

# SYMPOSIUM T

## Nanostructuring Materials with Energetic Beams

April 22 – 23, 2003

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\* Invited paper

**9:00 AM \*T1.1**

**NANOPORES AND NANOWIRES: A CHALLENGE FOR NEW PROPERTIES AND APPLICATIONS.** Roger Legras, Université Catholique de Louvain, Unité de Physique et de Chimie des Hauts Polymères

Filling the nanosized holes of polymer membranes with various combinations of metals or conductive polymers can produce nanocomposite materials with tailor-made electrical, magnetic or chemical properties. As sophisticated filter devices, these have many potential applications such as use in shielding microwave ovens and mobile phones in artificial noses or in lab-on-a-chip. Nanocomposites are materials which contain one or more components in shapes such as cylinders, rods or networks with nano-size dimensions. The enormous versatility of possible combinations of such composites, which can have both electrical conductivity and non-conductivity components, offers a real possibility of obtaining materials that combine metal-like properties with non-metallic characteristics. In addition, changes in the features and type of nanostructured component can substantially affect the composite's electrical, magnetic and chemical properties. If a thin polymer film such as polycarbonate is bombarded with energetic heavy ions (accelerated by a cyclotron), holes are burned out and after track-etching a membrane is produced. As filters, most membranes rely on several different mechanisms to enhance contaminant removal. Track-etched membranes, in contrast, capture particles by simple, direct sieving, so particles larger than the pore size cannot pass through. The existing production technology for these membranes has been significantly enhanced to allow the reliable and reproducible manufacture of nanoporous membranes with nanosized holes. They are currently used within the ultra-filtration market as molecular filters for biological applications such as water purification. Now these polymer membranes can be made with a well-controlled pore size and shape in the form of regular, periodic arrays. The nanopores can be filled with another organic or inorganic material, such as a polymer or metal. If a different material, such as an electrically conductive polymer, is deposited in the holes, nanotubes with enhanced conductivity are obtained. Filling the nanoholes with a magnetic material such as iron, cobalt or nickel or a combination of these metals by electrochemical deposition, produces a membrane with nanoparticles or wires of either one metal or with multi-layers of different metals. Various combinations of polymers and metals are produced and characterised with the aim of using such membranes to design devices in which the electrical and magnetic properties can be studied, controlled and so optimised. The nanoparticles produced by using these templates take the form of rods or tubes. The metal nanowires embedded in these polymer films are regularly spaced and densely packed. Since the volume and degree of their electrical insulation and magnetism is controllable and can be fine-tuned, they have good potential for use in high-density data storage. They also absorb certain wavelengths of electromagnetic radiation similar to those used in microwave ovens and communications. Consequently, potential applications include sophisticated devices such as microwave filters for making shields for microwave ovens and mobile phones. The deposition of conductive polymer nanotubes in the membrane will lead to the preparation of chemical detectors (artificial nose) and biosensors.

**9:30 AM \*T1.2**

**NANO-WIRE FIELD-EFFECT TRANSISTOR IN ETCHED ION TRACKS OF FLEXIBLE MATERIALS.** Jie Chen, Siegfried Klaumünzer and Rolf Könenkamp, Hahn-Meitner Institut, Berlin, GERMANY.

Fast heavy ions generate continuous-amorphised cylindrical tracks in polymers. Chemical etching rates in this degraded material are typically several orders of magnitude larger than in the surrounding intact material. As a consequence deep narrow cylinders with diameters in the range from 30 to 3000 nm and large aspect ratios can be prepared in thin self-supporting polymer foils. Semiconductor deposition on etched ion tracks of flexible substrates is an area of intense research activity since it will open new applications for a host of consumer products. We present detailed results on the preparation of nano wire field effect transistor and first results of their operation. In this work we have pursued a route that promises devices which are robustly embedded in a flexible matrix, and whose dimensions may be reduced well below the 100 nm limit. Our nano-wire transistors are embedded in sandwich flexible foils with one gate, one source and one drain. The advantages of our device are (1) a very high packing density which can be obtained without nano-lithography, and (2) a robust structure in which most of the mechanical forces due to

bending, stretching and compression are taken up by the high flexibility of the plastic template rather than by the semiconductor device.

**10:30 AM T1.3**

**FORMATION OF WELL-DEFINED NANOCOLUMNS BY ION TRACKING LITHOGRAPHY.** T.E. Felter, Lawrence Livermore National Laboratory, Livermore, CA; R.G. Musket, Musket Consulting, El Dorado Hills, CA; J. Macaulay, Multibeam Systems Inc., Santa Clara, CA; and R.J. Contolini, Novellus Systems, Tualatin, OR.

Low dimensional systems on the nanometer scale afford a wealth of interesting possibilities including highly anisotropic behavior and quantum effects. Nanocolumns permit electrical and mechanical contact, yet benefit from two confined dimensions. This confinement leads to new optical, mechanical, electrical, chemical, and magnetic properties. We construct nanocolumn arrays with precise definition and independent control of diameter, length, orientation, areal density and composition so that geometry can be directly correlated to the quantum physical property of interest. The precision and control are products of the fabrication we use which starts with an ion of sufficient energy to "track" a dielectric such as a film applied uniformly onto a substrate. The energy loss of the ion alters chemical bonding in the dielectric along the ion's straight trajectory. A suitable etchant quickly dissolves the latent tracks leaving high aspect ratio holes of small diameter (~10nm) penetrating the film up to several microns thick. These small holes are interesting and useful in their own right and can be made to any desired size by continuing the etching process. Moreover, they serve as molds to be filled electrochemically. After this deposition, the mold material can be removed leaving the columns firmly attached to the substrate at the desired orientation. A variety of structures from simple to complex can be envisioned with these techniques. As examples, we have created arrays of Ni and of Pt nanocolumns (~60 nm diameter and ~600 nm long) oriented perpendicular to the substrate. The high aspect ratio and small diameter of the columns enables easy observation of quantum behavior, namely efficient electron field emission and Fowler Nordheim behavior. This work was performed under the auspices of the U.S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

**10:45 AM T1.4**

**ION BEAM SCULPTING OF COLLOIDAL NANOMATERIALS.** T. van Dillen,<sup>a</sup> M.J.A. de Dood,<sup>a</sup> C.M. van Kats,<sup>b</sup> D.L.J. Vossen,<sup>a,b</sup> D. Fific,<sup>a</sup> A. van Blaaderen,<sup>a,b</sup> P.R. Onck,<sup>c</sup> E. van der Giessen,<sup>c</sup> A. Polman<sup>a</sup>. <sup>a</sup>FOM-Institute AMOLF, Amsterdam, THE NETHERLANDS; <sup>b</sup>Debye Institute, Utrecht University, Utrecht, THE NETHERLANDS; <sup>c</sup>Groningen University, Groningen, THE NETHERLANDS.

Nanometer and micrometer-sized colloidal silica particles show a dramatic anisotropic plastic deformation upon MeV ion irradiation, changing their shape from being spherical to ellipsoidal. Nonspherical colloidal particles have a wide range of applications in nanophotonics, nanomagnetism and nanolithography, which leads to various questions about the physical origin of this deformation process. As part of the detailed study into this process, we have irradiated colloidal silica particles with 0.5-4 MeV Xe ions to ion fluences as high as  $10^{16}$  ions/cm<sup>2</sup> at various sample temperatures between 77 and 500 K. It is found that the deformation strongly depends on the ion fluence and energy, as well as on the sample temperature. Small colloids (40 nm diameter) show a smaller deformation rate than large colloids (1000 nm). Pre-annealed silica colloids (1020 K) show a three times lower deformation rate.

Silicon also deforms under ion irradiation: micron-sized crystalline Si (100) pillars, made by deep reactive ion etching and pre-amorphized with 3 MeV Xe, were irradiated with 30 MeV Cu ions and showed dramatic expansion perpendicular to the ion beam. In contrast, pillars that were not pre-amorphized did not show this lateral expansion. Supplementary insight is offered by a finite-element continuum model of the transient plastic flow during the thermal spikes caused by the incident ions. Our model consists of a viscoplastic cylinder in an elastic surrounding medium. We will compare our results with an analytical model for anisotropic deformation derived by Trinkaus et al. Finally we show that ion irradiation-induced deformation can be used to tune the shape of a colloidal mask for nanolithography. For colloids of 1400 nm in diameter, the mask opening can be continuously tuned over the range from 0 to 350 nm.

**11:00 AM T1.5**

**ANISOTROPIC DEFORMATION OF MESOPOROUS SILICA FILMS AND COLLOIDS.** A.M. Vredenberg, G. Sengers, A. van Blaaderen, Debye Institute, Utrecht University, NETHERLANDS; S. Eiden, University of Konstanz, GERMANY.

We have used 30 MeV Si irradiation to anisotropically modify the pore structure of mesoporous silica. Films or particles of this material are finding a wide range of applications in e.g. catalysis, (bio)sensing, (nano)electronics, (nano)photonics. This is due to the large control over the pore size (2-20 nm) and structure (lamellar, 2D hexagonal, 3D cubic) that is inherent to the synthesis, which is based on self-assembly of surfactant molecules. Further tuning the shape of the pores (e.g. ellipsoidal instead of cylindrical) would greatly increase the potential of mesoporous materials. The goal of this work was to investigate if this can be achieved by anisotropic deformation induced by swift heavy ion irradiation. We have irradiated (1) 800 nm thick films and (2) 1 mm spherical colloids of mesoporous silica with 30 MeV Si up to a dose of  $2 \times 10^{15}/\text{cm}^2$ . The colloids deform anisotropically (i.e. they flatten in the direction of the ion beam) without total collapse of the pore structure. The films densify (i.e. their thickness decreases) with a concurrent change in the ordering distance between horizontal pores in the vertical direction. Already at  $5 \times 10^{13}/\text{cm}^2$  a 10% change in the out-of-plane lattice parameter of the pore structure is observed with low angle XRD, indicating a change of cylindrical pore symmetry to an ellipsoidal one. We will present results of an XTEM study, and discuss the dependence on ion beam dose.

SESSION T2: FOCUSED ION BEAMS OF HIGH AND  
LOW ENERGY  
Tuesday Afternoon, April 22, 2003  
City (Argent)

**1:30 PM \*T2.1**

**PROTON BEAM NANO-MACHINING: END STATION DESIGN AND TESTING.** Jeroen A. van Kan, Andrew A. Bettioli, Frank Watt, Research Centre for Nuclear Microscopy, Physics Department, National University of Singapore, SINGAPORE.

A new nuclear nanoprobe facility has been developed. This facility is the first dedicated to proton beam micro- and nano- machining. The design and performance of the facility, which is optimized for direct-write lithography with 2-3 MeV protons, is described here. The proton beam micromachining (PBM) facility (or P-beam writer) utilizes the Oxford Microbeams high demagnification lenses (OM52) in a high excitation triplet configuration, operating at a reduced image distance to enhance the system demagnifications ( $60 \times 228$ ). The PBM chamber is equipped with an annular detector for Rutherford backscattering measurements, a secondary electron detector for electron imaging, and a pin diode for scanning transmission ion microscopy mapping. The sample is mounted on a computer controlled Burleigh Inchworm XYZ stage which has a travel of 25 mm for all axes with a 20 nm closed loop resolution. The system has been designed to be compatible with Si wafers up to  $6''$ . The first tests show that the proton beam can be focused down to  $35 \times 75 \text{ nm}^2$ , the best performance in the world so far. For PBM, the proton beam is focused and scanned over a suitable resist material. The penetrating power of the protons allows 3D structures of high aspect ratio to be produced directly. The laterally induced secondary electrons produced by the primary proton beam have a very short range, which depends on the proton beam energy, and is typically less than 10 nm. This property ensures even exposures with  $\approx \text{nm}$  smoothness. The first test structures will be presented, including line structures of 60nm width machined in a 2 micron thick PMMA layer on silicon. In addition, we will present preliminary results of the use of the PBM method in micro-fluidics (electrophoresis channels) and micro-photonics (micro-lenses, micro-waveguides and photonic band gap crystals).

**2:00 PM T2.2**

**GENERATING NANOSCALE ARRAYS USING SELF-ORGANIZED POROUS-ALUMINA ION IMPLANTATION MASKS.** L.E. Rehn, P.M. Baldo, B.J. Kestel, J. Hiller, A.W. McCormick and R.C. Birtcher, Materials Science Division, Argonne National Laboratory, Argonne, IL.

Using two-step anodizing recipes available in the literature, we fabricated self-supporting ordered ion-implantation masks that are several  $\text{mm}^2$  in area and approximately  $2 \mu\text{m}$  thick. SEM micrographs reveal self-organized structures with straight open pores, 30-150 nm in diameter, which extend completely through the mask. As reported previously, the pore diameter and spacing depend critically upon the anodizing parameters, e.g., the type of acid and its molality, the applied voltage and the solution temperature. Ion-milling procedures were developed for opening the bottoms of the anodized pores. The resulting implantation masks appear quite robust during exposure to ion beams of 1-MeV He, Ne, and Kr. The steps necessary to fabricate the implantation masks, including opening the pores, will be described. Measurements of the angular dependence of the transmitted ion current will be reported; these results are consistent with the physical dimensions of the opened pores. Alignment

procedures will be discussed, and TEM images of implanted arrays will be shown. This research was supported by the U.S. Department of Energy, Office of Science, BES-Materials Science, under Contract No. W-31-109-Eng-38.

**2:15 PM T2.3**

**THERMAL STABILITY OF Pt NANOWIRES MANUFACTURED BY Ga<sup>+</sup> FOCUSED ION BEAM (FIB).** B.J. Inkson, Dept of Engineering Materials, Sheffield University, Sheffield, UNITED KINGDOM; G. Dehm, T. Wagner, MPI fuer Metallforschung, Stuttgart, GERMANY.

Pt nanowires have been produced by the deposition of Pt thin films in a commercial Ga focused ion beam (FIB) system, followed by cross-sectional sputtering to form electron transparent Pt nanowires. The thermal stability of FIB manufactured Pt wires on Ti has been investigated by in-situ thermal cycling in a TEM. The Pt wires recrystallize with heating in vacuum, and real-time videos of the in-situ nucleation, growth and coarsening of Pt nanocrystals are presented. The influence of Ga impurities in the Pt wires from the FIB processing is discussed.

**2:30 PM T2.4**

**ULTRA-RAPID NANO-SCALE PATTERNING OF PMMA USING A Ga<sup>+</sup> FOCUSED ION BEAM: TOPOGRAPHIC CONTROL AS A FUNCTION OF SPUTTERING TEMPERATURE.** Yajing Liu, Timothy Herlihy, Robert Hull, Department of Materials Science & Engineering, University of Virginia, Charlottesville, VA.

A 30 keV Ga<sup>+</sup> focused ion beam (FIB) is used to create nano-scale topographic patterns in Poly(methyl methacrylate)(PMMA) thin films at speeds up to  $5 \times 10^4$  features/second. The patterned features are about 60 nm in diameter and 5 nm deep using a 1pA beam with a diameter of 10nm. TEM observation of the irradiated material did not show material densification, implying that the material is removed rather than compacted. The material removal rate thus corresponds to about  $10^3 (\mu\text{m})^3/\text{nC}$  (or about  $10^4$  atoms/ion), orders of magnitude higher than typical physical sputtering rates (of order 1-10 sputtered atoms per incident ion) under these conditions. The effect of focused ion beam experimental parameters including incident angle, feature dwell time, ion current density and ion beam upon the material removal rate have been studied; under all examined conditions observed sputtering rates remain at least one-two orders of magnitude higher than typical values (i.e.  $0.1 - 1 (\mu\text{m})^3/\text{nC}$ ) with the substrate held at room temperature. A more significant effect was observed in varying the substrate temperature from  $-80$  to  $200^\circ\text{C}$ . When the substrate temperatures are lower than the glass transition temperature of the PMMA, depressed features (i.e. holes) are created with anomalously high sputtering yields. When the substrate temperatures are higher than the glass transition temperature of the PMMA, the ion-exposed regions then become raised hillocks with similar lateral dimensions to the holes created at lower temperatures. We speculate that ion-enhanced chemical modification and unzipping reactions are responsible for these complex topographies. In any event, these observations enable FIB-generated topographical patterns to be created at orders of magnitude faster rates than previously envisioned. Using this approach we have successfully demonstrated high-speed fabrication of topographic patterns as masters for micro-contact printing ( $\mu\text{CP}$ ), with resolution comparable to molding from conventional inorganic masters.

SESSION T3: NANOSTRUCTURES AND  
NANOSTRUCTURE SURFACES  
Tuesday Afternoon, April 22, 2003  
City (Argent)

**3:15 PM \*T3.1**

**NANOPHASE FORMATION UNDER ION BEAM: RADIATION EFFECTS VERSUS ION IMPLANTATION.** Lumin Wang, University of Michigan, Dept of Nuclear Engineering & Radiological Sciences, Ann Arbor, MI.

Nanostructures obtained with ion beam processing can be divided into two general types, i.e., those consist of significant amount implanted ion species and those do not. Nanostructures of the latter type are the results of radiation effects. Various kinds of nanostructures have been produced in ceramic or semiconducting materials under energetic beam irradiation as results of radiation effects rather than the implanted species. Irradiation of single crystalline materials with energetic particles can result in microstructures composed of nanophases (nanodots, nanolayers, nanofibers or a three-dimensional nanoparticle assembly) either with the original composition and crystal structure or with altered structures and compositions. The phenomenon has been observed with in situ transmission electron microscopy during ion beam

irradiation in all kinds of materials including metals, semiconductors and ceramics. Nanostructures may be induced by radiation effects with ion fluences several orders of magnitude lower than that typically required for ion implantation based method, thus resulting in much smaller local strain and stress. Examples of nanostructure formation with both methods in either crystalline or amorphous materials will be demonstrated. Plausible mechanisms responsible for the formation of radiation induced nanostructures include radiation-induced defect accumulation, recrystallization, structural order and disordering, chemical segregation and decomposition, etc. These mechanisms will be discussed with examples of experimental observations.

**3:45 PM T3.2**

Abstract Withdrawn.

**4:00 PM T3.3**

**ION-BOMBARDMENT INDUCED PATTERN TRANSFORMATION ON SELF-ORGANIZED SEMICONDUCTOR SURFACES.** S. Abermann, C. Teichert, C. Hofer, Institut für Physik, Montanuniversität Leoben, AUSTRIA; T. Bobek, S. Facsco, H. Kurz, Institut für Halbleitertechnik, RWTH Aachen, GERMANY; K. Lytovich, E. Kasper, Institut für Halbleitertechnik, Universität Stuttgart, GERMANY.

Self-organization of nanostructures in semiconductor heteroepitaxy has been established as an efficient route towards large-scale nanopatterning of surfaces [1]. Recently, it has been demonstrated that ion erosion of compound semiconductors may also result in well ordered dot patterns [2]. Here we use atomic-force microscopy to explore the ion bombardment induced morphology evolution of self-organized SiGe films grown on Si(001) under subsequent ion bombardment. Whereas in most of the cases a smoothening of the surface is observed, for a checkerboard array of {105} pyramids and pits [3] the nanopattern could indeed be transformed into the Si substrate. The details of the pattern transition and its dependence on ion energy are discussed in the framework of current theories of ion bombardment induced pattern formation. Research supported by the FWF, Austria (No. P14009-TPH). [1] C. Teichert, Phys. Rep. 365, 335 (2002) [2] S. Facsco, et al. Science 285, 1551 (1999); F. Frost, et al., Phys. Rev. Lett. 85, 4116 (2000) [3] C. Teichert et al., Thin Solid Films 380, 28 (2000).

**4:15 PM T3.4**

**NANOFABRICATION AND CHARACTERIZATION OF DIAMOND PROBES.** E. Kneedler, Y.C. Wang, T. Naef, R. Linder, and S. Ningen, FEI Company; P. Deshayé, Norsam Technologies.

The physical properties of the probe are integral to scanning probe microscopy (SPM). In metrology applications, the geometry and stiffness are particularly important. In many cases the tip-sample interface affects the measurement as well. Diamond is a favorable material for SPM probes because of its high stiffness and wear-resistance. Furthermore the chemically inert surface is usually desirable to minimize tip-sample interaction. To achieve the sharp geometries needed for high-aspect measurements, focused ion beam (FIB) milling is often the preferred method for diamond. However, the FIB process leaves a damage layer consisting primarily of amorphous carbon, compromising the probes stiffness and wear-resistance, and altering its surface chemistry. Removing the damage layer enables a new process for producing pure nano-scale diamond structures of practically any geometry. Nitric acid etch has previously been reported for top-down FIB-milled diamonds used for conduction SPM[1]. Reactive oxygen plasma etching offers an alternative removal method with potentially greater control. We present transmission electron microscopy and SPM data characterizing the performance of untreated and plasma-etched FIB-milled diamond tips. These tips represent a more complex geometry necessary for critical dimension metrology.

[1] A. Olbrich et al, J. Vac. Sci. Technol. B 17(4), Jul/Aug 1999, p. 1570.

SESSION T4: POSTER SESSION  
ION- AND ELECTRON-BEAM INDUCED  
NANOSTRUCTURES  
Tuesday Evening, April 22, 2003  
8:00 PM  
Golden Gate (Marriott)

**T4.1**

Abstract Withdrawn.

**T4.2**

**POTENTIALITIES OF FORMATION OF ANNULAR AND TUBULAR ARRAYS OF NANOPRECIPITATES IN THERMAL SPIKES FROM SWIFT HEAVY IONS.** Alexander E. Volkov, Michael V. Sorokin, Denis N. Korolev, Institute of General and Nuclear

Physics, Russian Research Centre, Kurchatov Institute, Moscow, RUSSIA.

Decay of solid solutions of impurity atoms in metals [1] and dielectrics [2] irradiated with swift heavy ions (SHI,  $E > 1 \text{ MeV/amu}$ ) results in nanosize precipitate nucleation, which correlates with the value of the electronic energy loss of the penetrating ions. For such high energies of ions the primary damage occurs mainly in the electronic subsystem of material and the damage produced by elastic recoils is of secondary importance. This effect manifests a new possibility of the monitoring of the precipitate formation and can be interesting as some alternative in manufacturing of low-dimension nanostructures. Subsequent energy transfer from the excited electrons to target atoms can result in subnanosecond temperature increase (thermal spike) at the surface and in the bulk of unirradiated material in nanometric region the vicinity SHI trajectory. Temperature dependence of the nucleation times of precipitates and islands has generally an abrupt minimum at the characteristic temperatures depending on the system parameters. A chance for the precipitation appears when the material/surface heating in SHI track results in those temperature values, where the nucleation time becomes shorter than the track cooling down time. We indicate the values of parameters of matrixes, surfaces, impurity atoms, projectiles, and irradiation conditions favorable for precipitations in the bulk and on the surface caused by SHI. It is shown that the region, where the maximum nucleation rates are possible, can be shifted from the track center. Initial inhomogeneity may result in appearance of nanosize tubular heterostructures along the projectile trajectories and/or ring-shaped nanoisland distribution on the surface near ion inlets. 1. Barbu A., Pareige P., Jacquet V., Nuclear Instr.Meth. B146, 278 (1998) 2. Valentin E., Bernas H., Ricolleau C., Creuzet F., Phys. Rev. Lett. 86 99 (2001).

**T4.3**

**MILLING, GROWING AND MOLDING WITH A FOCUSED ION BEAM.** Paul F.A. Alkemade, Valerie G.M. Sivel, Henny W. Zandbergen, Delft Univ of Technology, Dept of Nanoscience, Delft, THE NETHERLANDS.

Increasing knowledge of the physics and chemistry of materials at sub-micrometer length scales and rapid instrumental developments drive developments in nanotechnology. Nowadays charged particle beams of nanometer widths are available for shaping materials. The physical processes that underlie this technique are ion milling, beam-assisted growth, and ion-beam molding. In this presentation I will elaborate the interplay of ion-beam milling, ion-assisted growth and molding. Besides sputtering and ion-assisted decomposition, crucial aspects are defect production, amorphisation, and viscous flow. To illustrate these processes, I will show examples of nanostructures shaped with a focused Ga<sup>+</sup> beam in a dual-beam FIB instrument. In particular, I will show that there is not a direct one-to-one relation between processing and shape. In order to form distinct nanostructures, one must make clever use of the physics.

**T4.4**

**ION INDUCED DISPLACEMENT OF GOLD NANOPARTICLES IN CORE-SHELL COLLOIDS.** Sjoerd Roorda<sup>a,b</sup>, Teun van Dillen<sup>a</sup>, Joan Penninkhof<sup>d</sup>, Bart Kooi<sup>c</sup>, Jeff de Hosson<sup>e</sup>, Christina Graif<sup>d</sup>, Alfons van Blaaderen<sup>a,d</sup>, Albert Polman<sup>a</sup>, <sup>a</sup>FOM-Institute for Atomic and Molecular Physics, Amsterdam, THE NETHERLANDS; <sup>b</sup>Université de Montreal, GCM, Département de physique, Montreal, CANADA; <sup>c</sup>University of Groningen, Groningen, THE NETHERLANDS; <sup>d</sup>Utrecht University, Utrecht, THE NETHERLANDS.

It is well known that irradiation of amorphous materials with swift heavy ions leads to shape deformations known as "hammering", a mechanism that has been attributed to Coulomb explosions, viscoelasticity, or momentum transfer. Distinguishing between the different alternative explanations is not clear-cut because the predicted dependence of the deformation on projectile mass, energy, and incident angle is quite similar for each model. In this work, core-shell colloidal particles, were irradiated with 30 MeV heavy ions. The core-shell colloids consisted of a 15 nm diameter gold core surrounded by a silica shell of typically 70 nm thickness. The gold core, which normally is located at the centre of the particle, has moved to a new position downstream from its original position. This movement can be as large as 15 nm and is not due to surface sputtering, creep, or flow of the silica, although these phenomena do occur to some extent. The shift in position of the gold core is always in the direction of the ion beam and attributed to the force exerted by the ions on the gold core as they slow down, thus providing support for the momentum transfer model.

**T4.5**

Abstract Withdrawn.

**T4.6**

1/f NOISE AND ELECTRICAL TRANSPORT STUDIES ON

200MeV Ag ION IRRADIATED  $\text{La}_{0.7}\text{Ce}_{0.3}\text{MnO}_3$  THIN FILMS. R.J. Choudhary and Ravi Kumar, Materials Science Division, Nuclear Science Centre, New Delhi, INDIA; Shahid Husain and J.P. Srivastava, Department of Physics, Aligarh Muslim University, Aligarh, INDIA; S.I. Patil, Department of Physics, Pune University, Pune, INDIA; S.P. Sanyal, Department of Physics, Brakhtullah University, Bhopal, INDIA.

The effect of 200 MeV Ag ion irradiation on the electrical transport and  $1/f$  conduction noise properties of pulsed laser deposited epitaxial thin films of  $\text{La}_{0.7}\text{Ce}_{0.3}\text{MnO}_3$  have been studied. The electrical transport measurements show sharp metal-insulator transition at 282K and magneto resistance around 30% at 1T magnetic field in the pristine film. The well characterized thin films were irradiated with 200 MeV Ag ion for various values of fluence in the range  $5 \times 10^{10}$  to  $1 \times 10^{12}$  ions/cm<sup>2</sup> using 15UD Pelletron Accelerator at Nuclear Science Centre, New Delhi. The electrical transport and the conduction noise as a function of temperature and frequency of unirradiated and irradiated thin films have been studied. The spectral density  $S_v$  (V<sup>2</sup>/Hz) of conduction noise show  $1/f$  dependence throughout the temperature range in the low frequency range upto 100Hz for unirradiated as well as irradiated samples. The systematic variation of transition temperature and resistivity has been observed as a function of ion fluence. For fluence value  $5 \times 10^{10}$  ions/cm<sup>2</sup> the transition temperature is increased to 294K and with the further irradiation it decreased and remain higher than the unirradiated sample upto the fluence of  $5 \times 10^{11}$  ions/cm<sup>2</sup>. The  $S_v$  (V<sup>2</sup>/Hz) of irradiated thin films is an order of magnitude higher than the unirradiated thin film. Whereas, the temperature coefficient of resistivity (TCR) values for irradiated films are higher by factor of 2 than that of the unirradiated thin film. The observed modifications in the noise properties of the irradiated thin films are consistent with the general expectation concerning the effect of swift heavy ion induced lattice defects.

**T4.7**  
ELECTRON BEAM INDUCED EFFECTS IN COPPER DOPED CHALCOGENIDE THIN FILMS. J.S. Romero and A.G. Fitzgerald, Carnegie Laboratory of Physics, Department of Electronic Engineering and Physics, University of Dundee, Scotland, UNITED KINGDOM.

Copper migration and surface expansion of irradiated areas is observed in copper coated amorphous germanium based chalcogenide thin films when an electron beam is focused in pulsed or continuous operation on the surface of these bilayers. Both phenomena can be explained using a simple model in which the population of D-centers is considered to increase upon electron irradiation. The increase in the population of D-centers is envisaged as a result of the bond-breaking nature of electron radiation and the constant presence of negative charge in the irradiated regions. Expansion of the surface ranging between 30-40% of film thickness has been achieved and dry patterning in one step of features with micron and submicron dimensions on chalcogenide thin films has been demonstrated.

**T4.8**  
CHEMICAL ENHANCED ELECTRON BEAM INDUCED DIRECT PATTERNING. Jianhua Wang, Kunming University of Science and Technology, Kunming, Yunnan, P.R. CHINA; D.P. Griffis, R. Garcia, P.E. Russell, Analytical Instrumentation Facility, North Carolina State University, Raleigh, NC.

Maskless or direct patterning of electronic material is an interesting topic when considering structures with nanometer sized feature. E-beam is also a promising tool for mask repairing to remove opaque defects. This work demonstrates the ability of electron beam direct writing on silicon oxide, Cr film and TaN film. With the help of XeF<sub>2</sub>, well defined pattern and profile can be obtained on these materials used for mask making in semiconductor industry. The writing process was revealed by controlled dose and after AFM images. Influence of electron beam energy, beam current, and gas supply on patterning rate was studied. Creature depth increases linearly with dose. Electron beam current and gas supply show saturation effect. Electron beam with energy of 5kv is more efficient on SiO<sub>2</sub>, compared to 20kv. But for Cr film, it is opposite.

**T4.9**  
DISCOVERY OF NANOBOLDERS AT THE SURFACE OF ELECTRON BEAM ANNEALED LOW-ENERGY CARBON IMPLANTED SILICON. Andreas Markwitz and W.J. Trompeter, Rafter Research Centre, Institute of Geological and Nuclear Sciences, Lower Hutt, NEW ZEALAND; Horst Baumann, Institute for Nuclear Physics, J.W. Goethe University, GERMANY.

The combination of accelerator based low-energy ion implantation and rapid thermal electron beam annealing (EB-RTA) under high vacuum conditions can lead to the formation of nanostructured surfaces and thin film growth. Previously, <sup>15</sup>N and <sup>22</sup>Ne ions were implanted in the energy range of 5 to 20 keV into crystalline silicon at room

temperature to create new surfaces with distinct properties. To form these nanostructured surfaces and thin films, a 20 keV electron beam was used as the heating device. The electron beam was raster scanned over the implanted regions using annealing parameters set-up by a computer controlled feedback system. The specimens were annealed for a few seconds at typically 1000°C (temperature gradient 5°C/s) under high vacuum conditions. Depending on the ion fluence used in the implantation process, characteristically different structures were formed with implanting gaseous ions. In this contribution, we report for the first time, high dose 10 keV low-energy implantations of carbon (<sup>13</sup>C) into crystalline silicon that have been subsequently annealed with EB-RTA. Atomic force microscopy (AFM) of the implanted regions revealed the appearance of nanoboulders at the surface of the specimens after annealing. The very well spatially separated nanoboulders have a height of up to 150 nm. Additionally, an elevated ring surrounding the implanted area was discovered. Ion beam analysis (IBA) measurements using the highly sensitive <sup>13</sup>C(d,p)<sup>14</sup>C reaction at 920 keV revealed that the implanted carbon is still present after the annealing process. Selected AFM and IBA measurements are presented providing evidence for the discovery of the nanoboulders on the surface of implanted and annealed silicon substrates.

**T4.10**  
SELECTIVE NANOSCALE ELECTRON BEAM STIMULATED ETCHING. P.D. Rack, S. Randolph, J. Kim, J. Fowlkes, Y. Choi, and David C. Joy<sup>†</sup>, Department of Materials Science and Engineering, <sup>†</sup>Department of Biochemistry and Cellular and Molecular Microbiology, The University of Tennessee, Knoxville, TN.

The ability to manipulate materials at the nanoscale is critical for the nanotechnology revolution that is occurring. Current techniques to selectively deposit or etch microscopic features utilize ion beam deposition and etching, laser ablative etching using far field and near field optics, and mechanical abrasion using a fine microtip. As an alternative approach to these techniques we are exploring nanoscale electron stimulated deposition and etching. In this paper we will focus on our recent progress on nanoscale electron-stimulated etching. The experimental set-up consists of a scanning electron microscope modified with a gas delivery system. To date we have etched a variety of materials (Si, SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>, Ta, TaN, W, Al, Cr, C, Cu, low dielectric constant SILK and Black Diamond, and photoresist) using fluorine-based chemistries. Beam energy studies of silicon etching reveals increased etching yield and an increase in the effective beam size at lower beam energies. These results are in qualitative agreement with the increased inelastic scattering cross-sections for dissociation and ionization at lower beam energy. Silicon dioxide trends are similar, however the process is believed to be facilitated by an oxygen electron stimulated desorption process. In this paper selective etching results will be presented and the relevant electron-solid, electron-gas, and gas-solid interactions will be discussed.

**T4.11**  
NANOMETER SCALE STRUCTURES GENERATED BY COMBINED CHEMICAL AND RADIATIVE EXPOSURE OF SOLID SURFACES. Tom Dickinson, Khin Nwe, and Steve Langford, Dept. of Physics, Washington State University, Pullman, WA.

Novel nanometer scale structures on insulating inorganic materials with wide bandgaps can be generated by simultaneous exposure with laser and electron beams plus reactive gases. Here we present our current research on the mechanisms involved in modification of surfaces of ionic crystals following exposure to UV excimer laser light, keV electron beams, and relatively low pressures of water vapor. Dramatic synergisms are observed due to bond weakening which arises from localized chemisorption. We are able to modify single crystal surfaces of alkali halides and calcium phosphates/carbonates either at the single atomic layer level or, with higher intensities, at the level of several microns. Arrays of cones and fractal structures with unique optical properties can readily be generated. Mechanisms for the material removal, redeposition, and restructuring will be presented. Finally, we show that in the presence of water vapor, the rates of both laser and electron beam induced decomposition/desorption increase by as much as an order of magnitude. We present evidence that this is a highly localized, defect mediated mechanism. We suggest that point defect clusters lead to atomic dimension step structures (e.g., kinks) that peel off due to bond weakening from sorbed water derived radicals. Supporting evidence from spectroscopic and microscopic data on exposed surfaces will be presented. The consequences of irradiating hydrated crystals will also be discussed.

**T4.12**  
NANOSTRUCTURE OF GOLD NANOWIRE FORMED BY ELECTRON BEAM IRRADIATION IN HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPE. Yu Zhu, Kathleen Dunn, and Alain E. Kaloyeros, UAlbany Institute for Materials and School of NanoSciences and NanoEngineering, University at Albany - SUNY, Albany, NY.

In this study, self-supporting nanowires were fabricated from electron-transparent gold films irradiated in a JEOL 2010F high-resolution transmission electron microscope (HRTEM). The gold films were prepared either by thinning a 100 nm evaporated gold film to 5 nm via low-angle sputtering with 2 keV Ar<sup>+</sup> ions while cooling the specimen to 77 K, or by evaporating a 5 nm gold film on a copper grid coated with amorphous carbon membrane. Both approaches resulted in films of suitable thickness for high-resolution imaging and subsequent melting by the electron beam in situ. To accomplish this, the films were subjected to high current densities (~600 A/cm<sup>2</sup>) by converging the electron beam to a diameter of ~10 nm for brief intervals in order to locally melt the gold film. Images were acquired between irradiations with a diverged electron beam. Strategic placement of such holes resulted in the formation of self-supporting bridges of gold within one grain or between two grains. Wires with diameters less than 10 nm and aspect ratios of 1-3 were fabricated. Bridges which were longer or had higher aspect ratios (length-to-width) were subject to amorphization and breakage. The orientation of the crystal planes within the bridges appeared to rotate relative to the axis of the wire, suggesting that there may be a preferred low-energy orientation for nanowires.

**T4.13**  
SUB-MICRON MAGNETIC NANOSTRUCTURE PRODUCED BY ELECTRON BEAM DIRECT WRITING ON CoC AND Co(TaC) SYSTEM. **Y. Zhao**, Data Storage Institute, SINGAPORE; ISML, ECE Department, National University of Singapore, SINGAPORE; T.J. Zhou, Data Storage Institute, SINGAPORE; J.P. Wang, Department of Electrical and Computer Engineering, MINT, University of Minnesota, Minneapolis, MN; T.C. Chong, Data Storage Institute, SINGAPORE; ISML, ECE Department, National University of Singapore, SINGAPORE.

We present a novel method to produce magnetic nano-structures by E-beam radiation induced nano-scale phase transition. Non-magnetic CoC and Co(TaC) films were patterned by electron-beam direct writing and the 250nm magnetic dot/line array were produced. This method of magnetic patterning has the potential application for nanoscale solid magnetic devices. The CoC films with thickness of 30nm and Co concentration of 40-80at.% were co-sputtered under room temperature. The magnetic measurements revealed that the films with the cobalt concentration less than 60at.% were non-magnetic. After annealing under 400°C for two hours, both Hc and Ms of all the films increased significantly and the highest coercivity reached 560 Oe when Co content equated to 60 at.%(Fig.1). However, such coercivity is somewhat low for applications in magnetic recording. Ta was selected to improve the magnetic properties of the films because it can enhance the segregation of Co and C. It was found that the film with 3% Ta doping could obtain the coercivity of 822 Oe after annealing, which was 45% higher than those without Ta doping(Fig.2.). Magnetic patterning of the Co<sub>60</sub>C<sub>40</sub> and (Co<sub>60</sub>C<sub>40</sub>)<sub>97</sub>Ta<sub>3</sub> films was realized by subjecting them to electron beam radiation using a focused 30keV E-beam with a current of 7.1nA and a dwell time of 0.75~15.2 seconds. As shown in Fig.3, for CoC films, the magnetic dots weren't formed until the dwell time increased to 3.8 seconds, and the dot size increased almost linearly with the square root of the dwell time. While for Co(TaC) thin films, the weak magnetic line was produced with the dwell time of 0.75 seconds, and the tripe domain was very clear when the dwell time was 1.9 seconds. It is obvious that the time of magnetic patterning (Co<sub>60</sub>C<sub>40</sub>)<sub>97</sub>Ta<sub>3</sub> films is much shorter than that of patterning Co<sub>60</sub>C<sub>40</sub> films thus more practicable in use.

SESSION T5: POSTER SESSION  
NANOTUBES AND NANOCRYSTALS  
Tuesday Evening, April 22, 2003  
8:00 PM  
Golden Gate (Marriott)

**T5.1**  
ATOMISTIC SIMULATION OF RADIATION DAMAGE TO CARBON NANOTUBE. **Xiumei Wang**, Fuzhai Cui, Dept of Materials Science and Engineering, Tsinghua Univ, Beijing, CHINA.

Damaging carbon nanotube upon energetic irradiation has been modeled with molecular-dynamics simulations. The angular dependence of the threshold energy of the primary knock-on atom (PKA) escaping from the tube is investigated in the initial PKA directions spanning half space. The average value of the threshold energy is calculated to be 19.3 eV. The simulations provided a detailed picture of the damaging processes, in which four mechanisms were revealed. The interactions between carbon atoms are described with the Tersoff mode modified to match a screened Coulomb potential at short range.

**T5.2**  
Abstract Withdrawn.

**T5.3**  
Abstract Withdrawn.

**T5.4**  
CATALYST NANOPARTICLES SYNTHESIZED WITH ION IMPLANTATION IN SILICON AS TEMPLATE FOR CARBON NANOTUBES GROWTH. **Ananta Adhikari**, Dept of Physics, SUNY-Albany, NY; Mengbing Huang, Dept of Physics and School of NanoScience & NanoEngineering, SUNY-Albany, NY; Katharine Dovidenko, School of NanoScience & NanoEngineering, SUNY-Albany, NY; Bingqing Wei, Robert Vajtai, and Pulickel Ajayan, Dept of Materials Science and Engineering, RPI, Troy, NY.

The growth of carbon nanotubes (CNTs) in chemical vapor deposition (CVD) process strongly depends on the properties (size, location, density etc.) of catalysts particles and their interactions with substrates. In this work, we investigated the role of catalyst nanoparticles prepared by ion implantation in the CVD growth of CNTs. Nanoparticles of catalyst (nickel) embedded in silicon are prepared by Ni ions implantation followed by annealing. The implanted doses of Ni were varied between 1e15-1e17 cm-2. Post-implantation annealing between 600-1000°C produced nanometer crystallites of nickel silicide in Si. As the second step, CNTs were grown on these Si substrates containing Ni nanoparticles, by CVD through decomposition of hydrocarbon. The morphology and various properties of the resultant CNTs were examined with a number of techniques including transmission electron microscopy (TEM), scanning electron microscopy (SEM), x-ray diffraction (XRD) and x-ray photoelectron spectroscopy (XPS). We discuss the effects of different parameters, e.g., the size and density of nanoparticles and their embedding substrates, on the formation of CNTs.

**T5.5**  
HIGH-ENERGY PROTON IRRADIATION OF SINGLE-WALLED CARBON NANOTUBES. **Vladimir A. Basiuk**, Kensei Kobayashi, Takeo Kaneko, Yokohama Natl Univ, Fac of Engineering, Dept of Chemistry and Biotechnology, Yokohama, JAPAN; **Elena V. Basiuk** (Golovataya Dzhyμβeeva), **Jose M. Saniger**, Universidad Nacional Autonoma de Mexico, Centro de Ciencias Aplicadas y Desarrollo Tecnológico, Mexico D.F., MEXICO; **Yoichi Negishi**, Yokohama Natl Univ, Instrumental Analysis Center, Yokohama, JAPAN.

Effects of different kinds of irradiation on carbon nanotubes (CNTs) are of interest from two main points of view: (1) for modifying their physical and chemical properties, by introducing structural defects into the side walls; (2) for defining to what extent CNT materials can be used under harsh radiation environments, associated with their possible use for nuclear applications and under open space conditions. Effects of electron and heavy ion irradiation on CNTs already received attention in recent publications. The open space is an abundant source of one more important type of high-energy charged particles, namely protons. Their size is intermediate between that of electrons and of heavier positively charged ions, so that any information on CNTs irradiation with protons would be a significant addition to our knowledge of the whole spectrum of radiation effects on this important class of materials. We report on TEM observation of morphological changes in single-walled carbon nanotubes (SWNTs) caused by their bombardment with 3MeV protons, in a wide range of irradiation doses. Evident morphological alterations were observed at >0.1 mC, such as curving of the nanotubes, a loss of their straight shape and formation of short pieces. During further irradiation (doses approaching 1 mC) SWNTs degraded into an amorphous material, although a significant fraction of them was present as pieces of different lengths. This means a loss of the nanotube physical integrity, and thus puts an upper limit of proton irradiation doses where SWNT materials can be used, regardless of their particular application. This limit, however, is very high if to compare flux values in our case and for those typical for near-Earth space conditions during "quiet Sun" periods. Even upon a direct exposure to proton space irradiation, SWNTs will undergo no detectable alterations for practically unlimited time.

**T5.6**  
THE LUMINESCENT PROPERTIES OF THE ION-IMPLANTATION-FABRICATED SiO<sub>2</sub>:nc-Si NANOSTRUCTURES ANNEALED AT HIGH TEMPERATURES. **David Tetelbaum**, Oleg Gorshkov, Alexandr Kasatkin, Sergey Trushin, Alexey Mikhaylov, Physico-Technical Research Institute of University of Nizhny Novgorod, Nizhny Novgorod, RUSSIA; **Daria Gaponova**, Institute for Physics of Microstructures of Russian Academy of Science, Nizhny Novgorod, RUSSIA.

The SiO<sub>2</sub>:nc-Si structures are extensively investigated in connection with a good prospect of their utilization in the silicon-based optoelectronics. Until now, rather little attention is paid to the influence of the variation of the annealing temperatures  $T_{ann}$  (at high ones) on the luminescent properties of this system. It was recently revealed that even rather small variation of the  $T_{ann}$  (1000-1100°C) strongly affects the behavior of the SiO<sub>2</sub>:nc-Si system [1]. Here, the results of the further investigation of this item are presented. The elevation of the  $T_{ann}$  from 1000°C (2 h) to 1100°C and higher ones leads to a sharp lowering Si<sup>+</sup> dose for which the maximum photoluminescence (PL) intensity is reached. The behavior of the system at the additional doping with phosphorus is also different for  $T_{ann} = 1000^\circ\text{C}$  and  $T_{ann} \geq 1100^\circ\text{C}$ , respectively. In the first case, the strong phosphorus-induced increase of the PL intensity takes place, whereas in the last case such an increase is not observed. The pointed out difference is interpreted on the base of the model which takes into account the Ostwald ripening. Their role is sharply enhanced with the  $T_{ann}$  elevation. It is shown that the model permits to explain the experimental data using the reasonable values of the parameters characterizing the process of the Si nano-inclusions growth and ripening. This work is supported by INTAS 00-0064 grant and the Russian Ministry of Education program "Scientific researches of higher school in the priority fields of science and technique, subprogram 205". [1] S.A. Trushin, A.N. Mikhailov, D.I. Tetelbaum, O.N. Gorshkov, D.G. Revin, D.M. Gaponova. Surface and Coatings Technology. 2002. V.158-159. P.717-719.

**T5.7**  
EFFECT OF ANNEALING ON THE CHARACTERISTICS OF SILICON IMPLANTED NANO-TRAP MEMORY. T.S. Kalkur, Microelectronics Research Laboratories, University of Colorado, Colorado Springs, CO; Nick Cramer, Elliott Philofsky and Lee Kammerdiner, Applied Ceramics Research, Colorado Springs, CO.

Silicon nano-crystal and nano-trap memories are attracting the attention of many investigators because of lower programming voltage, longer retention time and improved endurance. In this paper, we are presenting the results of silicon nano-trap memory fabricated by implanting low energy high dose silicon into gate oxide of thickness 300 Å. The gate oxide was grown by dry oxidation. Capacitance versus Voltage (C-V) characteristics of MOS (metal oxide silicon) structures with silicon implanted samples annealed in nitrogen environment at a temperature of 950 C show a memory window depending on the applied DC bias voltage. A memory window of 3V was obtained for an applied bias voltage between +10 V and -10V. Annealing of the MOS structures in a furnace at a temperature of 800 C for 30 minutes in oxygen resulted in complete loss of memory window. Annealing the samples rapid thermally in oxygen environment at 800 C for 30 seconds resulted in a memory window of about 3V for a programming voltage of plus or minus 14 volts with improved C-V characteristics. We will present preliminary results on the characteristics of implanted silicon nano-trap MOSFET non-volatile memory devices.

**T5.8**  
EFFECT OF ENERGETIC BEAMS ON Si NANOSTRUCTURES IN SiO<sub>2</sub>. Gregory Kachurin, Svetlana Yanovskaya, Vladimir Volodin, Anton Gutakovskiy, Institute of Semiconductor Physics SO RAN, Novosibirsk, RUSSIA.

The influence of ion, electron and intense light beams on Si nanocrystals (Si-ncs), embedded in SiO<sub>2</sub> layers, was studied using HREM, photoluminescence and Raman spectroscopy. The Si-ncs were formed by implantation of  $\sim 10^{17}\text{cm}^{-2}$  Si ions in thin SiO<sub>2</sub> layers followed by annealing at 1100°C. Due to the quantum-size confinement, the as-prepared Si-ncs emitted strong red light under 488 nm laser excitation. Subsequent irradiations of the samples with the keV-range He and B ions and with 400 keV electrons resulted in structural transformations quite different from that of bulk Si. Single atomic displacements produced by low doses of light energetic particles caused an increase in Si-ncs number in the layers. That was the case only when the post-implantation annealing temperatures exceeded 1000°C. Further irradiation with the light ions and fast electrons amorphized Si-ncs, presumably via accumulation of mobile point defects by the Si-ncs boundaries. Amorphization occurred when about 20% of atoms were displaced due to elastic losses while the ionization losses did not play any role. Unlike bulk Si, more than 1000°C annealing was required to re-crystallize the amorphized nano-inclusions. Si-ncs may be formed by annealing of Si-implanted SiO<sub>2</sub> layers with the intense light pulses of sec- and msec-duration, which provided the samples heating up to 1200-1400°C. However, the subsequent furnace anneals at 800-900°C for 30 min resulted in disappearance of Si-ncs, as revealed by HREM and Raman data. All the observed peculiarities are ascribed to the strong influence of the Si-ncs boundaries and their surface tension on the structural transformations. Photoluminescence and Raman spectroscopy have shown the Si-ncs consist of 2-3 nm crystalline core and of  $\sim 1$  nm-thick

outer shell. Possible effect of the outer shell on amorphization and crystallization of Si nano-inclusions is evaluated.

**T5.9**  
PHASE TRANSITION AND THERMAL BEHAVIOUR IN NANOSTRUCTURED ZRO<sub>2</sub> AFTER HEAVY ION IRRADIATION. Wolfram Berky, Horst Hahn and Adam Georg Balogh, Darmstadt University of Technology, Institute of Materials Science, Darmstadt, GERMANY.

As it was verified by RBS, XRD and TEM measurements, heavy ion irradiation of nanocrystalline zirconia causes a monoclinic - tetragonal phase transition at room temperature. This new phase could be important for several applications, e.g. fuel cells, oxygen sensors, etc. To investigate the thermal stability of the irradiation induced tetragonal phase, samples have been prepared by the inert gas condensation technique and were sintered at temperatures of about 1000 C. After preparation samples were irradiated by 4 MeV Kr ions at room temperature and the stability of the new phase was studied by x-ray diffraction measurements. The heavily irradiated samples (fluences of about  $1 \times 10^{16}$  ion/cm<sup>2</sup> and above) showed a thermal stability up to the sintering temperature, whereas samples irradiated with lower fluences showed only a residual amount of this phase of about 5 vol.% at temperatures of 400-500 C. No significant change in the grain size of both phases could be observed. The grain size of the monoclinic phase was approximately 60 nm, and that of the tetragonal phase about 20 nm. Beside the thermal stability of the tetragonal phase an ageing stability within a time period of several years was also observed.

**T5.10**  
MATRIX SEEDING OF SEMICONDUCTOR NANOSTRUCTURES USING ION BEAMS. X. Weng, W. Ye, S. Clarke, R.S. Goldman, Dept of Materials Science and Engineering; A. Daniel, R. Clarke, Applied Physics; J. Holt, J. Sipowska, A. Francis, Dept of Chemistry; V. Rotberg, Dept Nuclear Engineering and Radiological Sciences; J. Phillips, Dept of Electrical Engineering and Computer Science, University of Michigan, Ann Arbor, MI .

Recently, the controlled nanoscale crystallization of amorphous solids has emerged as a promising means for producing technologically useful nanocomposite materials. In the case of semiconductors, the controlled formation of crystalline structures on the nanoscale provides an opportunity for producing new materials with unique electronic and optical properties. In this work, we are exploring the matrix seeding of narrow-gap nitride nanostructures in nitrogen ion implanted GaAs and InAs. Low-energy implantation followed by rapid thermal annealing (RTA) results in the formation of 2-3 nm sized amorphous precipitates in a crystalline matrix. Photoluminescence spectral features related to these nanostructures are not apparent. On the other hand, high-energy implantation results in an amorphous layer, with or without crystalline remnants. When the ion-beam-synthesized amorphous matrix contains crystalline remnants, subsequent RTA leads to the formation of 2-4 nm ZB-GaN-rich crystallites within the amorphous regions. When this matrix is a continuous amorphous layer, subsequent RTA leads to the formation of 4-5 nm ZB-GaN-rich crystallites in an amorphous matrix [1]. As the annealing temperature increases from 750 to 850°C, the average crystallite size increases while the size distributions are self-similar and the volume fraction remains constant. Thus, the crystallite coarsening process is considered to be governed by Ostwald ripening, with an activation energy,  $1.0 \pm 0.3$  eV, presumably due to N or As diffusion via gallium vacancies. Photoluminescence spectroscopy reveals a significant emission at  $\sim 1.27$  eV, apparently related to the GaAsN nanostructures. The lowering of the fundamental bandgap of these crystallites is likely due to the incorporation of As in GaN. We will discuss the effects of dopants on the crystallization processes and compare the effects of RTA and pulsed laser annealing on the nanostructure nucleation and coarsening mechanisms. [1] X. Weng, S.J. Clarke, W. Ye, S. Kumar, R.S. Goldman et al., J. Appl. Phys. 92, 4012 (2002).

**T5.11**  
LUMINESCENCE AND MICROSTRUCTURE OF SILICON NANOCRYSTAL ARRAYS. C.A. Ryan, A. Meldrum, Dept. of Physics, University of Alberta, Edmonton, AB, CANADA; C.W. White, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

The strong visible photoluminescence (PL) of silicon nanocrystals has, in recent years, been the focus of considerable research interest. Nanocrystals produced by silicon implantation of a fused silica wafer followed by high-temperature thermal processing show a broad and comparatively intense PL band. The light emission, centered in the near infrared and extending well into the visible part of the spectrum, has potential applications for the development of photonic materials based on silicon nanostructures. Here, we report on our attempts to

create luminescent arrays of Si nanoparticles. Silica microspheres containing embedded silicon nanocrystals were formed by ion implantation and annealing of commercially produced SiO<sub>2</sub> spheres with a nominal diameter of 5 micrometers. Arrays of luminescent microspheres were successfully fabricated, without significant coagulation or destruction of the silica spheres. However, a substantial deformation on the surface of the spheres that occurred during the high-flux implantation prevented the development of resonant cavity modes in the luminescence spectra. Resonant modes could clearly be observed for pre-implanted and annealed SiO<sub>2</sub> wafers with a layer of pristine microspheres subsequently deposited on the implanted surface. These combined results suggest that at lower ion fluxes, the development of luminescent microsphere superlattices, with novel optical properties due to resonant mode coupling to the light emission from silicon nanocrystals, may be possible if the deformation effects can be reduced or eliminated.

SESSION T6: METALLIC AND MAGNETIC  
NANOSTRUCTURES  
Wednesday Morning, April 23, 2003  
City (Argent)

**9:00 AM \*T6.1**

**METAL-SEMICONDUCTOR PHASE TRANSITION IN NANOSCALE VANADIUM DIOXIDE PRECIPITATES FORMED IN SILICA AND SAPPHIRE BY ION IMPLANTATION.**  
R. Lopez<sup>a,b</sup>, L.A. Boatner<sup>b</sup>, L.C. Feldman<sup>a,b</sup>, R.F. Haglund, Jr.<sup>a</sup> and T.E. Haynes<sup>b</sup>; <sup>a</sup>Vanderbilt University, Nashville, TN; <sup>b</sup>Oak Ridge National Laboratory, Oak Ridge, TN.

The study of solid-state phase transitions at nanometer length scales provides new insights into the effects of size on the mechanisms of structural transformations. Such research also opens the door to new applications since the physical properties of nanoparticles change with particle size or through the interaction of the nanoparticles with a surrounding host matrix. For applications such as optical memories, the hysteresis that is characteristic of the phase transition is particularly relevant. Here, we describe the formation of vanadium dioxide precipitates in silica and sapphire substrates by stoichiometric ion implantation and thermal processing. We observe 10-100 nm-size VO<sub>2</sub> nanoparticles with shapes that vary from spheroids to needles. The metal-semiconductor transition of the VO<sub>2</sub> precipitates shows features that differ in each substrate. The effects of nanocrystal morphology, size, and host interaction on the phase transition were characterized by transmission electron microscopy, x-ray diffraction, Rutherford backscattering, infrared optical measurements, and doping experiments.

Of particular interest are the enhanced size-dependent hysteresis and the surface plasmon resonance induced by dielectric confinement when the particles reach the metallic state. This resonance amplifies the optical contrast in the range of near-infrared optical communication wavelengths. The VO<sub>2</sub> nanoparticles in Al<sub>2</sub>O<sub>3</sub> present a 'rounded' and narrow hysteretic transition, while the nanoparticles embedded in SiO<sub>2</sub> exhibit a sharper transition with up to 50 K of hysteresis – one of the largest values ever reported for this transition. This hysteresis and the transition temperatures are correlated with the size of the precipitates; this size dependence can be understood within the framework of heterogeneous nucleation theory. Doping the particles with ions such as tungsten and titanium alters the transition temperatures and the hysteresis width over a wide range of values, and provides further clues to the mechanism of the phase transition. Nonlinear and ultra-fast optical measurements on VO<sub>2</sub> films have shown that the semiconductor-to-metal transition is the fastest known solid-solid transformation. The VO<sub>2</sub> nanoparticles will be shown to retain this bulk property, transforming in times shorter than 150 fs.

**9:30 AM T6.2**

**STRUCTURES OF ENERGETIC ION IRRADIATED CARBON NANOTUBES.** Ananta Adhikari, Dept of Physics, State University of New York, Albany, NY; Mengbing Huang, Dept of Physics and School of NanoScience & NanoEngineering, State University of New York, Albany, NY; Katharine Dovidenko, School of NanoScience & NanoEngineering, State University of New York, Albany, NY; Bingqing Wei, Robert Vajtai, Pulickel Ajayan, Dept of Material Science and Engineering, Rensselaer Polytechnic Institute, Troy, NY.

Carbon nanotubes (CNTs) have unique structures and hence a range of fascinating properties. If the structures can be controllably tailored by post-synthesis techniques such as irradiation, for example by introducing controlled number of defects, it may lead to better control in electronic applications. Ion irradiation offers a unique way to investigate defect structures of CNTs and their influences on CNTs' various properties. The understanding of energetic ion beam effects on CNTs is of great technological importance, since CNTs are also promising for applications in space exploration. However, there is only

limited experimental work on ion irradiation and most of this focused on electron irradiation. In this work we study radiation effects of energetic ions (proton and argon) on CNT structures. Both single wall nanotubes (SWNTs) and multi-wall nanotubes (MWNTs) were irradiated with ions of energy in keV-MeV to doses in the range of 1e12-1e16 cm<sup>-2</sup>. A comparison in CNT structures and chemical environments between the irradiated and un-irradiated samples was made based on materials characterization including transmission electron microscope (TEM), scanning electron microscope (SEM), x-ray photoelectron spectroscopy (XPS), and x-ray diffraction (XRD). The amount of defects (e.g., vacancies) created in the CNTs was correlated with the nuclear and electronic energy loss of ion beams. Effects of post-irradiation annealing on defect structures of CNTs were also examined.

**9:45 AM T6.3**

**ION BEAM MANIPULATION OF NANO-SIZED ARTIFICIAL SOLID-STATE MICROSTRUCTURES IN SOME IMMISCIBLE BINARY METAL SYSTEMS.** Z.C. Li and B.X. Liu, Tsinghua Univ, Dept of Materials Science and Engineering, Beijing, CHINA.

We developed a new scheme namely ion beam manipulation, i.e. interface-assisted ion beam mixing, for fabricating artificial solid-state microstructures in the metal-metal multilayers, in which the individual layer thickness was down to about 2 nm, differing from typical thickness of 5-8 nm in the conventional ion beam mixing [1]. In the scheme, the interfacial free energy was a controllable parameter and designed to be adequate for elevating the multilayers up to a highly energetic level. Another controllable parameter was the irradiation dose, which was executed with fine intervals for tracing the detailed structural evolution, prior to the formation of the new microstructure. We report, in this paper, some examples observed in the equilibrium immiscible Ag-Co, Ag-Ni and Pd-Ru systems to show that the scheme was indeed under a controllable manner with the two adjustable parameters. In the Ag-Co system, a highly energetic ordered layered structure was obtained and identified to consist of two overlapping fcc lattices, corresponding to a new magnetic state of Co atom with an average magnetic moment of 2.84  $\mu_B$ , which was about twice of the equilibrium value and probably the largest one ever observed. Interestingly, the similar ordered layered structure was reproduced in the Ag-Ni system [2]. In the Ru-Pd system, unique structural evolution was observed with increasing the irradiation dose, i.e. the initial polycrystalline Pd and Ru first transformed gradually into a single crystalline fcc phase, and then turned into a well grown ordered structure, which, however, showed an apparent tendency to transform back to the same fcc phase upon over-irradiation [3].

**10:30 AM \*T6.4**

**MAGNETIZATION DYNAMICS OF IRRADIATION-FABRICATED PERPENDICULARLY MAGNETIZED DOTS INSIDE A SOFTER MAGNETIC MATRIX.** T. Devolder, M. Belmeguenai, C. Chappert, Institut d'Electronique Fondamentale, UMR CNRS 8622, Universite Paris-Sud, Orsay, FRANCE; H. Bernas, Centre de Spectrometrie Nucleaire et de Spectrometrie de Masse, UMR CNRS 8609, Universite Paris-Sud, Orsay, FRANCE; Y. Suzuki, National Institute of Advanced Industrial Science and Technology, Electronics Institute, Tsukuba, JAPAN.

Obtaining reproducible magnetization switching within the sub-nanosecond regime and the deep sub-micron range is currently one of the most challenging tasks in nanomagnetism. We have previously shown that temperature-assisted helium ion irradiation is a very powerful tool to tune the magnetic properties of thin films, notably their magnetocrystalline anisotropy. Helium ion irradiation through nanofabricated masks has also been used to produce sub-micron planar nanostructures. We have recently tested perpendicularly magnetized dots in a matrix of weaker magnetic anisotropy. Static magnetometry measurements and Magnetic Force Microscopy showed that magnetization reversal in these dots is nucleation-free and proceeds by a very specific domain wall injection from the magnetically "soft" matrix, which acts as a domain wall reservoir for the "hard" dot, thus inducing a remarkably weak coercivity dispersion. We now generalize this approach by a new type of irradiation-fabricated magnetic device, designed to achieve high switching speeds, typically below 100 ps. The speed is obtained through the use of a very high effective magnetic field, and high resulting precession frequencies. During magnetization reversal, the effective field incorporates a significant exchange field, storing energy in the form of a domain wall surrounding a high magnetic anisotropy nanostructure's region of interest. Promising applications to magnetic storage are anticipated.

**11:00 AM T6.5**

**ION BEAM STABILIZATION OF FePt NANOPARTICLE ARRAYS FOR MAGNETIC STORAGE MEDIA.** J.E.E. Baglin, A.J. Kellock, W. Volksen, IBM Almaden Research Center, San Jose, CA; S. Sun and C.B. Murray, IBM T.J. Watson Research Center, Yorktown



Self assembled layers of monodisperse magnetic nanoparticles offer attractive possibilities for high density disk storage media. Sun et al.[1] displayed ordered planar arrays of 4 nm particles of FePt, prepared via a simple wet-chemical process, followed by dip-coating. In such arrays, each particle is decorated by organic ligands that serve to isolate the particles from each other, without which they would spontaneously agglomerate, even at room temperature. Any such agglomeration would destroy the spatial selectivity of the recording medium. In order to create a functional storage medium, the soft organic binder that separates the FePt particles by about 2 nm must be hardened, to provide a rigid layer, and also to prevent agglomeration of the particles during the subsequent high temperature annealing that is required to grow the high-anisotropy fct magnetic phase of FePt. We have achieved the conversion of the organic binder into a hard, continuous diamondlike carbon barrier between the nanoparticles, using ion irradiation after the introduction of various organic binders by ligand exchange during deposition of the nanoparticle layer. We report on the hardness and composition of the layer, and its ability to prevent agglomeration of nanoparticles under high temperature annealing. We also report on the ability of the DLC layer to inhibit subsequent oxidation of Fe at the particle surfaces. In a further application, we describe the technique of producing patterns of isolated rafts of such particles, by ion irradiation hardening of only selected areas, using a stencil mask for the ion beam, allowing the remainder of the coating to be removed by solution. [1] S. Sun, C.B. Murray, D. Weller, L. Folks and A. Moser, *Science* 287, 1989 (2000).

#### 11:15 AM T6.6

POST-GROWTH ORDERING OF MAGNETIC INTERMETALLIC ALLOY NANOSTRUCTURES BY ION IRRADIATION. H. Bernas<sup>a</sup>, D. Halley<sup>b</sup>, K.-H. Heinig<sup>c</sup>, J.-Ph. Attane<sup>b</sup>, D. Ravelosona<sup>d</sup>, A. Marty<sup>b</sup>, P. Auric<sup>b</sup>, C. Chappert<sup>d</sup>, Y. Samson<sup>b</sup>; <sup>a</sup>Centre de Spectrometrie Nucleaire et de Spectrometrie de Masse, CNRS-Universite Paris XI, Orsay, FRANCE; <sup>b</sup>CEA Grenoble, DRFMC Service de Physique des Materiaux & Microstructures, (assoc. with Universite J. Fourier), Grenoble, FRANCE; <sup>c</sup>FZ-Rosendorf, Inst. Ionenstrahlphysik & Materialforschung, Dresden, GERMANY; <sup>d</sup>Inst. d'Electronique Fondamentale, CNRSUniversite Paris XI, Orsay, FRANCE.

We trigger and control the chemical disorder-to-order transformation in ultrathin films of FePd, an intermetallic ferromagnet by combining He ion irradiation and thermal mobility below 600K. Kinetic Monte Carlo simulations demonstrate that the initial directional short range order determines order propagation. Under irradiation, the initially weak magnetic anisotropy reaches the highest values known for FePd films as magnetic ordering rotates from in-plane to perpendicular to the film plane. Applications to ultrahigh density magnetic recording are suggested.

#### 11:30 AM T6.7

ION IMPLANTATION SYNTHESIS AND CHARACTERIZATION OF COBALT NANODOTS. L.G. Jacobsohn, M. Nastasi, M.E. Hawley and D.W. Cooke, Materials Science & Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Recently, special attention has been paid to magnetic nanodots due to their possible application in data storage, logic devices and magnetic sensors. All these applications rely on the possibility of controlling the size and shape of the nanodots. Cobalt nanodots were obtained by ion implantation at 35 KeV into fused silica for doses from  $8 \times 10^{15}$  to  $1 \times 10^{17}$  Co/cm<sup>2</sup> that corresponds to a projected range + straggling of  $27 \pm 9$  nm according to TRIM. After implantation the samples were annealed in vacuum at 900°C for 2 and 10 hours. They were characterized on their chemical content as well as on actual Co depth distribution by means of 2 MeV He<sup>+</sup> Rutherford backscattering spectrometry (RBS). The presence of Co nanodots was positively identified by the presence of a Mie resonance band. No absorption band was found in the as-implanted samples. The analysis of the optical data provided the average size and the volume fraction occupied by the nanodots. For the annealed  $1 \times 10^{17}$  at/cm<sup>2</sup> implanted samples, an average dot radius of 5 nm was found while for the annealed  $3 \times 10^{16}$  at/cm<sup>2</sup> samples the average radius was 12 nm. Within the detection limits of the spectrophotometer, no nanodots were formed in the  $8 \times 10^{15}$  at/cm<sup>2</sup> annealed samples. The kinetics of the formation of Co nanodots is viewed as a competition between nucleation and diffusion in the SiO<sub>2</sub> matrix. Magnetic force microscopy (MFM) measurements investigated the magnetization component normal to the surface of the samples revealing randomly distributed structures with defined magnetization in the case of annealed samples.

#### 1:30 PM \*T7.1

ENERGY TRANSPORT IN METAL NANOPARTICLE PLASMON WAVEGUIDES. Stefan A. Maier, Pieter G. Kik, and Harry A. Atwater, California Institute of Technology, Thomas Watson Laboratory of Applied Physics, Pasadena, CA; Sheffer Meltzer, Elad Harel, Bruce E. Koel, and Ari A.G. Requicha, University of Southern California, Laboratory for Molecular Robotics, Los Angeles, CA.

The ultimate miniaturization of optical devices to spatial dimensions approaching the molecular scale will require structures that guide electromagnetic energy with lateral confinement below the diffraction limit of light, which is not possible using conventional dielectric optical or photonic crystal waveguides. We have shown theoretically that arrays of closely spaced metal nanoparticles can work as plasmon waveguides that guide electromagnetic energy on the nanoscale. Such structures could potentially be fabricated and their optical properties tuned using ion beam irradiation methods. We report on FDTD calculations of energy guiding in locally excited plasmon waveguides consisting of spherical noble metal nanoparticles. The simulations allow the determination of the dispersion relation and group velocities for energy transport and quantitatively confirm the results of point-dipole calculations. Using ellipsoidal nanoparticles with a 3:1 aspect ratio, group velocities up to 0.2c are predicted for longitudinal excitations in waveguide geometries that can be fabricated using electron beam lithography. We also present the results of optical characterization of the guiding properties of plasmon waveguides consisting of closely spaced gold and silver nanoparticles fabricated using electron beam lithography. Far-field spectroscopy confirms the existence of longitudinal and transverse collective modes of excitation and allows for the estimation of the dispersion relation and group velocities. Measurements of the polarization-dependent absorption confirm that the collective modes arise from near-field optical interactions. Using the tip of a near-field optical microscope as a local excitation source and fluorescent polystyrene nanobeads as detectors, we present experimental evidence for energy transport over a distance of about 0.5 micron for plasmon waveguides consisting of silver rods with a 3:1 aspect ratio and a center-to-center distance of 80 nm. This is the first direct evidence for localized energy transport in nanoparticle plasmon waveguides.

#### 2:00 PM T7.2

THE OPTICAL PROPERTIES OF SELENIUM NANOCRYSTALS FABRICATED BY ION IMPLANTATION. D.O. Henderson, R. Mu, M.H. Wu, A. Ueda, Chemical Physics Laboratory, Department of Physics, Fisk University, Nashville, TN; A. Meldrum, Dept. of Physics, University of Alberta, Edmonton, CANADA; C.W. White, Oak Ridge National Laboratory, Solid State Division, Oak Ridge, TN; M. Jaksic, Rudjer Boskovic Institute, Zagreb, CROATIA; B. Vlahovic, Department of Physics, North Carolina Central University, Durham, NC.

Silica windows were implanted with selenium at an energy of 330 KeV with ion doses ranging from  $1 \times 10^{16}$  to  $1 \times 10^{17}$  ions cm<sup>-2</sup>. Rutherford backscattering measurements were performed to determine the depth profiles for selenium implanted in both substrates. The implanted substrates were annealed in an atmosphere consisting of 5% H<sub>2</sub>+95% Ar at temperatures from ranging from 400 to 1000°C for one hour. The optical transmission spectra of implanted substrates showed a continuous red shift in the band edge as the annealing temperature increased from 400 to 1000°C. Specifically, at a transmission level of 75%, the unannealed sample implanted with  $1 \times 10^{17}$  ions cm<sup>-2</sup> indicated an absorption corresponding to 3.0 eV while the sample annealed at 1000°C showed an absorption corresponding to 2.7 eV. The annealing induced red-shift is attributed to the formation of Se nanocrystals with a size range that affects quantum confinement of the exciton. This interpretation is qualitatively consistent with the transmission electron microscopy measurements which showed that the nanocrystals increased in size from ~5 nm for a sample annealed 600°C to ~10 nm for the sample annealed at 1000°C. Moreover, the band edge showed a decrease with an increasing ion dose, which is also consistent with size dependence on ion dose, i.e., increasing the ion dose renders larger size particles.

#### 2:15 PM T7.3

A NEW PATHWAY FOR Si NANOCRYSTAL FORMATION: OXI-REDUCTION INDUCED BY IMPURITY IMPLANTATION. L.G. Jacobsohn, J.K. Lee, D.W. Cooke, M. Nastasi, Materials Science & Technology Division, Los Alamos National Laboratory, Los Alamos, NM; A.R. Zanatta, Instituto de Física de São Carlos, Universidade de São Paulo, São Carlos, SP, BRAZIL.

In this work we show the feasibility of producing silicon nanocrystals by means of a new method based on the oxi-reduction of silicon dioxide induced by the presence of an impurity. The choice of

magnesium as the impurity relies on its thermodynamic behavior since it has a high probability of forming an oxide while the formation of Si-based compounds is unlikely. The samples were obtained by low energy ion implantation into fused silicon dioxide with a dose of  $1 \times 10^{17}$  Mg/cm<sup>2</sup> and were annealed in vacuum at 900°C for 2 or 10 h. Rutherford backscattering spectrometry (RBS) provided the actual depth distribution as well as the amount of implanted Mg. For the 10 h annealed sample, Raman scattering spectroscopy showed a sharp peak at 523 cm<sup>-1</sup>, suggesting the existence of large Si nanocrystals. No Raman peak was observed in the case of 2 h annealing. The presence of Si nanocrystals is also supported by the presence of an absorption band around 340 nm that corresponds to the  $\Gamma_{25} - \Gamma_{15}$  direct transition. Photoluminescence (PL) results show a broad band starting around 1.8 eV with increasing intensity for lower energies that is a fingerprint for the presence of Si nanocrystals. The analysis of the PL data in the framework of quantum confinement theory supports the existence of large (10-30 nm) Si nanocrystals.

### 2:30 PM T7.4

LUMINESCENCE EFFICIENCY OF SILICON NANOCRYSTALS IN SiO<sub>2</sub>: EFFECTS OF EXCITATION SPECTRUM. A. Hryciw, K. Chow, A. Meldrum, Dept. of Physics, University of Alberta, Edmonton, AB, CANADA; C.W. White, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

Silicon nanocrystals formed by ion implantation and annealing of fused silica wafers show a strong, broad photoluminescence (PL) peak, typically centered at a wavelength between 720 and 850 nm. This luminescence has been extensively investigated and trial device structures based on these materials have been built. However, relatively few studies also report the optical absorption spectra. In fact, the absorbance of these specimens is typically quite low (often < 10%) at wavelengths greater than 450 nm (i.e., the pump wavelengths typically used for PL studies). This suggests that in numerous studies of Si nanocrystals produced by ion implantation, only a small fraction of the nanocrystals may be responsible for the observed PL at the typical pump wavelengths. In this study, we investigated how the spectrum and PL intensity depend on the power of the pump laser, for a range of excitation wavelengths. We find that the samples show a saturation behavior and blueshifted emission spectra at high pump fluences, which we attribute to the dynamics of the excitation/recombination processes at different energies. During CW laser excitation, the specimen absorbance is altered, but only at wavelengths longer than the excitation wavelength. These data can provide reasonable estimates of the absorption coefficient and quantum efficiency of highly luminescent silicon nanocrystals embedded in SiO<sub>2</sub>.

### 3:15 PM T7.5

NOVEL NANOCRYSTALS FORMED IN SILICON AND SILICON DIOXIDE BY PRECIPITATION OF ION IMPLANTED METALS AT NANOCAVITIES. J.S. Williams, M.J. Conway, M. Petravic and J. Wong-Leung, Department of Electronic Materials Engineering, Research School of Physical Sciences and Engineering, Australian National University, Canberra, AUSTRALIA.

Ion implantation is a convenient method to introduce nanocavities into semiconductors. In this study we have formed them in  $< 100 >$  Si by one of two methods: i) H ion implantation to high dose, followed by annealing to 850°C, to form a band of nanocavities  $\sim 10$ -20 nm in diameter at the end of ion range, and ii) Si ion implantation and annealing, also to 850°C, to form much smaller nanocavities (up to 5 nm diameter) at depths up to about half the ion range. Ion irradiation was subsequently used to shrink the cavities to a required narrow size distribution. In some cases the Si containing the nanocavities was oxidized: subsequent analysis (by metal decoration) confirming that the nanocavities survived within SiO<sub>2</sub>. Samples of both Si and SiO<sub>2</sub> containing nanocavities were then implanted with high doses of metals (Cu, Au and Zn) and annealed to diffuse and precipitate metals in nanocavities as either metal or silicide nanocrystals. Rutherford Backscattering and transmission electron microscopy were used to study cavity decoration and nanocrystalline phase and structure. In a further series of experiments, Zn particles in Si were oxidized in an attempt to form wide band gap ZnO nanocrystals in SiO<sub>2</sub>. Results indicate that decoration of nanocavities in Si and SiO<sub>2</sub> with metals introduced by ion implantation is a very attractive method for forming novel metal and semiconductor nanocrystals of a desired size in these materials. Possible formation mechanisms will also be discussed.

### 3:30 PM T7.6

LIBERATION OF ION IMPLANTED GERMANIUM NANOCRYSTALS FROM A SILICON DIOXIDE MATRIX VIA HYDROFLUORIC ACID VAPOR ETCHING. I.D. Sharp<sup>a,b</sup>, Q. Xu<sup>a,b</sup>, C.Y. Liao<sup>a</sup>, J.W. Ager III<sup>a</sup>, J.W. Beeman<sup>a</sup>, K.M. Yu<sup>a</sup>, D. Zakharov<sup>a</sup>, Z. Liliental-Weber<sup>a</sup>, E.E. Haller<sup>a,b</sup>, <sup>a</sup>Materials Science Division, Lawrence Berkeley National Laboratory, Berkeley, CA; <sup>b</sup>Department of Materials Science and Engineering, University of

California, Berkeley, CA.

We present the results of experiments to liberate germanium (Ge) nanocrystals from silicon dioxide (SiO<sub>2</sub>) matrices by hydrofluoric acid (HF) vapor etching. Ge nanocrystals were fabricated in 5000 nm thick wet oxide layers on silicon (Si) substrates by multi-energy implantation of mass separated Ge ions followed by thermal annealing at 850°C for 30 minutes. Raman spectroscopy and Transmission Electron Microscopy (TEM) confirmed the existence of nanocrystalline monoisotopic Ge clusters within the SiO<sub>2</sub> matrices. Raman spectra exhibited the expected isotope shifts and the characteristic asymmetric lineshapes due to the phonon confinement effect. TEM images revealed a range of Ge nanocrystal diameters between 2 and 8 nm, with an approximate average diameter of 5 nm. Twinning was observed within some individual nanocrystals. In order to free Ge nanocrystals, samples were etched in HF vapor to selectively remove the SiO<sub>2</sub> matrix and expose the Ge nanocrystals. Rutherford Backscattering Spectrometry (RBS) data showed that approximately 75% of implanted Ge remained on sample surfaces after complete removal of the oxide layer. Raman spectroscopy further confirmed that crystalline Ge remained on the sample surface after etching to the Si/SiO<sub>2</sub> interface. RBS data showed that Ge was successfully removed from etched surfaces using an ultrasonic methanol cleaning procedure. Thus, liberated Ge nanocrystals are expected to be accessible for a wide range of manipulation processes and direct characterization techniques.

### 3:45 PM T7.7

METALLIC NANORODS BY MeV ION BEAM INDUCED PLASTIC DEFORMATION OF CORE-SHELL COLLOIDS. Sjoerd Roorde<sup>a,b</sup>, Teun van Dillen<sup>a</sup>, Joan Penninkhof<sup>a</sup>, Bart Kooi<sup>c</sup>, Jeff de Hosson<sup>c</sup>, Christina Graf<sup>d</sup>, Alfons van Blaaderen<sup>a,d</sup>, Albert Polman<sup>a</sup>; <sup>a</sup>FOM-Institute for Atomic and Molecular Physics, Amsterdam, THE NETHERLANDS; <sup>b</sup>Universite de Montreal, GCM, Departement de physique, Montreal, CANADA; <sup>c</sup>University of Groningen, Groningen, THE NETHERLANDS; <sup>d</sup>Utrecht University, Utrecht, THE NETHERLANDS.

A new class of materials, core-shell colloidal particles, was irradiated with 30 MeV Si and Se ions to fluences of  $2\text{-}5 \times 10^{14}$ /cm<sup>2</sup>. The core-shell colloids consisted of a 15 nm diameter gold core surrounded by a silica shell of typically 70 nm thickness. High-resolution transmission electron microscopy was used to study the particle morphology after irradiation. At sufficiently high fluences, the silica shell deforms into an oblate ellipsoid (pancake) with its short axis along the ion beam. This anisotropic plastic deformation is known to occur on pure silica colloidal particles. The gold core also deforms, but into a prolate shape (rod) with the long axis along the ion beam direction. The deformation of the gold core is thought to be a consequence of the deformation of the silica shell, since it is observed that thinner shells lead to smaller core-deformations. Anisotropic nanorods such as these deformed core-shell particles have unique optical properties. The peak in the absorption spectrum normally attributed to a surface plasmon resonance shifts to longer or shorter wavelengths, depending on the relative orientation of the gold nanorods long axis and the polarisation of the incident light.

### 4:00 PM T7.8

THE STRUCTURAL AND OPTICAL PROPERTIES OF Zn AND ZnO NANOCRYSTALS IN SiO<sub>2</sub> AND CaF<sub>2</sub> SUBSTRATES. Y.C. Liu<sup>a,b</sup>, H.Y. Xu<sup>a</sup>, Y.M. Lu<sup>b</sup>, D.Z. Shen<sup>b</sup>, Henderson<sup>c</sup>, C.W. White<sup>d</sup>, and R. Mu<sup>c</sup>; <sup>a</sup>Advanced Functional Material Research Center, Northeast Normal University, Changchun, CHINA; <sup>b</sup>Key Laboratory of Excited State Processes, CAS, Changchun, CHINA; <sup>c</sup>Chemical Physics Laboratory, Fisk University, Nashville, TN; <sup>d</sup>Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

Ion implantation technique was employed to implant zinc ions into crystalline CaF<sub>2</sub> and amorphous SiO<sub>2</sub> substrates. Both zinc colloids and ZnO nanocrystals could be formed depending upon the post thermal annealing conditions. When the sample was annealed under reducing atmosphere, the absorption band at  $S \sim 5.3$  eV for zinc implanted in SiO<sub>2</sub> substrate was attributed to zinc metal colloids. The absorption peak in the 4.3 - 4.7 eV region was due to the formation of ZnO nanocrystals when the sample is annealed under an oxidizing environment. X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) study also suggested ZnO nanocrystal formation. For zinc implanted in CaF<sub>2</sub> substrate, the formed ZnO nanocrystals have (002) preferential orientation. Photoluminescence (PL) spectra exhibited UV and green emissions from the zinc implanted samples annealed under an oxygen atmosphere. No green emission is observed for ZnO formed in CaF<sub>2</sub> substrate. However, an additional emission was observed at  $\sim 420$  nm which may be due to F centers created by ion beam damage.

### 4:15 PM T7.9

MeV ION BEAM INDUCED ANISOTROPIC PLASMON

## RESONANCE OF SILVER NANOCRYSTALS IN GLASS.

J.J. Penninkhof and A. Polman, FOM Institute AMOLF, Amsterdam, THE NETHERLANDS; L.A. Sweatlock and H.A. Atwater, Harvard University, McKay Laboratory of Applied Science, Cambridge, MA; A.M. Vredenberg, Utrecht University, Utrecht, THE NETHERLANDS; B.J. Kooi, University of Groningen, Groningen, THE NETHERLANDS.

We demonstrate a technique for synthesizing silver nanocrystal-doped glass with strongly anisotropic optical properties. Silver ions are introduced in soda lime glass by a silver-sodium exchange process. Subsequently, spherical nanoparticles are formed by 1 MeV xenon irradiation. The samples are then subjected to a 30 MeV silicon ion beam at 77 K under an angle of 60 degrees off normal.

Optical transmission data show polarization-dependent plasmon bands of silver, with a maximum plasmon peak shift occurring for polarizations parallel and orthogonal to the irradiation axis. The splitting can be tuned by varying the ion fluence. For the highest fluence ( $1 \times 10^{16}$  Si/cm<sup>2</sup>) plasmon peaks are observed in the near infrared (890 nm) and in the UV (380 nm), for different polarizations. Transmission electron microscopy indicates a two-fold effect of the MeV silicon ion irradiation: 1) individual Ag nanocrystals change shape from spherical to slightly prolate, with the long axis along the ion beam direction, and 2) arrays of aligned silver nanoparticles are formed along the ion tracks.

We will present high-resolution electron microscopy results showing the distribution, orientation and aspect ratio of the nanocrystals. In addition, the results of full field electromagnetic simulations of the plasmonic interaction between ellipsoidal particles and particle arrays, using finite difference time domain calculations, will be discussed. When combined with a suitable mask technique, this method can be used to write metallo-dielectric optically anisotropic micropatterns that may find applications in nonlinear photonic devices, photonic crystals and fabrication of subwavelength "plasmonic" guiding structures.

### 4:30 PM T7.10

ION BEAM MODIFICATION OF METALLIC NANOCLUSTERS IN SiO<sub>2</sub>. Giovanni Mattei, Valentina Bello, Giovanna De Marchi, Chiara Maurizio, Paolo Mazzoldi, Moreno Parolin, Cinzia Sada, INFN - Univ of Padua, Dept of Physics, Padua, ITALY; Giancarlo Battaglin, INFN - Univ of Venice, Dept of Physical Chemistry, Venice, ITALY.

Composite materials made by mono-elemental or metal alloy nanoclusters embedded in SiO<sub>2</sub>-based matrices exhibit peculiar nonlinear optical properties which are function of the cluster size and composition. Sequential ion implantation in glass has demonstrated to be a very effective technique to obtain such composite materials. In this work we report on the use of ion beam irradiation to induce transformation (either compositional or structural) on metallic nanoclusters in SiO<sub>2</sub>. An elemental selective de-alloying in bimetallic Au-Cu and Au-Ag nanoclusters prepared by sequential ion implantation has been found upon irradiation with light ions or thermal annealing in oxidizing atmosphere. For instance, in the Au-Ag system, irradiation with He, Ne, Ar or Kr ions promotes a preferential extraction of Au from the alloy, resulting in the formation of Au-enriched satellite nanoparticles around the original Au<sub>x</sub>Ag<sub>1-x</sub> cluster. A systematic investigation on the role played by the irradiation parameters (i.e., dose, dose-rate, energy of the implanted ions) has been carried out. A correlation between the nuclear component of the energy released by the irradiating ions and the size of the satellite clusters is found. On the contrary, thermal annealing is found to promote preferential extraction of the less noble metal: in the case of Au-Cu system the preferential interaction of the incoming oxygen with copper promotes Cu<sub>2</sub>O formation, therefore extracting Cu from the alloy. Effect of ion irradiation on other systems (like In nanoclusters in SiO<sub>2</sub>) will be presented, showing a strong influence on the nanocluster size distribution.

SESSION T8: POSTER SESSION  
FUNDAMENTALS AND APPLICATIONS  
Wednesday Evening, April 23, 2003  
8:00 PM  
Golden Gate (Marriott)

### T8.1

SCALING BEHAVIOUR IN THE PENETRATION OF ENERGETIC SIZE-SELECTED CLUSTERS IN GRAPHITE. Carlos S. Navarro and Roger Smith, School of Mathematics and Physics, Loughborough University, UNITED KINGDOM; Sirapat Prantontep and R.E. Palmer, Nanoscale Physics Laboratory, University of Birmingham, UNITED KINGDOM.

Molecular dynamics (MD) simulations and experiments have been carried out to determine the penetration depth for implantation of

Ag, Au and Si clusters incident normally on graphite in the energy range 0.5-5 keV. The mean penetration depth for Ag<sub>n</sub> clusters is found to be linearly dependent on the impact velocity but there is a transition to a more linear scaling of depth with energy as the cluster size increases. Si cluster impacts induce deeper damage below the hole in which the cluster resides. An explanation for this behaviour is given in terms of the different nature of the collision cascades over picosecond time scales. The MD results are in good agreement with experimental measurements of the penetration depth.

### T8.2

Abstract Withdrawn.

### T8.3

METAL AND SEMICONDUCTOR CLUSTERS: THE ROLE OF THE ION ENERGY IN THEIR FORMATION AND REACTIVITY. Elsa Thune and Petra Reinke, Universität Göttingen II, Physikalisches Institut, Göttingen, GERMANY.

The rapidly growing interest in the deposition of size-selected nanoclusters on surfaces is motivated both by technology and by basic physical questions. Due to their finite size the small particles have totally different material properties compared to their bulk crystalline counterparts. Clusters have been deposited by a monoenergetic mass selected ion beam with low energies (10-500 eV) on amorphous carbon substrates, which are used to minimize the influence of surface crystallography and ion induced structural changes. Gold clusters were deposited as a model system to study the ion energy dependence, the temporal evolution and the influence of the temperature on the cluster distribution. For impact energies below 100 eV, surface processes dominate the clusters nucleation and growth. If higher energies are used, an increasing number of ions is implanted below the surface and different processes control the clusters formation. When the energy increases above 350 eV, the clusters size drastically drops below 5 nm. In a second set of experiments, clusters of different elements (Copper, Cobalt, Aluminum) are bombarded with reactive ion beams (Oxygen, Nitrogen), which will allow the formation of a wide variety of insulator, semiconductor and core-shell clusters. It will be discussed whether control over the progression of the reaction from the cluster surface to the core can be achieved by varying the ion energy. The clusters are analyzed with different methods such as Atomic Force Microscopy (AFM), Transmission Electron Microscopy (TEM) and X-Ray Photoelectron Spectroscopy (XPS) to determine their size distribution, composition and structure.

### T8.4

SPUTTER NANOWIRES FROM SOS SIMULATIONS.

Maria Stepanova and Steven K. Dew, University of Alberta, Dept. of Electrical and Computer Engineering, Edmonton, AB, CANADA.

We report a Monte-Carlo simulation of nanostructures created on surfaces under grazing-angle ion bombardment. We have developed an advanced solid-on-solid (SOS) model that describes the evolution of surface morphology due to sputter instability [R.M. Bradley and J.M.E. Harper, J. Vac. Sci. Technol. A 6, 2390 (1988)] and performed quantitative simulations for Cu bombarded by 1 keV Ar ions at 80° incidence. At early stages of the ion bombardment, we observe well-defined wire-like structures with the wavelength 2-5 nm, aligned parallel to the ion beam plane. Each wire also has a lateral 1-3 nm pattern, which appears to play an important role in causing wire coalescence at later stages of bombardment. After  $\sim 10^{17}$  ions/cm<sup>2</sup> hit the surface, we often observe a crossover to 10-50 nm sized ripples. To elucidate relevant control parameters, we have performed a series of model simulations playing with the spatial distribution of deposited energy and the surface relaxation. For the latter, we have considered the Mullins diffusion [W.W. Mullins, J. Appl. Phys. 28, 333 (1957)] and an atomistic relaxation mechanism that reduces the surface curvature [L.J. Friedrich, D.S. Gardner, S.K. Dew, M.J. Brett, and T. Smy, J. Vac. Sci. Technol. B 15, 1780 (1997)]. We demonstrate that both the ballistics and the relaxation are critical for the self-organized surface configuration. We compare our simulations with those from other authors and with experiments.

### T8.5

Abstract Withdrawn.

### T8.6

LIGHT-INDUCED MICROSTRUCTURAL ORDERING: STUDY OF LITHIUM-INCORPORATED COBALT OXIDE FILMS ON ALUMINIUM AND GLASS SUBSTRATE. Mahua Das, S.A. Shivashankar, Indian Institute of Science, Materials Research Centre, Bangalore, INDIA.

Photocrystallization is a phenomenon whereby an amorphous matrix undergoes structural changes to micro- or nano-crystals upon irradiation with photons. Topologically, a crystalline phase embedded in an amorphous matrix is expected to be advantageous over a fully

crystalline phase, in applications such as the cathode material in thin film lithium-ion rechargeable batteries. As this particular microstructural feature provides a high surface area and enables smooth contacts, it may be expected to result in fast ion conduction between cathode and electrolyte, while retaining the electrochemical property associated with the specific crystal structure of the material. Amorphous films of lithium-incorporated cobalt oxide were obtained on glass and aluminum substrate using a sol gel spin coating technique. The photocrystallization and microstructural changes in these films upon irradiation with unpolarised light were investigated using optical microscopy, XRD, SEM, and TEM analysis. The results of these investigations, as well as their relevance to lithium-ion battery applications will be presented.

**TS.7**  
OPTICAL PROPERTIES OF NANOBUBBLES INDUCED BY HIGH-ENERGY-HE-ION-MULTI-IMPLANTATIONS IN SILICON. Elisa Leoni, Gabrielle Regula, Rachid El Bouayadi, Maryse Lancin, Bernard Pichaud, TECSN-CNRS, Marseille, FRANCE; Esidor Ntsoenzok, CERI-CNRS, Orleans, FRANCE.

Silicon samples were implanted 12 times with helium ions at energies ranging from 0.8 MeV to 1.9 MeV using steps of 0.1 MeV with flux maintained between  $5 \times 10^{12}$  and  $1 \times 10^{13}$  He cm<sup>-2</sup>s<sup>-1</sup>. The dose was  $5 \times 10^{16}$  He cm<sup>-2</sup> for all energies but 0.8 MeV ( $3 \times 10^{16}$  He cm<sup>-2</sup>). This implantation sequence was carried out to obtain a  $5 \mu\text{m}$  wide dense band of bubbles from  $2.25 \mu\text{m}$  under the surface, down to  $7.5 \mu\text{m}$ , according to Monte Carlo simulations. During implantation, the temperature did not exceed 373K. After the multiple-implantations, the samples were cut into pieces and annealed at low (973K) or high temperature (1173K) for two hours under either neutral atmosphere (Ar) or argon hydrogen mixture. The samples were studied by cross section transmission electron microscopy (XTEM), photoluminescence (PL) and secondary ion mass spectroscopy (SIMS). An unexpected bubble distribution through the depth was observed by XTEM. The densities of the bubbles and their average diameter as measured by XTEM yield a porosity of about 5% and a mean distance between bubbles of about 10 nm. Such a bubble pattern can thus play the role of a porous structure. Moreover silicon remains crystalline and a low density of extended defects was observed by XTEM. This was confirmed by PL measurements in the infra-red range which show a broad band in the D1 region and still an efficient excitonic recombination. This indicates that carrier lifetime is not strongly reduced. Such a nano-structured silicon could be a good candidate for light emission in the visible range.

**TS.8**  
STUDY OF THE EFFECTS OF HEAVY-ION RADIATION ON THE NANOSTRUCTURE AND COMPOSITION OF SULFUR-DOPED NANOCOMPOSITE CARBON THIN FILMS. Oscar O. Ortiz, Department of Chemical Engineering, Polytechnic University, San Juan, PR; Nadia M. Medina-Emmanueli, Department of Chemistry, Pontifical Catholic University, Ponce, PR; Kathleen E. Kristian, Department of Chemistry, Swarthmore College, Swarthmore, PA; Adolfo González, Joel De Jesús, Iris M. Vargas, Department of Physics, University of Puerto Rico, San Juan, PR; Juan A. González, Department of Physics and Electronics, University of Puerto Rico, Humacao, PR; Brad R. Weiner, Department of Chemistry, University of Puerto Rico, San Juan, PR; Gerardo Morell, Department of Physical Sciences, University of Puerto Rico, San Juan, PR.

The compositional and microstructural transformations induced by simulated space radiation (i.e., heavy ions) on sulfur-incorporated nanocomposite carbon (nC:S) were investigated by Raman Spectroscopy (RS) and Scanning Electron Microscopy (SEM). Two identical sets of nC:S films were prepared in a hot filament chemical vapor deposition (HFCVD) system using CH<sub>4</sub>, H<sub>2</sub> and H<sub>2</sub>S. Films with various sp<sup>3</sup> C and sp<sup>2</sup> C bonding distributions were present within each set, which were obtained by varying the substrate temperature (300-900°C), the CH<sub>4</sub> concentration (0.3 and 2.0%) and the H<sub>2</sub>S concentration (0 and 500 ppm). One set of films was submitted to a 20 Krad dose of energetic Si and Fe ions at the NASA space radiation simulation facility hosted in Brookhaven National Laboratory's Alternating Gradient Synchrotron (AGS). All the films showed the characteristic diamond (tetragonal sp<sup>3</sup> C) band at around 1332 cm<sup>-1</sup> and the graphitic (trigonal sp<sup>2</sup> C) D and G bands at around 1350 and 1590 cm<sup>-1</sup>, respectively, evidencing their composite nature. Films with only submicron particles showed no significant morphological changes after being exposed to the ion radiation, while microcrystalline diamond particles underwent dramatic morphological improvement upon irradiation. Detailed quantitative analysis of the RS spectra indicated that significant changes in the aggregation state of sp<sup>2</sup> C and in the fraction of sp<sup>3</sup> C took place. The sp<sup>2</sup> C component showed a definite tendency towards aggregation upon irradiation, regardless of whether its total fraction was increasing or decreasing. However, the changes in the sp<sup>3</sup> C component were found to depend on the films' initial carbon bonding distribution. Films that

were initially rich in sp<sup>3</sup> C became less rich in sp<sup>3</sup> C and richer in sp<sup>2</sup> C after irradiation, and vice versa. These results suggest that stable nanocomposite carbon materials can be prepared, which would remain unchanged in their average composition while indefinitely absorbing ionizing radiation. Such behavior goes beyond the concept of radiation hardness and into radiation insensitiveness. These results are understood in terms of a carbon bonding interconversion hypothesis and indicate the possibility of fabricating carbon-based radiation buffers for protecting personnel and equipment from radiation.

**TS.9**  
ELECTRON FIELD EMISSION PROPERTIES AND MICROSTRUCTURES OF ION BEAM SYNTHESIZED AND MODIFIED SiC THIN LAYERS. W.M. Tsang, S.P. Wong, W.Y. Cheung, N. Ke, Dept of Electronic Engineering and Materials Science and Technology Research Centre, The Chinese Univ of Hong Kong, Hong Kong SAR, CHINA; J.K.N. Lindner, Univ of Augsburg, Institut für Physik, Augsburg, GERMANY.

It was reported several years ago that good electron field emission (FE) properties with a very low turn on field of about  $1 \text{ V}/\mu\text{m}$  could be obtained from ion beam synthesized (IBS) SiC/Si heterostructures [1]. More recently, by correlating the FE properties with the surface morphology and local surface conductivity, it was demonstrated that there were two distinct electric field enhancement mechanisms for electron FE from these IBS SiC samples [2]. Inspired by these understandings, modification of the IBS SiC layers by metal implantation was performed aiming at improving the FE properties. The SiC layers were synthesized by implanting carbon ions into Si wafers using a metal vapor vacuum arc (MEVVA) ion source at various energies and doses. Modification of the SiC layers was performed by tungsten implantation also by the MEVVA source. Characterization of the implanted samples were performed using atomic force microscopy (AFM), conducting AFM, Fourier transform infrared absorption spectroscopy, x-ray diffraction, x-ray photoelectron spectroscopy, and transmission electron microscopy. Excellent field emission properties with an ultra-low turn-on field of  $0.35 \text{ V}/\mu\text{m}$  have been achieved in some of these samples exhibiting a nanocomposite structure prepared under appropriate implantation and annealing conditions. The details of the FE properties, the structures and their dependence on the processing conditions will be presented and discussed. This work is supported in part by the Research Grants Council of Hong Kong SAR (Ref. CUHK4200/01E) and by the Germany-Hong Kong Joint Research Scheme of RGC, Hong Kong SAR and DAAD, Germany. [1] D. Chen et al, Appl. Phys. Lett. 72, 1926 (1998). [2] W.M. Tsang et al, Appl. Phys. Lett. 81 (2002) (in press).

**TS.10**  
IMMEDIATE MIXING OF DEPOSITED Co ATOM ONTO THE Al (001) SUBSTRATE WITH LOW INCIDENT KINETIC ENERGY OF 0.1 eV. Seung-Cheol Lee, Kwang-Ryeol Lee, Korea Institute of Sci. and Tech., Future Tech. Res. Div., Seoul, KOREA; Sang-Pil Kim, Yong-Chae Chung, Hanyan University, Dep. of Ceramic Eng., Seoul, KOREA.

Since the thickness of thin films for advanced devices becomes thinner down to a nanometer scale, understanding the atomic structure of the interface is essential for improving the device performances. Conventional models for thin film deposition have concentrated on the diffusion, nucleation and crystal growth of adatoms on substrate, without considering atomic scale interaction between the adatoms and the substrate. However, in a nanoscale multilayer, the atomic scale interaction with the substrate atoms to form an intermixing or alloying layer would be significant. In the present work, we investigated the interfacial structure evolution in Co/Al binary system, where Co-Al B2 compound is thermodynamically stable in wide range of temperature and composition. Classical molecular dynamics simulation using EAM potential was conducted on the initial stage of Co or Al deposition on single crystal Al at room temperature. In this simulation, Co or Al atoms of an incident kinetic energy 0.1 eV were deposited on Al (001) surface. We observed that over 90% of incident Co atom mixed with the Al substrate immediately after the deposition. When 2 ML of Co atoms are deposited, highly symmetric B2 structure was observed. On the contrary, only 4% of mixing is observed in the case of Al homoepitaxy. The activation barrier for intermixing with the Al (001) substrate was calculated to be about 0.6 eV, which is much larger than the incident kinetic energy. High degree of mixing could be understood in terms of the local acceleration of deposited Co atom near Al surface. In the case of Al homoepitaxy, increase in the kinetic energy by the local acceleration was much smaller than the activation barrier for the exchange of the Al atoms. Considering the high deposition flux on MD simulation, the formation of high symmetric and directional structure was also quite interesting. The detailed mechanism and structure evolution will be addressed.

**T9.1**  
AMORPHOUS CALCIUM PHOSPHATE NANOPOWDERS MADE FROM RADIO FREQUENCY PLASMA SPRAYING. Rajendra Kumar, Philip Cheang and K.A.Khor, Advanced Materials Research Centre, Nanyang Technological Univ, SINGAPORE.

Predominantly amorphous nano powders of calcium phosphate were produced by radio frequency plasma and subsequently characterised by particle-sizing SEM, TEM and XRD. Quantitative phase analysis was carried out by the Rietveld method. The starting feedstock comprised of hydroxyapatite powders spray dried from suspension. The input parameters were varied accordingly to produce predominantly amorphous calcium phosphate powders with Ca/P ratio close to 1.67. Results showed that as the plate power was increased and flow rate of feedstock was decreased the as-sprayed powders produced were largely amorphous. The size of the powders varied from 10-200nm depending on process parameters. SEM and TEM revealed spherical particles even smaller than 50nm. These powders are thought to have excellent bio-resorbability and are targeted for use in gene and drug delivery technologies. These nano sized powders are also envisioned to enhance mechanical properties of bulk products sintered by appropriate techniques.

**T9.2**  
INCREASING THE FRACTURE TOUGHENESS OF SILICON WITH ION BEAMS. J.G. Swadener, M.I. Baskes, M. Nastasi, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Our recent molecular dynamics (MD) calculations reveal that ion beams can be used to form nanostructured regions with greatly increased fracture toughness. In the MD simulations, ion implantation produced displacement spikes leading to the formation of clusters of disordered atoms. The presence of these disordered regions allowed silicon to deform plastically as a crack approached, blunting the crack tip and arresting crack growth. The MD calculations predict that fracture toughness can be increased by up to a factor of 3. This newly discovered toughening mechanism can also explain earlier experimental observations of increased fracture toughness in irradiated ceramics. In order to corroborate the MD results, silicon specimens have been implanted with Ne ions. Although detailed quantitative calculations are still on going, preliminary results show toughening in the implanted specimens that is consistent with the MD predictions. The combined experimental and atomistic method has enabled us to examine the details of fracture at the atomic scale, revealing how the fracture process converts energy from potential energy in the strained lattice into kinetic energy, surface energy and energy loss through distortions of the crystalline lattice.

**T9.3**  
PREPARATION OF CRYSTALLINE GALLIUM OXIDE RIBBONS ON GALLIUM ARSENIDE BY PLASMA IMMERSION ION IMPLANTATION AND RAPID THERMAL ANNEALING. H.P. Ho, S.P. Wong, Dept of Electronic Engineering, The Chinese University of Hong Kong, HKSAR, CHINA; K.C. Lo and P.K. Chu, Dept of Physics and Materials Science, City University of Hong Kong, HKSAR, CHINA.

We report for the first time the synthesis of gallium oxide ribbons by plasma immersion ion implantation of nitrogen/oxygen mixture followed by rapid thermal annealing. Transmission electron microscopy reveals that the long (>100 microns) ribbons are single crystals with very uniform size, approximately 0.1 micron in thickness and 1 micron in width. While a more detailed study on the formation mechanism is currently underway, we believe that our results might lead to the preparation of ordered materials which might be useful for sensor or photonic applications.

**T9.4**  
MAGNETITE/NICKEL AND MAGNETITE/COBALT MULTILAYER NANOSTRUCTURES OBTAINED BY PULSED LASER DEPOSITION. Monica Sorescu, Duquesne University, Department of Physics, Pittsburgh, PA; Agnieszka Grabias, Institute for Electronic Materials Technology, Warsaw, POLAND; Lucian Diamandescu, Institute of Atomic Physics, Bucharest, ROMANIA.

Multilayers containing magnetite have recently attracted considerable interest, since the construction of a magnetite-based all-oxide spin valve was proposed. In this paper we present a conversion electron Mossbauer spectroscopy (CEMS) study of magnetite/nickel and

magnetite/cobalt multilayers produced by pulsed laser deposition (PLD) and we follow the evolution of the site magnetic fields and populations as function of layer thickness. PLD was performed with an excimer laser at a wavelength of 248 nm and a pulse width at half maxima of 8 ns. A repetition rate of 10 Hz was used between laser pulses and an energy per pulse of 450 mJ was delivered. The deposition was performed using iron, nickel and cobalt targets in an oxidizing atmosphere. The multilayers obtained were magnetite/nickel and magnetite/cobalt, with a total thickness of 100-120 nm. Si (111) substrates were used in all cases. The CEMS spectrum of a magnetite/nickel multilayer was analyzed considering two sextets, corresponding to the tetrahedral (A) and octahedral (B) magnetic sublattices, typical of the magnetite structure. The dependence of the hyperfine magnetic fields of the two sublattices was derived as a function of the layer thickness. The magnetic hyperfine field of the tetrahedral sites is maximum for a layer thickness of 5 nm and reaches a minimum for a layer thickness of 10 nm, with saturation at large thicknesses. The site populations were also determined as functions of the layer thickness. The population of the tetrahedral site is minimum for a layer thickness of 10 nm and correspondingly, the population of the octahedral site is maximum for this thickness. In addition, we note that the magnetite structure is stoichiometric for this thickness, with an 1:2 areal ratio. The two magnetic sublattices become equally populated at large thicknesses. The results obtained for the magnetite/cobalt multilayers were in qualitative agreement with those obtained for the magnetite/nickel multilayers, with the exception of the presence of a pure iron layer in their structure.

**T9.5**  
Ge/Si QUANTUM DOTS NANOSTRUCTURE GROWN FROM ION-MOLECULAR BEAMS. A.V. Dvurechenskii, Z.V. Smagina, R. Groetzschel, A.K. Gutakovskii, Institute of Semiconductors Physics of the Siberian Branch of the Russian Academy of Science, Novosibirsk, RUSSIA. Research Center Rossendorf, Dresden, GERMANY.

Recently the effects of low energy (~100 eV) ion irradiation on surface islands nucleation and growth were found in the studies of Ge molecular beam epitaxy (MBE) on Si(100) and (111) with pulsed self-ion beam action on the growing layer. The irradiation results in reduction of critical thickness of pseudomorphic Ge layer at which there is a transition from layer-by-layer to three-dimensional growth. Reduction of the average island sizes, their dispersion and increase of their density is revealed [1]. Here we present the results of crystallinity investigation of Ge quantum dots embedded in Si structures grown with low energy ion irradiation. Structures were grown by MBE as the sequence of layers of Si on Si(100), Ge of 4-10 monolayers thickness and Si cap layer over Ge film. The substrate temperature varied in the range of 300-500°C with Ge heteroepitaxy. The pulsed (0.5 s) Ge low energy ion irradiation turned on at fractional monolayer coverage ≥0.8 for each grown layer. Beside pulsed action, continuous beam irradiation studied in similar growth regimes. The cap layer of 150 nm Si was grown at 500°C by common MBE (no irradiation). Normal incidents Rutherford back-scattering/channeling technique with 1.8 MeV He<sup>+</sup> ions has been used to study the yield from Si and Ge layers. The perfect structure with a yield of 2.5% was found in a mode of a pulse irradiation with number of deposited monolayers ≤5 at temperature 350°C. For lower temperature 300°C the yield exceeded 5% in similar structures. The enlarged yield was found also in the structures formed with continuous beam irradiation for 300-350°C temperatures of substrate. TEM studies show defect free Ge dots and Si layers for initial stage (≤5 monolayers) of heteroepitaxy in pulsed irradiation growth mode at ≥350°C. Continuous beam irradiation was found to induce dislocation around Ge dots. This work was supported by the RFBR Grant 02-02-16020 and INTAS-2001-0615. 1. A.V. Dvurechenskii, V.A. Zinoviev, Z.V. Smagina, JETP Letters, 2001, Vol.74, p.267-269.

**T9.6**  
ION-INDUCED RIPPLE FORMATION IN Cu(001). Wai Lun Chan, Niravun Pavenayotin, Eric Chason, Brown Univ, Division of Engineering, Providence, RI.

Ripple formation under off normal sputtering is a well known phenomena. Although a linear instability theory proposed by Bradley and Harper accounts for many of the observed phenomena on amorphous and semiconductor surfaces, it is not as successful for the case of metals. In contrast with the model, experiments on metals surface at low temperature have shown that the wavelength of the ripples may depend on ion fluence and the orientation of the ripples may depend on crystallographic direction instead of ion beam direction. In the present work, we have used in situ light scattering to measure the evolution of the surface of Cu(001) during sputtering. This technique enables us to measure the power spectral density of the surface over a range of spatial frequencies. We observe that ripples with a wavelength that is independent of time can indeed be produced on Cu(100) surfaces at higher ion flux and temperature than

previously used. The amplitude of the ripple is observed to increase exponentially during the early stages of growth. This work was supported by the U.S. Department of Energy under contract DE-FG02-01ER45913.

#### **T9.7**

Abstract Withdrawn.

#### **T9.8**

ARRAYS OF SUB-100-nm BLISTERS AND CRATERS PRODUCED BY LOW keV ION IMPLANTATION. O. Moutanabbir, N. Desrosiers, G.G. Ross, B. Terreault, INRS-EMT, Université du Québec, Varennes, Québec, CANADA.

Cavity formation, blistering and flaking induced by hydrogen and/or helium implantation followed by annealing have actual and potential applications in impurity gettering, and in silicon-on-insulator and molecular sieve fabrication. We have found that using low keV ions to produce such structures with sub-100 nm dimensions poses particular problems which challenge the usual models that were inferred from higher energy processing. This work aims at improving our understanding of the underlying mechanisms and increasing the efficiency of the process in terms of ion dose and annealing conditions. We implanted (100), (110) and (111) silicon crystals with 5 or 8 keV H, D and/or He ions at doses of  $1 \times 10^{16}$  to  $1 \times 10^{17}$  cm<sup>-2</sup>. We used Atomic Force Microscopy to quantitatively characterize the surface morphology, Thermal Desorption Spectrometry as a fingerprint of gas-lattice interaction, and the resistivity as an indication of defect evolution. The critical blistering doses and optimal annealing conditions have been determined for each ion singly or in combination. H and D ions behave remarkably differently: while a dose of  $6 \times 10^{16}$  D cm<sup>-2</sup> induced abundant blistering, no blisters were observed with the same H dose, due to insufficient defect production by low-keV H implantation to stably trap H. The density, size and morphology of the blisters and craters depend on the crystal orientation. Co-implantation of H and He decreases radically the critical blistering dose, and the order of implantation has a crucial effect on formation of blisters. No blisters were observed in samples implanted with H before He, while blisters and craters were very successfully produced by He implantation before H contrary to the belief that H implantation would "prepare the ground" for blistering by nucleating platelets parallel to the surface. The resistivity measurements show neat correlations with the morphology and the gas evolution, and in particular with the order of the implantation. Though convenient as an empirical signature of the underlying events, the resistivity is difficult to interpret physically, because it depends on both the carrier density, through the charge state of the H associated vacancies, and the carrier mean free path, through the nature and density of defects.