

SYMPOSIUM O

Ion Beam Synthesis and Processing of Advanced Materials

November 27 – 29, 2000

Chairs

David B. Poker

Oak Ridge National Laboratory
MS 6048
Oak Ridge, TN 37831
865-576-8827

Steven Moss

Aerospace Corp
MS M2-253
Los Angeles, CA 90009-2957
310-336-9216

Karl-Heinz Heinig

Forschungszentrum Rossendorf
Dresden, D-01314 GERMANY
49-351-2603288

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* Invited paper

SESSION O1: FUNDAMENTAL INTERACTIONS
AND MATERIALS MODIFICATION

Chair: David B. Poker
Monday Morning, November 27, 2000
Room 311 (Hynes)

8:45 AM *O1.1

ION BEAM MATERIALS INTERACTIONS: 20th CENTURY PHYSICS, 21st CENTURY REQUIREMENTS. L.C. Feldman, Department of Physics and Astronomy, Vanderbilt University, Nashville TN.

The basic interactions that govern the interaction of energetic ion beams with solids have their roots in the atomic and nuclear physics of the last century. The description of the ion-solid interaction provides a valuable quantitative guide to statistically meaningful quantities such as ranges, range straggling, sputtering coefficients and damage intensity and profiles. This talk will first review the underlying fundamental description of the ion-solid interaction and describe our understanding of the basic processes. New classes of experiments will be described (high ionization states, multiple ionization, cluster beams, finely focused ion beams) that require a non-linear description of the interaction and require substantial modifications to the existing theory. Atomic level materials modification places an even greater need for an atomic scale description of matter. The interaction of impurities, ion beam created defects and the control of these processes on the nanoscale, require improved experimental and theoretical approaches. As an example, our new infra-red absorption experiments, on extraordinarily low concentration hydrogen implants into silicon, give a description of the defect configuration around the individual implanted ion, providing new insights into fundamental interactions of the implantation process and the defect-impurity interaction. Co-workers: M. Budde, G. Leupke, C. Parks Cheney and N. H. Tolc.

SESSION O2: FUNDAMENTALS AND DEFECT
KINETICS I

Chair: Karl-Heinz Heinig
Monday Morning, November 27, 2000
Room 311 (Hynes)

9:15 AM *O2.1

IMPROVING THE UNDERSTANDING OF ION-BEAM-INDUCED DEFECT FORMATION AND EVOLUTION BY ATOMISTIC COMPUTER SIMULATIONS. Matthias Posselt, Forschungszentrum Rossendorf, Institute of Ion Beam Physics and Materials Research, Dresden, GERMANY.

During ion implantation ballistic processes lead to atomic displacements. The as-implanted defect structure is formed in subsequent athermal, rapid thermal and thermally activated processes. The knowledge of the type and the amount of defects created by a single ion impact is important for the physical understanding of ion beam processing in advanced technologies. In the present contribution a recently developed combination of time-ordered computer simulations based on the binary collision approximation (BCA) with classical molecular dynamics (MD) calculations is applied to examples relevant to Si technology. The method allows an effective calculation of depth profiles and the total number of different defect species (vacancies, self-interstitials and complex defects) formed on average per incident ion, and investigations on the temporal evolution of the defect structure up to several 100 ps after ion impact. The results obtained allow a microscopic interpretation of the phenomenological model employed in conventional BCA simulations to describe the enhanced dechanneling of implanted ions due to damage accumulation during implantation. In particular the explicit dependence of the dechanneling effect on the ion mass can be explained. Furthermore, the combined atomistic simulations can be applied to get more realistic initial conditions for the simulation of defect kinetics during post-implantation annealing than used so far.

9:45 AM O2.2

MODELING TRANSIENT ENHANCED DIFFUSION OF BORON FROM MEDIUM TO SUB-KEV ENERGIES. S.A. Centoni, M.-J. Caturla, T. Lenosky, B. Sadigh, S. Theiss, T. Diaz de la Rubia, Lawrence Livermore National Laboratory, Livermore, CA; J.J. Jimenez-Rodriguez, Universidad Complutense de Madrid, SPAIN.

Predictive modeling of dopant profiles under different temperature conditions are of great interest to the semiconductor industry. We have developed a model for defect migration and defect evolution in silicon using parameters obtained from ab initio calculations and from experiments whenever available. This model has been successfully applied to predict transient enhanced diffusion of boron for medium implant energies ($\approx 40\text{keV}$) and low doses (10^{13} to 10^{14} ions/cm²). New ab initio simulations have shown that di-interstitial silicon

clusters have a small migration energy ($\approx 0.5\text{eV}$). We have included this new migrating species in the model and compared with our previous results for this medium energy regime. However the most interesting processes for future devices are in the sub-keV implant regime and high doses. This low implant regime presents many challenges to device modeling. On one hand defects produced at these energies are closer to the surface, and therefore surface recombination plays a major role. On the other hand, defect profiles, the starting point of these simulations, are normally computed by binary collision models such as Marlowe. These models, however, are not reliable for energies lower than 1keV, and therefore, other more accurate methods, such as empirical molecular dynamics, are necessary in order to extract the defect profile for these energies. We have applied our model to sub keV implant energies. In order to do this we have computed the damage using molecular dynamics simulations and used this as input for the kMC model. This work was carried out under the auspices of the US Department of Energy by Lawrence Livermore National Laboratory under contract W-7405-Eng-48.

10:30 AM *O2.3

UNDERSTANDING ION BEAM SYNTHESIS OF NANO-STRUCTURES: MODELING AND ATOMISTIC SIMULATIONS. M. Strobel, Forschungszentrum Rossendorf, Dresden, GERMANY and CNR-IMETEM, Catania, ITALY; K.-H. Heinig, and W. Möller, Forschungszentrum Rossendorf, Dresden, GERMANY.

High-dose ion implantation is an established tool to synthesize buried low-dimensional nanostructures, which have become the basis of numerous technological applications. Depending on implantation parameters like ion fluence, ion flux and substrate temperature as well as on surface patterning, nanoclusters, nanowires and layers can be formed in a rather controlled manner. According to our current understanding of ion beam synthesis the evolution of nanostructures is determined by the competition between ballistic and thermodynamic effects. In general, standard thermodynamics, i.e. the phase transition from a supersaturated solid-solution to second-phase precipitates via homogeneous nucleation and growth, gives a reasonable explanation for a variety of observed nanostructures. However, under particular conditions ballistic processes can modify dramatically the phase-ordering kinetics, thus offering a means to achieve novel properties of nanostructures. Discussing various examples of ion beam synthesis it is shown, that kinetic 3D lattice Monte-Carlo simulations allow for an effective yet accurate modeling by combining collisional mixing with the standard thermodynamical model of phase transitions.

11:00 AM *O2.4

OPEN VOLUME DEFECTS IN SILICON: THEIR PRODUCTION AND PROPERTIES UNDER ION IRRADIATION. J.S. Williams, J. Wong-Leung, M.J. Conway, M.C. Ridgway, M. Petracic, Department of Electronic Materials Engineering, Research School of Physical Science and Engineering, The Australian National University, Canberra, ACT, AUSTRALIA; F. Fortuna, M.-O. Ruault, H. Bernas, Centre de Spectrometrie Nucleaire et de Spectrometrie de Masse, Centre National Recherche Scientifique, CNRS, Orsay, FRANCE.

Open volume defects in silicon generated by ion irradiation are currently of considerable interest both technologically and scientifically. For example, nanocavities, introduced by H- or He-implantation followed by annealing, are highly efficient gettering sites for fast diffusing metals. When the metal concentration is high, second phase precipitation can occur at cavities and the formation and dissolution of such a phase can be controlled by the availability and/or removal of silicon interstitials. In such cases, the system can exhibit non-equilibrium behaviour and require long annealing times to achieve thermal equilibrium. Furthermore, when nanocavities are irradiated with silicon ions, they can act as sinks for defects. Under certain conditions preferential amorphisation can occur around cavities. Voids can also be generated in silicon by direct self ion implantation and annealing. In this case, the separation in depth of the vacancy and interstitial excesses, resulting from displacement collisions, leads to interstitial clusters beyond the ion range and vacancy clusters at about half the ion range. During annealing the interstitial clusters coarsen into extended defects and the Rp/2 defects into voids. In this paper, many of the above processes are demonstrated and discussed. In particular, the interaction of metals with both nanocavities and voids is emphasised in an attempt to better understand the evolution of open volume defects under ion irradiation. Ion beam analysis and transmission electron microscopy are the main characterisation methods used.

11:30 AM *O2.5

EVOLUTION OF EXCESS VACANCY DEFECTS IN MEV IMPLANTED SILICON. V.C. Venezia, Lucent Technologies, Bell Labs, Murray Hill, NJ; L. Pelaz, Univ of Valladolid, Valladolid, SPAIN; H.-J. Gossmann, Lucent Technologies, Bell Labs, Murray Hill, NJ; T.E. Haynes, Oak Ridge National Laboratory, Oak Ridge, TN; A.

Agarwal, Eaton Corp., Beverly, MA; C.S. Rafferty, Lucent Technologies, Bell Labs, Murray Hill, NJ.

High-energy ion implantation into crystalline silicon creates a net defect distribution that is characterized by a concurrent excess of silicon interstitial defects, near the projected ion range (R_p), and an excess of vacancy defects near half the projected range. I will discuss the evolution of the region of excess vacancy defects created in silicon by a 2 MeV Si implant; from a large free vacancy supersaturation to the formation of stable vacancy clusters. Free vacancy supersaturations were measured with Sb dopant diffusion markers. Since the diffusion of Sb in silicon is mediated by vacancies, an enhanced Sb diffusion is related to an enhanced vacancy concentration. The Sb markers samples used in this work contained a buried silicon dioxide layer. The buried oxide was used to isolate the excess vacancy region from the excess interstitial region, making it possible to study the evolution of the excess vacancies independent of the interstitial defects near R_p . Stable clusters of vacancies were depth profiled using the Au labeling technique. This technique is based on the observation that Au is trapped at open volume defects, such as vacancy clusters, in silicon. Correlating the free vacancy supersaturation to the presence of vacancies in clusters, I show that a large free vacancy supersaturation dominates the region near R_p during the early stages of annealing, when interstitial-vacancy recombination is occurring. Afterwards, very stable vacancy clusters remain, which have a vacancy-binding energy that is similar to the formation energy of thermally generated vacancies. These clusters set a free vacancy concentration that is close to equilibrium. Results have been applied to an atomistic simulation of defect interactions to develop a predictive model of excess-vacancy evolution in MeV Si implanted silicon.

SESSION O3: FUNDAMENTALS AND DEFECT KINETICS II

Chair: Karl-Heinz Heinig
Monday Afternoon, November 27, 2000
Room 311 (Hynes)

1:30 PM O3.1

COORDINATION STRUCTURE OF IMPLANTED Mn IONS IN SILICA GLASS. Kohei Fukumi, Akiyoshi Chayahara, Hiroyuki Kageyama, Kohei Kadono, Naoyuki Kitamura, Hiroshi Mizoguchi, Yuji Horino and Masaki Makihara, Osaka National Research Institute, Dept of Optical Materials, Osaka, JAPAN.

When metal ions are implanted in glass, the implanted ions are present as metastable state at low local concentration. In this study, the valence and the coordination structure of Mn ions have been studied in silica glass implanted with 1×10^{15} to 2×10^{17} Mn^{+} ions cm^{-2} at energy of 180 keV by x-ray absorption spectroscopy. It was found that Mn ions were present as divalent state. The Mn-O bond length was 212 pm. Most of Mn ions were coordinated by 4-5 oxygen ions in the glass. The coordination polyhedra around Mn ions were much distorted. Manganese oxide crystals were, however, not formed in the glass. The formation of crystalline phases by post-annealing has been also studied.

1:45 PM O3.2

EFFECTS OF ELECTRONIC ENERGY DEPOSITION ON 2D-NANOPARTICLE DISTRIBUTION UNDER HIGH-FLUX Cu^{-} IMPLANTATION INTO INSULATORS. N. Umeda, Tsukuba Univ, Tsukuba, JAPAN; N. Kishimoto, Y. Takeda, C.G. Lee, National Research Institute for Metals, Tsukuba, JAPAN.

Application of negative ions attains efficient and accurate implantation into insulators, under a surface-charging-free condition. This advantage leads to applicability of a wide dose-rate range. High-flux Cu^{-} ions of 60 keV cause spontaneous formation of nanoparticles embedded in silica glasses, with a variety of particle morphology. The nanoparticle distribution is significantly different from the projected ion profile. The purpose of this paper is to study contribution of electronic energy deposition on the atomic rearrangement. Negative Cu ions of 60 keV irradiate substrates of silica glass(KU-1) at various dose rates up to $260 \mu A/cm^2$, at a total dose of 3×10^{16} ions/ cm^2 . Effective beam load is changed by using different contact-apertures (2 and 12 mm hole(s)) or different heat sinks. To compare with pure thermal effects, post-implantation annealing is also applied to the Cu-implanted specimens. Subsequently, nanoparticle morphology is examined by cross-sectional TEM. The nanoparticle distributions are compared with the prediction by the TRIM code. High-flux Cu^{-} implantation into the silica glass causes a bimodal distribution of Cu nanoparticles, i.e., particles of 10-20 nm in diameter around the projected range (~ 45 nm) and the smaller ones in the deeper region. The main peak of the Cu atomic density shows characteristic behaviors, i.e., dose-rate dependent narrowing and shallowing, with increasing dose rate.

Radiation-enhanced diffusion under a directional driving force plays an important role in the atomic rearrangement. There are two possible cases of the driving forces; nuclear(damage)- or electronic energy depositions. Since the atomic profile varies with changing the effective beam load, the atomic redistribution is not ascribed to athermal migration processes such as the inverse Kirkendall effect, but is associated with electronic-energy deposition including a possibility of beam heating effects. Electronic-energy effects and contribution of the beam heating are discussed in comparison with the thermal annealing effect.

SESSION O4: MATERIALS WITH NOVEL ELECTRICAL, OPTICAL, AND MAGNETIC PROPERTIES

Chair: Steven C. Moss
Monday Afternoon, November 27, 2000
Room 311 (Hynes)

2:00 PM *O4.1

ION IMPLANTATION OF OXIDES FOR OPTICAL APPLICATIONS. Chris Buchal Institut fuer Schicht- und Ionentechnik (ISI-IT), Forschungszentrum, Juelich, GERMANY.

Ion implantation of oxides causes damage, which is influencing the optical index. It also introduces donors and acceptors within the bandgap of the oxide, which change the photorefractive properties significantly. Luminescent ions in oxides are very sensitive to defects in their vicinity. We also discuss the impact excitation of rare earth ions, especially Erbium and Terbium, in oxides and the observed electroluminescence spectra. Hot electron impact excitation is characterized by high excitation cross sections.

2:30 PM O4.2

OPTICAL AND STRUCTURAL CHANGES OF Fe IMPLANTED SAPPHIRE. C. Marques, E. Alves, M.F. da Silva, J.C. Soares, Instituto Tecnológico e Nuclear, Sacavém, PORTUGAL and CFNUL, Lisboa, PORTUGAL; C. McHargue, Center for Mat. Processing, University of Tennessee, Knoxville, TN.

Single crystalline colorless $\alpha-Al_2O_3$ samples were implanted with several fluences of Fe^{+} ions in the range of 1×10^{16} to 5×10^{17} cm^{-2} at room temperature. Optical absorption and excitation measurements were carried out before and after annealing in reducing and oxidizing atmospheres. The structural changes were studied with RBS/channeling and x-ray diffraction. After implantation the damage induces a brownish coloration in the samples for fluences below 2×10^{17} cm^{-2} . The optical spectra are characterized by an absorption band centered at 200 nm. This band is strongly reduced after annealing at 1100°C in reducing atmosphere and a new well-defined band develops around 350 nm. This new band shifts to lower values with the implanted fluence, which is an indication of its correlation with the dimensions of the iron precipitates formed in the implanted region. The existence of these precipitates was confirmed by x-ray diffraction and TEM. The samples implanted with fluences above 1×10^{16} cm^{-2} annealed in oxidizing atmosphere display different optical spectra, with respect to the annealed in reducing atmosphere, characterized by an increase in the intensity of the peak at 200 nm. Luminescence measurements show the presence of F and F^{+} emission bands in the samples. The existence of these defects can be explained by the need of charge compensation and strain release due to the formation of mixed iron oxide and metallic precipitates. We will correlate the presence of these bands with the formation and dimension of those precipitates.

3:15 PM *O4.3

MeV ION BEAM DOPING OF DIAMOND. Steven Prawer and Russel Walker, University of Melbourne, Parkville, Victoria, AUSTRALIA; Cecile Uzan-Saguy and Rafi Kalish, Solid State Institute, Technion, Haifa, ISRAEL.

Diamond-based semiconductor devices offer the promise of operation at high temperatures, and under extreme radiation conditions. An essential step in the drive towards operational diamond-based electronic devices is the ability to controllably and reproducibly dope the diamond. Ion implantation is the method of choice for such doping because it offers precise control of the dopant concentration and spatially selective doping is achievable using standard masking techniques. However, compared to silicon, the doping of diamond is complicated by the tendency of the diamond to relax to graphite upon thermal annealing. Furthermore, even if graphitization can be avoided, the compensation of dopants by residual defects has proved in the past to be a limiting factor in obtaining very high mobility material. In this paper, we present a scheme for the effective doping of diamond using MeV implantation. For MeV implantation the doped layer is deeply buried under a cap of undamaged diamond and so the

scheme includes a method using pulsed laser irradiation for making electrical contact to the buried layer. Raman spectroscopy combined with molecular dynamics simulations are used to identify the defects responsible for the compensation/trapping of charge carriers. We show that a boron doped layer fabricated by the MeV implantation scheme has, after suitable annealing and removal of these compensating/trapping defects, very high mobility and low compensation ratio. In fact, its electrical properties are very similar to those of natural boron-doped type IIA diamond. The applicability of this scheme to the problem of creating n-type diamond layers is discussed.

3:45 PM O4.4

ION BEAM ASSISTED PULSED LASER DEPOSITION OF CoCrPt/Cr MAGNETIC RECORDING MEDIA. Mutsuhiro Shima, Caroline A. Ross, MIT, Dept. of MS&E, Cambridge, MA.

Longitudinal magnetic recording media are widely produced by sputtering Co-alloy magnetic films onto a Cr underlayer film. The crystallographic texture of the Cr underlayer governs the structure and magnetic properties of the overlying Co-alloy films, and greatly depends on the deposition conditions. It is commonly found that sputtered Cr films grow with (110) texture at low temperatures and (200) texture above about 200°C, but the exact mechanism for this is not completely understood. We have used pulsed laser deposition (PLD) to investigate Cr film texture formation. The advantage of using PLD over other deposition techniques includes the possibility of concurrent ion beam bombardment to modify the film properties. Ion beam bombardment is one of the most significant thin film process parameters in controlling the microstructure, particularly the film texture formation and grain size distribution. We have deposited Co-alloy/Cr films using PLD and have found that the texture of the Cr underlayer films is sensitive to the substrate temperature and low energy Ar ion bombardment (> 50 eV). For instance, Cr(002) texture is developed at 350 - 500°C and increased coercivity is observed from the Co-alloy layers, while Cr(110) texture is formed at 300°C and below. A Cr(002) peak is not observed from films grown at 400°C with low energy Ar ion bombardment, but ion bombardment during room temperature deposition promotes a Cr(002) texture. A moderate amount of ion bombardment could therefore be effective in promoting the Cr(002) texture at low deposition temperatures. In this work, we will report the effect of low energy ion bombardment on the Cr microstructure and texture formation, using He, Ne and Ar ion beams, and will describe how the magnetic properties of the Co-alloy film are modified by ion bombardment.

4:00 PM O4.5

MODIFYING THE STRUCTURE AND PROPERTIES OF CARBON NANOTUBES BY Ga⁺ IRRADIATION. B.Q. Wei, G. Ramanath, P.M. Ajayan, Department of Materials Science and Engineering, Rensselaer Polytechnic Institute, Troy, NY; H. Lezec, T.W. Ebbesen, ISIS, Laboratoire des Nanostructures, Strasbourg, FRANCE; P. Kohler-Redlich, Max-Planck-Institute für Metallforschung, Stuttgart, GERMANY.

We report the effects of Ga⁺ ion irradiation on the structure and electrical properties of multi-walled carbon nanotubes (MWNTs). Based upon our results, we demonstrate that ion irradiation is a potentially powerful tool for tailoring the properties of nanotubes for future nanoelectronics device applications. Arc-evaporated MWNTs were irradiated with a 10^{12} - 10^{17} cm⁻² dose of 30-130 keV Ga⁺ ions. At low ion doses and energies, the outer shells of the MWNTs remain intact, while the inner layers reorganize into highly ordered pillbox-like ~5-nm-dia nanocompartments of varying lengths between 2-20 nm. Increasing the dose above an energy-dependent threshold value results in the gradual disordering of the graphitic shells and destroys the nanocompartments. The disordered graphitic shells collapse into the hollow, thereby transforming the nanotubes into a homogenous amorphous rod. Apart from local bending or fracturing of the graphitic planes at isolated locations, high-energy (e.g., 130 keV) Ga⁺ ions do not have any observable effect on the MWNT structure. Four-point probe measurements of individual nanotubes show that the pristine MWNTs are semiconducting, with a band gap $E_g = 194 \pm 20$ meV. Ga⁺ irradiation decreases both the electrical resistivity and the band gap. The changes in electrical properties of nanotubes are explained in terms of the local defect generation and implantation in nanotubes. Possible applications of ion-irradiated nanotubes are also discussed.

4:15 PM O4.6

ION BEAM INDUCED GRAPHITIZATION OF TETRAHEDRAL AMORPHOUS CARBON: ELECTRICAL AND MECHANICAL CHANGES. R.J. Hohlfelder, D.R. Adkins, B.L. Doyle, T.A. Friedmann, P.G. Kotula, W.K. Schubert, J.P. Sullivan, Physical & Chemical Sciences Center, Sandia National Laboratories, Albuquerque, NM.

We have investigated structural changes produced by the ion beam

irradiation of non-hydrogenated tetrahedral amorphous carbon ("amorphous diamond", a-D) films. The as-deposited films, grown by pulsed laser deposition, consist primarily of tetrahedrally bonded (sp³) carbon. The films are correspondingly diamond-like, displaying extremely high hardnesses and elastic moduli. The films also have high compressive stresses in their as-deposited state, but stress can be completely relaxed or made slightly tensile by thermal annealing with no adverse effect on other properties. These properties make the material attractive for a number of applications, including flexural plate wave (FPW) sensors, which we have successfully fabricated. Graphitization, the transformation of carbon tetrahedral (sp³) bonds to planar (sp²) bonds, can be induced by ion bombardment. This transformation is associated with a large increase in electrical conductivity and with changes in residual stress and elastic modulus of the a-D films. We have used high resolution EELS measurements, electrical properties measurements, and bulge testing (pressurization of free-standing membranes), to study the structural, electrical, and mechanical changes associated with ion beam irradiation.

4:30 PM O4.7

ION-BEAM PROCESSING OF AND THIN-FILM GROWTH ON POLYMERIC MATERIALS: COMPARISON OF COMPUTATIONAL SIMULATIONS AND EXPERIMENTAL RESULTS. Susan B. Sinnott, Boris Ni, Robert Mania, Univ of Kentucky, Lexington, KY; Muthu B.J. Wijesundara, and Luke Hanley, Univ of Illinois-Chicago, Chicago, IL.

Polyatomic ions and neutrals are expected to play a critical role in plasma-induced surface chemistry, especially in the deposition of polymeric films from plasmas. In this work low-energy CH₃⁺ and C₃H₅⁺ ions are impacted on polystyrene and polyethylene surfaces in classical molecular dynamics simulations. The simulations use a many-body, reactive empirical bond-order potential coupled to long-range Lennard-Jones potentials. The results are used to quantify the effect of the unique chemistry and structure of the incident ions, incident energy, and surface structure on the results. Some of the simulation predictions are compared to experimental data for 25 - 100 eV CF₃⁺ and C₃F₅⁺ ion-modification of a polystyrene surface, as analyzed by x-ray photoelectron spectroscopy. The simulations predict that CH₃⁺ and C₃H₅⁺ behave in a similar fashion to their fluorocarbon analogs, remaining mostly intact and either embedding or scattering from the surface without reacting at 20 eV. At 50 and 100 eV, the simulations predict fragmentation most or all of the time. The simulations also show that the chemical products of the collisions depend significantly on the structure of the incident isomer. Finally, the effect of the polymer surface structure is examined.

4:45 PM O4.8

ENHANCEMENT OF POROSITY AND SURFACE ROUGHNESS OF CURED PHENOLIC RESIN BY ION IMPLANTATION. R.L. Zimmerman, D. Ila, and A.L. Evelyn, Center For Irradiation of Materials, Alabama A&M University, Normal, AL; D.B. Poker and D.K. Hensley, Oak Ridge National Laboratory, SMAC, Oak Ridge, TN.

We will present our most recent results using ions such as O, Fe, Zn, and Au at energies between 50 keV to 2 MeV to increase the roughness and porosity of the partially and fully cured precursor phenolic resins. The fully cured phenolic resins is called Glassy polymeric carbon (GPC), which is chemically inert and biocompatible that it is why it is used for medical applications, such as heart valves and other prosthetic devices. The ion implantation process enhances biological cell/tissue growth on, and to increase tissue adhesion to, prosthetic devices made from GPC. In our previous publications, we have shown that the increased porosity is also used for GPC biomaterials which are used for Drug Delivery devices. The porosity of the ion implanted partially and fully cured precursor phenolic resins were measured by introducing lithium from a molten LiCl salt into each sample and then by using (p,α) nuclear reaction analysis (NRA) we measured the concentration of Li retention in the pre- and post-implanted samples. The surface roughness were measured using optical microscopy. The curing process was monitored using micro-Raman microscopy. We have correlated the NRA measurements of increased pore availability with the observations of increased surface roughness.

SESSION O5: POSTER SESSION

Chairs: Karl-Heinz Heinig, Steven C. Moss and David B. Poker

Monday Evening, November 27, 2000
8:00 PM

Exhibition Hall D (Hynes)

O5.1

SECONDARY ION MASS SPECTROMETRY (SIMS) WITH GAS

CLUSTER ION BEAMS. Noriaki Toyoda^{1,3}, Jiro Matsuo², Takaaki Aoki², Isao Yamada², David B. Fenner³, Richard Torti³, Jim Greer³ and Allen Kirkpatrick³. ¹Massachusetts Institute of Technology, Material Processing Center, Cambridge, MA; ²Ion Beam Engineering Experimental Lab., Kyoto University, Kyoto, JAPAN; ³Epion Corporation, Billerica, MA.

Secondary Ion Mass Spectrometry (SIMS) has been used extensively to study the depth profile of dopants in semiconductors because of its low detection limit and relatively good depth resolution. However, the reduction of depth resolution due to ion-mixing by energetic primary ions is substantial. To improve the depth resolution, an ultra-low energy ion beam should be used, although it is difficult to get a high-sputtering yield at low impinging energy. Also, to get a high secondary ion signal in the low-energy regime, primary ions typically bombard the target at an oblique angle, which sometimes causes roughening of the surface and degradation of depth profiles. Instead of using atomic or small molecular-ion beams, larger molecular ions have also been used as the primary ion. By comparison, the Gas Cluster Ion Beam (GCIB) technique will be a good candidate for the primary ion of SIMS because it is a low-energy ion beam and shows a much higher sputtering yield than monomer ions with the same acceleration energy. For clusters of 2000 atoms and acceleration of 20 kV, each atom has an average of only 10 eV. This will help to reduce ion mixing and improve the depth resolution. Moreover, GCIB has excellent surface smoothing effects at normal incidence. Usually, the surface roughness can be reduced below 1nm with GCIB, therefore, there will be no degradation at high ion dose. In this study, an Ar cluster beam is used as the primary ions for SIMS. Preliminary experimental results are reported by analyzing with Residual Gas Analyzer and Secondary Ion Mass Spectrometer. In addition to the experimental result, Molecular Dynamics (MD) simulations are performed to clarify the kinetics of primary and target atoms in the solid. Project at Epion supported by U.S. DoC-NIST ATP and by U.S. NSF SBIR.

O5.2

SIMULATIONS AND ARGON-CLUSTER ION SMOOTHING OF SURFACES. D.B. Fenner, Epion Corp, Billerica MA; D.W. Dean, Studsvik Scandpower, Inc., Newton MA; L.P. Allen and J. Hautala, Epion Corp, Billerica MA; P.B. Mirkarimi, Lawrence Livermore National Laboratory, CA.

The impact of weakly-bound clusters of argon atoms is known to reduce the fine-scale roughness of many solid surfaces. With engineering development this may lead to a technology that will have a wide scope of application in microelectronic and photonic manufacturing. Empirically, bombardment with such energetic cluster ions is known to result in an exponential decrease in roughness toward an asymptotic value as the ion dose accumulates. The impact of each cluster is an isolated event and creates a small crater in the surface with a volume proportional to the cluster kinetic energy. Associated with the impulse of the impact event, a small amount of the surface material will be evaporated and a small amount transported laterally. Nonplanar features on the surface will be eroded stochastically at large impact areal density. The argon clusters for smoothing are typically a few thousand atoms, with diameters of several nanometers, and at a dose density such that a few thousand collisions have occurred within a crater diameter. Here we report comparison with a phenomenological model having a rate equation for the surface height undergoing multiple cluster impacts. With suitable but simple assumptions the time evolution of an initially nonplanar surface can be simulated. Qualitatively, both experiment and simulation show that sharp steps and asperities are rapidly eroded. Randomly rough surfaces show roughness reduction to be increasingly rapid at higher spatial frequencies up to that approximately corresponding to the cluster size. In an effort to better understand the mechanism of smoothing empirically and by way of simulation, we have also used argon clusters to erode gold balls, about 35-nm diameter, dispersed at a low density over an extremely smooth silicon-wafer surface. The work at Epion was supported by NIST-ATP.

O5.3

THE FATE OF A CLUSTER COLLIDING ONTO A SUBSTRATE - DISSIPATION OF TRANSLATIONAL KINETIC ENERGY OF THE CLUSTER. Seiichi Takami, Kenji Yajima, Ken Suzuki, Momoji Kubo, Akira Miyamoto, Tohoku Univ, Dept of Materials Chemistry, Sendai, JAPAN.

The fate of clusters emitted onto a substrate falls into several categories including repulsion, soft landing, migrating, and explosion, depending on the interaction between them. These dynamic behaviors of the clusters control thin-film formation processes such as cluster ion beam deposition and sputtering. This collision process involves the dissipation of translational kinetic energy of clusters into thermal energy as well as deformation energy of both the substrate and clusters. We studied the dissipation of the energy during the collision processes using molecular dynamics simulation. The simulation

elucidated that the activation of thermal vibrational energy of the substrate immobilized the emitted cluster on the substrate. We will discuss the importance of phonon coupling between the cluster and substrate.

O5.4

RADIATION DAMAGE EFFECTS IN TiO₂ THIN FILM ON TITANIUM DUE TO ION IMPLANTATION OF GOLD AT VARYING ENERGIES AND DOSES. Nancy Ruzycski, and G. Glass¹, U. Diebold, D.L. Ederer, W.A. Hollerman¹ and P. Flemming², Tulane University, Department of Physics, New Orleans, LA. ¹Acadiana Research Lab, University of Louisiana at Lafayette, Lafayette, LA; ²Oak Ridge National Laboratory, Oak Ridge, TN.

TiO₂ is a common catalyst used in industrial applications. While several studies have looked at the effects of low energy ion sputtering and implantation on the thin film characteristics, few studies have been undertaken on the effect of MeV implantations at varying energies and ion fluences. The thin films were characterized with XPS and found to be TiO₂, with Si and SiO impurities. Rutherford Backscattering (RBS) was used to profile the implanted thin films of TiO₂ e-beam deposited on the diamond polished titanium samples. Samples were implanted with gold ions at fluences of 4 X 10¹², 4 X 10¹³, 4 X 10¹⁴, 4 X 10¹⁵ ions/cm² and energies of 1.5, 3.0, 6.0 and 8.33 MeV. The RBS results of the study indicate that the oxygen in the thin films was relatively unchanged by the ion implantation showing a 16% difference in oxygen area, while the titanium and silicon regions showed little difference when analyzed both by dose and energy. There were no changes in the concentration ratio of titanium to oxygen in the samples. An additional two samples implanted at fluences of 2.5 X 10¹⁵ and energies of 1.5 and 8.32 MeV were analyzed using Soft X-Ray Spectroscopy. The Total Electron Yield (TEY) and Partial Fluorescence Yield (PFY) graphs at the oxygen K-edge indicate that the films in the samples are similar in nature and unchanged by the ion implantation. This was confirmed by the emission spectrum for the oxygen K-edge. The emission spectra for the Titanium L-edge shows that the unimplanted samples were more TiO like than TiO₂ prior to implantation and that the high energy implantation changed the sample more nearly to TiO₂ with the low energy implantation showing a lesser effect. An additional sample was analyzed using Soft X-ray Spectroscopy for the Silicon L-edge with the result that the unimplanted sample was SiO like prior to implantation and more SiO₂ like after implantation.

O5.5

MEASUREMENT OF SPUTTERING COEFFICIENT DURING HIGH DOSE SELF- ION IMPLANTATION INTO SILICON USING A GOLD MARKER. X.F. Zhu, Department of Electronic Materials Engineering, Research School of Physical Science and Engineering, Australian National University, Canberra, AUSTRALIA and Frederick Seitz Materials Research Laboratory and Department of Materials Science and Engineering University of Illinois at Urbana, IL; J.S. Williams, Department of Electronic Materials Engineering, Research School of Physical Science and Engineering, Australian National University, Canberra, AUSTRALIA; Ian Robertson, Frederick Seitz Materials Research Laboratory and Department of Materials Science and Engineering University of Illinois at Urbana, IL.

Sputtering is a very important parameter during high dose implantation in determining whether there is a net loss (by erosion) or gain (by deposition) of material. Therefore, the sputtering coefficient must be measured in order to interpret the evolution of the implanted structures during high dose implantation. The sputtering coefficient is not only a function of the mass ratio for target and ion, ion energy, and ion incidence angle from the normal of the wafer, but can also depend on local structure and bonding effects. In high dose implantation cases, it is often not straightforward to calculate sputtering coefficients and it is necessary to resort to measurement. For self ion irradiation, the sputtering coefficient can be difficult to measure accurately since this usually requires a marker layer to be placed below the irradiated region. In this paper, the sputtering coefficient for the 40 keV Si self-implantation to high dose was determined by a Au marker technique. A three -step process was established for the routine measurement. The three steps include Au marker preparation via Au implantation into a buried a-Si layer and subsequent epitaxial regrowth to segregate Au into a narrow layer, high dose self ion implantation, and finally Rutherford backscattering/channeling measurement of Au profile. This measurement procedure has proven to be consistent and accurate. For a huge dose of 10E18 cm-2 Si ions at 40 keV, the sputtering coefficient is less than 1, resulting in the effective accumulation (deposition) of several hundred layer of excess Si.

O5.6

VACANCY CONCENTRATIONS IN ION-IRRADIATED Cu₃Au. Luncun Wei, C.P. Flynn and R.S. Averback, Materials Research Laboratory, University of Illinois at Urbana-Champaign, Urbana, IL.

By measuring resistivity change under different ion flux irradiation in epitaxially grown Cu_3Au thin film, the absolute vacancy concentration in Cu_3Au is calculated by fitting to mean field theory of the experimental data. At 607K, data yields thermal vacancy concentration of 1×10^{-8} .

O5.7

KINETICS STUDY OF Si NANOCRYSTALS ION BEAM SYNTHESIZED IN SiO_2 . C. Bonafos, M. Carrada, B. Colombeau, G. Ben Assayag, A. Clavierie, CEMES/CNRS Toulouse, FRANCE; B. Garrido, M. Lopez, A. Perez-Rodriguez, J. R. Morante, EME-Dep. d'Electronica, Univ. de Barcelona, Barcelona, SPAIN.

Most studies concerning group IV (Si, Ge) nanocrystals synthesized by ion implantation in SiO_2 have shown that a link exists between the observed physical properties and the characteristics of the populations of nanoparticles. These characteristics are their size-distribution, density and volume fraction. In order to establish this link, we have studied the influence of the initial Si excess and annealing conditions (temperature, duration) on the characteristics of a Si nanocrystal population. Different Transmission Electron Microscopy (TEM) imaging conditions have been tested and Dark-Field conditions have been shown to be the most adequate for a quantitative study. The kinetic behavior of Si nanoparticles has then been studied by coupling TEM measurements and atomistic simulations. During annealing, the growth of these nanoparticles is very slow when increasing the annealing time while their size significantly increases when increasing the initial Si excess. The activation energy for the precipitate growth has been deduced from the isochronal evolution and has been evaluated to be around 2.8 eV. The increase of the size when increasing the initial Si excess is predicted by the simulations only when taking into account interaction effects between particles via their diffusion field.

O5.8

THE LOSS KINETICS OF SUBSTITUTIONAL CARBON IN $\text{Si}_{1-x}\text{C}_x$ REGROWN BY SOLID PHASE EPITAXY. Yong Jeong Kim, Tae-Joon Kim, Byungwoo Park, Seoul National Univ, School of MS&E, Seoul, KOREA.

Heteroepitaxial $\text{Si}_{1-x}\text{C}_x$ materials have been extensively investigated for bandgap engineering due to available applications to electron channel by conduction-band offset. Epitaxial layers of $\text{Si}_{1-x}\text{C}_x$ ($x = 0.008-0.016$) were synthesized using ion implantation and solid phase epitaxy (SPE), and the loss kinetics of substitutional carbon was investigated. As annealing temperature and time increase, more carbon atoms diffuse from substitutional to interstitial sites, eventually forming nano-size SiC precipitates. The activation energy for the loss of substitutional carbon into interstitial sites was obtained over a temperature range of 700-1040°C using high-resolution x-ray diffraction (HR-XRD) and Fourier transform infrared spectroscopy (FTIR). With HR-XRD analysis, the substitutional carbon contents were obtained from the dynamic simulations of x-ray rocking curves, and activation energy of 2.91 ± 0.18 eV was obtained. The activation energy was also measured using FTIR with the integrated peak intensity at 607 cm^{-1} range to quantify the fraction of substitutional carbon. Both methods yield similar activation energies for the loss kinetics. Rutherford backscattering spectrometry (RBS) channeling spectra and cross-sectional transmission electron microscopy (XTEM) studies show that rapid thermal annealing (RTA) looks more suitable for SPE regrowth of $\text{Si}_{1-x}\text{C}_x$ than vacuum furnace annealing, leading to greater structural perfection for the regrowth of implanted layers. In addition, the effects of carbon concentration on the loss kinetics will be discussed.

O5.9

PROPERTIES OF GALLIUM DISORDER AND GOLD IMPLANTS IN GaN. W. Jiang, W.J. Weber, S. Thevuthasan, Pacific Northwest National Laboratory, Richland, WA.

Epitaxial single-crystal GaN films on sapphire were implanted 60° off the $\langle 0001 \rangle$ surface normal with 1.0 MeV Au^{2+} ions over a range of fluences from 0.4 to 53.8 ions/nm^2 at 180 and 300 K. The implantation damage was studied in-situ using conventional Rutherford backscattering spectrometry in channeling geometry. The relative disorder at the damage peak on the Ga sublattice for Au^{2+} implantation at room temperature exhibits four stages that consist of gradual disorder accumulation below 1 dpa, a rapid disorder increase between 1 and 6 dpa, an intermediate stage between 6 and 20 dpa, and a rapid amorphization process at higher doses. Room temperature migration of Au implants in GaN is observed during ion implantation. As a result of thermal annealing at 870 K for 20 min, some Au implants in GaN diffuse into the amorphized surface region, while the remaining Au atoms stay near the mean ion-projected-range and may be trapped at defects in the crystal structure. Full amorphization in GaN at the damage peak requires a dose of about 40 dpa at 300 K, which is about two orders of magnitude higher than for SiC under the

similar irradiation conditions. The result suggests a high mobility of both Au implants and Ga defects in GaN. Additional results regarding the locations of the gold implants in the crystal structure, obtained from ion channeling analysis along different crystallographic axes, will also be presented and discussed.

O5.10

DYNAMICAL X-RAY DIFFRACTION ANALYSIS OF SOLID PHASE EPITAXY GROWTH OF $\text{Si}_{1-y}\text{C}_y$ HETEROSTRUCTURES. J. Rodriguez-Viejo, Z. el Felk, Grupo de Fisica de Materiales I, Dep Fisica, Univ Autonoma de Barcelona, Bellaterra, SPAIN.

We have studied the formation of partially strain-relaxed $\text{Si}_{1-y}\text{C}_y$ films obtained by Solid Phase Epitaxy (SPE). The strain in the $\text{Si}_{1-y}\text{C}_y/\text{Si}(100)$ heterostructures was measured fitting the triple crystal X-ray rocking curves using dynamical X-ray diffraction theory. Carbon depth profiles were monitored by Secondary Ion Mass Spectrometry (SIMS). Si implantation ($180 \text{ keV}, 5 \times 10^{15} \text{ Si at cm}^{-2}$) at liquid nitrogen temperature forms a buried amorphous layer. HRTEM shows that annealing at temperatures close to 650°C results in epitaxial films with significant defect recovery and the formation of microtwins and stacking faults along 111 planes. C implantation, at $60 \text{ keV}, 7 \times 10^{15} \text{ cm}^{-2}$ and 450°C , in the preamorphized Si wafers results in the growth of $\text{Si}_{1-y}\text{C}_y$ epitaxial films with a low amount of substitutional carbon ($y \sim 0.1\%$). In those films X-ray rocking curves show the existence of interference fringes on the left hand side of the 004 Si peak indicating the presence of tensile strained Si layers. This is related to Si interstitials produced during the implantation process. Rapid thermal annealing at 750°C induces epitaxial films with a carbon content close to 0.5%. At this temperature HRTEM reveals the existence of rod-like 113-defects. The amount of interstitial Si also decreases drastically with increasing temperature. At 950°C , both a complete recovery of the Si defects and a significant loss of substitutional carbon is observed. A complete analysis using dynamical X-ray diffraction theory of the amorphization, defect formation and strain in the heterostructures grown by SPE is presented.

O5.11

OPTICAL PROPERTIES OF HIGH DOSE ION IMPLANTED THIN LAYERS OF METAL CLUSTERS EMBEDDED IN SILICA GLASS. P.S. Chung¹, S.P. Wong¹, W.Y. Cheung¹, N. Ke¹, W.K. Lee², C.W.

Chan². ¹Department of Electronic Engineering. ²Department of Physics The Chinese University of Hong Kong Shatin, NT Hong Kong, CHINA.

Thin layers of metal clusters in silica glass were formed by high dose ion implantation of Ag, Ni and Cu using a metal vapor vacuum arc (MEVVA) ion source. The ion distribution and the layer thickness were determined by Rutherford backscattering spectroscopy. The cluster size and distribution was observed by transmission electron microscopy. The linear and nonlinear optical properties of the implanted samples were studied using spectroscopic ellipsometry and the z-scan technique. The effective complex index of refraction in the wavelength range from $0.35 \mu\text{m}$ to $1.6 \mu\text{m}$ of the implanted layers consisting of metal nanoclusters embedded in silica were deduced by fitting of the ellipsometric spectra using Maxwell-Garnett effective media approximation. The nonlinear refractive index of the implanted samples was determined by the z-scan method, using self mode-locked Ti: sapphire laser delivering 130fs long, linearly polarized pulses at a wavelength of 700nm at 76MHz. Values of n_2 up to $0.595 \text{ cm}^2/\text{gW}^{-1}$ were measured in the case of a sample co-implanted with Cu and Ni ions. The details of the optical properties, the microstructures, their correlation, and their dependence on the implantation condition will be reported and discussed. This work is supported in part by the Research Grants Council of Hong Kong SAR (Ref. No.: CUHK374/96E).

O5.12

NANOPARTICLES OF METALLIC COBALT AND NICKEL PREPARED BY ION IMPLANTATION INTO SiO_2 . O. Cántora-González, C. Estournés, M. Richard-Plouet, J.L. Guille, Groupe des Matériaux Inorganiques Institut de Physique et Chimie des Matériaux de Strasbourg (UMR7504 CNRS-ULP-ECPM) Strasbourg, FRANCE; D. Muller, J.J. Grob, Laboratoire PHASE (UPR292 CNRS) Strasbourg, FRANCE; A. Traverse, LURE (UMR130 CNRS), Université de Paris-Sud, Orsay, FRANCE.

The structural and magnetic properties of nano-sized particles of transition metals (Co and Ni) implanted into amorphous SiO_2 are investigated. The SiO_2 substrates used were either as grown on a silicon (001) wafer under dry or wet O_2 condition or a piece of bulk silica glass. The metals were implanted as singly charged atoms energized to 30 or 160 keV. In all cases, particles of metals were observed by diffraction and micrography using transmission electron microscopy (TEM) on freshly prepared samples. Two regions were observed; the first region which is close to the surface is characterized

by nanometric fcc nickel or fcc and hcp cobalt and the second region has sub-nanometric particles. The thickness of the first zone increases with the energy of the charged atoms and the size of the particles varies from 2 to 6.5 nm. XANES and EXAFS at the K-edge of the transition metal reveal the presence of both metallic and metal-oxide particles; the latter essentially present in the second region may be due to free oxygen diffusion during implantation. A marked difference is observed for the two metals in the different substrates. When Co^+ is implanted, the amount of the two types of particles depends on the nature of the substrate; the more OH groups present in the glass the larger is the content of metal particles. This behavior is not clear-cut for the case of nickel.

In every conditions (energy of implanted ions and substrate) the nickel particles behave as super-paramagnets at 300 K. On the other hand, cobalt particles behave as ferromagnets for an implanted energy of 30 keV and super-paramagnetic for 160 keV. These results are as expected for the determined particle sizes and crystal systems using TEM.

O5.13

MODIFICATION OF THE ELECTRONIC PROPERTIES OF $a\text{-Si}_{1-x}\text{C}_x\text{:H}$ BY Fe^+ ION IMPLANTATION. T. Tsvetkova, S. Balabanov, Inst. Solid State Physics, Sofia, BULGARIA; B. Amov, Ch. Angelov, Inst. Nucl. Res. & Nucl. Energy, Sofia, BULGARIA; J. Zuk, D. Maczka, M. Curie-Sklodowska University, Lublin, POLAND; G.J. Adriaenssens, K. Jakubovskii, University of Leuven, BELGIUM.

The electrical and optical properties of hydrogenated amorphous silicon carbide ($a\text{-Si}_{1-x}\text{C}_x\text{:H}$) alloy films, modified by Fe^+ ion implantation ($D = 10^{16} - 10^{17} \text{ cm}^{-2}$), have been investigated. Optical transmission spectra were measured in the visible range (400-900 nm) and a considerable absorption edge shift to the longer wavelength region was registered, together with a well-defined decrease of transmittance over the whole measured range. These effects are increased with the dose and are similar for samples with carbon contents of $x = 0.08$ and $x = 0.15$. Room temperature (RT) surface electrical conductivity is also increased by Fe^+ implantation and the effect is most pronounced for the highest doses. The temperature dependence of the surface conductivity was measured in the temperature range from RT to $\sim 250^\circ\text{C}$ and the electrical conductivity activation energy was determined. The activation energy is considerably reduced by the implantation and the effect is again strongest for the highest doses. The above results lead to the general conclusion that high-dose Fe^+ implantation in $a\text{-Si}_{1-x}\text{C}_x\text{:H}$ affects both the localized states deep in the gap and the shallow states in the band tails, thus effectively reducing the optical bandgap while also modifying the whole spectrum of charged defect states. The support of the Bulgarian Academy of Sciences, of the Flemish FWO, and of M. Curie-Sklodowska University, Lublin, Poland, is gratefully acknowledged

O5.14

GOLD NANO-CLUSTER FORMED BY MEV ION IMPLANTATION INTO Bi_2TeO_5 . A. Kling, M.F. da Silva, J.C. Soares, Instituto Tecnológico e Nuclear, Sacavém, Portugal and Centro de Física Nuclear da Universidade de Lisboa, Lisboa, PORTUGAL; P.F.P. Fichtner, L. Amaral, F.C. Zawislak, Instituto de Física, Universidade Federal do Rio Grande do Sul, Porto Alegre, RG, BRAZIL; I. Földvári, Research Laboratory for Crystal Physics, Hungarian Academy of Sciences, Budapest, HUNGARY.

Bismuth tellurite (Bi_2TeO_5) has recently emerged as new non-linear optical material with interesting properties for opto-electronic applications. On the other hand the formation of noble metal nanoclusters which exhibit important third-order non-linear optical properties in transparent materials is of high technological importance. This work presents the first studies on the formation of Au clusters in this material. The gold is introduced into Bi_2TeO_5 by implanting single crystalline samples with 1 MeV Au^+ ions at room temperature with a dose of $1 \times 10^{16} \text{ cm}^{-2}$. RBS/channeling measurements showed a full amorphization of the implanted layer although the optical absorption did not significantly increase - in contrast to most other optical materials. The Au-implanted samples were subjected to annealing in a conventional furnace at temperatures ranging from 400°C to 700°C in air. While heat treatment at 400°C causes the crystal to turn gray higher temperatures induce an intense blue color in the implanted area. Studies of the optical absorption within the visible range show for the sample annealed at 400°C just an increase of the absorption without prominent features. In contrast, samples annealed above 400°C reveal a strong and broad absorption peak with the maximum located at 610 nm. This is a clear indication for the formation of Au clusters in bismuth tellurite under these heat treatment conditions. On the other hand RBS/channeling measurements show that the recovery of the crystal lattice appears to be hampered for the samples annealed at temperatures above 400°C .

O5.15

PALLADIUM ISLANDS FORMATION ON SILICON CARBIDE FOR SENSING PROPERTIES ENHANCEMENT. Claudiu I. Muntele, D. Ila, Center for Irradiation of Materials, Alabama A&M University, Normal, AL; D.J. Larkin, NASA - Glenn Research Center, Cleveland, OH; D.B. Poker, and D.K. Hensley, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

Using silicon carbide as a semiconductor substrate for hydrogen sensor prototyping has been an ongoing research at the Center for Irradiation of Materials of Alabama A&M University for the past few years. The goal has been to design and to prototype a device that can operate at elevated temperatures (500°C), using palladium as an active element (catalyst). We used both low energy implantation of palladium and deposition as methods of adding the active element to the silicon carbide host material. The implantation was performed at energies between 50 eV and 1 keV at both room temperature and at elevated temperature. This allowed us to produce palladium precipitates just a few atomic layers below the surface, as well as deep inside the substrate. The elevated temperature implantation helps formation of palladium nano-clusters near the surface of the silicon carbide. This procedure was chosen in order to minimize the recovery time after each response to hydrogen. We will present the results during this meeting.

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O5.16

CHEMICAL EFFECTS IN ION IMPLANTATION INDUCED QUANTUM WELL INTERMIXING. Todd W. Simpson, Paul G. Piva, Ian V. Mitchell, Univ. of Western Ontario, Dept. of Physics & Astronomy, London, ON, CANADA.

Ion implantation followed by rapid thermal annealing is used to induce layer intermixing and thus selectively blue-shift the emission wavelength of InP-based quantum well hetero-structures. The magnitude of the observed blue-shift has been studied previously as a function of ion fluence and ion mass. The dependence on ion mass is well established, with heavier ions producing a larger shift. We show, for the first time, that chemical effects also play a significant role in determining the induced blue-shift. Data are presented from the implantation of the similar mass ions; aluminum, silicon and phosphorus. The P-induced blue shift displays a monotonic increase with fluence, consistent with previous studies; however, the fluence dependence of Al- and Si-induced blue-shifts both deviate significantly from the behaviour for P. These results have important implications for attempts to scale intermixing behaviour with ion mass.

O5.17

MODIFICATION OF LITHIUM-PHOSPHORUS OXYNITRIDE ELECTROLYTE BY ION IMPLANTATION. Yoon Sang Cho, Kyong-Hee Joo, Byoungsoo Kim, Kwang Ok Jeong, Jeongmin Oh, Byungwoo Park, Hun-Joon Sohn, Tak Kang, Seoul National Univ, School of MS&E, Seoul, KOREA; Young-Shin Park, Joo Yeal Oh, Samsung Advanced Institute of Technology, Suwon, KOREA.

The miniaturization of electronic devices can be accelerated with thin-film battery technology for power sources of micro devices. A lithium phosphorus oxynitride (Lipon) electrolyte synthesized by sputtering Li_3PO_4 in pure N_2 has an ionic conductivity of $2(\pm 1) \times 10^{-6} \text{ S/cm}$ and excellent stability with lithium anode. The nitrogen incorporation into the electrolyte results in higher degree of cross links, causing increased ionic conductivity. As nitrogen concentration increases in the Lipon electrolyte, it improves both ionic conductivity and chemical stability. However, when Lipon films are deposited by sputtering, it has been reported that nitrogen concentration cannot exceed 6 at.%. As the nitrogen concentration increases above 6 at.% by nitrogen implantation, the Li conductivity of Lipon electrolyte can be improved. We will report the enhancement of ionic conductivity by ion implantation into sputter-deposited Lipon films. The effects of ion dose, implantation temperature, ion-dose rate, and post annealing will also be discussed.

O5.18

MODIFICATION OF CARBON-RELATED FILMS WITH ENERGY BEAMS. Hiroshi Naramoto, Yonghua Xu, Kazumasa Narumi, Jiri Vacik, JAERI, Advanced Science Research Center, Takasaki, JAPAN; Shunya Yamamoto, JAERI, Dept. Mater. Develop., Takasaki, JAPAN; Kiyoshi Miyashita, Gunma Pref. Industrial Tech. Res. Lab., Maebashi, JAPAN.

Directed energy beams (ion and pulsed laser beams) were employed to control the carbon allotrope states through the electronic excitation and/or nuclear collisions. The carbon films obtained were

characterized as the irradiation effect after carbon deposition or as the simultaneous effect between carbon deposition and ion irradiation. The characterization was made mainly with micro-Raman spectrometry, x-ray diffraction and ion beam analysis. Ion beam deposition of mass-selected $100\text{eV }^{12}\text{C}$ ions at RT on Si resulted in highly sp^3 bonded amorphous carbon films, with larger optical band gap (2.53eV) and resistive feature for graphite formation up to 973K under He gas environment. The carbon films prepared at higher temperature than 723K did not show the refractory feature. On the contrary, ion beam assisted deposition (C_{60} deposition and 5keV Ar^+ irradiation) realized the highly sp^3 bonded amorphous carbon films in the broad temperature range up to 973K different from the ion beam deposition films. Ion irradiation with 360keV Ar^{4+} on C_{60} films induced different carbon states depending on the irradiation doses: graphite formation for $1 \times 10^{14} / \text{cm}^2$ and sp^3 bonded amorphous state for more than $1 \times 10^{15} / \text{cm}^2$. The different carbon states described above will be discussed from the view point of carbon allotrope conversion with energy beams including laser beams.

O5.19

NITRIDATION OF Si USING AMMONIA CLUSTER BEAM.
Hiroshi Saito, Masakazu Ohishi, Minoru Yoneta, Okayama University of Science, Dept of Applied Physics, Okayama, JAPAN.

Metal- and semiconductor-nitrides are expected, on one hand, for materials resistant high temperatures, on the other hand, are for photo-electronic devices in ultraviolet to blue wavelength region. Usually nitrides can be synthesized only at very high temperatures, giving unrecoverable damage to underlying structure. The use of nitrogen or nitrogen-related cluster beam will give an answer to this problem. Gas-cluster beams are extensively used for the surface modification of many kinds of metals as well as insulators. Also extremely shallow doping, such as B, can be achieved only by using gas cluster beam. In contrast, almost no application to material synthesis or growth has been reported so far. The present report concerns with the nitridation of Si. A small sized gas-cluster beam source intended to install to conventional molecular beam epitaxy chamber is developed. Ammonia is used as source material. Typical clusters have a size consisting of about 1000 ammonia molecules, which after ionized are accelerated up to $2.5\text{-}10\text{kV}$. Ion current of about 1mA is obtained. This ammonia clusters are irradiated on Si surface at room temperature. X-ray photoelectron spectroscopy measurements showed that a new side-band appeared at the high energy side of Si(2s) and Si(2p) lines. This side-band is due to Si having bonds with nitrogen atoms. Nitridation rate defined by the side-band intensity divided by the sum of the side-band and the main band intensities amounts up to 80% under the cluster energy of 2.5 and 5kV . Further increase in the cluster energy, the rate decreases, probably due to etching effect by higher energy clusters.

O5.20

ENHANCING ADHESION BETWEEN METAL AND LINEAR SATURATED POLYMERS BY A keV Ar^+ IRRADIATION IN O_2 ENVIRONMENT. S. Han, K.H. Kim, Y.W. Beag, and S.-K. Koh, Thin Film Technology Research Center, Korea Institute of Science and Technology, Seoul, KOREA.

Polyethylene (PE), Polyvinylidene fluoride (PVDF), Polytetrafluoroethylene (PTFE) were irradiated by a keV Ar^+ ion beam in an O_2 environment. Polymeric surfaces were activated by energetic ions and reacted upon oxygen gases. Surface energies of PE, PVDF, PTFE were increased by the formation of polar functional groups. Newly formed functional groups such as $-(\text{C}=\text{O})-\text{O}-$, $-(\text{C}=\text{O})-$, etc were detected by the XPS study. Adhesion of metal/linear saturated polymer interface was enhanced due to the formation of polar functional groups induced by ion irradiation in O_2 environment. Enhanced adhesion of inert metal/polymer can be explained by donor-acceptor interaction between metal and the polar functional groups. In case of PVDF and PTFE, the surface reaction is a surface oxidation accompanied with preferential detachment of fluorine from the irradiated surface. However, PTFE surface has changed into filament-type rough surface with ion dose. Enhanced adhesion of Cu thin film on PTFE irradiated with high ion doses can be attributed to mechanical interlocking and surface functionality coincidentally. After tape peel test, metal films on raw specimens were buckled up while metal films on the irradiated polymer were not taken off from the irradiated surface.

O5.21

AN EFFECT OF RADICAL TERMINATION PROCESS ON THE AGING PROPERTY OF PLASMA POLYMERIZED ACETYLENE FILMS. K.-H. Kim, D.J. Choi¹, S. Han, Y.W. Beag, S.-K. Koh, Thin Film Technology Research Center, Korea Institute of Science and Technology, Seoul, KOREA; ¹Dept. of Ceramic Engineering, Yonsei Univ., Seoul, KOREA.

It is inevitable in the plasma polymerized films that the surface

chemical state changes with time (aging phenomenon) should occur. The aging property of plasma polymerized film surface results from the chemical reaction of residual free radicals in plasma polymer with environments. The changes of chemical state on plasma polymer with time leads to degradation of hydrophilicity. In order to prohibit the aging of hydrophilicity of plasma polymer, we have developed the new post plasma treatment process which uses double mesh type electrode. With this post-treatment, the surface of plasma polymer maintains to be hydrophilic after long time, comparing to plasma polymer without post-treatment. It may be considered that radical in plasma polymer surface would be terminated by post-treatment, resulting in long term durability of hydrophilicity. In this study, plasma polymer on the Al substrate were deposited by DC plasma using precursor as acetylene and nitrogen mixture. Changes in wettability of plasma polymerized films were determined by contact angle. We investigated with XPS and FT-IR the changes of chemical state on plasma polymerized film surface as a function of time and without post-treatment, respectively. Mechanism of radical termination by post-treatment will be discussed.

O5.22

ION BEAM SURFACE PROCESSING OF POLYMERS FOR OPTICAL APPLICATIONS. Rafael Rodriguez, Jose A. García, Rafael Sanchez, Centro de Ingeniería Avanzada de Superficies, Pamplona, SPAIN; Alejandro Perez-Rodriguez, Blas Garrido, J.R. Morante, EME, Dept. Electronica, Universitat de Barcelona, Barcelona, SPAIN.

Polymeric materials have a strong interest in the optical industry, for the fabrication of passive optical components as lenses. Substitution of inorganic lenses in spectacles by polymeric ones allows for significant reduction of cost, weight and fragility. The main drawback of these materials is related to their lower surface hardness, which leads to a lower lifetime of the devices. This work is devoted to the study of the ion beam surface processing of ophthalmic polymers, aiming to the optimisation of their tribological properties. For this, light ions (H, deuterium, N, Si) have been implanted at different doses (in the range between 5×10^{14} and 1×10^{16} ions/ cm^2) and energies (100keV , 150keV), to enhance cross-linking between polymeric chains induced by inelastic interaction of the implanted ions with electrons. Several surface mechanical tests have been performed, including Micro and nanoindentation measurements. These have been correlated with the analysis of surface morphology (down to nanometric level, by AFM), as well as with the optical and chemical characterisation of the samples (by FTIR and visible-UV spectroscopies). The results obtained show a strong surface hardness enhancement (up to 200%), which inversely correlates with the mass of the implanted ion. Besides, the low density of the target polymeric lattice allows for a relatively high thickness (in the range of microns) of the surface region modified by the implantation process. These data give interest to the surface hardening processing of polymeric materials by ion implantation.

O5.23

SURFACE CHEMICAL STATE INDUCED BY IRRADIATION OF 80keV AND 1keV Ar^+ ION. Y.W. Beag, S. Han, K.-H. Kim, and S.-K. Koh, Thin Film Technology Research Center, Korea Institute of Science and Technology, Seoul, KOREA; K.H. Chae, C.-N. Hwang, Atomic Scale Surface Research Center, Dept of Physics, Yonsei Univ, Seoul, KOREA.

Energetic particle irradiation give rise to chemical changes in polymeric materials. Free radical generation, bond severance, molecule emission, and formation of new chemical bond can be considered. Polyvinylidene fluoride (PVDF) was irradiated by 1 and 80keV Ar^+ ion beam. In case of two different irradiated PVDF, chemical state of the irradiated surface was changed into fluorine deficient and unsaturated chemical species increased with ion doses. Ion-induced carbonization and crosslinking between activated chains has been researched for 1 and 80keV ion irradiation. Energy and ion dose dependences of the surface chemical state changed by Ar^+ ion beam irradiation have been studied using XPS. Also, composition changes on the irradiated PVDF surface were represented with ion doses.

O5.24

IN-SITU X-RAY PHOTOELECTRON EMISSION CHARACTERISTICS OF PVDF BY A keV Ar^+ ION IRRADIATION. Y.W. Beag, S. Han, and S.-K. Koh, Korea Inst of Science and Technology, Seoul, KOREA; Y.-S. Lee, School of Information Communication & Computer Engineering, Taejon National Univ of Technology, Daejeon, KOREA; and C.-N. Hwang, Atomic Scale Surface Research Center, Dept of Physics, Yonsei Univ, Seoul, KOREA.

Polyvinylidene fluoride (PVDF) exhibits unique chemical/physical characteristics between polyethylene and polytetrafluoroethylene. PVDF were irradiated by Ar^+ ion beam and introduced into XPS chamber without exposing to atmosphere. Some chemical bonds on PVDF surface were severed and resulted in new functional groups. Composition ratio of fluorine on the irradiated PVDF was reduced to

18%. -CF₂- bonds were reduced and -C(H···F)- bonds increased with Ar⁺ ion dose because fluorine detached by Ar⁺ ion beam might be bounded within some severed polymeric chains. New chemical state on the irradiated PVDF can be explained not by thermodynamically equilibrium phase, but by non-equilibrium phase. -CF₂ and -CF₃ bonds were slightly formed on the irradiated PVDF surface. XPS result of surface state can reveal small crosslinking and severance of carbon backbones. A keV ion beam irradiation can result in small damage during surface modification process.

O5.25

OPTICAL PROPERTIES AND MICROSTRUCTURE OF SILICON-ON-INSULATOR FORMED BY LOW-DOSE OXYGEN IMPLANTATION. L. Lin, Y.H. Yu, M. Chen, J. Chen, Z.X. Lin, X. Wang, Ion Beam Laboratory, Shanghai Institute of Metallurgy, Chinese Academy of Sciences, Shanghai, CHINA.

Silicon-on-insulator (SOI) have been formed by oxygen implantation with a low-dose window of $3.5\text{-}6.5 \times 10^{17} \text{cm}^{-2}$ at 100-160 keV, and consequently annealed at a high temperature of 1324°C for 5 hours in argon and oxygen atmosphere. The composition and microstructure of SOI have been investigated by RBS, XTEM, HRTEM and AFM respectively. The results indicate that the dose and energy of oxygen implantation should be carefully chosen in order to form continual buried oxide layer with silicon islands free. The silicon islands in BOX were investigated and the formation mechanism is discussed. Optical properties of SOI structures have been studied by IR reflection spectra in the range of 400 to 5000 cm⁻¹. The experimental IR reflection spectra have been fitted by calculating the complex dielectric function of SOI structures based on effective medium theory (EMT), in which the buried oxide layer is consisted of homogeneously distributed silicon islands. The fitting of the experimental spectra using our model is quite satisfactory, which means the model in our simulation is reliable in explaining the optical properties of SOI structures formed by low-dose oxygen implantation.

O5.26

PHYSICAL ORIGIN OF THE DOSE WINDOW IN SIMOX PROCESSES. T. Motooka, M. Arita, H. Yoshida, Y. Iriguchi and Y. Ikoma, Kyushu University, Dept of MS&E, Fukuoka, JAPAN.

Silicon-on-insulator (SOI) is a useful structure to enhance existing Si-CMOS technology and Separation by IMplantation of OXYgen (SIMOX) is one of the major methods to provide the SOI structure. It is experimentally well known that there exists a dose window for implanted oxygen to obtain continuous buried oxide (BOX) layers after high-temperature annealing. For example, the dose of 180 keV O⁺ ions must be around $4 \times 10^{17} \text{cm}^{-2}$ to form continuous high-quality BOX layers. However, it is not well understood how the dose window is determined. We have investigated the formation process of BOX layers for the analysis of a possible mechanism controlling the dose window based on cross-sectional transmission electron microscopy (XTEM), X-ray photoelectron spectroscopy (XPS), and infrared (IR) spectroscopy measurements combined with computer simulations. Depth distribution of the as-implanted oxygen atoms was experimentally determined by XTEM and XPS and the depth distribution was in good agreement with that obtained by TRIM calculations. From these results, we have proposed that there exists a critical oxygen concentration, approximately $3.5 \times 10^{22} \text{cm}^{-3}$ for the formation of continuous BOX layers. Oxidation processes during high-temperature annealing of as-implanted samples were also analyzed using IR spectra combined with molecular orbital calculations based on Si₁₂₃O_x (x=1-4) clusters. Calculated vibrational spectra of the Si-O stretching modes well reproduced the IR data of the annealed samples with elevated temperatures. From the cluster model calculations, it is suggested that O₂ can be formed during O⁺ ion implantation for the doses larger than the critical dose which may lead to the formation of Si islands in the BOX layer.

O5.27

MECHANISMS OF BUBBLE AND VOID COARSENING IN SILICON DURING HIGH TEMPERATURE ANNEALING. V.M. Vishnyakov, S.E. Donnelly, G. Carter, Joule Physics Laboratory, University of Salford, Manchester, UNITED KINGDOM.

Transition metal impurities in silicon devices can be effectively getter by voids. Voids are created in a two-stage process, consisting of the production of helium bubbles by ion implantation and the subsequent removal of the gas by annealing at temperatures above 700°C. During annealing not only is the helium removed from the silicon but also the mean bubble size (and thus the mean void size at higher temperatures) is increased. It is commonly believed that this coarsening occurs through the coalescence of moving bubbles and voids. We have analysed void size distributions and void depth distributions following annealing at temperatures from 500 to 1100°C. In addition we have studied the behaviour of individual bubbles and voids dynamically during the annealing of plan-view and

cross-sectional specimens in a high temperature TEM stage. Bubble/void coarsening begins at temperatures as low as 500°C and continues throughout the temperature range studied. Our data indicate that bubble and void movement appears to play little part in the coarsening process. Instead bubbles and later voids appear to grow - presumably by the acquisition of vacancies and/or vacancy clusters.

O5.28

IRRADIATION EFFECTS OF SILICON DIOXIDE FILMS IMPLANTED BY CARBON IONS. Y.H. Yu, J. Zhao, Ion Beam Laboratory, Shanghai Institute of Metallurgy, Chinese Academy of Sciences, Shanghai, CHINA; S. Jin, IMEC, Kapeldreef, Leuven, BELGIUM.

Microstructures of the carbon-implanted silicon dioxide films were characterized by transmission electron microscopy (TEM) and Fourier transform infrared (FTIR), aiming to investigate the irradiation effects in the interaction of energetic carbon ions with silicon dioxide. As revealed by the cross sectional TEM (XTEM) images, bubbles are created by carbon ion implantation. The plan-view investigations indicate that Si and SiC nanocrystals are formed in the as-implanted sample, and that the nanocrystals exist in a thin layer above the bubble layer. The FTIR results indicate that some Si-H based species are created by carbon ion implantation. Both physical and chemical effects play an important role in the structural modifications of silicon dioxide by the interaction between the carbon ions and silicon dioxide.

O5.29

SYNTHESIS OF CONTINUOUS SmSi₂ LAYERS ON Si BY SAMARIUM ION IMPLANTATION USING METAL VAPOR VACUUM ARC ION SOURCE. X.Q. Cheng, H.N. Zhu and B.X. Liu, Dept of Materials Science & Engineering, Tsinghua Univ, Beijing, CHINA.

Samarium ion implantation was conducted to form Sm-disilicide films on silicon wafers using a metal vapor vacuum arc ion source [1,2] and the continuous SmSi₂ layers were directly obtained with neither in-situ heating nor post-annealing. The Si wafers were implanted with 40 keV Sm ions with three current densities, i.e. 8.8, 17.6 and 35.2 μA/cm², to the nominal doses ranging from 5×10^{16} to 2×10^{17} ions/cm². The temperature rises of the Si wafers were measured by a thermal couple during implantation. X-ray diffraction analysis indicated that SmSi₂ layers were formed by implantation under three conducted current densities, which correspondingly heated the Si wafers up to the temperature rises of 190, 290 and 390°C, respectively. Scanning Electron Microscopy analysis revealed that the formed SmSi₂ layers were continuous without any surface pitting defects, which were often observed on those formed by solid state reaction. The optimal experimental parameters for synthesizing the SmSi₂ layers of good crystalline quality were found to be an ion current density of, or higher than, 17.6 μA/cm², which corresponded to a formation temperature around 290°C, and an implantation dose greater than 1×10^{17} ions/cm². Besides, random Rutherford backscattering measurements confirmed that the thickness of the formed Sm-disilicide layers was about 15 nm and the SmSi₂/Si interface was more or less sharp. The formation mechanism of the SmSi₂ on Si was also discussed.

References:

1. I.G. Brown, J.E. Gavin and R.A. MacGill, Appl. Phys. Lett. 47, 358 (1985).
2. D.H. Zhu et al., Appl. Phys. Lett. 62, 2356 (1993).

O5.30

FOCUSED ION BEAM TECHNIQUES - TOOLS FOR ENHANCEMENT OF DEPTH SENSING INDENTATION TEST METHODS Pavel Trtik¹, Peter J.M. Bartos¹, Clive M. Reeves², ²Scottish Enterprise, Glasgow, Scotland, UNITED KINGDOM.

This paper provides a summarised review of applications of focused ion beam (FIB) techniques applied for enhancement of depth sensing indentation (DSI) test methods. Nanotechnology based DSI test methods are increasingly important tools for characterisation of mechanical properties of materials. By utilising the nanoscale resolution of the ion column, the FIB techniques show a large potential for the improvement of depth sensing indentation test methods. The applications can be categorised into the following areas: (i) Nano/micromachining of diamond probes - diamonds, the hardest naturally occurring material, shaped into a three-sided pyramid are usually used as indenter tips for DSI test methods. FIB milling techniques show potential for production of the tip whose depth-area-function is closer to the ideal shape than that produced by conventional methods. Production of other specially shaped diamond probes, such as those that are suitable for push-out tests of sub-10 μm-diameter filaments in fibre-reinforced composites, is also presented. The influence of the FIB milling process on the mechanical properties of the FIB-machined diamond probes is discussed. (ii) as an impression into the surface of the tested material. In its

cross-sectioning mode, the FIB workstation enables the material to be etched out in such a manner that the previously unavailable but very important observation of the microstructure of the material underneath the indent can be carried out. Such cross-sections reveal entirely new information about the 'third dimension' of the microindents and about the microstructure of the sub-surface material surrounding the indent. (iii) Pre-indentation preparation of the specimens - three-dimensional objects of various sizes, such as beams, cantilevers, etc., can be FIB-machined in the structure of the specimen. Such a nano/microbeam or a nano/microcantilever is then loaded in indentation apparatus. Since the dimensions of the micromachined object can be measured with a high precision and the deflection vs. load diagram is monitored during the DSI experiment, the elastic modulus of the material of microbeam/microcantilever can be derived from the classical bending theory of the beam

SESSION O6: ION BEAM INDUCED SLICING AND FOCUSED ION BEAM APPLICATIONS

Chair: Tony E. Haynes
Tuesday Morning, November 28, 2000
Room 311 (Hynes)

8:30 AM *O6.1

AN EFFICIENT PROCESS FOR SEPARATION OF THIN-FILMS IN BULK SiC UTILIZING H⁺-ION-CUTTING TECHNIQUES.

O.W. Holland, Oak Ridge National Laboratory, Solid State Division, Oak Ridge, TN.

Thin-film transfer by H⁺-ion implantation offers exciting possibilities for forming heterostructures other than silicon-on-insulator (for which it was developed). However, as the technique is extended to include a wider range of materials and their combinations, it becomes important to understand the mechanism(s) responsible for ion-induced separation of thin layers from bulk material. In particular, there is a need to understand and optimize this process for a new class of semiconductors (i.e., wide band gap materials) targeted to replace silicon in a variety of device applications including power electronics. SiC, a leading contender for this role, would benefit greatly from this technology, if for no other reason than cost. Substantial savings would be realized if thin, single-crystal films of this material could be efficiently transferred to cheaper substrates (e.g., polycrystalline SiC). An extensive study of the separation mechanism was done in SiC with the intent to optimize implant processing to affect efficient separation while maintaining the quality of the transfer layer. A wide range of material and irradiation parameters were examined including processing temperature, sample orientation (i.e., channeled vs. random implantation), polytype differences, ion energy and dose, etc. Also, the effects of ion-induced damage were also studied by coimplantation in which other ion types were used to intentionally introduce defects into the lattice. The results will be discussed and, in most cases, compared with those obtained in Si under similar processing conditions. In most cases, the processing efficacy was evaluated by gauging the amount of exfoliation produced under given conditions.

9:00 AM O6.2

ION-BEAM INDUCED SLICING OF SINGLE CRYSTAL OXIDE THIN FILMS. S. Thevuthasan, V. Shutthanandan, W. Jiang, W.J. Weber, Pacific Northwest National Laboratory, Richland, WA.

Precise cleaving of single crystal oxide films with known thickness using hydrogen implantation and subsequent annealing was investigated using SrTiO₃ and Y-ZrO₂ single crystals. Rutherford backscattering and channeling (RBS/C) techniques, nuclear reaction analysis (NRA) and scanning electron microscopy (SEM) have been used in this study to investigate the effects of hydrogen implantation and subsequent annealing on the profiles of disorder and hydrogen content. Under the right set of conditions, cleavage of thin films due to hydrogen bubble formation can be carried out in these materials. Samples were irradiated at a temperature of 120 K with 40 keV H⁺ ions to fluences in the range of 5.0x10¹⁶ to 1.0x10¹⁷ H⁺/cm². Isochronal annealing for 20-minute time periods was performed at temperatures from 370 to 870 K (±5 K) at 100 K intervals under high vacuum conditions. After each annealing step, each sample was cooled down to 300 K, and the RBS/C and hydrogen NRA measurements were carried out. Annealing the 5.0x10¹⁶ H⁺/cm² irradiated SrTiO₃ sample above 570 K, resulted in the cleavage of the entire irradiated surface. However, due to a significant loss of hydrogen in the implanted region of Y-ZrO₂ compared to SrTiO₃, cleavage of surface layer was not observed in 5.0x10¹⁶ H⁺/cm² irradiated Y-ZrO₂ up to an annealing temperature of 870 K. Annealing both SrTiO₃ and Y-ZrO₂ samples irradiated to 1.0x10¹⁷ H⁺/cm² up to 870 K results in remnants of ruptured hydrogen blisters on the entire surface.

9:15 AM O6.3

BURIED OXIDE PRECIPITATES IN A Si WAFER DUE TO He

ION IMPLANTATION AND HIGH-TEMPERATURE OXIDATION.

Sadao Nakashima, NTT Telecommunications Energy Labs, Atsugi, JAPAN; Jyoji Nakata, Kanagawa University Faculty of Science, Hiratsuka, JAPAN; Junzou Hayashi, NTT Basic Research Labs, Atsugi, JAPAN; Kazuo Imai, NTT Telecommunications Energy Labs, Atsugi, JAPAN.

A new method for producing buried oxide using helium (He) ion implantation, not oxygen, followed by high-temperature oxidation has been developed. He ion doses ranging from 3.3 × 10¹⁶ to 1 × 10¹⁷ cm⁻² were implanted into Si wafers at 55 keV. The implanted wafers were then annealed at 1200°C to form voids around the depth corresponding to the projected range of the implanted ions. Next, oxidation was performed at temperatures between 1200 and 1350°C to diffuse some of the oxygen atoms into the Si. The wafers were analyzed using transmission electron microscopy (TEM) and Rutherford backscattering spectrometry (RBS). The density of the formed voids was approximately 1 × 10¹⁰ cm⁻². The diameter of the voids depended on the ion dose and ranged from 20 to 70 nm. After oxidation at 1200°C, thermal oxide grew on the surface of the voids as well as on the wafer and completely filled the voids, which led to the formation of buried oxide precipitates. When oxidation was performed at temperatures higher than 1300°C, these precipitates grew to more than 100 nm, and some began to coalesce. RBS analysis revealed that the number of oxygen atoms in the buried precipitates was 4.4 × 10¹⁶ cm⁻², 20 times more than the number of oxygen atoms that diffused into void-less Si. An enhanced precipitation model of the buried oxide will be presented at the symposium. We have demonstrated that thermal oxide grows at the interface between Si and voids formed by He ion implantation and that the size of the resulting oxide precipitates increases during high-temperature oxidation. We hope to obtain a continuous buried oxide layer by optimizing He ion implantation conditions and subsequent annealing and oxidation processes.

9:30 AM O6.4

CO-IMPLANTATION INDUCED THERMAL STABILITY ENHANCEMENT OF HELIUM IN INDIUM PHOSPHIDE.

Todd W. Simpson, Ian V. Mitchell Univ. of Western Ontario, Dept. of Physics & Astronomy, London, ON, CANADA.

The thermal stability of ³He implanted into single crystal indium phosphide is measured via the d(³He,p) nuclear reaction. An enhancement in the stability of the implanted helium is observed when hydrogen ions are co-implanted. It is known that, in silicon, the exfoliation process is enhanced by co-implantation of hydrogen and helium ions and the effect is attributed to chemical interactions between the two implanted ions. However, we also show that, in InP, the stability of implanted helium is enhanced still more efficiently with the co-implantation of phosphorus ions. It is concluded that the effect of co-implantation, in InP, is to provide diffusion-inhibiting defects to minimize the out-diffusion of helium during the ramp to the annealing temperature where relatively stable voids can form.

10:15 AM O6.5

Cu IMPURITY GETTERING IN Si CAVITIES OBSERVED BY POSITRON BEAM ANALYSIS. H. Schut, A. van Veen and S.W.H. Eijt, Delft University of Technology, Interfaculty Reactor Institute, Delft, THE NETHERLANDS.

The reduction in device dimensions and the increasing complexity of IC technology puts limits to the levels of unwanted (metallic) impurities, such as Fe, Ni and Cu, in e.g. Si. Impurity gettering in cavities is a method proposed to reduce the deteriorating effects of the impurities on the performance of the devices. From earlier work it is known that in Si nano cavities can be generated by implantation of keV light-ions (H and He) at doses beyond 10¹⁶ cm⁻² followed by an annealing at typically 1100 K. At this temperature the irradiation induced defects have clustered to nanometer sized cavities while the implanted atoms have dissolved and permeated to the surface. A method to detect the formation of such cavities and the modification of their internal interfaces is the positron beam annihilation (PBA) technique. Trapping and subsequent annihilation of positrons in the cavities is observed by a significant change of the so-called S and W annihilation parameters. In bulk materials these relate to the annihilation of positrons with valence and core electrons, respectively. The trapping at Si-cavities opens a channel for the formation of Positronium (Ps) with annihilation characteristics clearly different from that of the bulk material. Furthermore, the annihilation of the Ps by pick-off reactions with electrons at the internal cavity surface enables the detection of impurities arriving at these cavities. In this study we present the results of PBA experiments on FZ-Si implanted with 30 keV He⁺ ions with doses ranging from 0.5 to 3 × 10¹⁶ cm⁻² followed by an anneal treatment at 1100 K under N₂ ambient. Copper is introduced by diffusing from the back-side of the wafer at 1000 K, again under N₂. Mapping of the S and W data, with the implantation dose as running parameter, shows the formation of cavities. After the

in-diffusion of the Cu the change in position of the characteristic S-W cluster points clearly demonstrate the arrival of Cu at the internal surface of the cavities.

10:30 AM **O6.6**

EFFECTS OF Ga-ION IRRADIATION ON CHEMICAL AND ELECTRICAL PROPERTIES OF MATERIALS PROCESSED BY A FOCUSED ION BEAM (FIB). Heinz D. Wanzelboeck, Alois Lugstein, Helmut Langfischer, Stefan Harasek, Emmerich Bertagnoli; Vienna University of Technology, Institute for Solid State Electronics, VIENNA, AUSTRIA.

Focused Ion Beam (FIB) technology allows to process materials within a lateral range below 100 nm. The feasibility to mechanically sputter as well as to direct-write nanostructures and the fact that Ga-ions are utilized is unique for this method. The focused Ga-ions are used to locally induce a chemical vapor deposition of volatile precursor molecules adsorbed on a surface. Local depositions of metals and dielectrics have been achieved on a sub- μm scale with a focused ion beam. This method is highly suitable for advanced microelectronic semiconductor fabrication. However, material specifications are narrow for these tailor-made applications. The effect of the Ga-ions implanted into the material both during sputtering and deposition has been realized as key parameter for the function of FIB processed microelectronic devices. For Si-based semiconductors Ga can be used as dopant intentionally implanted into a Si substrate to locally modify the conductivity of Si. The effect of locally confined Ga implantation on the function of a working electronic device has been demonstrated with an exemplary device. The penetration depth and the distribution of Ga have been exploited both theoretically by simulation and experimentally by profiling with secondary ion mass spectroscopy (SIMS). For the deposition of advanced materials for microelectronic processing the Ga is considered as a disadvantageous impurity. Implanted Ga is a main reason for the degradation of quality differing from bulk properties. The influence of process parameters on the amount of impurities in ion beam synthesized materials has been investigated by spectroscopic analysis. The effect Ga on the conductivity of ion beam deposited metal structures and the resistance and conductivity of ion beam synthesized oxides has been investigated by electrical test structures such as Van-der-Pauw structures and MIM capacitors. The advantages of focused ion beam processing and the limitations are illustrated by examples.

SESSION O7: METASTABLE PHASES, PLASTIC FLOW, AND PATTERNING OF SURFACES I

Chair: David M. Follstaedt
Tuesday Morning, November 28, 2000
Room 311 (Hynes)

10:45 AM **O7.1**

SPONTANEOUS FORMATION OF NANOMETER-SCALE SELF ORGANIZED STRUCTURES IN Cu-Ag ALLOYS UNDER IRRADIATION. Raúl A. Enrique, Slim Zghal, Pascal Bellon, University of Illinois, Dept. of MS&E and Frederick Seitz Materials Research Laboratory, Urbana, IL.

Ion-beam irradiation can be used as a processing tool to synthesize metastable materials. A particular case is the preparation of solid solutions from immiscible alloys, which have been achieved for a whole range of systems. In this process, increased solubility is obtained through the local mixing induced by each irradiation event, which if occurring at a high enough frequency can outweigh species segregation by thermal diffusion. Therefore, the resulting microstructure forms in far from equilibrium conditions, and theoretical results for these kind of driven alloys have shown that novel microstructures exhibiting self-organization can develop. Since most previous experimental studies of ion-beam mixing were based on x-ray diffraction and Rutherford backscattering, we carry out an investigation that includes the direct observation of the resulting microstructures by TEM and STEM, using both cross-section and plan-view samples. To this purpose, we prepare Ag-Cu multilayered thin films which we subject to 1 MeV Kr^+ -ion irradiation at temperatures ranging from 77 K to 473 K. We observe two different phenomena occurring at different length scales: On the one hand, regardless of the irradiation temperature, the grains grow under irradiation until reaching a size limited by the film thickness (~ 200 nm). On the other hand, the distribution of species inside the grains is greatly affected by the irradiation temperature. At intermediate temperatures, a semi-coherent decomposition is observed at a scale of ~ 10 nm. This nanometer-scale decomposition phenomenon appears as an evidence of patterning, and poses the interesting proposition of using ion-beam irradiation as a route to synthesize nanostructured materials with novel optical and magnetical properties.

11:00 AM **O7.2**

SPONTANEOUS CRYSTALLINE MULTILAYER FORMATION IN Ni IMPLANTED Al AT 100 K. Alexandre Cuenat, Aicha Hessler-Wyser, Rolf Gotthardt, IGA-DP-Ecole Polytechnique Federale Lausanne, SWITZERLAND and Max Döbeli Ion Beam Physics, PSI c/o ETHZ, Zürich, SWITZERLAND.

The microstructure evolution of Al implanted with Ni at 5 MeV and at 100 K to a local concentration of 25 at.% is described. For lower Ni concentration, it has been previously observed that $\text{Al}_{0.75}\text{Ni}_{0.25}$ amorphous precipitates are formed together with a high dislocation density. When the Ni concentration reaches 25 at.%, a new crystalline multilayered microstructure is observed without further annealing. Rutherford Backscattering Spectrometry (RBS) experiments are conducted to determine the Ni profile inside the sample after the implantation. In addition, Transmission Electron Microscopy observations have been carried out to determine the microstructure of the high dose implantations. These observations reveal the presence of well defined crystalline layers separated by sharp interfaces. To our knowledge, it is the first time that such a structure is observed without further annealing of the implanted sample. A series of models are discussed, which describe the formation of the crystalline multilayer. It is argued that its formation is the result of a recrystallization front produced by the exothermal amorphous to crystal transformation.

11:15 AM ***O7.3**

SPONTANEOUS PATTERN FORMATION DURING LOW ENERGY ION BOMBARDMENT. Eric Chason, Michael Scarpulla, Division of Engineering, Brown University, Providence, RI; Jonah Erlebacher, Michael Aziz, Harvard University, Cambridge, MA; J.A. Floro and M.B. Sinclair, Sandia National Laboratories, Albuquerque, NM.

Sputtering of surfaces by collimated, low energy ion beams results in spontaneous pattern formation in many systems. By varying the ion beam and the material parameters, these patterns can be organized in one or two dimensions with length scales ranging from 10's to 1000's of nanometers. In order to explore the mechanisms which control the pattern formation, we have used in situ light scattering to measure the evolution of sputtered Si(001) surfaces. The results are interpreted within a linear instability model originally proposed by Bradley and Harper that includes the dependence of the sputter yield on the local surface morphology. Monte Carlo simulations have also been developed that include the surface-dependent sputter yield. In addition to describing pattern formation, these results provide insight into fundamental surface kinetic processes during ion bombardment. Portions of this work were performed at Sandia National Laboratories and supported by the United States Department of Energy under contract DE-AC04-94AL8500.

SESSION O8: METASTABLE PHASES, PLASTIC FLOW, AND PATTERNING OF SURFACES II

Chair: David M. Follstaedt
Tuesday Afternoon, November 28, 2000
Room 311 (Hynes)

1:30 PM ***O8.1**

ION IRRADIATION-INDUCED PLASTIC DEFORMATION OF COLLOIDAL PARTICLES. T. van Dillen^a, E. Snoeks^a, W. Fukarek^b, M.L. Brongersma^a, A. van Blaaderen^{a,c}, and A. Polman^a; ^aFOM-Institute AMOLF, Amsterdam, THE NETHERLANDS; ^bResearch Center Rossendorf, Dresden, GERMANY; ^cDebye Institute, Utrecht University, Utrecht, THE NETHERLANDS.

Colloidal particles with a diameter in the 100-1000 nm range show a dramatic plastic deformation under MeV ion irradiation. The originally spherical particles turn into oblate ellipsoids with size aspect ratios as large as 5, and by using subsequent irradiations under different angles, prolate (sugar-shaped) particles can be made as well. The typical deformation rate is 100% per 10^{16} ions/cm². When the ion range is chosen less than the colloid diameter, particles with non-ellipsoidal shape can be made as well. The polydispersity in colloid size (3%) is not affected by the irradiation. We have used 1-16 MeV Xe and Au irradiation of silica colloids, at temperatures ranging from 90-700 K, to study this phenomenon in detail. It is found that the deformation: 1) is linear with ion fluence, 2) is only slightly flux-dependent in the flux range studied, 3) decreases with temperature, and 4) increases for increasing ion energy; it is almost linearly proportional to the average electronic energy loss. Experiments on TiO_2 and ZnS colloids, and on ZnS/SiO₂ core-shell particles were also performed and show a clear deformation effect as well. Metallic Ag colloids and polycrystalline Al_2O_3 do not show deformation under the conditions studied. A tentative model to explain the deformation behavior will be presented.

Several applications of these anisotropic ion-beam modified particles will be demonstrated. First, we have fabricated metallic nanodots by using an ion-beam deformed hexagonal two-dimensional colloidal crystal as a lithographic mask. Second, we have brought the irradiated particles in suspension to study the ordering of the ion-beam modified particles into a nematic liquid crystalline phase that has so far only been predicted by computer simulations. Third, we have shifted the optical bandgap of a three-dimensional colloidal photonic crystal by MeV ion irradiation.

SESSION O9: SURFACE MODIFICATION, SUCH AS
HARDNESS AND TEXTURE

Chair: O. Wayne Holland
Tuesday Afternoon, November 28, 2000
Room 311 (Hynes)

2:00 PM *O9.1

ION BEAM ASSISTED TEXTURE EVOLUTION DURING THIN FILM DEPOSITION OF METAL NITRIDES. Bernd Stritzker, Institut für Physik, Universität Augsburg, Augsburg, GERMANY; and Bernd Rauschenbach, Institut für Oberflächenmodifizierung, Leipzig, GERMANY.

Ion beam assisted deposition, i.e. the bombardment of thin films with a beam of energetic particles has become a highly developed tool for the preparation of thin films. This technique provides thin films and coatings with modified microstructure and properties. In this paper examples are presented for the modifying the structure: in-situ modification of the texture during the ion assisted film growth and the ion beam enhanced epitaxy. The biaxial alignment of TiN on Si(111) films prepared by nitrogen ion beam assisted deposition at room temperature was studied. The bombardment perpendicular to the surface of the substrate causes an {001} alignment. A 55° ion beam incidence produces both {111} orientation relative to the surface and {100} orientation relative to the ion beam. This results in a totally fixed orientation of the crystallites. The texture evolution is explained by the existence of open channelling directions and minimization of the elastic deformation energy. Epitaxial, hexagonal gallium nitride films were grown on c-sapphire by low-energy nitrogen ion assisted deposition (≤ 25 eV). The ion energy was chosen to be less than the bulk displacement energy to avoid the formation of ion-induced point defects in the bulk. The results show that GaN films with a nearly perfect {0002} texture are formed which have superior crystalline quality than films grown without ion assisted bombardment. The mosaicity, the defect density and the surface roughness are reduced. The growth mode changes from island growth to layer-by-layer growth.

2:30 PM O9.2

NANOSCALE PATTERN FORMATION IN Pt THIN FILMS DUE TO ION BEAM INDUCED DEWETTING. Xiaoyuan Hu, Karsten Albe, Kai Nordlund, David Cahill, Robert Averback, University of Illinois at Urbana-Champaign, Dept of Materials Science and the Materials Research Laboratory, Urbana, IL.

Dewetting induced by heavy ion irradiation is observed for 3 nm thick Pt films on SiO₂ substrates bombarded by both 800 and 20 keV Kr⁺. The evolution of film morphology and roughness is characterized by atomic force microscopy and scanning electron microscopy. Pt films dewet from the substrate at irradiation doses in the range 2×10^{14} - 6×10^{15} cm⁻². The dewetted films break into patterned structures. The lateral correlation lengths of the patterned structures are 30-60 nm, increasing with the irradiation dose. At the highest dose ($\sim 2 \times 10^{16}$ cm⁻²), the patterns disappear due to sputtering, and the remaining Pt form nanoparticles partially embedded in the SiO₂ substrates. The dewetting process is relatively insensitive to ion energy. Molecular Dynamics simulations of single ion impact of Pt films on C substrates have been carried out to show local melting and detachment of the Pt films. We propose that the dewetting is nucleated by molten zones created by the impacts of individual Kr⁺ ions. However, the characteristic feature size of the pattern formation is determined not only by the molten zone sizes. Irradiation of Pt/Ti alloys produces smaller size pattern structures and suggests that decreasing the interface energy reduces the characteristic feature size.

3:15 PM *O9.3

RELATING NANOSTRUCTURES TO MECHANICAL PROPERTIES IN IMPLANTED MATERIALS. David M. Follstaedt, James A. Knapp, Samuel M. Myers and Gary A. Petersen, Sandia National Laboratories, Albuquerque, NM.

Mechanical properties of ion-implanted layers can be accurately assessed by using nanoindentation to probe the response of the layer on its substrate and finite-element modeling to account for effects of the substrate. For Ni implanted with 8 at.% of O or Al O to form

oxide precipitates, exceptionally high yield strength is found: 4.6 /-0.5 GPa, versus 0.75 GPa for heavily damaged Ni-implanted Ni and 0.15 GPa for unimplanted Ni. We account for this strength by using the fine sizes (1-5 nm) and high densities of precipitates in dispersion hardening theory, which predicts 4.4 GPa. Moreover, yield strengths for as-implanted Ni with oxides and for annealed material, as well as values obtained earlier for Al with fine oxides, all vary with precipitate size and density in accordance with this theory, even though the shear moduli of Ni and Al differ by 3x. We are also using this approach to assess the properties of amorphous Si formed by self-ion implantation. This problem is made both complicated and interesting by a phase change of crystalline (diamond-structured) Si to a denser metallic phase at ~ 12 GPa pressure. By modeling both unimplanted and amorphous Si as isotropic solids, we obtain a yield strength of 4.3 GPa and Young's modulus of 140 GPa for amorphous Si, versus 5 GPa and 180 GPa for crystalline Si, respectively. Indentations (>1 μ m depth) through the 0.57 μ m-thick implanted layer produce much less cracking and fracture than for unimplanted Si, indicating that the amorphous phase is more ductile. Discontinuities in material response during withdrawal of the indenter, possibly indicating re-transformation of metallic Si back to semiconducting Si, are much reduced with the amorphous Si layer. This work supported by Division of Materials Sciences, Office of Basic Energy Sciences of the United States Department of Energy under contract DE-AC04-94AI85000.

3:45 PM O9.4

Ar CLUSTER ION BOMBARDMENT EFFECTS ON SEMI-CONDUCTOR SURFACES. Toshio Seki, Kazumichi Tsumura, Jiro Matsuo, Gikan H. Takaoka, Ion Beam Engineering Experimental Laboratory, Kyoto University, Kyoto, JAPAN; Isao Yamada, Laboratory of Advanced Science and Technology for Industry, Himeji Institute of Technology, CAST, Ako, JAPAN.

New surface modification processes have been demonstrated using the gas cluster ion irradiations because of their unique interaction between cluster ions and surface atoms. For example, high quality ITO films could be obtained by O₂ cluster ion assisted deposition at room temperature. It is necessary to understand the role of cluster ion bombardment during film formation for the further developments of this technology. Variable Temperature Scanning Tunneling Microscope (VT-STM) in Ultra High Vacuum (UHV) allows us to study ion bombardment effects on surfaces and nucleation growth at various temperatures. The irradiation effects between Ar cluster ion and Xe monomer ion were compared. When a Si(111) surface with Ge deposited a few Å was annealed to 400°C, it was observed that many islands of Ge were formed. The surface with the Ge islands was irradiated by these ions. In the STM image of cluster-irradiated surface, large craters with diameter of about 100Å were observed, while only small traces with diameter of about 20Å were observed in monomer-irradiated surface. The number of Ge atoms displaced by one Ar cluster ion impact was much larger than that by one Xe ion impact. This result indicates that Ar cluster ion impacts can enhance the physical modification of Ge islands with low ion dose. When the sample irradiated with Ar cluster was annealed at 600°C, the hole remained, but the outer rim of the crater disappeared and the hemispherical damage in the target was removed. The depth of hole became shallower with decrease of the impact energy. These results indicate that low damage and useful surface modification can be realized using the cluster ion beam.

4:00 PM O9.5

TIME-RESOLVED X-RAY SCATTERING STUDY OF Co SURFACE EVOLUTION DURING ION IRRADIATION AND HOMOEPITAXY. O. Malis, Applied and Engineering Physics, Cornell Univ, Ithaca, NY; R.L. Headrick, Cornell High Energy Synchrotron Source, Cornell Univ, Ithaca, NY; J.M. Pomeroy, Dept of Physics, Cornell Univ, Ithaca, NY; J.D. Brock, Applied and Engineering Physics, Cornell Univ, Ithaca, NY.

The sputter-erosion of Co(0001) single crystal with Ar⁺ ions in the 100 to 750 eV energy range was investigated using *in-situ* time-resolved x-ray scattering. At temperatures above 300°C the surface remains relatively smooth, erosion evolving through a layer-by-layer or step flow mechanism. Below 250°C the surface develops a pattern of mounds and pits with a characteristic wavelength. The time, ion energy and temperature dependence of this wavelength were studied in detail. Epitaxial Co thin films thermally evaporated on sapphire were also sputtered through in order to synthesize self-assembled arrays of Co nanoclusters with a narrow size distribution. The degree of local order within the Co dot arrays was examined using atomic force microscopy.

SESSION O10: ION BEAM SYNTHESIS OF
NANOSTRUCTURES AND THIN LAYERS I

Chair: Alkiviathes Meldrum

Tuesday Afternoon, November 28, 2000
Room 311 (Hynes)

4:15 PM *O10.1

Si NANOCRYSTAL MEMORIES: A CRITICAL DEVICE PERFORMANCE COMPARISON OF ION BEAM SYNTHESIS AND RIVAL TECHNIQUES FOR FLOATING GATE FABRICATION. Harry A. Atwater, Michele Ostraat, Mark Brongersma, Elizabeth Boer, L. Douglas Bell, Richard C. Flagan, Thomas J. Watson Laboratory of Applied Physics, California Institute of Technology, Pasadena, CA; Jan de Blauwe, Martin Green, Gary Weber, Jeff Bude, Andreas Kerber, Fred Clemens, and Young Kim, Lucent Technologies, Murray Hill, NJ.

Silicon nanocrystal-based floating gate MOS field effect devices have potential for terabit/cm² density nonvolatile memory applications. Device performance and reliability are strongly dependent on nanocrystal layer properties (such as crystal size distribution, density, and co-planarity) and nanocrystal insulation. In addition, the nanocrystal layer fabrication technique has to be simple, 8 wafer compatible, cleanroom compatible and well controlled. We have fabricated nanocrystal memory devices in a conventional MOS ULSI process with channel lengths from 0.2-10 μm with a Si nanoparticle floating gate fabricated by four different nanocrystal synthesis methods, including ion implantation, direct chemical vapor deposition, silicon-rich oxide deposition and aerosol Si nanocrystal deposition. Dense, nearly co-planar nanocrystal layers have been obtained in all cases, and were investigated by cross-sectional transmission electron microscopy and photoluminescence characterization. Nanocrystal layers were deposited on 4-5 nm thermally-grown tunnel oxides and were encapsulated by a deposited high temperature oxide via a SiH₄/N₂O chemical vapor deposition process. Key device characterization features, with memory programming by either Fowler-Nordheim tunneling or channel hot electron injection and Fowler-Nordheim tunneling as the erase mechanism, will be discussed. Threshold voltage windows obtained for typical program/erase voltages and times, and threshold window closure effects observed after more than 10⁹ program/erase cycles will be compared. Transient, disturb, retention, and endurance characteristics for the different devices will be presented. In summary, the prospects for ion implantation synthesis of nanocrystal floating gates will be critically assessed.

4:45 PM O10.2

SYNTHESIS OF SPATIALLY CONTROLLED NANOSTRUCTURES BY ION IMPLANTATION IN V-GROOVES ON (001)Si SURFACES. T. Müller, K.-H. Heinig, B. Schmidt, A. Mücklich, W. Möller FZ Rossendorf, Institut für Ionenstrahlphysik und Materialforschung, Dresden, GERMANY.

The fabrication of more and more miniaturized electronic and photonic devices requires spatially controlled formation of nanostructures. Examples are electronic devices based on semiconducting nanowires, and photonic devices based on chains of metallic clusters in dielectrics which conduct light by the coupling of surface plasmons. In this contribution the synthesis of spatially controlled Ge nanowires and nanoclusters by Ge⁺ ion implantation in oxidized V-grooves on (001)Si surfaces will be presented. The V-grooves were prepared by lithography SiO₂ patterning and anisotropic wet Si etching. Dry oxidation at 1000°C results in a 200nm thick silicon dioxide layer covering the V-groove. The whole wafer having V-grooves was implanted with 2.10 × 10¹⁶ Ge⁺ cm⁻². The implanted Ge accumulates within the silicon dioxide at the bottom of the V-grooves which has been proven by XTEM-EDX mapping. Modeling and simulation has shown that Ge accumulation is caused by the specific V-groove geometry and enhanced by forward sputtering, and redeposition. During subsequent annealing the accumulated Ge forms a nanowire by precipitation, ripening and coalescence, whereas the lower Ge content in the side walls can be oxidized by a very low concentration (a few ppm) of moisture in the annealing atmosphere. Kinetic lattice Monte Carlo simulations of the nanowire formation process show growth instabilities and self-organization phenomena.

SESSION O11: POSTER SESSION
Chairs: Karl-Heinz Heinig, Steven C. Moss and
David B. Poker
Tuesday Evening, November 28, 2000
8:00 PM
Exhibition Hall D (Hynes)

O11.1

NOVEL METASTABLE ALLOYS AND THIN-FILMS FORMED USING PULSED INTENSE ION BEAMS. T.J. Renk and P.

Provencio Sandia National Laboratories, Albuquerque, NM; M.O. Thompson, Cornell University, Ithaca, NY; N. Kishimoto, National Research Institute for Metals, Tsukuba, JAPAN; and K. Kasuya, Tokyo Institute of Technology, Yokohama, JAPAN.

Materials processing applications are being investigated on the 700 kV RHEPP-1 ion beam facility at Sandia National Laboratories. Surface modification for property improvement is being investigated in the fluence range 1-5 J/cm². Surface mixing of pre-applied 0.1-2 μm thick coatings using melt-resolidification cycles has led to improvement in hardness and corrosion resistance of various metals. At 5-20 J/cm² fluences, ablation and thin-film synthesis is possible. Thin-films of ablated carbon, YBCO, and rare earth-doped SiO₂ are being investigated for mechanical and electrical properties. Titanium based alloys, with and without sputter-deposited transition metal coatings, were treated using N and Ar/C beams, producing cooling rates of 10⁸ - 10⁹ K/sec. We investigated changes in the microstructure of treated samples, using electron diffraction (SAD) and both bright field and dark field cross-sectional TEM (XTEM). Compositional changes were measured by nanobeam energy dispersive spectroscopy (EDS). EDS and XTEM observations of Pt-overcoated Ti-2 and Ti-5 substrates are consistent with incorporation of Pt in solid solution at concentrations well above equilibrium values, in micron-sized single crystal structures. These effects appear to be contributing to improved hardness. Preliminary study of treated sputter-deposited Nb coatings are being investigated, and show there may be some mixing. The micron scale metastable phase crystals do not appear at the treated surface if the transition metal coating is not present. Below the melt region, whether a transition metal is present or not, the normal micron scale Ti microstructure has been modified to 10-500 nm-scale due to a high density of defects. There is a difference between N and Ar/C beam effects, the former leading to enhanced dislocations, the latter showing more martensitic features in the treated layer. In both cases, the dislocation density is very high below the melt zone and reaches peak values at 10 and 100 micron depths, indicating that ion beam effects occur well below the melt zone.

This work was supported by the U.S. Department of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

O11.2

MODEL OF PHASE FORMATION COBALT DISILICIDE PRODUCED IN SILICON BY ION BEAM SYNTHESIS.

Yurii N. Parkhomenko, Alex V. Bozhenov, Kirill D. Chtcherbatchev, Vadim T. Bublik, Moscow State Institute of Steel and Alloys, Dept of Semiconductor Materials and Devices, Moscow, RUSSIA; Albert Romano-Rodriguez, University of Barcelona, Barcelona, SPAIN.

In order to understand the phase formation processes which occur during ion beam synthesis of CoSi₂, in this work we implanted cobalt ions in (001) silicon wafers (resistivity 4.5 Ohm•cm) with doses from (1)•10¹⁶ to (3,5)•10¹⁷ ions/cm⁻². The implantation energy were 50-180 keV, and the current density were 5-100 μA/cm². After implantation, the samples were annealed in a conventional furnace in a nitrogen ambient for 30 minute at temperatures varied within the range from 600 to 1150°C. The detailed characterization of both as-implanted and annealing samples was performed by XRD, SIMS and TEM. It is revealed, that depending on implantation conditions the relative amount of aligned A-type and twinned B-type precipitates varies. The following model of phase formation and competitive growth of precipitates with the different morphological form not contradicting set of experimental data is offered. During high dose implantation a supersaturated solid solution forms. In this solid solution because of fluctuations of concentration a phase of a cobalt disilicide coherent with a matrix - precipitates A-types form. The further growth of precipitates up to 1-2 nm results to an elastic strain, which is removed because of origin of twinned dislocations. Therefore the aligned precipitates A-type transformed in twinned B-type. Consequently, growth on non-coherent plane {110} twinned precipitates up to approximately 10-15 nm results to the relaxation of elastic strains through formation on the interfaces of misfit dislocations. The magnification of a role of surface energy of large precipitates leads to dissolving of B-type precipitates and growth of A-type precipitates, which are more energy favourable.

O11.3

STUDIES OF COMPOSITIONAL PATTERNING IN ALLOYS UNDER IRRADIATION BY KMC SIMULATIONS. Raúl A. Enrique and Pascal Bellon, University of Illinois, Dept. of MS&E and Frederick Seitz Materials Research Laboratory, Urbana, IL.

There is a collection of immiscible binary systems, e.g., Ag-Cu, Ag-Ni, Cu-Fe, etc., for which metastable solid solutions can be stabilized by

irradiation. The degree of mixing that can be obtained depends on the relative strength of two competing mechanisms: thermally-activated decomposition on one hand, and irradiation-induced mixing on the other. As a result, we are faced to a far from equilibrium steady-state arising from competing dynamics. In contrast to the equilibrium situation, for these driven systems the steady-state depends explicitly on the details of the interplay between the two kinetics, which can give rise to interesting phenomenologies. In particular, by means of a mean field modeling, we have shown [1] that the fact that atomic relocations due to irradiation can be of a greater range than the atomic motion due to thermal diffusion has implications on the resulting microstructure: for large enough relocation distances, the alloy can exhibit self-organized compositional patterning. In this work, we study the problem by means of kinetic Monte Carlo simulations of an immiscible binary alloy on a FCC lattice, undergoing thermal diffusion by a vacancy mechanism and random atomic exchanges of a finite range. The simulations show that self-organized structures can indeed be stabilized by irradiation, confirming the mean-field results, and in agreement to recent experimental results. We also use the simulations to investigate the nature of the resulting solid solution, and we find that a latent form of patterning is already evident in the structure factor $S(k)$, which we rationalize in terms of nonequilibrium theories of driven alloys.

[1] R.A. Enrique and P. Bellon, Phys. Rev. Lett. **84**, 2885 (2000).

O11.4

PLASTIC FLOW AND MIXING OF Ag INTO SiO₂ DURING Xe IRRADIATION. R.C. Birtcher, Materials Science Division, Argonne National Laboratory, Argonne, IL; S.E. Donnelly, Jule Physics Laboratory, University of Salford, Manchester, UNITED KINGDOM; L.E. Rehn, Materials Science Division, Argonne National Laboratory, Argonne, IL; and L. Thomé, Centre de Spectrométrie Nucléaire et de Spectrométrie de Masse, Orsay-Campus, FRANCE.

In situ TEM has been used to observe a thin Ag layer sandwiched between two SiO₂ layers during Xe bombardment at room temperature. Irradiation-induced plastic flow of the Ag film enlarged pre-existing pinholes and separated the film at grain boundaries. The fractional coverage of the Ag film reduced exponentially with ion dose as the Ag thin film transformed into three-dimensional microcrystals having diameters greater than 30 nm. This plastic flow process is also observed in freestanding Ag specimens during heavy ion irradiation. In addition to plastic flow, Ag atoms were ballistic injected into the SiO₂ where they precipitated into 2 - 3 nm in diameter nanoclusters. Both effects are greatly enhanced by simultaneous electron and ion irradiation.

O11.5

SYNTHESIS OF B-C-N THIN FILMS BY ION-BEAM-ASSISTED DEPOSITION AND THEIR MECHANICAL PROPERTIES.

Akihito Matsumuro, Yoshimasa Kato, Nobuhide Ohta, Nagoya Univ, Dept of Micro System Engineering, Nagoya, JAPAN.

B-C-N films have been synthesized by ion-beam-assisted deposition technique, in which boron and carbon were evaporated by electron beam and a mixed nitrogen and argon ion beam was simultaneously irradiated onto silicon (100) substrates. The ratio of argon ions to nitrogen ions was varied by the flow rate ratio of Ar and N₂ gases fed into the ion source. In this experiment, the influence of the ratio on the mechanical properties and the microstructure were investigated. Nano-indentation studies show the maximum hardness up to 23 GPa at the gas ratio of 25-50%. The films prepared under the appropriate conditions indicated low friction coefficient of 0.04-0.08 against a sapphire pin and excellent wear resistance. The existence of cubic-phase B-C-N crystalline in the film which has been predicted as one of new hard materials was revealed by transmission electron microscopy image. It was concluded that the superior mechanical properties prepared at the optimum gas ratio could be attributed to the appearance of the new cubic phase.

O11.6

NITRIDE FORMATION IN ALUMINIUM BY MULTIPLE ENERGY ION IMPLANTATION. Daniel Heyden, G.K. Wolf, University of Heidelberg, Physikalisch-Chemisches Institut, Heidelberg, GERMANY; G. Walter, University of Frankfurt-Main, Institut für Kernphysik, Frankfurt am Main, GERMANY.

Aluminium and many Al-alloys represent a group of light metals showing on one hand a good corrosion resistance but on the other hand they are very soft and therefore not appropriate for technical applications requiring high hardness and good wear resistance. Ion beam techniques represents a powerful tool for surface treatment and find more and more attention in modification of surface properties. In the last years also the surface treatment of aluminium and Al-alloys by nitriding with different ion beam techniques was applied for improving the surface properties. However the main problem for ion beam nitriding is the natural aluminium oxide surface layer which

acts as a diffusion barrier for the implanted nitrogen and therefore for the formation of a thick aluminium nitride layer. In order to improve the hardness, the wear resistance and the resistance against pitting corrosion by formation of aluminium nitride, we describe the performance of multiple energy nitrogen implantation experiments in Al. The implantations were performed with the 300kV ion implanter at GSI-Darmstadt. The energy of implanted N-14 and N-15 ions was 60keV, 110keV and 150keV at different substrate temperatures (RT, 300°C and 500°C). The ion fluence per energy step ranging from 5 - 8e17 N/cm². The total fluence was kept constant at 1,5*10e18 N/cm². The modified surface layer were analysed by RBS and NRA for nitrogen depth- profiling. The phase formation and microstructure was investigated with XPS depth-profiling and GXR measurements. In addition, hardness, wear and corrosion measurements were performed. The results by multiple energy nitrogen ion implantation will be presented with respect to the beam parameters and implantation conditions. The diffusion processes and the mechanism of phase formation will be discussed in detail.

O11.7

DEPOSITION OF DIAMOND-LIKE CARBON FILMS USING PLASMA BASED ION IMPLANTATION (PBII) WITH BIPOLAR PULSES. Soji Miyagawa, Yoshiko Miyagawa, Nat. Ind. Res. Inst. of Nagoya, Nagoya, JAPAN; Hayato Miyagawa, Inst. of Ind. Sci., Univ. of Tokyo, JAPAN.

Plasma based ion implantation (PBII) is a rapidly developing technique for surface modification of three-dimensional workpieces. In this work, a PBII processing with bi-polar high voltage pulses has been proposed, where a pulsed glow discharge plasma is produced by a positive pulse supplied directly on a target, and ions in the plasma are implanted into the target by the following negative high voltage pulse. Diamond-like carbon (DLC) coatings are of technological interest for enhancing wear resistance and corrosion resistance of metals, and it has been expected to be applied for a coating of large areas with more complex surface geometry. The DLC coatings using PBII were performed with an Ar-plasma sputter-cleaning step at 2kV, a carbon ion implantation at 20kV using PBII of methane plasma, and then a DLC deposition which employs a low pulsed bias voltage using acetylene plasma. A time resolved plasma density and the spatial profiles of the pulsed glow discharge plasma were measured by a Langmuir probe in a boxcar mode, and DLC films are synthesized under optimal conditions of the pulsed plasma. After the DLC coating, the composition and the structure of the films were measured by RBS, ERD, a micro Raman spectrometer and X-ray photoelectron spectrometer. The time and the spatial resolved measurements of the pulsed plasma reveal that the plasma density reaches less than one tenth of maximum value at 50 micro sec after the plasma ignition and increases in space as it approaches to the target. The deposition rate of DLC films reaches maximum values at the pulsed bias voltage of 5kV and the hydrogen concentration was within from 20 to 27 atomic percent.

O11.8

EFFECT OF SUBSTRATE MATERIALS ON MECHANICAL PROPERTIES AND MICROSTRUCTURE OF CARBON NITRIDE FILMS PREPARED BY ION-BEAM-ASSISTED DEPOSITION.

Hideobu Ohta, Akihito Matsumuro, Nagoya Univ, Dept of Micro System Engineering, Nagoya, JAPAN; Yutaka Takahashi, Mie Univ, Dept of Mechanical Engineering, Mie, JAPAN.

We have been reported that the argon ion assist is the effective method which improve the tribological properties and hardness when carbon nitride films were produced on silicon substrate by ion-beam-assisted deposition (IBAD). The carbon nitride film had excellent wear properties and 20 GPa of hardness. This result suggests that the carbon nitride films prepared by IBAD were expected as the engineering materials. In this study, the changes in the tribological properties, hardness, adhesion and microstructures of the carbon nitride films by IBAD with argon ion assist on several substrate materials have been investigated. Carbon was evaporated by electron beam. Nitrogen and argon ion beams were irradiated simultaneously. The conventional aluminum alloy (7075), high-carbon chrome bearing steel, pure titanium plate (99.5%) and silicon (100) wafer were used as substrates. Pin-on-disk type tribotester was used for characterization of tribological properties. The hardness was measured by the nano-indentation system. The adhesion between the films and substrates measured by scratch tester. The microstructure of interfaces between the carbon nitride films and substrates were studied by Auger electron spectroscopy (AES) analysis, X-ray photoelectron spectroscopy (XPS) analysis and cross-sectional transmission electron microscopy (TEM) observation. The correlation between mechanical properties and microstructure was clarified.

O11.9

STUDIES ON TITANIUM NITRIDE COATINGS - EFFECT OF ION BOMBARDMENT. K. Deenamma Vargheese and G. Mohan Rao

Department of Instrumentation Indian Institute of Science, Bangalore, INDIA.

Ion bombardment during thin film growth is known to cause structural and morphological changes in the deposited films and thus affecting the film properties. These effects can be due to the variation in the bombarding ion flux or their energy. We have deposited titanium nitride films by two distinctly different methods, viz. Electron Cyclotron Resonance (ECR) plasma sputtering and bias assisted reactive magnetron sputtering. The former represents low energy (typically less than 30 eV) but high density plasma (10^{11} cm^{-3}), where as, in the latter case the ion energy is controlled by varying the bias to the substrate (typically few hundred volts) but the ion flux is low (10^9 cm^{-3}). The deposited titanium nitride films are characterized for their structure, grain size, surface roughness and electrical resistivity. TiN films deposited on Silicon (100) substrates have been investigated. The films deposited by ECR plasma are highly (100) oriented with grain size of about 25 nm, while the magnetron sputtered films have random orientation and larger grain size of about 70 nm. Also, the ECR sputtered films showed a highly smooth surface with a surface roughness of 0.25 nm. The electrical resistivity of the ECR sputtered films was slightly higher (80 micro ohm.cm) than the magnetron sputtered films (70 micro ohm.cm). Due to high reactivity of ECR plasma, the ECR sputtered films have higher degree of oxygen contamination. These effects clearly bring out the difference between the high dense ion flux bombardment and high energy ion bombardment on the growing film. ECR sputtered films have been further studied for application as diffusion barriers in copper metallization of silicon. There was no detectable diffusion upto a temperature of 700°C. This was evident from the resistance measurements and RBS depth profiling studies.

O11.10
MECHANICAL PROPERTIES OF AlN THIN FILMS PREPARED BY ION BEAM ASSISTED DEPOSITION. Shuichi Miyabe, Toshiyuki Okawa, Yoshihisa Watanabe, and Yoshikazu Nakamura, National Defense Academy, Dept. of Materials Science and Engineering, Kanagawa, JAPAN.

Aluminum nitride (AlN) thin films were prepared by the ion beam assisted deposition method. The effects of deposition conditions on the mechanical properties were studied by changing the nitrogen ion beam energy from 0.1 to 1.5 keV and the substrate temperature from room temperature to 200°. The mechanical properties were studied by a nano-indentator and the microstructures were observed by a scanning electron microscope (SEM). The nano-indentation tests reveal that the film hardness decreases with increasing the ion beam energy, typically from 23 GPa to 0.1 keV ion beam to 14 GPa at 1.5 keV ion beam. Observations of the film cross section by SEM display the films prepared with low energy ion beam show a columnar structure and the films prepared with high energy ion beam show a granular structure. These results suggest that the difference in the film hardness is related to the difference in the film microstructure. Compared with the films prepared on the substrate of room temperature and 200°C kept at 0.2 keV ion beam, the remarkable difference in hardness is not discerned between both films.

O11.11
STRUCTURE, NANO-HARDNESS AND TRIBOLOGICAL PROPERTIES OF CHROMIUM NITRIDE THIN FILMS DEPOSITED ON SILICON USING ION-BEAM-ASSISTED-DEPOSITION METHOD. C.-H. Ma, Z.-K. Xu, Haydn Chen, Department of Materials Science and Engineering and Frederick Seitz Materials Research Laboratory, University of Illinois at Urbana-Champaign, Urbana, IL.

Highly (200) oriented Chromium Nitride (CrN) thin films have been grown on Si by Ion-Beam-Assisted-Deposition (IBAD) method. A number of different processing conditions, such as substrate temperature, ion (N₂ and N⁺) to metal (Cr) ratio, ion beam incident angle and energy, have been selected to enhance the growth of CrN phase vis-a-vis the Cr₂N phase. Preferred orientations and nano-hardness were measured. Amount of residual stress as well as grain size were found to change with deposition parameters. The optimal conditions to grow CrN/Cr₂N films with a dense columnar structure and high hardness were discussed. Microstructure of grown films were examined using XRD, X-TEM, SEM and AFM techniques and are correlated to their mechanical, thermal and electrical properties. Keywords: IBAD, Chromium Nitride, Preferred orientation

O11.12
MODELING FOR THE DIAMOND LIKE CARBON FILM SYNTHESIS BY PBII WITH METHANE PLASMA. Yoshiko Miyagawa, Soji Miyagawa, Nat. Ind. Res. Inst. of Nagoya, Agency of Industrial Science and Technology, Hayato Miyagawa, Inst. of Ind. Sci., Univ. of Tokyo, JAPAN.

The properties of DLC film can be characterized by the hydrogen concentration and by the relative fractions of sp² and sp³ bonded carbon. The energy of the impinging ion together with the ion/neutral arrival ratio plays a crucial role for the properties of the film synthesized by the plasma based ion implantation (PBII). Dynamic Monte Carlo simulations with the binary collision approximation have been applied to the synthesis of DLC films by PBII. We take as representative carbon carriers energetic CH₃⁺ ions and CH₃ radicals, which are abundant at sufficiently low pressure. The direct chemical incorporation of the radicals, like CH₃ reacting with a diamond surface, is too low for the deposition of DLC films, so that the other reaction mechanisms should be responsible. The assumptions used in the calculation were: (a) Complete dissociation of CH₃⁺ ions into one C atom and three H atoms with identical velocities upon bombarding the surface. (b) A unity and only one mono layer sticking of CH₃ radicals on the surface. (c) Incorporation of H and C atoms under the surface induced by binary collisions with energetic CH₃⁺ ions. (d) Release of H atoms by the dissociation of CH₃ radicals on the surface. (e) Release of the displaced H atom after the subsequent collision cascade. Effects of ion/neutral arrival ratio and ion energy on the deposition rate, the mixing layer thickness and the hydrogen content in the deposited film are presented. Preliminary calculation for sp³/sp² ratio is also presented.

O11.13
IMPLANTATION OF Ta IONS INTO A COPPER SINGLE CRYSTAL (100), (110), (111). Alexander D. Pogrebnyak, Vladimir V. Bondarenko, Sumy Institute for Surface Modification, Sumy, UKRAINE.

High-dose ion implantation of tantalum ions into a copper single crystal has been investigated. Irradiation was performed in three different directions: (100), (110), (111). The rate of a dose accumulation was 10¹⁶ ion/cm² per minute and current density - tens of mA/cm² during ion implantation. Studies of an element composition of copper sample surfaces, which were carried out with the help of Rutherford backscattering spectrometry, showed that tantalum distribution is typical for high dose ion implantation and depends on a plane of a treatment. Maximum in concentration profile of tantalum atoms was observed on the surface for all of the samples due to processes of sputtering, which is of great importance in case of high dose ion implantation. Surface concentration of the tantalum atoms was twice as much higher for plane (100) than for (111). Plane (100) is a plane of the closest packing in FCC lattice of a copper. So a penetration of the tantalum ions in this direction is the most difficult and therefore the surface concentration is higher. Such difference in tantalum concentration can be seen in pictures of surfaces obtained with the help of scanning electron microscope. There are many crystallites on the surface of the copper single crystal (100). These crystallites weren't present on other sample surfaces evidently due to low atom concentration of the tantalum. Tantalum implantation was accompanied by surface carbonization and oxidation. Carbon and oxygen atoms were presented in the residual atmosphere of vacuum chamber because of poor vacuum (10⁻³ Pa). Concentration of carbon and oxygen atoms on the surface reached 50 at.%. Microhardness tests showed that surface hardness increased for all of the treated samples. Microhardness values for them was ~30% higher than for original samples. During the corrosion studies also was observed corrosion resistance enhancement for implanted sample. Mass coefficient of corrosion was one order lower in case of the irradiated sample.

O11.14
TEXTURED Cu(111) FILMS PREPARED BY ION BEAM ASSISTED DEPOSITION ON Si(111), Ti(111)/Si(111) AND Ti(010)/Si(111) SUBSTRATES. H.C. Mu, Y.H. Yu, Ion Beam Laboratory, Shanghai Institute of Metallurgy, Chinese Academy of Sciences, Shanghai, CHINA; E.Z. Luo, B. Sundaravel, S.P. Wong, I.H. Wilson, Department of Electronic Engineering, Chinese University of Hong Kong, Hong Kong, CHINA.

(111) oriented Cu films have been prepared by ion beam assisted deposition (IBAD) on Si(111), TiN(111)/Si(111) and Ti(010)/Si(111) substrates. The influence of the IBAD parameters, such as the incident angle of bombarding ion beam (61537) and ion/atom arrival rate ratio (R), on the texture of the Cu(111) overlayer on Si(111) substrate have been investigated. It was found that the development of the texture of the Cu(111) overlayer shows no apparent correlation with the variation of the incident angle of the bombarding ion beam while it could be improved gradually with increasing R up to 0.4 and was degraded with the further increase of R. The great effect of the texture of underlayer Si(111), TiN(111) and Ti(010) on the texture of IBAD derived Cu(111) overlayer are demonstrated. The Cu(111) films on Ti(010) underlayer show the best texture behavior on account of the high lattice match between them compared to that at TiN(111) and Si(111) underlayer. (200) 61542;-scan test also indicates the existence of the twin boundaries in Cu(111) overlayer, which is demonstrated by TEM images.

O11.15

NANO-PROCESSING WITH GAS-CLUSTER ION BEAMS.

Isao Yamada, Laboratory of Advanced Science and Technology for Industry, Himeji Institute of Technology, CAST, Ako, JAPAN; Jiro Matsuo, Ion Beam Engineering Experimental Laboratory, Kyoto University, Kyoto, JAPAN; Allen Kirkpatrick, Epion Corporation, Bellerica, MA.

Gas cluster ion beam (GCIB) processes use a beam of ions consisting of a few hundreds to thousands of atoms generated from various kinds of gaseous materials. In GCIB process, non-linear collisions occurring during the impact of accelerated cluster ions upon substrate surfaces produce fundamentally low energy bombarding effects at very high density. These bombarding characteristics can be applied to (i) shallow ion implantation, (ii) high yield sputtering, cleaning and smoothing, and (iii) low temperature thin film formation. These characteristics can facilitate new industrial applications which would not be possible by traditional ion beam processing. R&D of gas cluster ion beam processing is now going on in several different fields. Possible industrial applications are discussed. These presently include (i) shallow ion implantation for ULSI applications, (ii) atomic scale surface smoothing and low damage processes on metals, dielectrics, superconductors, and diamond films for SOR and X-ray lithography, non-spherical plastic lens mold surfaces, SiC surfaces, SIMOX, MOS gate oxide in advanced VLSI, (iii) very high yield sputtering and etching processes, (iv) high quality thin multi-layer films for reliable and durable optical filters, transparent conductive films (ITO).

O11.16

ION RADIATION DAMAGE EVOLUTION IN RECONSTRUCTED SURFACES. O. Rodríguez de la Fuente, M.A. González, J.M. Rojo, Departamento de Física de Materiales, Universidad Complutense, Madrid, SPAIN.

Ion beam surface modification is becoming increasingly more important for scientific and technological purposes. A better knowledge of radiation damage evolution during ion bombardment and the role that experimental conditions play on the final morphology is required. In this work we present an STM study of surface evolution during low-energy ion bombardment in reconstructed Au(001) as a function of the dose, ranging from the initial stages to very high doses. Bombardment with similar doses of Pt(001) show the same general behaviour. Apart from vacancy islands, other defects related to the hexagonal-like reconstruction are reported. At very low doses the main features are 2D dislocation dipoles, which act as nucleation and growth sites for rectangular vacancy islands after subsequent bombardment. When these islands are large enough but still below a nominal removal of one monolayer, perpendicular reconstruction domains and unreconstructed patches with square (001) symmetry become visible. Extending the bombardment to very high doses results in coalescence of vacancy islands and formation of multi-level structure. It is shown that raising the temperature during ion bombardment results in very large vacancy islands, while lowering the ion fluence results in fewer exposed levels and a smoother surface. Some annealing processes are also studied at room temperature in the high-dose configurations, such as vacancy and adatom islands disappearance or transitions from non-reconstructed to reconstructed patches.

O11.17

FORMATION OF METAL NANOCRYSTALS IN SILICON BY IMPLANTATION WITH HIGH DOSES HEAVY IONS.

Christo Angelov, Institute for Nuclear Research and Nuclear Energy; Maria Kalitzova, Institute for Solid State Physics, Sofia, BULGARIA.

The present paper addresses the process of Ion Beam Induced Crystallization (IBIC) in amorphized silicon (a-Si) layers produced by high dose implantation of large mass, low solubility species, Pb^{+} and Bi^{+} . (100) oriented Si wafers have been implanted at room temperature (RT) with 50 keV Pb^{+} and Bi^{+} ions to doses ranging from 5×10^{13} to $1 \times 10^{18} \text{ cm}^{-2}$ at a constant ion current density of $10 \text{ } \mu\text{A} \cdot \text{cm}^{-2}$. The resulting structures have been evaluated by Conventional Transmission Electron Microscopy (TEM), High Resolution Transmission Electron Microscopy (HRTEM) and Rutherford Backscattering Spectroscopy (RBS) in combination with appropriate computer simulations. The kinetics of ion beam induced crystallization of new phases and precipitate evolution in the a-Si have been studied as a function of implant dose. It has been established that the front of new phase crystallization (cubic Pb and hexagonal Bi nanocrystals) starts at certain sites within the a-Si layer corresponding to the peaks in the Pb^{+} and Bi^{+} implant profiles. As the dose is increased, the formation of Pb and Bi precipitates occurs closer to the surface. Computer data processing of the HRTEM images of the nanocrystals so formed has been carried out, and the Nygren theoretical model for recrystallization via precipitation in a liquid phase of species having a low melting point has been experimentally confirmed for the Bi:Si system.

O11.18

METAL-ALLOY NANOCLUSTER FORMATION IN SILICA GLASS BY SEQUENTIAL ION IMPLANTATION. Giancarlo Battaglin, Elti Cattaruzza, Francesco Gonella, INFIM, Venice Univ, Dept of Physical Chemistry, Venice, ITALY; Giovanni Mattei, Chiara Maurizio, Paolo Mazzoldi, INFIM, Padua Univ, Dept of Physics, Padua, ITALY; Francesco D'Acapito, INFIM, GILDA-CRG, ESRF, Grenoble, FRANCE.

Sequential ion implantation of two metal species in silica glass may give rise to the formation of alloy metal nanoclusters. This depends on the thermodynamics of compound formation, in terms of Gibbs free energy balance, and on the physics of ion-matrix interaction. Composite materials with peculiar optical properties can be therefore fabricated, with application in integrated-optical devices. In the presented experiment, several transition elements implantations in fused silica are realized (Cu Ni, Au Cu, Au Ag, etc.). The resulting systems, in some cases after annealing in either reducing or oxidizing atmospheres, are studied by optical absorption spectroscopy, transmission electron microscopy, X-ray photoelectron spectroscopy, and extended X-ray absorption fine structure spectroscopy. The formation of different alloy nanoclusters is evidenced, with detailed information on the clusters crystalline structure and on the chemical environment of the dopant species.

O11.19

NANO-CRYSTALS IN STRONTIUM ION IMPLANTED CUBIC ZIRCONIA. Sha Zhu, Shixin Wang, Lumin Wang, Rodney C. Ewing, University of Michigan, Department of Nuclear Engineering and Radiological Sciences, Ann Arbor, MI.

Yttria stabilized zirconia (YSZ, cubic fluorite structure) is a promising candidate material as both an inert matrix fuel for "burning" excess plutonium in reactors and a nuclear waste form for direct geologic disposal. In this study, 400 keV strontium ions have been implanted into YSZ in order to study the effects of fission product incorporation in YSZ. The ion implantation was conducted at room temperature. The ion fluence reached $1 \times 10^{21} \text{ ions/m}^2$ which gives a peak displacement damage level of $\sim 330 \text{ dpa}$ and a peak Sr-concentration of $\sim 11.6 \text{ at.}\%$. In situ plan-view and ex situ cross-sectional transmission electron microscopy (TEM) was completed to investigate the microstructure changes caused by the implantation. No evidence of amorphization was detected after the highest dose although a high density of defect clusters was observed as early as $1 \times 10^{20} \text{ ions/m}^2$ by in situ TEM. Cross-sectional TEM revealed crystalline precipitates of a few tens of nanometers after annealing of the implanted bulk sample at 1000°C for two hours. The nano-crystals were concentrated in a narrow band of $\sim 80 \text{ nm}$. The nano-crystalline precipitates are isometric SrZrO_3 ($a \approx 0.409 \text{ nm}$). The orientation relation between the matrix and precipitates, as determined by selected area diffraction pattern, was: $[011]_{\text{YSZ}} // [111]_{\text{SrZrO}_3}$. The lattice mismatch was compensated by interface dislocations, as shown by high resolution TEM. The size and density dependence of the nano-crystals on annealing was also determined.

O11.20

EVOLUTION OF ION BEAM SYNTHESIZED Au NANO-CLUSTERS IN SiO_2 UNDER ION IRRADIATION. Bernd Schmidt, Karl-Heinz Heinig, Arndt Mücklich Research Center Rossendorf, Institute of Ion Beam Physics and Materials Research, Dresden, GERMANY.

Au nanoclusters have been synthesized in a 500 nm thick SiO_2 layer on (001)Si by 330 keV implantation of $2 \times 10^{16} \text{ Au}^+ \text{ cm}^{-2}$ and subsequent annealing at $T = 1000^\circ\text{C}$ for 1 h in dry oxygen. XTEM images show around the projected ion range $R_p = 100 \text{ nm}$ crystalline Au clusters of a mean size of 5 nm with a broad size-distribution resembling roughly the LSW distribution of diffusion controlled Ostwald ripening. The mean nanocluster size as well as the nanocluster size distribution can be changed drastically by high-energy ion irradiation. High-energy Au^+ ions of 4.5 MeV with $R_p = 1000 \text{ nm}$ have been used in order to avoid Au^+ implantation in the nanocluster region at 100 nm depth. For irradiation temperatures in the range of RT... 200°C and for fluences of $0.5 \dots 1.5 \times 10^{16} \text{ Au}^+ \text{ cm}^{-2}$ the observed narrowing of the width of the size-distribution as well as the decrease in the mean nanocluster size will be compared with recent theoretical predictions on inverse Ostwald ripening under ion irradiation.

O11.21

METALLIC NANOPARTICLE FORMATION IN ION-IMPLANTED SILICA: DEPENDENCE ON THE ANNEALING ATMOSPHERE. J.C. Cheang-Wong, A. Oliver, J. Roiz, L. Rodríguez-Fernández, J.M. Hernández, J.G. Morales, E. Muñoz and A. Crespo, Instituto de Física, Universidad Nacional Autónoma de México, MEXICO.

Composites formed by nanometer-sized metallic clusters embedded in

glass matrices exhibit interesting optical properties, and are promising candidates for applications in different material-science fields. These metallic nanoparticles can be synthesized within silica glasses by ion implantation followed by thermal annealing. Several factors such as the ion fluence, the radiation damage induced by the ion implantation, and the subsequent thermal treatment conditions, can determine the particle size at superficial layers of the ion-implanted silica glass. High-purity silica samples, with an OH content less than 1 ppm and a total impurity content less than 20 ppm, were implanted with 2 MeV Cu, Ag and Au ions at various fluences (0.7, 3.0, and 5.0×10^{16} ions/cm²). The samples were then annealed in either a reducing or an oxidizing atmosphere at temperatures ranging from 300°C to 1000°C, and characterized by optical absorption, electron paramagnetic resonance (EPR) and Rutherford backscattering measurements. The evolution of both the nanoparticle formation and of the B₂ and E' point-defects generated by the ion implantation is discussed as a function of the thermal annealing conditions.

O11.22

GOLD NANOCLUSTER CONTAINING DIELECTRIC FILMS DEPOSITED BY ION BEAM ASSISTED DEPOSITION. S. Schiestel, C.A. Carosella, G.K. Hubler, S. Qadri and D. Knies, Naval Research Laboratory, Washington, DC.

Au nanoclusters in a dielectric film were deposited by simultaneous evaporation of Au and the dielectric matrix in the presence of an Ar ion beam. Defects, radiation enhanced diffusion and segregation, and ballistic mixing are some of the processes that play a role in the final size and size distribution, shape and orientation of the nanoclusters. In this contribution we will present the potential and limits of ion beam assisted phase separation on control of nanocluster sizes, size distribution, shape and orientation in the model system of gold in a dielectric matrix such as silica. The displacements per atom (DPA) resulting from the IBAD process are varied from 0.0 to 2.0, by controlling the argon ion beam energy (50 - 400 eV) and ion-atom arrival rate. Cluster size, size distribution, orientation and shape are investigated by X-Ray diffraction; Au concentration and profiling are found by Rutherford backscattering. We observe various preferred orientations of Au nanocrystals (nc's) that depend on the DPA level. Two nm Au crystals form with (111) orientation at a DPA of 0.05; this orientation is maintained for post-annealing temperatures less than 800°C. At and above 800°C annealing, the Au particles have mainly (200) orientation. The particles are also elongated in the (200) direction. At DPA's > .05, the gold clusters have a more round shape and are randomly oriented. These results are compared to gold clusters deposited at elevated temperatures. X-ray diffraction also reveals that the Au clusters formed with IBAD are highly strained; these strains relax with increasing annealing temperature.

O11.23

KINETICS OF ION BEAM SYNTHESIS OF Sn AND Sb CLUSTERS IN SiO₂ LAYERS. S. Spiga, S. Ferrari, M. Fanciulli, Laboratorio MDM-INFM, Agrate Brianza, ITALY; B. Schmidt, K.H. Heinig, R. Grötzschel, A. Mücklich, Research Center Rossendorf Inc., Institute of Ion Beam Physics and Materials Research, Dresden, GERMANY; G. Pavia, STMicroelectronics, Agrate Brianza, ITALY.

The study of the formation of metallic and semiconducting nanocrystals embedded in SiO₂ layers is gaining interest, mainly due to the promising technological applications, such as memory devices and single electron transistors. Recently, the ion beam synthesis of nanocrystals in SiO₂ has been demonstrated and single electron effects have been observed at low temperature. However, several problems, mainly related to the control of the cluster-size distribution and cluster position in the oxide layer, remain to be solved. In this contribution, we report on the kinetics of formation of metallic nanocrystals in SiO₂. Sn and Sb clusters have been formed in 100 nm and 20 nm thick SiO₂ layers by ion implantation with different energies (12 ÷ 100 keV) and doses ($5 \cdot 10^{14}$ ÷ $2 \cdot 10^{16}$ ions/cm²) followed by furnace or rapid thermal annealing. The depth distribution of implanted Sn and Sb after annealing have been studied by dual-beam Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS), Rutherford backscattering spectroscopy (RBS) and cross-sectional TEM. The evolution of nanoclusters has been related to the implantation dose and energy, oxide thickness, and annealing conditions. The optimal conditions for the formation of crystalline clusters have been addressed. In thicker oxides, the formation of Sn and SnO_x cluster bands at different depth in the SiO₂ has been observed. This can be influenced by the processing ambient during annealing. The influence of the annealing atmosphere becomes stronger for thinner oxides. Differences in the chemical reactivity of Sn and Sb nanoclusters will be discussed. Mössbauer spectroscopy on ¹¹⁹Sn implanted samples has been used to determine the Sn atomic local environment.

O11.24

LITHIUM NANOCLUSTER FORMATION IN Li-ION IMPLANTED

MgO. A. van Veen, M.A. van Huis, A.V. Fedorov, H. Schut, C.V. Falub, S.W.H. Eijt, F. Labohm, Delft University of Technology, Interfaculty Reactor Institute, Delft, NETHERLANDS; B.J. Kooi, J.Th.M. de Hosson, University of Groningen, Materials Science Centre, NETHERLANDS.

The optical effects of lithium clusters produced by ion implantation and subsequent annealing show a very large dependence on annealing temperature. In this study photon absorption and a variety of techniques is applied to reveal structure and morphology of the precipitates. In this way the relation between photon absorption and precipitates is clarified. Monocrystals of Mg(100) were implanted with 1.0×10^{16} ⁶Li ions cm⁻² at an energy of 30 keV. The samples were thermally annealed in air in steps up to 1200 K. After each step Doppler broadening Positron Beam Analysis (PBA) was applied to monitor the depth profile of the implantation defects. The evolution of the depth profile of lithium was followed by the method of Neutron Depth Profiling (NDP) using the reaction ⁶Li (n,α) ³H. During the annealing hardly any change is observed in the location of the lithium implantation peak at 150 nm (peak concentration 1 at %). Only after annealing to 1200 K the majority of the lithium has left the crystal and optical absorption effects have disappeared. In the as-implanted sample absorption is centered at 580nm (V-centers); at 750 K an absorption band develops from 400 to 600 nm; at 950 K the maximum absorption is centered at 450 nm; at 1100 K there is a shift of the maximum to 560 nm. Cross-sectional TEM pictures taken at 950 K revealed 2-4 nm size semi-coherent nanoclusters at the implantation depth of Li. In addition, cubic voids of smaller size are observed. Positron beam analysis showed a considerable increase of annihilations with low momentum electrons in the implanted zone. This is in line with the formation of voids as well as Li-precipitates. A high resolution method for measuring electron momentum distributions (2D-ACAR coupled to an intense positron beam) gave evidence for the presence of metallic lithium inclusions.

O11.25

PROPERTIES OF InAs NANOCRYSTALS IN SILICON FORMED BY SEQUENTIAL ION IMPLANTATION. A. Tchebotareva, J.L. Brebner, S. Roorda, Université de Montréal, Dept of Physics, Montréal, CANADA; C.W. White, Oak Ridge National Laboratory, Oak Ridge, TN.

Optical and structural properties of InAs nanocrystals fabricated by co-implantation of In and As ions in Si-c (100), followed by thermal annealing are investigated. In the first sample named Si/AsIn the implantation of As ions was followed by In ion implantation, whereas in the second sample named Si/InAs the order of implantation was inverted. RBS spectra of these samples taken before and after annealing show that the depth profiles of implanted ions depend strongly on the order of implantation. XRD measurements confirm the presence of InAs crystallites oriented along the crystallographic axes of the silicon matrix irrespective of the order of implantation. Alignment and strain in the InAs nanocrystals were investigated through ion channelling in (100), (110), and (111) directions. These results, as well as those of photoluminescence and optical absorption measurements indicate that the average size and size distribution of the resultant nanocrystals are strongly dependent on the order of ion implantation. Research sponsored by the U.S. Department of Energy under contract DE-AC05-00OR22725 with the Oak Ridge National Laboratory, managed by UT-Battelle, LLC.

O11.26

HIGH-FLUENCE IMPLANTATION OF ERBIUM INTO SILICON-GERMANIUM ALLOYS: STRUCTURAL AND THERMAL PROPERTIES. Vladimir Touboltsev, Jyrki Räisänen, Department of Physics, University of Jyväskylä, FINLAND; Erik Johnson, Allan Johansen, Leif Sarholt, Oersted Laboratory, Niels Bohr Institute, Copenhagen, DENMARK.

Recently the optical properties of Er incorporated into semiconductors have attracted a great deal of attention due to potential applications in optoelectronics. Among the various semiconductors, Si based materials are of particular interest as luminescence from Er in its trivalent state at a wavelength of 1.54 μm corresponds to the minimum in optical absorption of silica. Incorporation of Er into Si-based chips is expected to provide the basis for optoelectronic integrated circuits with considerably improved performance. However, the terminal solid solubility of Er in Si-based materials is very limited. Consequently, Si-Er optoelectronic devices produced by near-thermal equilibrium methods (e.g. doping during MBE growth) show insufficient emission during room temperature operation. The band gap energy of the host semiconductor is an important parameter for optimization of the emission. In a Si_{1-x}Ge_x host the band gap energy and thus the energy transfer can reach acceptable levels when the Ge concentration x is changed. Therefore, Si_{1-x}Ge_x alloys form a promising material for fabrication of advanced optoelectronic devices with high integrability in conventional Si technology. Since ion

implantation by nature is a non-equilibrium process, it can be used to extend the concentration of optically active Er atoms in $\text{Si}_{1-x}\text{Ge}_x$ alloys well beyond the terminal equilibrium solid solubility limit. High-quality crystalline $\text{Si}_{1-x}\text{Ge}_x$ ($x=0.10$ and 0.25) alloys were implanted with 70 keV Er^+ ions at temperatures of 350°C and 550°C to a fluence of 10^{15} cm^{-2} . In-situ Rutherford backscattering/channeling analysis supplemented with transmission electron microscopy showed that as-implanted alloys were in form of ternary solid solutions with a peak Er concentration of 1 at.% without any trace of Er-Si or Er-Ge phases precipitation. In the samples implanted at 350°C Er atoms were found to be distributed randomly in the heavily damaged host matrix. Post-implantation annealing at different temperatures up to 600°C showed that the solid phase epitaxial regrowth of the damaged layers strongly depends on both the Ge concentration in the alloys and the temperature of annealing. Along with the recrystallization of the damaged matrix, annealing was observed to induce simultaneous removal of nearly all the implanted Er as the recrystallization front progresses towards the surface. In contrast, high temperature implantation at 550°C led to spontaneous recovery of the alloys crystallinity and incorporation of the major fraction of implanted Er atoms on regular tetrahedral interstitial sites in the host matrix.

O11.27

GROWTH AND CHARACTERIZATION OF ERBIUM SILICIDE SYNTHESIZED BY METAL VAPOR VACUUM ARC ION IMPLANTATION. X.W. Zhang, W.Y. Cheung, S.P. Wong, Department of Electronic Engineering, The Chinese University of Hong Kong, Hong Kong, P.R. CHINA.

Recently, erbium silicide has attracted considerable attention because its use in fabrication of interesting new devices such as metallic base transistor or tunable infrared detector, also in conventional microelectronics technology as ohmic or rectifying contact or low-resistance interconnect. In this paper, erbium silicide layers were formed by high-dose Er implantation into p-type Si (111) substrates with a metal vapor vacuum arc (MEVVA) ion source. The implantation was performed at an extracted voltage of 60 kV at various beam currents to doses ranging from 2×10^{17} to $6 \times 10^{17}\text{ cm}^{-2}$. Annealing was carried out in argon atmosphere at various temperatures by rapid thermal annealing. The characterization of the as-implanted and annealed samples was performed using Rutherford backscattering spectrometry and x-ray diffraction experiments. The results showed that ErSi_{2-x} layers were directly formed by MEVVA implantation without post-annealing. The electrical properties of the erbium silicide layers were studied before and after annealing using resistivity and Hall effect measurements from 30 to 400 K . Results of the microstructures and properties of erbium silicide layers and their dependence on the preparation and annealing conditions will be presented. This work is partially supported by the Research Grants Council of Hong Kong SAR (Ref. No. CUHK4405/99E).

O11.28

INFLUENCE OF CARBON ON ERBIUM LATTICE LOCATION IN Si:Er. Xiaotang Ren and Mengbing Huang Department of Physics, State University of New York at Albany, Albany, NY.

The luminescence intensity of Si:Er is known to strongly depend on impurities (e.g. carbon) in silicon. In this work, we investigate the effect of carbon doping on the lattice location of Er atoms in Si. A Czochralski-grown Si(100) wafer is first amorphized to a depth of $\sim 0.23\mu\text{m}$ by Si ions implanted to a dose of $\sim 1 \times 10^{15}/\text{cm}^2$ at liquid nitrogen temperature. Carbon is then implanted into the amorphous silicon, and the silicon is recrystallized via solid phase epitaxial growth (SPEG) at 600°C . After the SPEG process, implanted carbon atoms are incorporated on silicon lattice sites. Finally the C-doped sample along with the control sample is implanted with 350-keV Er ions ($R_p = 0.12 \pm 0.02\mu\text{m}$) at 300°C to various doses. Rutherford backscattering (RBS) and nuclear reaction analysis (NRA) in conjunction with channeling techniques are applied to detect the lattice location of Er and C atoms after thermal annealing. The results are correlated with the photoluminescence measurement, providing insight into the effect of carbon on the enhancement of luminescence in Si:Er.

O11.29

VISIBLE PHOTOLUMINESCENCE OF Gd-DOPED SiO_2 FILMS FABRICATED BY PULSED ION BEAM ABLATION WITH RHEPP-1. N. Kishimoto, National Research Institute for Metals, Tsukuba, JAPAN; T.J. Renk, Sandia National Laboratories, Albuquerque, NM; M.O. Thompson, Cornell University, Ithaca, NY; and K. Kasuya, Tokyo Institute of Technology, Yokohama, JAPAN.

Rare-earth elements in solids have attracted much attention in magnetic and optical properties due to the characteristic electronic states of partially filled 4f shells. One of the key issues for photonic

applications is to obtain efficient "blue emission" at room temperature. Among the lanthanide elements, photoluminescence (PL) of Gd has been less studied because the relevant energy gap is too large ($>4\text{ eV}$) for optical or electrical excitation. To achieve non-equilibrium doping of Gd into a SiO_2 film, we have used pulsed ion beam ablation on the 700 kV RHEPP-1 facility at Sandia National Laboratories. Thin films of Gd-doped SiO_2 were fabricated by intense carbon-beam ablation at $5\text{-}10\text{ J/cm}^2$ fluences. Photoluminescence was measured by a luminescence-lifetime detection system (Hamamatsu Photonics Co. Ltd.) which consisted of an N_2 -laser for excitation (wavelength 337 nm , pulse width 300 ps) and a streak scope. The lifetime resolution was about 5 psec . The Gd/ SiO_2 film as deposited showed intense visible luminescence at room temperature. The blue-green emission was clearly discernible by eyesight, e.g., the intensity is comparable with porous Si. A broad band in the spectrum ranged from 370 nm to 550 nm and roughly consisted of two broad peaks around 420 nm and 480 nm . The lifetime is about 4 ns with a weak dependence on wavelength. The ordinary energy diagram of Gd3 does not seem to allow optical transitions of 337 nm -excitation nor the PL observed. The broad spectrum and the short lifetime suggest that the origin of PL is ascribed to defect-associated Gd atoms, not to isolated ones. Annealing behaviors and the mechanism of PL will be discussed.

O11.30

RECOVERY OF OPTICAL AND STRUCTURAL PROPERTIES AFTER ANNEALING OF ION IMPLANTED GaN. Igor Usov, Nalin Parikh, Brian Stoner, Univ of North Carolina, Dept of Physics and Astronomy, Chapel Hill, NC; Darren Thomson, Robert Davis, North Carolina State Univ, Dept of Material Science and Engineering, Raleigh, NC; Jaime Freitas, George Braga, Naval Research Laboratory, Washington, DC; Stephen Withrow, Oak Ridge National Laboratory, SMAC, Oak Ridge, TN.

We report on the recovery of optical and structural properties of Na-implanted GaN films after annealing at temperatures up to 1100°C . The annealing was performed both in a rapid thermal annealing (RTA) furnace under a nitrogen ambient as well as in a microwave plasma enhanced chemical vapor deposition (MPECVD) apparatus with an ammonia/hydrogen plasma ambient. The GaN samples annealed in the RTA furnace were encapsulated with a thin film of AlN to protect the surface from decomposition at elevated temperatures. In the case of MPECVD annealing, the plasma was comprised of nitrogen and hydrogen ions and excited neutrals; this overpressure of atomic nitrogen prevented loss of nitrogen from the GaN surface. The optical and structural properties of the implanted films were studied before and after annealing by low temperature photoluminescence and channeling Rutherford backscattering spectroscopy. Both the atomic nitrogen and hydrogen overpressures appear to play an essential role in the recovery of radiation damage and the activation of implanted impurities in GaN. Annealing in the MPECVD reactor significantly reduces the implantation-induced disorder and restores the luminescence properties to that of the un-implanted material. In addition, the Na impurity related peaks observed after RTA annealing appear to be completely passivated as a result of annealing in the ammonia/hydrogen plasma as a result of diffusion of hydrogen into the GaN near-surface region.

O11.31

ION BEAM RADIATION EFFECTS ON InAs/GaAs SEMICONDUCTOR QUANTUM DOTS. Jie Zhu, Department of Physics, State University of New York at Albany, Albany, NY; Mengbing Huang, Department of Physics, State University of New York at Albany, Albany, NY.

Self-assembled quantum dots (QDs) have attracted significant attention, because of their potential applications in novel electronic and optical devices. Understanding radiation effects on QDs is important in order to extend QDs in space applications. In this work, we study ion beam radiation effects on InAs/GaAs quantum dots. The luminescence and the strain field associated with the dots are examined for various conditions (e.g. ion species, ion doses and annealing temperatures). The results are discussed in terms of interactions between defects and quantum dots.

O11.32

ION BEAM SYNTHESIS BASED FORMATION OF Si- AND Ge RICH THERMALLY GROWN SILICON DIOXIDE LAYERS FOR MEMORY APPLICATIONS. T. Gebel¹, H.-J. Thees², J.V. Borany¹, M. Wittmaack², K.-H. Stegemann² and W. Skorupa¹. ¹Institute of Ion Beam Physics and Materials Research, Forschungszentrum Rossendorf, Dresden, GERMANY; ²Zentrum für Mikroelektronik GmbH, Dresden, GERMANY.

Ion beam synthesis (IBS) is a versatile instrument to circumvent obstacles dictated by the thermal equilibrium. The basic steps to perform ion beam synthesis include ion beam irradiation with

stoichiometric doses into a target kept at a certain temperature followed by another dedicated annealing step. On the other hand the actual EEPROM's basing on floating gate transistors are driven to their limits of performance. An advanced very promising approach to circumvent these problems bases on the concept of Si-and Ge-nanoclusters embedded into silicon dioxide layers using IBS. We have performed room temperature implantation of high fluences ($3.9 \times 10^{15} \text{ cm}^{-2}$) of silicon and germanium into silicon dioxide layers with a thickness in the range 20-30 nm. This was followed by an annealing step at 950°C for 30 s using Rapid Thermal Annealing (RTP). The microstructure and the electrical properties were investigated using a variety of methods. It will be shown that this type of processing leads to the formation of nanoclusters and the formation of trapping centres within the silicon dioxide layers. First device tests look promising.

O11.33

ION-PLATING DEPOSITION OF MgO PROTECT LAYER FOR AC-PLASMA DISPLAY PANEL. Kazuo Uetani, Yasuhiro Koizumi, Koichi Nose, Yasushi Ihara, ShinMaywa Industries, Ltd., R&D Center, Hyogo, JAPAN; Hiroshi Kajiyama, Akira Kato, Ken-ichi Onisawa, Tetsuro Minemura, Hitachi, Ltd., Hitachi Research Laboratory, Ibaraki, JAPAN.

Plasma display panel (PDP) is a self-emitting flat panel display using a glow discharge of a Ne Xe gas mixture. The ac-type PDP has stripe electrodes embedded in a stacked structure of a dielectric layer and a MgO protecting layer. The top-most MgO is all responsible for a stable discharge and an operation at low bias voltages. The high resistance to ion sputtering and high secondary emission by ion bombardment is strongly desired for the protect layer. The goal of this paper is to improve the quality of the MgO for PDP using the advanced ion-plating apparatus developed by ShinMaywa Industries, Japan. We deposited the MgO films with thickness up to 500 nm at a deposition rate of 1-10 nm/s and a substrate temperature of 450 K. RF and DC bias-voltage were directly applied to the substrate holder so as to sustain a stable plasma at an Ar gas pressures smaller than 2×10^{-2} Pa. We observed the surface morphologies in a sub-micron scale using an atomic force microscopy. Film texture, crystal orientation, surface area, temperature desorption characteristics and secondary electron yield of the MgO films were measured. So far the MgO protect layers have been manufactured by an electron beam (EB) method. Thus we referred the quality of EB films deposited at the same conditions. We confirmed that the MgO films deposited in this paper were structurally and electrically improved so much, compared with the EB films. The MgO films had the (111), (220), and (200) orientations depending on the deposition conditions. The surface was excellently smooth and flat. Crystal grains uniformly dotted at high density. We suppose that the successes are due to an efficient excitation of impinging clusters in the advanced ion-plating method employed.

SESSION O12/R7: JOINT SESSION ION BEAM SYNTHESIS OF NANOSTRUCTURES AND THIN LAYERS II

Chair: J. C. Soares
Wednesday Morning, November 29, 2000
Room 311 (Hynes)

8:30 AM *O12.1/R7.1

NANOCOMPOSITE MATERIALS FORMED BY ION IMPLANTATION: RECENT DEVELOPMENTS AND FUTURE OPPORTUNITIES. A. Meldrum, Univ of Alberta, Dept. of Physics, Edmonton, Alberta, CANADA.

Materials consisting of nanocrystalline precipitates embedded in a solid host have been utilized for over 2000 years for the formation of various types of colored glass. In "gold-ruby" glass, for example, colloidal precipitates of gold scatter light by the Mie process to produce a deep red color. More recently, materials of this type have been found to exhibit large third order optical nonlinearities. In the case of semiconductor nanoparticles, numerous novel electronic effects (i.e., blue shifting of the band gap and the presence of discrete transitions) have been found to occur. Chemical techniques in particular have been used to produce highly luminescent and monodispersed solutions of semiconductor nanoparticles, and new biological and electronic applications are being actively explored. The properties of these "nanomaterials" depend directly on the size of the particles, as well as on their crystal structure, shape, and orientation. By controlling these various parameters, it should be possible to tailor the electronic and magnetic properties of nanocomposite materials for specific applications. Ion implantation, in particular, is a highly versatile and flexible technique for forming many nanocrystal compositions embedded in a variety of selected host materials. The nanocrystals are protected from the environment and their electronic

and magnetic properties can be easily measured. By tailoring the microstructure of the particles (average size, orientation, crystal structure, etc.), it should then be able to control the electronic and magnetic properties of the system, and potentially, to develop these materials for use in new kinds of sensing or information technology devices. In the work completed to date, we have demonstrated that ion implantation is a viable and versatile technique for forming embedded nanoparticles of various compositions, and that the processing parameters can be modified to tailor the microstructural properties of the composite. Obtaining narrow size distributions remains a problem; however, in new experiments we are investigating means to obtain more monodispersed nanoparticles. The implantation method, its versatility, usefulness, and flexibility, as well as its inherent problems and possible solutions will be discussed.

9:00 AM O12.2/R7.2

ION BEAM ASSISTED NUCLEATION OF NANO-CRYSTALS. D. Ila, R.L. Zimmerman, and C. Muntele, Center for Irradiation of Materials, Alabama A&M University, Normal, AL; S. Schiestel, C.A. Carosella and G.K. Hubler, Naval Research Laboratory, Washington, DC; David B. Poker and Dale K. Hensley, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

There is a threshold concentration in an implanted sample, after which annealing will tend to cause spontaneous nucleation of nanoclusters. This has also been observed in samples prepared by ion beam assisted deposition. Similarly, there is a threshold implantation dose, after which some of the implanted species will spontaneously form nanoclusters. In our recent work, we have used the energy deposited due to the electronic excitation by post-implantation irradiation to induce the nucleation of nanoclusters. This process was used to reduce the threshold implantation dose by at least two orders of magnitude. In this presentation, we are applying a similar technique, post-irradiation electronic excitation, to films produced by low-energy, ion beam assisted deposition (IBAD). Gold and silica (co-deposited at various concentrations and temperatures) are grown in the presence of a 50 eV Ar ion beam, then post-irradiated. The resultant Au-nanocluster formation was observed and studied using optical spectroscopy, X-ray diffraction and TEM. Research sponsored by the Center for Irradiation of Materials, Alabama A&M University and the Division of Materials Sciences, U.S. Dept. of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

9:15 AM O12.3/R7.3

ION BEAM ENHANCED FORMATION AND LUMINESCENCE OF Si NANOCCLUSERS FROM α -SiO_x. Jung H. Shin, Se-Young Seo, and Johan Sun, Dept. of Physics, KAIST, KOREA; T.G. Kim and C.N. Whang, Dept. of Physics, Yonsei University, Seoul, KOREA; J.H. Song, Advanced Analysis Center, KIST, Seoul, KOREA.

Si nanocrystals, due to quantum confinement effects, possess optical and electrical characteristics which allow realization of Si-based devices with novel functionalities. A widely used method to prepare a dense and robust array of well-passivated Si nanocrystals is precipitating them out of SiO_x ($x < 2$). While this method has the advantage of being compatible with the standard Si processing technology, it often requires long anneals at temperatures in the excess of 1100°C, which is undesirable from the process point of view. The anneal temperature can be lowered by decreasing the value of x , but this often results in larger sized Si clusters. In this paper, we show that by ion-irradiating SiO_x films prior to anneal, both the cluster formation rate and the luminescence intensity can be greatly enhanced. SiO_x film with x of 0.58 was deposited by electron cyclotron resonance enhanced chemical vapor deposition of SiH₄ and O₂. Prior to anneal, some samples were implanted with 380 keV Si to a dose ranging from $5.74 \times 10^{15} \text{ cm}^{-2}$ to $5.74 \times 10^{15} \text{ cm}^{-2}$. All films were hydrogenated after anneals to passivate defects. Ion-irradiated films already display substantial luminescence in the 900 nm region after an anneal of only 1 min at 900°C, while virtually none can be detected from the non-irradiated film. Both films luminesce with a similar spectrum when annealed for 30 min at 1000°C, but the luminescence intensity from the ion-irradiated film is several times larger. Based on the effect of the irradiation dose and the ion specie, we rule out chemical effects due to the implanted ions, and propose that irradiation damage greatly accelerates nucleation of small Si clusters from the α -SiO_x matrix.

9:30 AM O12.4/R7.4

NANOSCALE LEAD-CADMIUM ALLOY INCLUSIONS IN SILICON. E. Johnson, A.S.B. Jensen, A. Johansen, V.S. Touboltsev, L.Sarholt, Niels Bohr Institute, University of Copenhagen, DENMARK; U. Dahmen, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA.

Cadmium rich nanoscale lead-cadmium alloy inclusions have been made in silicon by sequential ion implantation and subsequently

analyzed by transmission electron microscopy (TEM) and Rutherford backscattering/channelling analysis (RBS). To ensure crystallinity of the silicon matrix the implantations were carried out at 600°C. This is well above the melting point of both lead and cadmium, and the inclusions were therefore liquid during their formation and growth. The overall shape of the inclusions is cuboctahedral and they have a two-phase microstructure consisting of nearly pure segments of lead and cadmium attached along internal planar interfaces parallel to {001} or {011} in the silicon matrix. The lead segments tend to grow in parallel cube alignment with the silicon matrix while the orientation of the cadmium segments is varied. Due to shrinkage of the inclusions during initial cooling and solidification in the rigid silicon matrix, the alloy inclusions also contain voids. They are faceted both externally towards the silicon matrix and internally towards the metals. The voids tend to have a well-defined location with respect to the inclusion/matrix interface, and in this respect the voids can be considered as a *third phase* with a shape defined by surface energies rather than interface energies. In-situ TEM and RBS experiments show that melting of the inclusions takes place in a two-stage process. The first stage is due to eutectic melting at a temperature around 160°C leading to a curved liquid/excess Cd interface, and this is followed by melting of the excess cadmium at around 200°C. During heating the voids gradually shrink until they become invisible around 650°C. During the following cooling the voids reappear around 450°C and increase in size as the temperature is decreased. Solidification of the inclusions into a three-phase structure with cadmium, lead and a void phase takes place in a two-stage process, which is reversed in comparison with the melting.

SESSION O13/R8: JOINT SESSION
ION-SOLID INTERACTIONS FOR
OPTOELECTRONICS/PHOTONICS AND
MICROELECTRONIC MATERIALS
Chair: Chris Buchal
Wednesday Morning, November 29, 2000
Room 311 (Hynes)

10:15 AM *O13.1/R8.1

THE LIMITATIONS AND ADVANTAGES OF USING ION BEAM SYNTHESIS TO FABRICATE PLASMONIC DEVICES.

Mark L. Brongersma, John W. Hartman, Stefan A. Maier, and Harry A. Atwater, Thomas J. Watson Laboratory of Applied Physics, California Institute of Technology, Pasadena, CA.

Integrated optics appears to face the fundamental limitation that structures for guiding and modulation of light must have dimensions comparable to the wavelength of light. Recently however, it was theoretically shown that this problem can be circumvented by "plasmonics", i.e., transport of electromagnetic energy along linear chain-arrays of closely spaced 10-50 nm diameter metal nanoparticles. This transport relies on the coupled near-field electrodynamic interaction between metal particles that sets up coupled plasmon modes. Calculations in the point dipole approximation indicate strong guiding of electromagnetic radiation and electromagnetic dispersion relations are obtained. Coherent propagation with a group velocity exceeding 0.1 c is possible in straight arrays, around sharp corners (bending radius $<\lambda$), and structures of more complex architecture. The transport properties are dependent on the frequency and polarization direction of the coupled plasmon mode. Fabrication of these structures requires considerable control over particle size, particle shape, and interparticle spacing. The sensitivity of the transport properties to fluctuations in these parameters is quantified. Based on this we will discuss the limitations and advantages of using ion beam synthesis over other techniques to fabricate what could be the smallest structures with optical functionality.

10:45 AM *O13.2/R8.2

SYNTHESIS OF (InGa)(AsN) NANOSTRUCTURES BY ION IMPLANTATION. S. Clarke, X. Weng, S. Kumar, and R.S. Goldman, Dept of Materials Science and Engineering; V.H. Rotberg, Dept of Nuclear Engineering and Radiological Sciences; S. Krishna and P.K. Bhattacharya, Dept of Electrical Engineering and Computer Science; J. Sipowska and A. Francis, Dept of Chemistry; A. Daniel and R. Clarke, Dept of Physics, University of Michigan, Ann Arbor, MI.

Mixed anion nitride-arsenide compound semiconductor alloys and nanostructures are promising for light emitting devices operating throughout the near infrared range. However, due to the large N-As size difference, a limited miscibility of InGaAsN on the anion sublattice is predicted. To date, only a few percent nitrogen has been incorporated into mixed anion nitride-arsenide materials synthesized by conventional epitaxial methods. Furthermore, limited studies have been performed using ion implantation. Therefore, we have investigated the synthesis of InGaAsN by N ion implantation into GaAs and InAs, using a variety of implantation and rapid thermal

annealing conditions. We have characterized the nitrogen content, alloy segregation, and optical properties of the implanted structures using Rutherford backscattering spectrometry (RBS), four-circle x-ray diffraction (XRD), cross-sectional transmission electron microscopy (XTEM), and Photoluminescence (PL) spectroscopy. RBS suggests a small overall percentage of nitrogen incorporation into the implanted GaAs and InAs samples. On the other hand, XRD suggests the formation of high nitrogen content GaAsN and InAsN alloys. Since XRD is very sensitive to coherent crystallite structure, we infer that the alloyed regions are forming small nitrogen-rich clusters. This is confirmed by XTEM, which indicates the formation of precipitates with 10-20 nm diameters. Furthermore, photoluminescence spectroscopy suggests that the nitrogen implantation plus rapid thermal annealing has lowered the fundamental band gap, consistent with theoretical predictions of band gap bowing in this system. Detailed investigations of the microstructure using high resolution and analytical electron microscopy will be discussed.

11:15 AM O13.3/R8.3

SYNTHESIS OF III-N_x-V_{1-x} THIN FILMS BY N ION IMPLANTATION. K.M. Yu, W. Walukiewicz, W. Shan, J. Wu, J. Beeman, J.W. Ager III, E.E. Haller, Materials Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, CA; M.C. Ridgway, Department of Electronic Materials Engineering, Research School of Physical Sciences and Engineering, Australian National University, Canberra, AUSTRALIA.

We have successfully synthesized dilute III-N_x-V_{1-x} alloys (with $x=0.001-0.005$) by nitrogen implantation followed by appropriate post implantation annealing treatments. For GaAs, optical investigations show that the fundamental band-gap energy decreases with increasing N implantation dose in a manner similar to that commonly observed in GaN_xAs_{1-x} alloys grown by molecular beam epitaxy or metal organic chemical vapor deposition. Although no N loss can be detected by secondary ion mass spectrometry, the N incorporation in the substitutional As site decreases at higher annealing temperatures. As inferred from the magnitude of the band gap shift, typically only ~12% of the implanted N is incorporated in the As sublattice after rapid thermal annealing at 800°C. This relatively low N substitution is improved when a group III (Al and Ga) element is co-implanted with N in GaAs. The co-implantation is believed to enhance N substitution on the As sites by creating a group III-element rich region. Results on the synthesis of dilute nitrides of other III-V materials such as Al_yGa_{1-y}N_xAs_{1-x}, GaN_xP_{1-x}, InN_xP_{1-x}. Using N-implantation will also be discussed.

11:30 AM O13.4/R8.4

DOPING OF GALLIUM NITRIDE BY ION IMPLANTATION. E. Alves^{a,b}, C. Liu^{a,b}, A. Sequeira^a, N. Franco^a, M.F. da Silva^{a,b}, J.C. Soares^{a,b}, T. Monteiro^c, ^aInstituto Tecnológico e Nuclear, Sacavém, PORTUGAL; ^bUniversidade da Lisboa, CFN, Lisboa, PORTUGAL; ^cUniversidade da Aveiro, Dept. Física, Aveiro, PORTUGAL.

With the help of modern growth techniques, the quality of epitaxial GaN films continues to improve. The background electron concentration decreases to the order of 10¹⁶ cm⁻³. This makes it possible to dope GaN by ion implantation. In this work, we report the structural, electrical and optical properties of ion implanted GaN. Potential acceptors such as Ca, Fe and Er were used as dopants in this study. Ion implantation was carried out at substrate temperatures of 30 and 550°C, respectively. The structure of GaN films before and after the implantation was characterized by Rutherford backscattering/channeling combined with particle induced X-ray emission, high resolution X-ray diffraction and Mössbauer effect spectroscopy for the samples implanted with Fe. Using ion channeling the lattice site location of the dopants has been determined. The electrical and optical properties of the ion implanted GaN films have been studied by Hall effect and photoluminescence measurements. The as implanted GaN films exhibit an expanded lattice. After annealing at 1000°C, the lattice distortion does not fully recover. The Er-related luminescence near 1.54 μm is observed under below band gap excitation at liquid helium temperature. The spectra of the annealed samples consist of multiline structures with the sharpest lines found in GaN until now. Angular scans along both [0001] and [1011] directions show that the implanted dopants occupy mostly the lattice site of Ga atoms. For Er the combination of photoluminescence and ion channeling results allowed the identification of different sites for the optical active Er-related centers.

11:45 AM O13.5/R8.5

CORRELATION BETWEEN STRUCTURAL AND OPTICAL PROPERTIES OF Si NANOCRYSTALS IN SiO₂: MODEL FOR THE VISIBLE LIGHT EMISSION. Blas Garrido, Manel Lopez, Alejandro Perez-Rodriguez, Joan Ramon Morante, EME-Departament d'Electronica, Universitat de Barcelona, Barcelona, SPAIN; Caroline Bonafos, Alain Claverie, CEMES-CNRS, Toulouse, FRANCE.

It is at large admitted that finding the correlation between the structural (average nanocrystal size) and optical properties (band-gap energies and light emission) is among the key factors to understand the emission mechanism of Si nanocrystals in SiO₂. This has not been possible up to now mainly because of the great difficulty of imaging large populations of Si nanocrystals embedded in SiO₂. We have solved this problem by developing a method in which high resolution electron microscopy (HREM) is used in conjunction with conventional TEM in dark field conditions. So, size-distribution histograms, band-gap energies and photoluminescence (PL) emission of Si nanocrystals ion beam synthesized in SiO₂ have been measured by independent and direct methods and correlated between them. The results have allowed to experimentally determine for the first time the Stokes shift between absorption and emission as a function of crystallite size. The experimental band-gap versus size correlates exceptionally well with the most accurate theoretical predictions. Moreover, the PL dependence versus size is parallel to that of band-gap energy. Consequently, the experimental Stokes shift is independent of nanocrystal size and is found to be 0.26 ± 0.03 eV. This is almost exactly twice the energy of the Si-O vibration (0.134 eV). These results suggest that the dominant emission for SiO₂ capped Si nanocrystals is a fundamental transition spatially located at the Si-SiO₂ interface with the assistance of a local Si-O vibration.

SESSION O14/R9: JOINT SESSION
SEMICONDUCTORS AND ELECTRONIC
MATERIALS

Chairs: Ian M. Robertson and William J. Weber
Wednesday Afternoon, November 29, 2000
Room 311 (Hynes)

1:30 PM O14.1/R9.1

MEDIUM-RANGE-ORDER IN ION-IMPLANTED AMORPHOUS SILICON AND DAMAGE MODEL. J.-Y. Cheng, Univ of Illinois, Dept of Materials Science Engineering, Urbana, IL; Murray Gibson, Argonne National Lab, Materials Science Div, Argonne, IL.

We have measured medium-range order in ion-implanted amorphous silicon, based on fluctuation electron microscopy. Low-energy self-ion implantation leads to a highly topologically ordered paracrystalline state. Thermal annealing greatly reduces the order and leaves a random network. The free energy change previously observed on relaxation is therefore associated with randomization of the network. Energy spike model is employed to understand the origin of medium-range order in a damaged zone.

1:45 PM O14.2/R9.2

DAMAGE PROCESSES IN GaN UNDER ION BOMBARDMENT. S.O. Kucheyev, J.S. Williams, C. Jagadish, The Australian National University, Dept of Electronic Materials Engineering, Research School of Physical Sciences and Engineering, Canberra, AUSTRALIA; J. Zou, The University of Sydney, Electron Microscope Unit and Australian Key Center for Microscopy and Microanalysis, Sydney, AUSTRALIA; G. Li, LED Expert Corporation, Kaohsiung County, TAIWAN ROC.

The structural characteristics of wurtzite GaN films bombarded under a wide range of implant conditions (ion mass and energy, ion dose, implantation temperature, and beam flux) are studied by Rutherford backscattering/channeling (RBS/C) spectrometry, transmission electron microscopy (TEM), and atomic force microscopy (AFM). Results show that ion-generated defects in GaN exhibit efficient dynamic annealing even during bombardment at liquid nitrogen temperature. Damage build-up proceeds via the formation of point defect complexes and a band of planar defects. These somewhat stable planar defects are characteristic of ion bombarded GaN. At high doses, such defects appear to act as "nucleation sites" for amorphization, but the surface of GaN seems to be a more effective "nucleation site" for amorphization. Results suggest that amorphization of GaN can be stimulated by local material decomposition or a high concentration of implanted species. GaN films amorphized by ion bombardment are often porous. Elevated temperature ion bombardment under some implant conditions is complicated by very efficient material erosion.

2:00 PM O14.3/R9.3

DETERMINATION OF THE DISTRIBUTION OF ION IMPLANTED BORON IN SILICON. T.-S. Wang, A.G. Cullis, Sheffield Univ, Dept of Electronic and Electrical Engineering, Sheffield, UNITED KINGDOM; E.J.H. Collart, A.J. Murrell, Applied Materials, Thermal Processing and Implant Division, Horsham, UNITED KINGDOM; M.A. Foad, Applied Materials, Transistor Doping and Junction Division, Santa Clara, CA.

Ion implantation is the key doping process for semiconductor device

fabrication and the most important p-type dopant in Si is B. It is essential that, especially for low energy implantation, both as-implanted B distributions and those produced by annealing should be characterized in very great detail to obtain the required process control for advanced device applications. Secondary ion mass spectrometry (SIMS) is ordinarily employed for this purpose. However, in the present studies, implant concentration profiles have been determined by direct B imaging with approximately nanometer depth and lateral resolution using energy-filtered imaging in the transmission electron microscope. The as-implanted B impurity profile is correlated with theoretical expectations: differences with respect to the results of SIMS measurements are discussed. Changes in the B distribution and clustering that occur after annealing of the implanted layers are also described.

2:15 PM O14.4/R9.4

CRYSTALLIZATION OF ISOLATED AMORPHOUS ZONES IN SEMICONDUCTOR MATERIALS. E.P. Hollar, I.M. Robertson, University of Illinois, Dept of Materials Science and Engineering, Urbana, IL; Igor Jenčič, Jozef Stefan Institute, Ljubljana, SLOVENIA.

Crystallization of spatially isolated amorphous zones in Si, Ge, GaP, InP and GaAs was stimulated thermally and by irradiation with electrons and photons. The amorphous zones were created by a 50 keV Xe⁺ implantation. Significant thermal regrowth occurred at temperatures greater than 523 K, 473 K and 200 K in Si, Ge and GaAs, respectively. Electrons with energies between 25 and 300 keV stimulated crystallization in all materials at temperatures between 90 and 300 K. For electron energies above the displacement threshold, the crystallization rate decreased as the electron energy decreased. As the electron energy decreased below approximately 100 keV, the crystallization rate unexpectedly increased with further decrease of electron energy. The crystallization rate was independent of temperature for all electron irradiations. Irradiation with a 532 nm green laser (2.33 eV) caused crystallization in Si at a rate comparable to a thermal anneal at 523 K and caused minimal crystallization in GaP (E_g = 2.26 eV). The electron and photon irradiation results are consistent with the model that crystallization is controlled by defects (dangling bonds and kinks) created by electronic excitation at the amorphous-crystalline interface.

2:30 PM O14.5/R9.5

INVESTIGATION OF IRRADIATION DAMAGE IN SILICON DIOXIDE POLYMORPHS USING CATHODOLUMINESCENCE MICROANALYSIS. Marion A. Stevens Kalceff, Faculty of Science, University of Technology, Sydney, NSW, AUSTRALIA.

Cathodoluminescence (CL) is the luminescent emission from a material, which has been irradiated with electrons. CL Microanalysis (spectroscopy and microscopy) enables the microstructural processes induced by irradiation to be investigated as CL provides unique high sensitivity, high spatial resolution information about the defect structure and distribution of defects in wide band gap materials (i.e. materials with poor electron conductivity). CL microanalysis allows the in situ monitoring of electron irradiation induced damage as well as the post irradiation assessment of damage induced by other energetic irradiation. CL microanalysis complements the average defect structure information available from techniques such as photoluminescence (PL) and electron spin resonance spectroscopy (ESR). Electron beam irradiation of wide band gap materials can produce a trapped charge distribution which induces an electric field. The irradiation induced localized electric field can result in the electromigration of pre-existing and irradiation induced mobile charged defects. These processes result in the micro-segregation of positively and negatively charged mobile defect species within the irradiated volume of specimen. Silicon dioxide polymorphs are irradiation sensitive technologically important materials which are used in many advanced applications that operate in irradiation environments. The electron beam radiation sensitivity of various silicon dioxide polymorphs and related materials (e.g. pure crystal quartz, pure silicon dioxide glasses and borosilicate glass) has been investigated. CL evidence for the radiolytic production and micro-segregation of irradiation induced defects will be presented and compared. Radiolytic processes can result in the formation of stable defects via the non-radiative relaxation of excitons (i.e. decay of electronic excitations producing atomic displacements). Radiolytic processes can occur at beam energies less than that necessary for momentum transfer (i.e. knock-on) processes to occur. In particular, new evidence for the differing spatial segregation of radiolytic oxygen molecules in various electron irradiated silicon dioxide polymorphs will be presented.

3:15 PM *O14.6/R9.6

INVERSE OSTWALD RIPENING AND SELF-ORGANIZATION OF NANOCCLUSERS DUE TO ION IRRADIATION. K.-H. Heimig, B. Schmidt, FZ Rossendorf, Institute of Ion Beam Physics and Materials Research, Dresden, GERMANY; M. Strobel, CNR-IMEM,

Catania, ITALY; H. Bernas, CSNSM, CNRS-IN2P3, Orsay, FRANCE.

Under ion irradiation collisional mixing competes with phase separation if the irradiated solid consists of immiscible components. If a component is a chemical compound, there is another competition between the collisional forced chemical dissociation of the compound and its thermally activated re-formation. Especially at interfaces between immiscible components, these processes far from thermodynamical equilibrium may lead to unexpected phenomena. In this contribution it will be shown how nanoclusters embedded in a matrix and the chemical composition at an interface evolve under irradiation. The inverse coarsening of clusters found almost 30 years ago by studies of reactor materials, can now be understood. The mathematical treatment of the competition between irradiation-induced detachment of atoms from clusters and their thermally activated diffusion leads to the Gibbs-Thomson relation with modified parameters. A drastic consequence results from the negative capillary length, which will be shown to be the reason for inverse Ostwald ripening. This theoretical prediction has been proven by kinetic Monte Carlo simulations and experimental studies of Au clusters in SiO₂ irradiated by MeV ions. The second unexpected phenomenon to be addressed is self-organization of nanoclusters in a δ -layer parallel to the Si/SiO₂ interface which has been found when at the interface the damage level was in the order of 1...3 dpa. It will be shown that the origin of the δ -layer is the chemical reaction of Si at the interface with free oxygen coming from the collisional forced chemical dissociation of SiO₂. Thus, a sub-stoichiometric SiO_{2-x} layer forms at the interface. During subsequent annealing this layer provides nuclei for a nanocluster δ -layer, which is of interest for non-volatile memory application.

3:45 PM O14.7/R9.7

IMPACT OF BORON, GALLIUM AND OXYGEN ON DEFECTS PRODUCTION IN SILICON. Aurangzeb Khan, Nethaji Dharmarasu, Masafumi Yamaguchi, Kenji Araki, Tuong K. Vu, Toyota Technological Institute, Nagoya, JAPAN; Tatsuo Saga, Sharp Corporation, Nara, JAPAN; Takao Abe, Shin-Etsu Handotai Co., Ltd, Gunma, JAPAN; Osamu Annzawa and Sumio Matsuda, National Space Development Agency of Japan, Tsukuba, JAPAN.

Recently, it has been observed that light or carrier injection induced degradation of Czochralski grown silicon (Cz - Si) is due to the formation of boron - oxygen complex. An approach to avoid the deleterious effects of the boron - oxygen complex is to use different dopants, such as Ga, which shows no light degradation in Si Solar cells. In order to clarify the potential of Ga doped Si solar cells for space applications in comparison with B doped Si cells, we report the results of comparison of radiation induced defects (1 MeV electrons) in n⁺ - p - p⁺ Si solar cells and single crystals doped with gallium or boron ranging in the concentration from 8×10^{14} to 5×10^{16} cm⁻³, together with the impact of oxygen on radiation induced defects. The purpose of comparison is also to clarify the effects of different impurities on microstructural changes, which occur in Si during radiation. The interesting new feature of our results is that the gallium appears to strongly suppress the radiation induced defects, especially hole level E_v 0.36 eV, which is thought to act as a recombination center. Similarly the dominant electron level at E_c - 0.18 eV in B doped Si (which act as a donor) has not been observed in Ga doped CZ grown Si. Present study also compare the isochronal annealing recovery of the photovoltaic parameters of both gallium or boron doped n⁺ - p - p⁺ Si solar cells solar cells and single crystal after 1 MeV electron irradiation and is correlated with changes in the Deep Level Transient Spectroscopy (DLTS) defects spectra. New aspects of radiation induced defects in Cz - Si solar cells and single crystals leads to the broad understanding of the effects of boron, gallium and oxygen on defect production in electron irradiated Si. We will give a through account of our study at the conference, including our latest results.

4:00 PM O14.8/R9.8

POST ANNEALING STUDIES OF C₆₀ ION IMPLANTED THIN FILMS. Nethaji Dharmarasu, Kannan L. Narayanan, Nabuaki Kojima, Yoshio Ohshita and Masafumi Yamaguchi, Toyota Technological Institute, Semiconductor Lab, Nagoya, JAPAN.

Recently ion implantation in fullerene C₆₀ has attained an enormous interest for its opto-electronic device applications. We have carried out multiple energy boron ion implantation in C₆₀ thin films to various doses. Physical properties like electrical, structural and optical properties were studied. The C₆₀ films were prepared on quartz and silicon substrates at a substrate temperature of 150 degree celsius by molecular beam epitaxy (MBE) technique. The conductivity type of the implanted films is found to be p-type and the conductivity measurements reveal the dramatic increase in the conductivity with ion implantation. The optical gap is found to decrease due to the implantation and it could be attributed to the formation of defect levels and dopant (acceptor). FTIR results indicate the structural

transformation of C₆₀ to amorphous carbon phase during implantation. These results could be due to both ion implantation induced damage and doping of boron ions. To delineate the ion implantation induced damage from doping effect, the thermal annealing experiments were carried out. The implanted films subjected to thermal annealing indicate the removal of the defects caused during the implantation. Ion implantation-induced defects are found to annihilate with the annealing temperature. Electrical conductivity and optical gap are determined in the post implanted films. The observation of the systematic increase in the conductivity of the annealed films is due to the removal of the defects and the formation of defect free boron impurity acceptor. Their mechanism will be presented at the conference.

4:15 PM O14.9/R9.9

UNUSUAL CHANGE IN COLUMNAR DEFECT MORPHOLOGY IN YBCO UPON ANNEALING. Y. Yan, M. Kirk, Materials Science Division, Argonne National Laboratory, Argonne IL; A. Petrean and L. Paului, Department of Physics, Western Michigan University, Kalamazoo, MI.

It is well established that the columnar defects produced by GeV heavy ion irradiation of YBCO consist of amorphous material whose diameter can fluctuate significantly along the ion path under the appropriate conditions of ion mass and energy. Recently it has been shown that local oxygen reordering can occur adjacent to the columns of amorphous material forming an associated nanotwinned structure. In this presentation we show evidence from transmission electron microscopy of an unusual change in this defect morphology upon annealing to 600°C. The disappearance of the nanotwinned, but not the larger scaled thermal twinned, structure is found. A removal of the large fluctuations in the diameter of the amorphous column, preserving a narrow continuous column, is surprisingly discovered. Correlations with magnetisation measurements demonstrate the greater efficiency of vortex pinning at 77 K by the annealed defect structure. Work supported by U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Science under contract #W-31-109-ENG-38

4:30 PM O14.10/R9.10

ION-INDUCED AMORPHOUS TRACKS IN LAYERED STRUCTURES WITH VARIOUS TYPES OF CONDUCTION. György Szenes, Eötvös Univ, Dept of General Physics, Budapest, HUNGARY.

Amorphous tracks induced by high-energy monoatomic and cluster ions are analyzed in mica (insulator), Y-Ba-Cu-O, Bi-Sr-Ca-Cu-O (HTCSs) and in GeS and MoS₂ (semiconductors) applying the author's thermal spike model. These solids have a layered structure and a strong anisotropy of the transport properties except mica, which is an insulator in any direction. The ion beams were perpendicular to the layers. Good quantitative agreement was found between the experimental R_e- S_e data (R_e - effective track radius, S_e - electronic stopping power) and the predictions of the model. Two parameters: the efficiency g and the initial Gaussian width of the thermal spike a(0) were determined from the analysis. In mica the parameters of track formation are very close to that in other covalent insulating crystals, and 0.17S_e is transferred to the thermal spike (g=0.17). We conclude that the layered structure does not modify the track diameter in mica. In Y-Ba-Cu-O, Bi-Sr-Ca-Cu-O, GeS and MoS₂ only about 1/3 of the excitation energy is involved in the initial logarithmic stage of track evolution (g≈0.17/3) and the derivative $dR_e^2/dlnS_e = a^2(0) \approx 20.25 \text{ nm}^2$ is equal to that in insulators. The results are explained by the high anisotropy of the electron effective masses m* and by the formation of a nearly isotropic electron velocity distribution after numerous elastic collisions. As m* values are about 10-30 times higher for the wavevector component k_z normal to the layers, the energy transfer to the lattice is highly efficient and fast for about 1/3 of the excitation energy. In GeS a second stage of track evolution starts at S_e=19.4 keV/nm with a²(0)=130 nm² and g=0.17. The high efficiency indicates that like in insulators, all excited electrons are involved in the energy deposition in this stage. The high value of a(0) is in agreement with the expectation of a broad thermal spike in semiconductors.

4:45 PM O14.11/R9.11

ION IMPLANTATION GENERATED NANOVoids IN Si AND MgO MONITORED BY HIGH RESOLUTION POSITRON BEAM ANALYSIS. S.W.H. Eijit, C.V. Falub, A. van Veen, H. Schut, P.E. Mijnarends, M.A. van Huis and A.V. Fedorov, Delft University of Technology, Interfaculty Reactor Institute, Delft, NETHERLANDS.

The formation of nanovoids in Si(100) and MgO(100) by ³He ion implantation has been studied. Whereas the voids are generally almost spherical for Si, in MgO nearly perfectly rectangular nanosize voids are created. It is shown that the existence of a threshold dose

for cavity formation is related to a competition in vacancy trapping between the outer sample surface and the largest, most stable cavities. Recently, the 2D-ACAR setup at the Delft positron research centre has been coupled to the intense reactor-based variable-energy positron beam POSH. This allows a new method of monitoring thin layers containing nanovoids or defects by depth-selective high-resolution positron beam analysis. The 2D-ACAR spectra of Si with a buried layer of nanocavities reveal the presence of two additional components, the first related to para-positronium (p-Ps) formation in the nanovoids, and a second likely related to unsaturated Si-bonds at the voids' internal surface. The positronium is present in excited kinetic states with an average energy of 0.3 eV. Refilling of the cavities by means of low dose ^3He implantation ($1 \times 10^{14} \text{ cm}^{-2}$) and annealing reduces the formation of Ps and its ACAR-linewidth due to collisions of Ps with He atoms in the voids. The possibilities of this new, non-destructive method to monitor cavity sizes and the evolution of defect and void layers will be discussed.