, wear reduction, and diffusion barrier applications. The compositional analysis of such typically both thick and nanoscaled materials is difficult, requiring methods of high spatial resolution as well as depth range. The PIII&D method is an ion implantation and deposition technique frequently used for enhancement of tribological properties. In the present study, the focus rests on the analysis of amorphous carbon / silicon composite multilayers, envisioned for stress reduction and crack propagation control, and amorphous carbon / TiC structures, which offer advantages in terms of adhesion and layer stress. Characterization was based on energy filtered transmission electron microscopy (EFTEM) to achieve high spatial resolution and good sensitivity with respect to small concentrations. However, quantification of measured EFTEM element distributions is not straight forward. Combination of the high spatial resolution of this technique with accurate standardless concentration determination by Rutherford backscattering spectrometry (RBS) and elastic recoil detection analysis (ERDA) for heavy and light elements results in highly resolved and accurate multielement concentration profiles. To achieve this, laterally integrated EFTEM profiles were compared to depth profiles from RBS and ERDA and normalized accordingly.

### OO14.2

Ion Beam Synthesis of Cubic Boron Nitride Hard Coatings at low Temperatures. <u>Hans Hofsaess</u>, Carsten Ronning and Soeren Eyhusen; 2nd Institute of Physics, University Goettingen, Goettingen, Germany.

Cubic boron nitride (c-BN) films have been deposited on (100) silicon substrates using ion beam deposition of  $^{11}\mathrm{B}$  and  $^{14}\mathrm{N}$  ions. We apply a novel two step process consisting of (i) low energy (500 eV) ion deposition at about 520 K for nucleation and growth of an initial 50-100 nm thick c-BN layer, followed by (ii) high fluence ion implantation of 5 - 30 keV B an N singly charged ions at about 370 K to synthesize thick and low stress c-BN films. The first step is required to nucleate c-BN on a thin intermediate textured turbostratic BN-layer. The ion energy and substrate temperature dependence of the subsequent ion implantation growth process was systematically investigated and the samples were analyzed using EELS, FTIR and TEM as well as SIMS profiles of  $^{10}{\rm B}$  tracers. We find a threshold temperature for c-BN growth increasing from room temperature at 5 keV to about 370 K at 30 keV. Based on the SIMS analyses we can explain this temperature dependence by efficient dynamic annealing of individual collision cascades during growth. The resulting c-BN film structure is columnar and exhibits a reduced stress as compared to films grown solely under the initial nucleation conditions.

### 0014.3

Investigation of effects of ion beam irradiation on properties of magnesium oxide films. <u>Yasuhiko Morimoto</u><sup>1</sup>, Yoshikazu Tanaka<sup>1</sup> and Ari Ide-Ektessabi<sup>2</sup>; <sup>1</sup>Graduate School of Engineering, Kyoto University, Kyoto City, Japan; <sup>2</sup>International Innovation

Center, Kyoto University, Kyoto City, Japan.

Magnesium oxide (MgO) films have attracted attention for applications as the protective layer of ac plasma display panels (ac PDPs). The properties of protective layer exposed directly to the discharge space influence the discharge characteristics and lifetime of PDPs. The required properties for the protective layer are high ion-induced sputtering resistance, high secondary electron emission coefficient, high insulation, and high transparency. The preparation technology of the MgO films with preferable characteristics is required in the production process. The MgO films are presently prepared using electron beam (EB) evaporation method. However, it is difficult to control the properties of the films using EB evaporation method, and the films are weak for the ion bombardment. In order to overcome these problems, the authors focused ion beam assisted deposition (IBAD) method. The ion bombardment of the growing films causes considerable changes in principal properties due to the effects of ion mixing, ion sputtering, and channeling. IBAD method has a lot of controllable parameters, which makes IBAD method highly reproducible and flexible. In this study, effects of ion beam irradiation during deposition on properties of MgO films were investigated. The crystal orientation, surface morphology, and composition of the films were analyzed by X-ray diffraction (XRD), atomic force microscope (AFM), and Rutherford backscattering spectroscopy (RBS), respectively. The authors developed the secondary electron analyzer to investigate the secondary electron emission coefficient. The experimental results show that the crystal structure, surface morphology, and density of the MgO films were greatly influenced by the ion bombardment, which influenced the secondary electron emission coefficient. The relationships between secondary electron emissivity and the other parameters were also discussed.

### 0014.4

Chemical Modification of Polystyrene through Fluorocarbon and Hydrocarbon Ion Beam Deposition. Wen-Dung Hsu and Susan B. Sinnott; Materials Science and Engineering, University of Florida, Gainesville, Florida.

Classical molecular dynamics simulations are used to study the effects of continuous hydrocarbon (HC) and fluorocarbon (FC) ion beam deposition on a polystyrene surface. Plasma processing is widely used to chemically modify surfaces and deposit thin films. It is well-accepted that polyatomic ions and neutrals within low-energy plasmas have a significant effect on the surface chemistry induced by the plasma. This research focuses on how FC ions compare to similarly structured HC ions in their direct modification of polymer surfaces to yield controlled chemistry and nanoscale structures. In particular, the detailed chemical modification that result from of the deposition of beams of polyatomic HC ions (C<sub>3</sub>H<sub>5</sub><sup>+</sup> and CH<sub>3</sub><sup>+</sup>) and FC ions  $(C_3F_5^+$  and  $CF_3^+)$  on polystyrene surfaces at experimental fluences is investigated. The simulations predict that the degree of modification is influenced by the size of the incident ions, their velocity, and the strength of the interionic bonds. For example, larger ions modify the surface to a smaller depth than smaller ions. In addition, HC dissociate more readily than FC ions during deposition. Consequently, HC ions are predicted to chemically modify the polystyrene to a greater extent than the FC ions. This work is supported by the National Science Foundation (Grant number CHE-0200838).

# 0014.5

Properties and structures of nanocrystalline ZrN thin film deposited by 90\$\phi\$-bend electromagnetic filtered vacuum arc. \(\text{Uei-Shin Chen}^1\), Han C. Shih\(^1\), Por-Chan Lin\(^2\) and Sen-Hung Hsueh\(^1\); \(^1\) Department of Materials Science and Engineering, National Tsing hua University, Hsinchu, Taiwan; \(^2\)Institute of Materials Science and Nanotechnology, Chinese Culture University, Taipei, Taiwan.

Microparticle-free nanocrystalline ZrN films (grain size  $\sim 7\text{-}13$  nm) were deposited on Si substrates using a 90ø-bend electromagnetic filtered cathodic vacuum arc system. The effect of bias voltage on the microstructure and property of ZrN films was investigated. A negative bias voltage ranging from 0V to -400V was applied to the substrate. Texture, roughness, packing factor, and electric resistivity were performed by X-ray diffraction, atomic force microscope, Rutherford backscattering spectrometer (RBS), and 4-point probe measurement. The N/Zr ratio can be calculated by the packing factor obtained by RBS. The elements composition of ZrN films on Si substrate were obtained by Auger electron spectrometer. Electric resistivity decreases from 0V to -250V and then increases from -250V to -400V. The residual stress of the ZrN films obtained from bending beam apparatus is ranging from -0.5GPa to -7.3GPa. These properties are correlated to the total energy delivery with different substrate bias voltage. The optimal recipe for synthesizing ZrN film as diffusion barrier layer is also demonstrated.

### 0014.6

Simultaneous deposition of ITO film on ion beam treated polymers. Shih Hsiu Hsiao<sup>1</sup>, Tetsuro Yamaguchi<sup>1</sup>, Satoshi Kobayakawa<sup>1</sup>, Yoshikazu Tanaka<sup>1</sup> and Ari Ide-Ektessabi<sup>2</sup>; <sup>1</sup>Mechanical Engineering and Science, Kyoto University, Kyoto, Japan; <sup>2</sup>International Innovation Center, Kyoto University, Kyoto, Japan.

Indium oxide doped with tin oxide, ITO, is extensively used to fabricate transparent conductive coatings. ITO thin film on flexible substrates has been used in applications including touch panel contacts, electrodes for LCD and electrochromic displays. ITO thin film can be deposited onto polymer substrates using electron-beam evaporation. Surface modification of polymer substrate improves its superficial properties, adhesion in particular, to meet specific requirements while retaining the good mechanical properties of the substrates. In this study, a system consists of a linear ion beam and an electron beam which have been developed in vacuum. The linear ion beam treats the polymer surface while simultaneously the ITO thin film is deposited onto the surface using electron-beam evaporation. We demonstrate that polymer surface treated with the ion beam has superior binding to both standard adhesives and the deposited ITO films. Moreover, linear ion beam source provides good uniformity of surface treatment and is very suitable for production-scale processes. An integrated electron-beam evaporation and linear ion beam system significantly increases the chemical compatibility of a polymer surface to an over-layer, and promotes adhesion of chemical adhesives by increasing the superficial area of ITO deposited film.

### 0014.7

Ion beam modification of polyimides with linear ion source. Tetsuro Yamaguchi<sup>1</sup>, Shih Hsiu Hsiao<sup>1</sup>, Yoshikazu Tanaka<sup>1</sup> and Ari Ide Ektessabi<sup>2</sup>; <sup>1</sup>Graduate School of Engineering, Kyoto University, Kyoto, Japan; <sup>2</sup>International Innovation Center, Kyoto University, Kyoto, Japan.

Polyimides are widely used in various industrial fields. Example products are flexible printed circuit (FPC), flexible display and electric paper. Adhesion between metal or ceramic thin films and polyimides is required for high resistance and long term endurance. Ion beam irradiation modifies surface nanomorphology and chemical composition, crystalline structure. These modifications are potential techniques to improve the adhesion. The large area of ion beam irradiation is required for mass production. However, in the present techniques, the irradiation area is not large enough to be applied in production lines. In this study, the authors employed a linear ion beam source to improve productivity and increase the adhesion. The linear ion beam source can irradiate large surfaces homogeneously. Also, it is easy to enlarge the ion beam source in a longitudinal direction. A deposition system with linear ion source was developed during this study. The deposition of Cu and ZnO was performed on the polyimides using this system. During the deposition, the ion beam irradiation was carried out for modification of interface between the thin films and polyimide. The chemical state of the interface was characterized using x-ray photoelectron spectroscopy (XPS). The surface nanomorphology was investigated by atomic force microscopy (AFM). In this talk, the performance of the deposition system will be discussed, and the characteristics of the modified polyimides will be investigated in detail.

### 0014.8

Layer-by-Layer Sputtering and Ultrathin Ion Implantation by Low-Energy Grazing Ion Bombardment.

Abdurauf Dzhurakhalov, S. E. Rahmatov, N. T. Teshabaeva and M. Yusupov; Theoretical Dept., Arifov Institute of Electronics, Tashkent, Uzbekistan.

Last decades a grazing ion bombardment finds more and more application in the analysis and modification of surface structures of solids. For nanotechnology, nanoscale analysis and modification the application of grazing angles of incidence of ions on the solid surface is of high importance. Grazing incidence ion bombardment can play an important role in the smoothing, preparation, and characterization of semiconductor surfaces. In present work the peculiarities of sputtering and implantation processes at 1-5 keV Be, Se grazing ion bombardment of GaAs(001) surface and their application for the modification of surface structures have been investigated by computer simulation in binary collision approximation. The sputtering yield of target atoms and depth distribution of implanted ions versus both polar and azimuth angle of incidence have been calculated. At grazing incidence the component of projectile velocity which is normal to the surface is comparatively small. As a result the ions of primary beam penetrate only in several nearest to surface atomic layers during the process of their movement in channels of along a surface. As results show in this case the contribution of primary knocked-on recoils from the first layer to sputtering yield dominates very much in all azimuthal angular range. The most atoms ejected from second and

third layers lose their energy at collisions with surface atoms, they can not overcome the surface potential barrier and that is why their contribution to the sputtering yield is insignificant. These results show that at grazing incidence the layer-by-layer removal of surface layers is possible. Such removing allows both the layer-by-layer surface analysis and the surface polishing in atomic scale. On the other hand at grazing incidence the preferential sputtering in case of compounds and ion implantation lead to change of a profile of composition and structure of thin layers on the surface. It is known that implantation of Be and Se into GaAs allows to make the acceptor and donor impurities in this semiconductor. It was shown that at grazing incidence the main peak of implanted depth distributions is considerably shallower than that for large angles of incidence. The range for Se is shallower than that for Be and the half-width of profile for Se is narrow. The calculated results allow to select the optimum conditions for obtaining implanted depth distributions with demanded shape in narrow near-surface area of crystals. It was observed also the azimuth angular dependencies have a strong correlation with the crystal orientation which can be explained by influence of various surface semichannels and channels forming in different directions on the GaAs(001) surface.

### 0014.9

Fabrication and Characterization of YBCO Coated Conductor on IBAD-MgO Template. HongSoo Ha<sup>1</sup>, H. K. Kim<sup>3</sup>, R. K. Ko<sup>1</sup>, H. S. Kim<sup>1</sup>, J. S. Yang<sup>1</sup>, K. J. Song<sup>1</sup>, S. S. Oh<sup>1</sup>, C. Park<sup>4</sup>, S. I. Yoo<sup>4</sup>, J. H. Joo<sup>2</sup> and S. H. Moon<sup>3</sup>; <sup>1</sup>Superconductor Research Group, Korea Electrotechnology Research Institute, Changwon, Kyeongnam, South Korea; <sup>2</sup>Sungkyunkwan University, Seoul, South Korea; <sup>3</sup>SuNAM Company, Seoul, South Korea; <sup>4</sup>Seoul National University, Seoul, South Korea.

Biaxially textured MgO films have been fabricated as buffered template for YBCO coated conductor using ion beam assisted deposition(IBAD). We had observed the RHEED pattern to measure in-situ biaxial texture of film surface. SrTiO3 buffer layer was deposited on the MgO template to make high quality YBCO coated conductor. We have obtained YBCO layer with better in-plane texture by optimizing the buffer layer. Characterization of buffer and YBCO layers was studied using XRD and SEM. Experiment details and results of IBAD-MgO template will be reported. This research was supported by a grant from Center for Applied Superconductivity Technology of the 21st Century Frontier R&D Program funded by the Ministry of Science and Technology, South Korea.

### 0014.10

Influence of Ion Doping with Donor and Acceptor Impurities on Photoluminescence of Defects and Si Nanocrystals in SiO2 Films. David Isaakovich Tetelbaum<sup>1</sup>, Alexey Mikhaylov<sup>1</sup>, Alexey Belov<sup>1</sup>, Dmitry Kambarov<sup>1</sup> and Daria Gaponova<sup>2</sup>; <sup>1</sup>Physico-Technical Research Institute of University of Nizhny Novgorod, Nizhny Novgorod, Russian Federation; <sup>2</sup>Institute for Physics of Microstructures of RAS, Nizhny Novgorod, Russian Federation.

It was established earlier [D.I. Tetelbaum et al. Phys. Sol. State. 46 (2004) 17] that ion doping with phosphorus and boron modifies essentially photoluminescence (PL) related to ion-beam-synthesized Si nanocrystals in SiO2 films. However, the nature of this effect is still controversial. In the present report we generalize the experimental data on P, B, and N ion implantation into SiO2 thermal and deposited films containing Si nanocrystals. The PL in the range of 350-700 nm caused by intrinsic and radiation-induced defects and the PL at 750 nm originated from quantum-size effect in Si nanocrystals was detected on the each stage of sample preparation. Impurity ions were implanted in a wide range of doses either before or after Si nanocrystals synthesis, which was performed at 1273 and 1373 K. It has been shown that implantation of impurity ions quenches the PL from preliminary introduced Si nanocrystals and radiative defects (oxygen-deficient and non-bridging oxygen hole centers). Subsequent annealing at 1273 K increases considerably the defect-related PL at the highest impurity concentrations and leads to the recovery or enhancement of the nanocrystal-related PL depending on the impurity kind. In particular, the most intensive PL at 750 nm (up to one order of magnitude) may be achieved at certain conditions of phosphorus doping. In order to explain the observed PL regularities, several physical and chemical factors such as defect stabilization at the formation of composite glasses, influence of impurities on Si nanocrystals nucleation and growth, donor and acceptor nature of impurities, formation of nonradiative defects are considered. The optimal conditions of ion doping needed for reaching maximal PL emission in the whole visible spectrum are determined. Support of Russian Ministry for Education and science, CRDF (BRHE REC-001 project, RUR1-1038-NN-03, and Y2-P-01-09 awards), and EU (FP6 505285 STREP) is kindly acknowledged.

### 0014.11

Influence of Neutron Transmutation Doping to the Optical

Properties of Ge nanocrystals Prepared by Ion implantation. Shaobo Dun<sup>1</sup>, Tiecheng Lu<sup>1,2</sup>, Qiang Hu<sup>1</sup> and Songbao Zhang<sup>1</sup>; <sup>1</sup>Department of Physics, Sichuan University, Chengdu, China; <sup>2</sup>International Center for Material Physics, Chinese Academy of Sciences, Shenyang, China.

Recently, considerable attention has been focused on semiconductor nanocrystals embedded into SiO<sub>2</sub> films for light emission and for future high speed/ low power consuming logic and memory devices. In this paper, Ge nanocrystals (nc-Ge) were doped by Neutron transmutation doping (NTD) technique. Ge ions were implanted into SiO<sub>2</sub> amorphous films with the fluence of  $1\times10^{17}$  and  $3\times10^{17}$  cm<sub>-2</sub> respectively. Some samples was annealed in reduction atmosphere at 800  $^0{\rm C}$  to form nc-Ge, and others were not done. Then all of them were irradiated in nuclear reactor with neuron fluence of  $2.2 \times 10^{18}$   $1.4 \times 10^{19} \, \mathrm{cm}^{-2}$ . Subsequently, these samples were annealed at first . Subsequently, these samples were annealed at first 400<sup>0</sup>C then 800 <sup>0</sup>C to eliminate defects and to prepare doped nc-Ge, respectively. The doped and un-doped nc-Ge were measured by using laser Raman scattering spectroscopy(LRS), transmission electron microscopy (TEM) and laser excited photoluminescence (LE-PL)spectrocopy, respectively. The results show that, neutron irradiation induces nc-Ge amorphous transition from crystalline to amorphous particles at a certain extend and result the intensity of nc-Ge decreasing of Raman peak's. With increasing annealing temperature from 400 to 800  $^{0}$ C, amorphous nano-particles induced by neutron irradiation gradually re-crystallize. From the PL results, a new strong, wide PL peak at about 700nm was observed after dopping. It may be result from doped nc-Ge. But as for amorphous Ge nano-particles samples, after neutron irradiation, no new PL peak around 700nm as well as other PL peaks appear even if it was annealed. It may be not doped by NTD. It may be due to impurities being separated and aggregated to the surface of the re-fabricated nanocrystals during annealing as for amorphous nano-particles. That is to say, the nanocrystals is easy to be doped by NTD, but amorphous nano-particles not.

### 0014.12

Microstructural Study of Metal and Metal Oxide
Nanoparticles Growth in Silica by Ion Implantation.
Alicia Oliver, Jesus Arenas, Ovidio Pena, Juan Carlos Cheang-Wong,
Luis Rodriguez-Fernandez, Alejandro Crespo-Sosa and Vladimir
Iglesias; Instituto de Fisica, Universidad Nacional Autonoma de
Mexico, Mexico, D.F., Mexico.

— Nanometer-sized metal particles embedded in a glass matrix exhibit peculiar optical properties, and are particularly promising candidates for applications in the fields of non-linear integrated optics and photonics. Shape, size, spatial distribution and ambient conditions are crucial parameters to control their optical behavior. In this work we present a microstructural study of Cu and Ag nanoparticles growth in silica by ion implantation at 2 MeV, and subsequent thermal annealing in a reducing atmosphere (RA=50%N2+50%H2) or in an oxidizing one (OA=air). The ion fluence (4-8X1016 ions/cm2) and the Cu and Ag depth profile distributions were determined by Rutherford Backscattering Spectrometry. The nanoparticles were characterized by HRTEM and computational techniques such as digital processing and image simulation. HRTEM images of the metal nanoparticles showed particles in the 2-30 nm range. The samples annealed in the RA presented a narrower size distribution than those annealed in the OA. Cu and CuO nanoparticles were observed in the Cu-implanted samples regardless the annealing atmosphere, but the amount of CuO nanoparticles was higher after the OA annealing. In the case of Ag implantation, Ag nanoparticles growth for both atmospheres, but only in the OA annealing appeared AgO and Ag2O3 oxide nanoparticles. Crystal lattice and angle measurements were consistent with the identification of metal and metal oxide nanoparticles. Cubic and pentagonal bipyramidal shapes were predominant for the Cu nanoparticles, while the CuO ones presented an irregular form. Cuboctahedral shapes were predominant in the Ag and AgO nanoparticles. The authors would like to thank Samuel Tehuacanero, Juan Gabriel Morales, Roberto Hernandez and Luis Rendon for technical assistance in the HRTEM studies, and Karim Lopez y Francisco Jaimes for the accelerator operation.

### 0014.18

The ion-beam-induced magnetic anisotropy and structural change of Co/Pt thin film. Se Hong Kim<sup>1</sup>, Gap Soo Chang<sup>2</sup>, Chung Nam Whang<sup>1</sup> and Kyung-Hwa Yoo<sup>1</sup>; <sup>1</sup>Institute of Physics and Applied Physics, Yonsei University, Seoul, South Korea; <sup>2</sup>Department of Physics and Engineering Physics, University of Saskatchewan, Saskatchewan, Canada.

Magnetic anisotropy is one of the most important themes in the magnetic thin film study, since the manipulation of it has been spotlighted as a promising technique for advanced technologies in magnetic sensors, ultrahigh density magnetic storage, and spintronic systems. Due to spin-orbit coupling, the magnetic anisotropy is

sensitive to changes in atomic environment and film structure, and therefore spin manipulation has been accomplished mostly in well-defined epitaxial thin films or superlattices. Also the mechanism of the formation of magnetic easy axis in ferromagnetic thin films does not become known entirely. In this research, we intend to manipulate the direction of magnetic easy axis in a ferromagnetic thin film by using the ion irradiation method that has attracted a lot of attention due to its ability in modifying magnetic structures, and study the mechanism of the easy axis formation in a viewpoint of crystallography. For the research,  $\mathrm{Co}/\mathrm{Pt}$  multilayered films with eight periods of alternating layers of Co (3.5 nm) and Pt (4.5 nm) which were fit to equiatomic composition were grown on Si(100) substrates by an electron beam evaporation method in a high vacuum. The samples were ion-irradiated with Ar<sup>+</sup> ions which have an energy of 80 keV by using Cockroft-Walton typed ion accelerator in a high vacuum. In particular, an external magnetic field of 2500 Oe parallel to the film plane was applied to the samples during the ion irradiation process in order to incite the magnetic elements to be aligned along the field direction. While the as-grown sample is magnetically isotropic, the ion-irradiated sample comes to have an easy axis parallel to the direction of the field applied during the irradiation process in the film plane. The domain structure on nanoscale with respect to the easy axis acquired can be observed by using Magneto-Optical Microscope Magnetometer and the in-plane crystal structure confirmed by x-ray diffraction pattern can give us information about the mechanism of this ion-beam-induced magnetic anisotropy.

### 0014.14

Optical Studies of 200 MeV Ag<sup>+15</sup> Ion Irradiated Co Implanted ZnO Thin Films. Sangwon Shin<sup>1</sup>, Basavaraj Angadi<sup>1</sup>, Y. S. Jung<sup>1</sup>, Wonkuk Choi<sup>1</sup>, Ravi Kumar<sup>3</sup>, Fouran Singh<sup>3</sup>, K. Jeong<sup>2</sup>, M. Wasi Khan<sup>4</sup>, J. P. Srivastava<sup>4</sup>, Jonghan Song<sup>1</sup> and Jong-Han Lee<sup>1</sup>; <sup>1</sup>Korea Institute of Science and Technology, Seoul, South Korea; <sup>2</sup>Physics and Applied Physics, Yonsei University, Seoul, South Korea; <sup>3</sup>Materials Science Division, Nuclear Science Center, New Delhi, India; <sup>4</sup>Physics, Aligarh Muslim University, Aligarh, India.

We present the results of photoluminescence studies at low temperature on 200 MeV  ${\rm Ag}^{+15}$  ion irradiated Co-implanted ZnO thin films are presented. The c-axis oriented epitaxial ZnO thin films were grown using plasma assisted MBE on (001)  ${\rm Al_2O_3}$  substrate and implanted with 80keV Co ions with fluence values  $1{\rm x}10^{16}$  to  $5{\rm x}10^{16}$  ions/cm², which show Co clusters. The Co clusters were dissolved using 200 MeV  ${\rm Ag}^{+15}$  ions irradiation with fluence  $1{\rm x}10^{12}$  ions/cm². The photoluminescence spectra of pure ZnO thin films were characterized by the I4 peak at 3.365 eV and the broad emission due to the vacancies and defect states 2.45eV. The Co-doped ZnO films show three sharp levels at 3.247eV, 3.211eV and 3.169eV due to  $t_{2g}$  and two shoulders at 2.975eV and 2.815eV due to the  $e_g$  levels of crystal field splitted Co d-orbitals lying in the band gap of ZnO. Their position is independent of the temperature, whereas, the I4 intensity is decreased with the increase in temperature due to the thermal quenching of this emission.

### 0014.15

Seoul, South Korea.

Ion-beam patterning of magnetic easy-axis in the direction of in-plane and out-of-plane. Jong-Han Lee<sup>1,2</sup>, Sangwon Shin<sup>1,3</sup>, Jonghan Song<sup>1</sup>, In-Hoon Choi<sup>2</sup> and Chungnam Whang<sup>3</sup>; <sup>1</sup>Advanced analysis center, Korea Institute of Science and Technology, Seoul, South Korea; <sup>2</sup>Materials Science and Engineering, Korea University, Seoul, South Korea; <sup>3</sup>Physics and applied physics, Yonsei University,

Recently, ion irradiation has been used to modify the extrinsic properties of magnetic films, such as magnetic anisotropy, coercivity, and magnetic exchange field. The ion irradiation process has the advantage in possibility of patterned magnetic media fabrication using stencil mask or fine lithographic process. In this work, magnetic patterning in the direction of in-plane and out-of-plane was demonstrated by ion irradiation, respectively. Out-of-plane patterning was performed in the epitaxial Cu/Ni(60Å)/Cu(002) possessing perpendicular magnetic anisotropy(PMA). Epitaxial Cu/Ni/Cu(002) is well-known as having a PMA in the Ni with thickness, ranging from 1.5 nm to 14 nm, due to strain caused by lattice mismatch between Ni and Cu layer. After ion irradiation, the strain in the Ni layer was relaxed and magnetic easy-axis was altered from the direction along the surface normal to in-plane. Cu/Ni(60Å)/Cu film was irradiated by 40 keV O ion with doses of  $5 \times 10^{15}$  ions/cm<sup>2</sup> through PMMA mask having  $10\mu \text{m} \times 10\mu \text{m}$  pattern size. After mask removal, it was confirmed that the sample has in-plane magnetization of irradiated area and perpendicular magnetization of unirradiated area by measuring MOKE in polar and longitudinal configurations. MFM measurement shows the magnetic pattern directly. In-plane patterning was performed in the Co/Pt multilayer. As-grown Co/Pt multilayer has no magnetic easy direction. After ion irradiation in the magnetic field along the in-plane direction, the film has magnetic easy-axis in

the applied external magnetic filed direction. In order to pattern the film with two different in-plane magnetic easy axis, additional ion irradiation was performed with an external magnetic field applied in the perpendicular direction to the magnetic easy axis of the film. PMMA mask with a 10  $\mu$ m x 10  $\mu$ m pattern size was used during ion irradiation. After mask removal, it was confirmed that the sample has dual easy-axis pattern in plane by measuring MOKE and MFM.

### 0014.16

Ton-beam nano-patterning by using porous anodic alumina as a mask. <u>Jong-Han Lee<sup>1,3</sup></u>, Sangwon Shin<sup>1,2</sup>, Jonghan Song<sup>1</sup>, In-Hoon Choi<sup>3</sup>, Chungnam Whang<sup>2</sup>, Jaeyong Lee<sup>2</sup> and Sunggu Lee<sup>2</sup> <sup>1</sup>Advanced analysis center, Korea Institute of Science and Technology, Seoul, South Korea; <sup>2</sup>Physics and Applied Physics, Yonsei University, Seoul, South Korea; <sup>3</sup>Materials Science and Engineering, Korea University, Seoul, South Korea.

Anodized aluminium oxide (AAO) with self-organized and ordered nano-hole arrays may be a good candidate for an irradiation mask to modify the properties of a nano-scale region. In order to use AAO as a mask for ion beam patterning, the ion beam transmittance of AAO should first be tested. In an AAO with a high aspect ratio (about 100), anodized from Al bulk foil, the ion beam transmittance was extremely low. However, when AAO with low aspect ratio (about 5), fabricated with thin film Al on SiO2, was irradiated with 80 keV Co ions, the Co ion transmittance was enormously improved. After selective etching of the unirradiated region, ion beam patterned 80 nm SiO<sub>2</sub> dot arrays have been fabricated. This shows a potential of AAC with a low aspect ratio for an ion beam patterning nano-mask. In order to demonstrate the ion beam nano-patterning, magnetic nano-patterning was performed. A Co/Pt multilayer film with a perpendicular magnetic anisotropy was ion irradiated through an AAO mask with a low aspect ratio, 460 nm height and 50 nm diameter, and the magnetic properties were investigated by magneto-optic Kerr effect (MOKE). The formation of a magnetic nano-pattern was confirmed by magnetic force microscopy (MFM).

Formation of Pt Nano-Wires and Dots on SiO<sub>2</sub> Substrates Using Ion Irradiation. <u>Kai Zhao</u>, Robert S Averback and David G Cahill; Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois.

We had shown previously that irradiation of thin Pt films on dielectric substrates with energetic ions leads to a dewetting process and nanoscale patterning. The patterns in this case had a characteristic length scale but they were not ordered. In the current work we use focused ion beam or lithography to create an array of Pt lines on the SiO<sub>2</sub> substrate and thereby control the nucleation sites for subsequent dewetting. Surprisingly, patterns are established in all three directions. The Pt lines, which are several tens microns in length, have initial widths between 100 nm and 5  $\mu m$  and thickness between 8 and 24 nm. Upon irradiation with ions, the wires begin to shrink, reaching widths less than  $\approx 25$  nm. Rims observed along the line edges indicate that the dewetting process is similar to that for liquids, although the temperature is 300 K. With continued irradiation, the Pt lines become unstable along their lengths, resembling very closely Rayleigh instability, which is normally found in liquid jets. The wavelength of the instability,  $\lambda_c$ , was  $\approx 10$  times the radius of the wire. This instability leads eventually to rows of nanodots with their spacing given by  $\lambda_c$ . Finally, it was also observed for the 1 MeV Kr<sup>+</sup> irradiations that surface region becomes patterned out-of-plane as well. Regions covered by the metal wires rise above the surface plane, while uncovered regions become recessed. The relative deformation is around 180 nm at the dose of  $2 \times 10^{16}$  ions/cm<sup>2</sup>. The total volume of material is conserved. These various observation are discussed in terms of a radiation induced viscosity and anisotropic shear deformation.

Formation of Nanolayers of Nanomaterials and Their Applications. Daryush ILA<sup>1</sup>, Paul Thevenard<sup>2</sup>, Robert L. Zimmerman<sup>1,3</sup>, Iulia Muntele<sup>1</sup>, Claudiu Muntele<sup>1</sup>, Bangke Zheng<sup>1</sup>, Zhigang Xiao¹ and A. Leslie Evelyn¹; ¹Physics, Alabama A&M University, Normal, Alabama; ²Physics, Universite Claude Bernard, Lyon, Villeurbanne CEDEX, France; ³Department of Physics and Mathematics, University of Sao Paulo, Ribeirao Preto, Sao Paulo,

Workers at the Center for Irradiation of Materials of Alabama A&M University have used various MeV ion beams to form composites of layers of nanocrystals (NC) in various substrates. The periodic layered films were produced either by MeV ion implantation followed by thermal annealing and/or by MeV ion irradiation, or by simultaneous deposition of various species followed by thermal annealing and/or by post MeV ion irradiation. The produced multilayer films have a periodic structure consisting of alternating thin film layers of the thicknesses between 20 and 50 Angstroms. The periodic layered thin

films were characterized by Rutherford backscattering (RBS) spectrometry. For optical properties we used UV/VIS/IR absorption photo-spectrometry. For thermoelectric properties of nano-layered structure we fabricated a  $3\omega$  method to measure thermal conductivity, used a Hall Effect system to measure the electrical conductivity and used a Seebeck coefficient measurement system to completely measure all physical properties of the produced multi-nano-layered structure in order to calculate the Figure of Merit for these nanolayered systems. The systems which we will present as examples for this presentations are 10 to 50 nanolayers layers of [insulator/metal NC-insulator/insulator/], and [insulator/semiconductor NC-insulator/ This presentation will focus on our last few years efforts on production of variable width optical filters as well as production of highly efficient thermoelectric materials.

### 0014.19

Nanostructure Formation in Focused Ion Beam Processed CaF<sub>2</sub>. Tianhua Ding<sup>1</sup>, Sha Zhu<sup>1</sup>, Wei Zhou<sup>1,2</sup> and Lumin Wang<sup>1,3</sup>; <sup>1</sup>Department of Nuclear Engineering and Radiological Science, University of Michigan, Ann Arbor, Michigan; <sup>2</sup>Precision Engineering

& Nanotechnology Center, Nanyang Technological University, Singapore, Singapore; <sup>3</sup>Department of Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan.

Focused Ion Beam (FIB) has been increasingly used as a tool for surface modification and nanofabrication. However, damage induced by FIB has not been well understood. We used a 30 keV  $\mathrm{Ga}^+$  focused ion beam to irradiate CaF<sub>2</sub> (100) surface and SEM to monitor the evolution of the surface patterns. We observed uniform thickness reduction of surface under normal-angle FIB irradiation and self-organized ripples with a wavelength of about 200 nm under off-normal FIB irradiation. The orientation of the ripples was perpendicular to the direction of ion beam projection, as predicted by the Bradley-Harper model. We managed to irradiate a CaF<sub>2</sub> foil of less than 10 micron in thickness until it was thin enough for TEM observation. FIB-induced voids of about 20 nm in diameter and CaO nano-particles were observed under the TEM in the areas by normal ion beam irradiation; however, no such voids or nano-particles were found in regions irradiated at large off-normal angle of 50 degree, indicating more severe irradiation damage at the normal angle. The observation of CaO nano-particles is very interesting, because it serves as evidence to show selective sputtering of F in the  $\text{CaF}_2$  compound. The selective sputtering left Ca on the irradiated surface and the Ca was oxidized when the sample was transferred from FIB to TEM. Furthermore, we report formation of three-dimensional superlattice of voids of 5 nm in size and 20 nm in spacing in the ripples after they were exposed to 200 keV electron beam inside the TEM. Such e-beam induced voids did not form in the regions irradiated by normal FIB.

## OO14.20

Deposition energy dependence in cluster-assembled thin film densities. Kristoffer Meinander, Tina Clauss and Kai Nordlund; Accelerator Laboratory, University of Helsinki, Helsinki, Finland.

Nanocluster deposition is a novel technique with immense possibilities for the growth of thin nanostructured films. Properties of films grown in this manner are greatly governed by the energy at which clusters are deposited. Even small differences in the energy per atom at which clusters impact a surface will result in large differences in the final films. Low-energy deposition of nanoclusters is an excellent means of producing films with grain sizes similar to the size of the deposited clusters themselves. Unfortunately, these films have a tendency towards being extremely under-dense. An increase in the energy of deposition will result in a gradual improvement of the film properties, due to a higher packing density between the atoms from individual clusters. At sufficiently high energies the resultant films exhibit extremely good mechanical quality as well as an improved adhesion to the underlying substrate. Although energetic cluster deposition is a seemingly perfect method for producing high quality thin films, it remains inadequate if films with nanocrystallinity are desired. Using molecular dynamics simulations we have deposited Cu nanoclusters on a smooth (100) Cu substrate at 300 K, and investigated the density and structure of the resulting films as a function of deposition energy. The cluster-assembled films were grown through sequential deposition of 586 atom nanoclusters, at energies ranging from 5 meV to 10 eV per atom in the cluster, for each individual film. Between each consecutive impact, clusters were relaxed on the surface for 150 - 200 ps. Film growth was continued until each film contained an excess of 50 clusters. Initial results show that the average density for cluster-assembled thin films increases logarithmically with increasing deposition energy. Films produced with deposition energies in the lower range of the studied region contained a multitude of voids, and therefore had low densities. At deposition energies above 1 eV/atom, voids were no longer present in the films, with surface roughness being the only contributor towards a lowering of the average film density. At very high energies the films were not only epitaxial, but also very smooth.

### OO14.21 Abstract Withdrawn

OO14.22

Chemical Behaviour and Corrosion Resistance of Medical Grade Titanium after Surface Modification by Means of Ion Implantation Techniques. Frank Schrempel<sup>1</sup>, Gerhard

Hildebrand<sup>2</sup>, Marion Frant<sup>2</sup>, Kaiyong Cai<sup>3</sup> and Klaus Liefeith<sup>2</sup>; <sup>1</sup>Institute of Solid State Physics, Friedrich-Schiller-University, Jena, Germany; <sup>2</sup>Department of Biomaterials, Institute for Bioprocessing and Analytical Measurement Techniques, Heilbad Heiligenstadt, Germany; <sup>3</sup>Institute of Materials Science and Technology, Friedrich-Schiller-University, Jena, Germany.

Interest in chemical modifications of bioinert metallic implants has been stimulated by the successful use of coatings such as hydroxyapatite, which provide an inorganic surface with physicochemical characteristics similar to those of bone and thus enhances osseointegration. Plasma spraying is currently the most common deposition technique, but the high processing temperature causes structural changes, stress peaks and delamination of the deposition in the body. Besides low-temperature sputtering and wet chemical treatments also ion implantation techniques are used for the bio-activation of titanium. This work presents data on the topographical, chemical and physicochemical surface composition and corrosion resistance of medical grade titanium and titanium after ion implantation. Pure commercial titanium (grade 2) according ISO 5832-2 has been implanted with 30 keV Na-, Ca- and P-ions and fluences between 1 x  $10^{15}$  cm<sup>-2</sup> and 4 x  $10^{17}$  cm<sup>-2</sup>. Some of the samples were heat treated at 600 °C for 40 min. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) was used for surface observations. The chemical composition was investigated using Rutherford backscattering spectrometry (RBS) and X-ray photoelectron spectroscopy (XPS). Physico-chemical investigations were carried out using contact angle measurements to determine the surface tension as well as the polarity of the modified titanium surfaces. The determination of zetapotentials were performed and the corrosion resistance was examined in simulated body fluid (SBF). Cyclic voltametry studies have been performed to quantify electrochemical parameters like the current density in the passive range as well as the corrosion current density under open circuit conditions. The results show that ion implantation using certain ions can be used to design tailor made titanium surfaces. Considering the P-implantations, the measured depth distribution of phosphorus agrees very well with simulation results. The maximum concentration is located at about 30 nm and the width is less than 40 nm. In contrast, for the implantation with Na and Ca, the measured depth distribution deviates remarkably from the simulation. The simulated distribution shows a maximum at 30 nm and a width of 30 nm. The measured ion concentration extending from the surface to a depth of about 80 nm and the shape of the distribution is similar to a diffusion profile. This finding is associated with a strong incorporation of oxygen with concentrations of about 60 atomic percent at the surface and the same shape as for the calcium distribution. According to the chemical changes different contact angles as well as zetapotentials have been detected for the ion implanted surfaces compared to pure titanium. The results of the performed electrochemical examinations indicate that the implantation has no significant negatively influence to the observed corrosion resistance in comparison to medical grade titanium.

0014.23

Materials Processing of Surfaces and Coatings by Intense Ion Beams for In-Body Applications. Timothy J. Renk, Sandia National Laboratories, Albuquerque, New Mexico.

We are investigating the processing of surface-modified materials and surface coatings for improved mechanical performance and biocompatibility for in-body use. Substrate materials include Ti-6Al-4V and Co-Cr-Mo alloys as well as Ultra High Molecular Weight Polyethylene (UHMWPE). Coatings include Hf, multi-layer and nanocomposite ceramics, and a-C layers produced by intense pulsed ion beams at the RHEPP-1 facility at Sandia National Laboratories. Such beams can modify near-surface properties by fast heating and cooling cycles (10<sup>9</sup>K/sec). Higher-fluence beams can be used to ablate and redeposit material from a target, in a manner similar to Pulsed Laser Deposition (PLD). A combination of the two processes is also being investigated, i.e. a deposited coating can subsequently be surface-modified at lower fluence. Both processes tend to produce nanocrystalline structures and metastable phases. Improvements in wear durability and enhanced biocompatibility have been demonstrated in a 1 micron Hf-rich layer deposited and surface-alloyed with a Ti-6Al-4V substrate. An ablated and surface-treated nano-laminate ceramic (ZrO2 and Y2O3 layers) shows promise as a hard coating for Co-Cr-Mo. The microstructure of treated samples has been investigated by cross-sectional high

resolution transmission electron microscopy (HRTEM), annular dark-field scanning TEM (STEM), and selected area electron diffraction (SAD). Compositional changes have been measured by nanobeam energy dispersive spectroscopy (EDS) and energy filtered TEM imaging. Treated layers are subjected to tribological and corrosion testing, to study the relation between the microstructures and friction and wear behavior. Latest results will be presented. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Co., under US DOE Contract DE-AC04-94AL85000.

0014.24

Continued Growth of Single-Walled Carbon Nanotubes from Open-ended SWNT Substrates. Myung Jong Kim 1.2, Erik Haroz 1.3, Hongwei Shan 1.3, Nolan Nicholas 1.2, Carter kittrell 1.3, Hua Fan 1.2, Wen-Fang Hwang 1.3, Sivaram Arepalli 4, Robert Wheeler 5, Eric Jung 6, T.J. Wainerdi 1, Robert Hauge 1.3 and Richard Smalley 1.3, 2; 1 CNL, Rice University, Houston, Texas; 2 Physics, Rice University, Houston, Texas; 3 Chemistry, Rice University, Houston, Texas; 4 NASA Johnson Space Center, Houston, Texas; 5 Wright-Patterson AFB, Daton, Ohio; 6 Materials Science and Engineering, University of Pennsylvania, Philadelphia, Pennsylvania.

We prepared a nanoscopically flat open-ended SWNT substrate from SWNT spun fibers by using the focused ion beam cutting technique followed by various etching and cleaning schemes. Deposited catalyst was docked to the open ends of SWNTs, and carbon feedstocks were catalyzed into continued single-walled carbon nanotube growth resembling 1D molecular epitaxy in both the cold wall furnace and the hot wall furnace setups. The data obtained from Raman spectroscopy indicates that the (n, m) structure of the newly grown SWNT was cloned from that of the pre-existing SWNT substrate. Such results lead us to believe that this method will provide us with a means of chirality-controlled SWNTs growth on a macroscopic scale using a fairy general and scalable setup in the future.

OO14.25

Highly Anisotropic Carbon Nanofibers via Inert Gas Sputtering of Graphite: How do They Grow? Steven C. Seel, Thomas A. Westrich and <u>Jerrold A. Floro</u>; Surface and Interface Sciences, Sandia National Labs, Albuquerque, New Mexico.

Argon ion sputtering of graphite substrates results in the formation of carbon nanofibers, which can have lengths up to 50 um while being only 50 nm in diameter, implying a 1000x preference for axial vs. radial growth. This extraordinary growth anisotropy is difficult to understand. Nanofibers are not erosional features – the thickness of carbon removed is less than a micron. These structures are NOT nanotubes; Raman spectroscopy indicates that nanofibers are a form of nanocrystalline carbon, consistent with transmission electron microscopy images and with a measurable electrical conductivity. Furthermore, nanofibers are formed at modest homologous temperatures, in the range 400-600C, where surface diffusivity should be limited. Indeed, when HOPG substrates are used, significant cone—like surface roughness forms first — indicative of low surface mobility — followed by the growth of nanofibers from the cone-tips. On polycrystalline POCO graphite, the already extremely rough surface actually locally smoothens prior to nanofiber formation, which tends to initiate near pores in the substrate before spreading across the entire substrate with increasing dose. On glassy carbon substrates, nanofibers will form, but only at macroscopic asperities (e.g, scratches and pits). We will briefly discuss various models for nanofiber growth including deposition from the sputtered carbon flux, field-enhanced growth, and stress-induced extrusion. This work was partially supported by the DOE Office of Basic Energy Sciences. Sandia is a multiprogram laboratory of the United States Department of Energy 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.

0014.26

Low Temperature Annealing Effects on Magnetron Sputtered Al/Ni(V) Reactive Coatings. Matthew P. Blickley<sup>1</sup>, J. Derek Demaree<sup>1</sup>, James K. Hirvonen<sup>1</sup> and Nicholas A. Soroka<sup>2</sup>; <sup>1</sup>U. S. Army Research Laboratory, Aberdeen Proving Ground, Maryland; <sup>2</sup>U. S. Military Academy, West Point, New York.

Reactive nanofoil coatings of magnetron-sputtered aluminum and nickel/vanadium bilayers were characterized for reliable debonding of carbon composite structures to be used in future Army applications. We determined the initiation energies of multilayer stacks (300 bilayers, each approximately 85 nm Al and 55 nm Ni/V) by discharging an electric spark from a fixed capacitor bank and varying DC voltage before and after low temperature annealing (90-250 C; 1 140 hr), which resulted in varying amounts of interfacial interdiffusion observable by Rutherford backscattering spectrometry (RBS). Measurable increases in initiation energy were observed with

increasing anneal times and temperatures, in agreement with the RBS results and existing literature discussion of reaction mechanisms. The initiation sensitivity was also impacted by varying the stoichiometric ratio of Al and Ni/V, also quantified by RBS. These results allow for the optimization of both reliable initiation and long term, low temperature storage stability of the final reactive coatings. \*The Pennsylvania State University. The research was performed under appointment to the U.S. Army Research Lab administered by Oak Ridge Institute for Science and Education under a contract between the U.S. Department of Energy and the Oak Ridge Associated Universities.

> SESSION OO15: Nanotubes and Nanowires: Beam Induced Formation and Modification Chair: Roger Webb Thursday Morning, December 1, 2005 Commonwealth (Sheraton)

### 8:30 AM \*OO15.1

Ion Irradiation-Induced Welding of Carbon Nanotubes to a Si (100) Surface. Jani Kotakoski and Kai Nordlund; Accelerator Laboratory, University of Helsinki, Helsinki, Finland.

Carbon nanotubes (CNTs) are one of the possible building blocks for electronic devices in the transition phase from traditional silicon based microelectronics towards the few-nanometer regime. Remaining problems in integrating CNTs to the existing technology is the low reactivity of the CNT walls. This leads to low conductance between CNTs and the other components. We examine the possibility to use low energy ion irradiation to solve this problem by increasing the number of chemical bonds in the contact regions. We have used classical molecular dynamics simulations with empirically fitted potentials to study the enhancement in the binding of a single-walled nanotube (SWNT) to a Si (100) surface at room temperature. The studied system consists of a silicon substrate with a trench, over which we have deposited a SWNT. Low irradiation doses and low energies (0.2 keV - 1.2 keV) were used to ensure that the irradiated SWNT will not be destroyed. We simulated the irradiation with silicon, carbon and neon ions. The number of bonds between the SWNT and Si surface and the damage caused to the SWNT were estimated by studying the positions of the atoms after each irradiation event. Our simulations indicate that ion irradiation will increase the number of covalent bonds between the SWNT and the Si substrate. When the irradiation dose and energies are low, the damage caused to the SWNT atomic network can be tolerable when compared to the improvement in the conductance of the contact regions. Furthermore, as the CNTs have high ability to heal the irradiation-induced damage, it is possible that the irradiation will not have a significant negative effect to the conductivity of the SWNT in a system of this type.

### 9:00 AM OO15.2

Ga Nanodot Fabrication by a Focused Ion Beam. Yajing Liu<sup>1</sup>, Jacob Thorp<sup>1</sup>, Pavan Aella<sup>1</sup> and S. Tom Picraux<sup>2,1</sup>; <sup>1</sup>Chemical & Materials Engineering, Arizona State University, Tempe, Arizona; MST-CINT, MS G756, Los Alamos National Laboratory, Los Alamos, New Mexico.

A focused ion beam (FIB) is a versatile tool for characterization and fabrication of materials and devices. In this presentation we demonstrate the use of a 30 keV dual beam FIB system for the formation of well-defined metallic Ga nanodots and explore the use of this approach for the seeded growth of precisely placed arrays of Si nanowires by the Vapor-Liquid-Solid (VLS) growth technique. FIB has the great advantage of site-specific implantation, micromachining and nanopatterning, and also has the capability for in situ monitoring of the process. By combining FIB patterning and Ga implantation into oxide layers followed by vacuum annealing, Ga nanodots are fabricated on SiO<sub>2</sub> grown on Si wafers. In this work, the Ga ion fluences in the patterned areas range in from  $10^{14}$  to  $10^{19}$  ions/cm<sup>2</sup> For a 30 keV Ga onto SiO<sub>2</sub> layers of 170 nm thickness, it is found that the nanodots are formed when the Ga fluence exceeds the threshold value  $3.4 \times 10^{16}$  Ga ion/cm<sup>2</sup>, and are formed in the range between  $3.4 \times 10^{16}$  ion/cm<sup>2</sup> (with the trench depth of 10 nm) and  $1.6 \times 10^{17}$  ion/cm<sup>2</sup> (with the trench depth of 50 nm). The diameter of the fabricated nanodots range from 30 nm to 100 nm, and the total number of the nanodots decrease with ion fluence. The presence, size, and patterned surface morphologies of these nanodots under the different systematic parameters are investigated, and their formation mechanisms are discussed. Locally adjusting the Ga fluence by programmed beam scans allows controlled placement of the Ga nanodots. Previously large Ga droplets have been successfully used as catalysts to seed the growth of Si nanowires by the VLS method. In the present work we discuss preliminary results for Si nanowire growth at 400 to 600°C using nanoscale Ga metallic dots and demonstrate the possibility of using this approach for forming nanowire arrays by well-defined Ga nanodot patterning.

# 9:15 AM <u>OO15.3</u>

Carbon Nanotube Growth from Nanoscale Clusters Formed by Ion Implantation. Yongho Choi<sup>1</sup>, Jennifer Sippel Oakley<sup>2</sup>

Andrew Rinzler<sup>2</sup> and Ant Ural<sup>1</sup>; <sup>1</sup>Electrical and Computer Engineering, University of Florida, Gainesville, Florida;  $^2$ Physics, University of Florida, Gainesville, Florida.

Controlled growth of carbon nanotubes remains one of the biggest challenges in bottom-up assembly of nanotube-based devices. One of the key components of carbon nanotube growth is the catalyst material used to nucleate the nanotubes. In this talk, we demonstrate that transition metal ions implanted into silicon dioxide thin films form nanoscale clusters which can act as catalyst for carbon nanotube growth. In particular, we implant iron ions at various doses and energies into silicon dioxide thin films thermally grown on silicon substrates. We then use chemical vapor deposition (CVD) to grow carbon nanotubes on these ion implanted substrates using methane as the precursor gas. We study the effect of ion implantation dose and energy, and anneal and growth time and temperature on the structural properties of the nanoscale clusters, as well as the carbon nanotubes nucleated from these clusters. In particular, we find that growth of low density, horizontal, and small diameter carbon nanotubes on silicon dioxide is possible using this nucleation technique. This makes it possible to electrically address individual nanotubes. We also study the diffusion of ion implanted iron in thermally grown oxide in order to better understand the microscopic mechanisms for nanoscale cluster formation. Since ion-implantation can be easily masked by lithography, this technique of nucleating nanotube growth opens up the possibility of controlling the origin of nanotubes at the nanometer scale over high aspect ratio topography. This level of control is not possible with the liquid solution-based catalyst material typically used. This technique of nucleating nanotube growth, which can easily be integrated with silicon processing and scaled to larger substrates, demonstrates the versatility of ion beam processing for nanomaterials growth applications.

9:30 AM  $\underline{\text{OO15.4}}$  Fabrication of Nanofibers on the Surface of Ge and GaSb by Focused Ion Beam Irradiation. Lumin Wang<sup>1,2</sup>, Wei Zhou<sup>1,3</sup>, Sha Zhu<sup>1</sup> and Tianhua Ding<sup>1</sup>; <sup>1</sup>Nuclear Engineering & Radiological Sciences, University of Michigan, Ann Arbor, Michigan; <sup>2</sup>Materials Science & Engineering, University of Michigan, Ann Arbor, Michigan; <sup>3</sup>Provision Engineering & Natural Science <sup>3</sup>Precision Engineering & Nanotechnology Centre, Nanyang Technological University, Singapore, Singapore.

Controlling the structure and morphology of materials at the nano-scale opens exciting opportunities for manipulating material properties with great flexibility and precision. We report a convenient and efficient way to produce nanofibers on the surface of Ge and GaSb using focused ion beam (FIB) irradiation. When a 30 keV  $\mathrm{Ga}^+$ focused ion beam was rastered on surfaces of  $\mathrm{Ge}(100)$  and  $\mathrm{GaSb}(100)$ , a network of nano-scaled voids first formed at a low ion fluence below 10x15 ions/cm<sup>2</sup> and the main surface feature quickly evolved into a high density of nanofibers standing on a porous structure when the ion fluence was increased to around 10x16 ions/cm<sup>2</sup>. This process was accompanied by an "abnormal" surface swelling to heights of tens of nanometers above the original surface level. It is also interesting to observe liquid-like nano-droplets on irradiated surface of GaSb and the gradual coarsening of these droplets with the further increasing of ion fluence. We provide experimental evidence to show that the droplets are amorphous Ga and attribute their formation to selective sputtering of Sb in the GaSb compound. Another noteworthy observation is the self-organized formation of nanofibers outside the ion beam irradiated surface areas. The fibers were found to have a fairly uniform diameter of about 20 nm and various lengths up to a few microns. It can be envisaged that formation of such long nanofibers outside the irradiated areas are related to initial nucleation of fibers due to irradiation damage from scattered ions and the subsequent growth due to redeposition of the materials sputtered away from the nearby areas.

> SESSION OO16: Structural Modifications IV: Defect Accumulation, Amorphization, Strain Engineering, Grain Orientation Control Chair: Hans Hofsss Thursday Morning, December 1, 2005 Commonwealth (Sheraton)

# 10:15 AM OO16.1

High Aspect Ratio Microstructures in LiNbO<sub>3</sub> Produced by Ion Beam Enhanced Etching. Frank Schrempel<sup>1</sup>, Thomas Gischkat¹, Holger Hartung², Ernst-Bernhard Kley² and Werner Wesch¹; ¹Institute of Solid State Physics,

Friedrich-Schiller-University, Jena, Germany; <sup>2</sup>Institute of Applied Physics, Friedrich-Schiller-University, Jena, Germany.

High aspect ratio micro- and nanostructures in electro-optic materials are of great interest due to the possibility to create ridge waveguides and crystals with photonic band gap structures. Photonic crystals are very attractive structures which show strong confinement of light and allow the generation of ultra-compact devices with low optical losses. They are based on a nanometric periodic arrangement of adjacent regions with strong different refractive indices. A widely used electro-optic material for the fabrication of integrated optical devices is lithium niobate (LiNbO<sub>3</sub>). In order to create photonic crystals in LiNbO<sub>3</sub>, the material has to be removed periodically with dimensions in the order of the magnitude of the wavelength. The achievement of nanometric structures in LiNbO<sub>3</sub> is a challenging problem, because the material is very resistant to standard etching technologies. A promising method to overcome this problem is the use of ion irradiation in the context of ion-beam-enhanced wet etching. However, in order to apply this technique successfully, the correlation between radiation damage and etching behaviour must be understood and controlled. This work presents data of damage evolution, etching rates, contrast and aspect ratios as a function of irradiation and etching conditions, like ion species, ion energy, ion fluence, caustic as well as irradiation and etching temperature. Single crystals of x-cut LiNbO<sub>3</sub> were irradiated at room temperature and 15 K using He-, Nand Ar-ions with energies between 20 and 800 keV. For the He-irradiation at room temperature complete amorphization in the depth region of maximum nuclear energy deposition is reached at  $\sim$ 2.0 dpa (displacements per target atom). In contrast  $\sim$ 0.4 dpa are sufficient to amorphize the LiNbO<sub>3</sub> in the case of Ar-irradiation. Irradiations at 15 K reduce the number of displacements per atom necessary for amorphization to  $\sim 0.4$  dpa and  $\sim 0.2$  dpa for He- and Ar-irradiation, respectively. To study the etching behavior  $\sim 400$  nm thick amorphous layers were generated via irradiations with He- and Ar-ions of different energies and ion fluences. Etching was preferentially performed in a 4% HF-solution at 40 °C. Whereas the perfect crystal possesses negligible etching, the etching rate of the amorphized regions amounts to ~80 nm/min. The influence of the ion species, the ion fluence, the irradiation temperature and subsequent thermal treatment on damage and etching of LiNbO<sub>3</sub> will be discussed. It is shown that high aspect ratio microstructures can be obtained applying the optimal irradiation and etching conditions.

### 10:30 AM OO16.2

Crystallographic orientation control of nanocrystals formed by FIB under a bending strain state. <u>Masaki Kubota</u>, Timothy P. Halford, Yutaka Kamibayashi and Yakichi Higo; Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Japan.

In addition to the strengthening effects expected from the generation of nanocrystals (NC's) within MEMS materials there is also the potential to utilize the magnetic properties of orientated NC's for data storage purposes. In order to achieve this, however, the capability to form NC's orientated in the desired crystallographic direction is necessary. Oriented Ni NanoCrystals (NC's), with a grain size of approximately 10 nm, have been formed in Ni-P amorphous alloy thin film using Focused Ion Beam (FIB) irradiation from a Ga ion source, at a potential of 40kV. The NC's formed in the irradiated area demonstrate an orientation relationship, with {111} parallel to the irradiated plane and <110> parallel to the projected ion beam direction. The in-plane orientation of these NC's has been varied by changing the angle that the irradiation is applied at. This technique has, however, proven to be insufficient to control the out of plane orientation of these NC's. In order to control the out of plane orientation of produced NC's, irradiation by FIB has therefore been completed whilst the specimen material is subjected to varying bending strains. In this way, irradiation is applied to the tensile surface of the material prior to removal of the strain condition. The process has then been repeated on the opposing sample surface without applying an external strain. In this way material with differing NC orientations on its opposing faces has been produced in a controlled manner. Examination, by Transmission Electron Microscopy (TEM), of the nanocrystalline microstructures produced in this manner shows that the application of a strain of  $1.2 \times 10-3$ during irradiation is insufficient to alter the crystallographic orientation of the formed NC's from that occurring without applying an external strain. NC's produced at this strain level do however show an increase in average NC size, to as much as 30nm, as well as a lower percentage area of NC's being formed. When the strain upon the sample is increased to values in excess of 2.3 x 10-3 the selected area diffraction pattern reveals the NC orientation relationship to have {112} parallel to the irradiated plane, <110> parallel to the projected ion beam direction and <111> parallel to the applied stress field. In all of these cases, exposure to irradiation without an applied strain on the reverse side of the sample has been shown to generate NC's orientated as previously described. This suggests that the surface

normal orientation of these NC's has been controlled by the application of a stress field.

### 10:45 AM <u>OO16.3</u>

Surface Modification of Silicon Nano Mechanical Structures by Carbon Ion Implantation for Post-fabrication Transformation to Silicon Carbide. Dinesh K. Sood<sup>1</sup>, Kumar R. Virwani<sup>2</sup>, Ajay P. Malshe<sup>2</sup> and Robert G. Elliman<sup>3</sup>; <sup>1</sup>School of Electrical & Computer Engineering, RMIT University, Melbourne, Victoria, Australia; <sup>2</sup>Department of Mechanical Engineering, University of Arkansas, Fayetteville, Arkansas; <sup>3</sup>Department of Electronic Materials Engineering, Australian National University, Canberra ACT 0200, Australian Capital Territory, Australia.

For applications in harsh environments such as, for example, high temperature MEMS for aerospace temperature, pressure and gas sensors, MEMS and NEMS sensors and actuators for bio chemical and mechanical implantable devices, applicability of widely used silicon is limited due to its incompatible properties. Hence, the need for high temperature materials such as silicon carbide that can withstand in harsh environment is critical. However, unlike silicon, fabrication and processing infrastructure for fabrication of nano and micro mechanical structures from silicon carbide and related materials is relatively scanty. In this work, to overcome this limitation, we have explored a novel but simple process hierarchy, where one can take advantage of using silicon fabrication infrastructure in the first step to fabricate the desired MEMS and NEMS devices, followed by post-fabrication C+ implantation to selectively transform silicon - to - silicon carbide retaining mechanical integrity. In the presented research, nanometer sized silicon cantilever beams were first fabricated using silicon on insulator (SOI) wafers with a box thickness of 193nm. PMMA (poly methylmethaacrylate) and MAA (Methaacrylic Acid) were used as e-beam resists, to define the cantilever structures. The fabricated nanoscale silicon beams [ $\sim$ 3000 nm long, 250 nm wide and 193 nm thick] were ion implanted with C+ ions at 30keV energy and doses of 5X1017 ions/cm2, 2.5X1017 ions/cm2 and 1X1017 ions/cm2, or at  $15 \mathrm{keV}$  energy and doses of  $3 \mathrm{X} 1017$  ions/cm2 and  $1.5 \mathrm{X} 1017$  ions/cm2. These modified structures were then studied using an atomic force microscope, scanning electron microscopy and Rutherford backscattering and channeling. It is observed that there is controlled transformation of silicon -to- silicon carbide. In addition to change in the chemical properties, mechanical integrity is retained and some unique changes in the stress profile of the mechanical structures are observed. A tentative model is proposed to explain these observations. This exploration may have major implications for nano and micro electromechanical systems and related devices.

### 11:00 AM <u>OO16.4</u>

Enhanced Biocompatibility of GPC by Ion Implantation and Deposition. Robert L. Zimmerman<sup>1,3</sup>, I. Gurhan<sup>2</sup>, Sergey Sarkisov<sup>1</sup>, Claudiu Muntele<sup>1</sup>, Daryush ILA<sup>1</sup>, Marcello G. Rodrigues<sup>3</sup>, F. Ozdal-Kurt<sup>4</sup>, B. H. Sen<sup>5</sup> and A. Leslie Evelyn<sup>1</sup>; <sup>1</sup>Physics, Alabama A&M University, Normal, Alabama; <sup>2</sup>Faculty of Engineering, Ege University, Izmir, Turkey; <sup>3</sup>Department of Physics and Mathematics, University of Sao Paulo, Ribeirao Preto, Sao Paulo, Brazil; <sup>4</sup>Faculty of Science, Celal Bayar University, Manisa, Turkey; <sup>5</sup>Faculty of Dentistry, Ege University, Izmir, Turkey.

Biocompatible Glassy Polymeric Carbon (GPC) is used for artificial heart valves and in other biomedical applications. Although it is ideally suited for implants in the blood stream, tissue that normally forms around the moving parts of a GPC heart valve sometimes loses adhesion and creates embolisms downstream. We have shown that silver ion implantation or surface deposition inhibits cell growth on GPC, and that implantation of MeV oxygen ions enhances cell adhesion, both are desirable improvements on current GPC cardiac implants. In vitro biocompatibility tests have been carried out with model cell lines to demonstrate that MeV ion bombardment can favorably influence the surface of GPC for biomedical applications.

### 11:15 AM $\underline{\text{OO}16.5}$

Gold Cluster Formation: An Investigation of the Regime of Low Au Ion Energy (20 to 300 eV). Petra Reinke<sup>1</sup>, James M. Howe<sup>1</sup>, Santhana K. Eswaramoorthy<sup>1</sup>, Elsa Thune<sup>2</sup> and Michael Buettner<sup>3</sup>; <sup>1</sup>Dep. of Materials Science and Engineering, University of Virginia, Charlottesville, Virginia; <sup>2</sup>Physik, Universitaet Regensburg, Regensburg, Germany; <sup>3</sup>Insitut fuer Physik, Universitaet Basel, Basel, Switzerland.

Many of the established processes in the fabrication of thin films employ plasma or ion beam assisted techniques to control film composition and structure. These processes involve a wide range of reactive species, with various kinetic energies. The regime of ion energies above 300 eV is well understood and can be described in the framework of codes like TRIM which simulate the progression of the collision cascade. In the low energy regime the impinging ion can only displace a few atoms and is implanted in a depth of 1 to 5 atomic

layers. Although the extent of damage caused by these ions is small, it occurs in a section of the growing film, which is highly relevant to the growth process. To improve the understanding of the role of low energy ions we investigate the formation and growth of Au clusters on amorphous carbon substrates. The use of a mass selected ion beam facility allowed to control the ion energies and afforded a narrow energy distribution (about  $5~{\rm eV}$ ). The Au energy was adjusted between 320 and 20 eV, a substantial structural modification of the amorphous carbon substrate layer surface can be excluded. The films were subsequently characterized with TEM, photoelectron spectroscopy (PES), and Rutherford Backscattering (Au inventory). The study concentrated on the effect of Au impact energy and subsequent annealing on the cluster size distributions. TEM and PES are established successfully as complementary methods to determine cluster sizes in the regime above and below 1.5 nm, respectively. The ion energy determines Au cluster size distribution and while 20 eV Au ions form relatively large clusters with an average diameter of about 4nm, the cluster size decreases by an order of magnitude for 320 eV Au . The size distributions are always narrow and thus a superior control of the cluster size can be achieved. The interplay between surface and bulk mobilities of Au atoms, the local environment during nucleation and differences in nuclei stability at the surface and in the bulk determine the cluster formation. The ion energy controls the primary bonding environment of the Au atoms and a qualitative model can be used to describe the link between ion energy and final cluster size distribution. When these clusters are exposed to elevated temperatures (<600C) the cluster growth adopts a more complicated pattern, which results from the interplay between surface and volume diffusivity and the presence of a Au atom reservoir below the surface. A simple model using the particle fluxes within the system can be used to describe the modification of cluster size distributions with temperature. These results indicate new pathways to the control of cluster size and impact on the interpretation and control of thin film deposition.

> SESSION OO17: Magnetic Materials: Material Synthesis for Spintronics, Sensors and Data Storage Chair: William Weber Thursday Afternoon, December 1, 2005 Commonwealth (Sheraton)

### 1:30 PM \*OO17.1

Ion Processing of Magnetic Materials. S. P. Wong, <sup>1</sup>Dept of Electronic Engineering, Chinese University of Hong Kong, Shatin, Hong Kong; <sup>2</sup>Materials Science and Technology Research Center, Chinese University of Hong Kong, Shatin, Hong Kong.

In the past decade, the magnetic storage industry has witnessed a very significant progress in terms of the rapid increase in storage areal density. The compound annual growth rate has reached 100% since the late 90/s. In this work, we shall present some of our explorations in applying ion beam techniques for the synthesis and processing of high anisotropy magnetic nano-composite thin films promising for these high density magnetic recording media applications. Several approaches we attempted will be described. The first is the direct synthesis of CoPt or FePt nanocrystals in a dielectric matrix such as SiO<sub>2</sub> by co-implantation of Pt and Co or Fe ions. In the second approach, a multi-source pulsed filtered vacuum arc deposition system was developed and applied to the synthesis of magnetic nano-composite thin films. These magnetic films can also be prepared by first producing a multilayer structure using the arc deposition system followed by thermal and ion beam treatments. This multi-layer deposition plus post treatment approach was demonstrated to have added advantages over the co-deposition approach. For examples, the ordering temperature of the high anisotropy phase can be lowered and it allows for more degrees of freedom to achieve the desired combination of grain size and coercivity values for practical applications. The relations among the microstructures, magnetic properties and the processing conditions will be analyzed and discussed. This work is partially supported by the Research Grants Council of Hong Kong SAR (ref. no.: CityU 2/04C) and the Germany-Hong Kong Joint Research Scheme sponsored by RGC of Hong Kong SAR and DAAD of Germany (ref. no.: G\_HK017/04).

### 2:00 PM OO17.2

Magnetic Behavior of Ion Irradiation Induced Laterally Patterned Buried Magnetic Layers. Bhupendra N. Dev<sup>1</sup>, O. M. Liedke<sup>1</sup>, K. Potzger<sup>1</sup>, J. Fassbender<sup>1</sup>, L. Bischoff<sup>1</sup>, R. Groetzschel<sup>1</sup>, F. Allenstein<sup>2</sup> and G. Beddies<sup>2</sup>; <sup>1</sup>Institute of Ion Beam Physics and Materials Research, Forschungszentrum Rossendorf, Dresden, Germany; <sup>2</sup>Technische Universitaet Chemnitz, Reichenhainer Strasse 70, 09126 Chemnitz, Germany.

Ion irradiation causes many interesting changes in magnetic behavior in layered systems. In multilayers showing giant magnetoresistance

and antiferromagnetic coupling, e.g. Co/Cu multilayers, ion irradiation causes significant suppression of antiferromagnetic coupling [1]. Ion irradiation induced magnetization reorientation occurs in Co/Pt multilayers [2]. Even ferromagnetic behavior can be obtained by ion irradiation of nonmagnetic multilayers like Pt/C with Fe impurities [3]. These multilayers are patterned along the depth of the sample. In the present work we have undertaken the studies of magnetic behavior of systems patterened laterally. We have chosen  $\mathrm{Si}(5~\mathrm{nm})/\mathrm{Ni}(t)/\mathrm{Si}$  samples (t = 5-10 nm) grown by electron beam evaporation under ultrahigh vacuum condition. Magneto-optic Kerr effect (MOKE) measurements show no ferromagnetic behavior for the t = 5 nm sample, as the buried Ni layer tends to be nearly a two-dimensional system. The sample with t = 10 nm shows ferromagnetism with a coercive field of 55 Oe. This sample was then patterned laterally with a focussed ion beam where the irradiated stripes undergo Ni-Si mixing destroying ferromagnetism in the Ni layer under the irradiated stripes. Thus, over the whole depth of the Ni layer a lateral nanoscale periodic structure of Ni/Ni(x)Si(1-x)/ is formed. Magnetic measurements have been performed as a function of width of the unirradiated Ni strips and their inter-strip separation. The results of magnetic measurements on such systems for Si/Ni/Si and Si/Co/Si by MOKE, MOKE microscopy and magnetic force microscopy will be presented. [1] M. Cai, T. Veres, F. Schiettekatte, S. Roorda and R.W. Cochrane, J. Appl. Phys. 95 (2004) 2006. [2] D. Weller, J.E.E. Baglin, A.J. Kellock, K.A. Hannibal, M.F. Toney, G. Kusinski, S. Lang, L. Folks, M.E. Best and B.D. Terris, J. Appl. Phys. 87 (2000) 5768. [3] B.N. Dev, S. Bera, B. Satpati, D.K. Goswami, K. Bhattacharjee, P.V. Satyam, K. Yamashita, O.M. Liedke, K. Potzger, J. Fassbender, F. Eichhorn and R. Groetzschel, "Nonmagnetic to magnetic nanostructures via ion irradiation" (Invited talk in the 31st International Conference on Micro- and Nano-Engineering, Vienna, Austria, 19-22 September, 2005).

### 2:15 PM OO17.3

Ferromagnetic Mn-Implanted Si for Spintronic Applications.

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Integrating spintronic device concepts with silicon may enable new possibilities for fabrication and integration with conventional devices. Ion implantation has been demonstrated as an attractive means in the synthesis of ferromagnetic group-IV semiconductors. In addition, theoretical calculations have predicted ferromagnetic ordering in Mn-doped group-IV semiconductors. This potential has motivated the search for a Si-based ferromagnetic semiconductor. We demonstrate that p-type and n-type Si (111) wafers can be made ferromagnetic above room temperature through Mn-ion implantation. 300-keV Mn+ ions were implanted at doses of (1-10)X1015 cm-2 reaching peak concentrations of (0.1-0.8) at.% as measured through SIMS profiling. The samples were held at 350 C during implantation to avoid amorphization. Ferromagnetic hysteresis loops were obtained using a SQUID magnetometer at temperature of (10-300) K, yielding a saturation magnetization of 0.1-0.7 emu/g-sample. The saturation magnetization increased by  ${\sim}2$  times after annealing at 800 C for 5 min. The Curie temperature is found >400 K with carrier concentration dependence. The crystal structure has been investigated by RBS in the channeling mode and by TEM cross-section images analysis. Here we report the effects of Mn concentration and post implant annealing on the strength of the ferromagnetism and on the crystal composition. These results will be discussed in comparison with other ion implanted or MBE grown group-IV ferromagnetic semiconductors.

### 2:30 PM Final Comments