SYMPOSIUM R
Microstructural Processes in Irradiated Materials

November 27 – 29, 2000

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*Invited paper
SESSION 11: IRRADIATED METALS  
Chairs: Gary S. Wad and Holmat Trinkaus  
Monday, Morning, November 27, 2000  
Room 310 (Hyne)  

8:30 AM R1.1  
MICROSTRUCTURAL CHANGES TO XENON NANOCLUSTERS IN ALUMINUM UNDER 1MEV ELECTRON IRRADIATION  
S.E. Donnelly, Joule Physics Laboratory, University of Salford, Manchester, UNITED KINGDOM; R.C. Birchler, C.W. Allen, Materials Science Division, Argonne National Laboratory, Ill.  
K. Furuya, M. Song, K. Masumi, National Research Institute for Metals, Tsukuba, JAPAN  

Inert gas implanted into metals generally form small nanometric sized clusters. The size and the shape of the inert gas implanted Xe, such nanostructures may form in the solid phase at temperatures as high as 600 K as a consequence of the high interface energy of such inclusions. In fcc metals, they are often observed to form as incommensurate, isotropic precipitates and to assume the cuboctahedral shape of matrix voids. This paper reports on recent work in which solid Xe nanostructures in Al have been subjected to 1 MeV electron irradiation in a high-voltage electron microscope. High-resolution images have been recorded on video tape in order to monitor the microstructural changes resulting from the electron irradiation. Inspection of the video recordings reveals that complex, rapid processes occur under the electron beam. These include the movement of small clusters, shape changes, coarsening, and the occasional apparent melting and resolidification of the Xe. Many of the changes to the nanostructures result from random alternations to the cluster shape due to interaction with the Frenkel defects created by the electron irradiation. The melting and resolidification behavior, however, is not yet fully understood and thought to result from complex interactions with extended defects in the aluminum.

9:00 AM R1.2  
THE EFFECT OF POINT DEFECT TRANSIENTS IN LOW-TEMPERATURE IRRADIATION EXPERIMENTS  
Roger E. Stiller, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN.  

The kinetic models used to simulate the response of materials to irradiation typically assume that the point defect concentrations are in equilibrium with the existing sink structure. This assumption is generally valid if (1) the response time of the point defect concentrations is much faster than the time scale over which the microstructure changes, and (2) point defect diffusion yields the quasi-steady-state point defect concentrations in a much shorter time than the duration of the experiment being simulated. For irradiation temperatures below about 250°C, the time required to obtain steady state point defect concentrations can become long enough to influence the irradiation response of the material. A point defect transient effect has previously been invoked to explain unexpectedly high creep rates observed in low-temperature irradiation experiments. The hydraulic tube irradiation facility in the High Flux Isotope Reactor (HFIR-HT) is being used to further explore the potential impact of the point defect transient effect. Irradiation times in the HFIR-HT can be precisely varied from seconds to days to achieve a range of doses at a relatively high dose rate. Small tensile samples of AISI 316 stainless steel and an ASME reactor pressure vessel steel are being irradiated in a series of experiments at temperatures between 60 and 300°C. Irradiations are being carried out to compare the effect of continuous irradiation for a fixed time with multiple, interrupted irradiation cycles that sum to the same total time. Differences in the damage accumulation under continuous and interrupted irradiation should appear when the total irradiation time is on the order of the time required for the vacancy concentration to reach steady state. The results of these experiments will be compared to model calculations, and the implications of the experiments for commercial reactor components will be discussed.

Research sponsored by the Division of Materials Sciences and Engineering and the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:15 AM R1.3  
MATRIX DAMAGE IN IRON. A. Nioul, M.L. Jenkins, Department of Materials, University of Oxford, Oxford, UNITED KINGDOM; M.A. Kirke, Materials Science Division, Argonne National Laboratory, Ill.

The matrix component of hardening of reactor steels under neutron-irradiation is due to the aggregation of irradiation-induced defects to form the exact shape of the target cluster. For a fcc lattice the answer is not clear, although suggestions have included interstitial and vacancy loops; kagome aggregates such as vacancy sponge; decorated microvoids; and solute-point defect clusters. In this paper we present some results of a study of matrix damage in pure iron supplied by BNFL Magnox Generation [‘Hawthorne’ alloys 1A and 2A-2A is the purer of the two], produced by neutron irradiation to a fluence of 5 x 10^20 n/cm^2 at 298°C. We have employed weak beam microscopy, which, with suitable modifications, has been shown capable of reliably identifying and sizing dislocation loops as small as 1-3nm. The matrix damage in both materials was found to consist of small [5-6 nm] dislocation loops. About 81% have Burgers vectors b = ca 100 >, and the remainder have b = n/a < 111 >. The loops in alloy 1A have a mean size d of 4.2 ± 0.3 nm, while those in 2A have a mean size of 4.5 ± 0.3 nm, while the number densities are about 8.5 x 10^10 m^-2 in alloy 1A, and 6.5 x 10^10 m^-2 in 2A. It can be seen that the loops can account for a large part but not all of the observed irradiation hardening. The loops are stable under thermal annealing to temperatures of at least 430°C. This and other indirect evidence suggests that their nature is interstitial.

9:30 AM R1.4  
INHOMOGENEOUS SWELLING NEAR GRAIN BOUNDARIES IN IRRADIATED MATERIALS: CAVITY NUCLEATION AND GROWTH SATURATION EFFECTS. S.L. Davidge, EURATOM/UKAEA Fusion Association, Culham Science Centre, Abingdon, Oxon, UNITED KINGDOM.

Inhomogeneous nucleation and growth of cavities observed near grain boundaries is an example of the failure of the standard rate theory to describe the kinetics of phase transformations in irradiated materials. Enhanced swelling observed near grain boundaries is believed to be associated with the competition between the diffusional growth of cavities and their destruction in collisions with mobile interstitial clusters. Swelling rates associated with the local cavity field are shown to be different in a radically different way as a function of the size of growing cavities. For a spatially homogeneous distribution of cavities this gives rise to the saturation of growth in the limit of large irradiation doses. The present work gives direct evidence for the growth of cavities in a material, and the effect on the kinetics of growth of cavities. The model describing the evolution of the population of cavities nucleating and growing near a grain boundary exhibits the formation of a characteristic profile of inhomogeneous swelling, where cavities situated in the vicinity of a grain boundary are able to reach substantially larger sizes than cavities growing in the interior region of the grain. The magnitude of swelling at maximum is found to be up to eight times higher than the value characterizing swelling in the grain interior. The dependence between the grain boundary and the maximum of the swelling profile is found to scale with the density of cavities N_0 = a^2 ~ N^β, where β is close to 1/3.

This work was funded by the UK Department of Trade and Industry and by EURATOM.

9:45 AM R1.5  
RADIATION-INDUCED PHASE INSTABILITIES AND THEIR EFFECTS ON HARDENING AND SOLUTE SEGREGATION IN PRECIPITATION-STRENGTHENED ALLOY 718. L.E. Thomas, B.P. Sencer and S.M. Bresnizer, Pacific Northwest National Laboratory, Richland, WA.

Radiation-induced displacement damage in metallic alloys can significantly modify the material microstructure and microchemistry, and hence severely degrade structural properties. An example of this behavior occurs in Fe- and Ni-base martensitic stainless steels, which are extensively used for structural components in commercial light-water reactors. Yield strength can increase by as much as five times under moderate dose (~5 dpa) irradiations. Over the same dose range, segregation of alloying and impurity elements prompts rapid changes in composition along grain boundaries. The combination of these effects can promote susceptibility to intergranular stress corrosion cracking (IGSCC) in reactor water environments. This paper will demonstrate how these detrimental changes can be altered by the presence of second-phase precipitates which are unstable under irradiation. Precipitation-hardened nickel-base alloy 718 is selected as an example where the strengthening phase is rapidly removed by irradiation. During neutron irradiation at 388°C, the Ni_3Nb γ/γ' particles present at γ' boundaries and dispersed within the matrix disappear after a few dpa. At higher doses, the γ' particles [present only in the matrix] also dissolve and precipitate. Matrix hardening is unaffected by disappearance of the γ/γ', but decreases as the original γ/γ' particles dissolve. Nano-probe compositional measurements with an analytical transmission electron microscope show that the softening coincides with compositional leaching near the particles rather than with the phase disappearances. Similar softening after initial hardening is observed in alloy 718 irradiated at low temperatures (~300°C) in a mixed spectrum of protons and spallation neutrons. In this case, both the γ/γ' and γ/γ' disappear after only 0.6 dpa. Delayed softening after the γ/γ' and γ/γ' disappearance is attributed to solute redistribution by further ballistic mixing. Since the primary radiation-induced changes are strength and segregation, mechanical properties and IGSCC resistance of an unstable alloy such as 718 may be improved during irradiation.
10:30 AM R1.6
EFFECT OF PERIODIC TEMPERATURE VARIATIONS ON THE MICROSTRUCTURE OF NEUTRON-IRRADIATED METALS.
S.J. Zinkle, N. Hashimoto and D.T. Hoelzer, Metall and Ceramics Div., Oak Ridge National Laboratory, Oak Ridge, TN.

Modest temperature excursions are expected to be a common occurrence in any commercial nuclear system due to scheduled startup and shutdown events. Some ion irradiation and low-dose neutron irradiation studies have found that these temperature excursions may exert a significant influence on the microstructural evolution, particularly if the temperature excursion coincides with the recovery Stage IV temperature range in different microstructures (e.g., dislocation annihilation of small vacancy clusters). In the present study, specimens of pure copper, CuCrZr, Type 316 stainless steel, 9Cr-3WV ferritic martensitic steel, and V-0Cr-4Ti were exposed to eight cycles of periodic temperature variations during neutron irradiation in the High Flux Isotope Reactor (HFIR) to a cumulative damage level of 4 to 5 displacements per atom. Specimens were exposed to a low temperature during the initial 10% of accrued dose in each of the eight cycles, and were exposed to a higher temperature during the remaining 90% of accrued dose in each cycle. Different specimens were exposed to low/high irradiation temperatures of 201/350°C and 300/500°C. The transmission electron microscopy results will be compared with the microstructure of companion specimens which were continuously maintained at 350°C and 500°C, respectively during the entire irradiation.

10:45 AM R1.7
IRRADIATION-INDUCED RECRYSTALLIZATION OF CELLULAR DISLOCATION NETWORKS IN URANIUM MOLYBDENUM ALLOYS.
J. Jonck and G.L. Holman, Argonne National Laboratory, Argonne, IL.

A rate-theory-based model is used to investigate the nucleation and growth of interstitial loops and cavities during low-temperature in-reactor irradiation of uranium-molybdenum alloys. Consideration of the dislocation structure takes into account the generation of forest dislocations and capture of interstitial dislocation loops. The theoretical description includes stress-induced glide of dislocation loops, direct production of interstitial loops/loops from the damage cascade, and accumulation of dislocations on cell walls. The loops accumulate and ultimately evolve into a low-energy cellular dislocation structure. The calculations indicate that nanometer-size bubbles are associated with the walls of the cellular dislocation structure. The accumulation of interstitial loops within the cells and of dislocations on the cell walls leads to increasing values for the rotation (misfit) of the cell wall into a subgrain boundary and the change in the lattice parameter as a function of dose. Subsequently, increasing values for the stored energy in the material are shown to be sufficient for the material to undergo recrystallization. The results of the calculations are compared with micrographs of the irradiated material.

11:00 AM R1.8
IDENTIFICATION OF DEFECTS IN FERRITIC/MARTENSITIC STEELS UNDER LOW DOSE IRradiation.
L. Kobeh, M. Victoria, Fusion Technology - Materials, Centre de Recherches en Physique des Plasmas, Ecole Polytechnique Federal de Lausanne, Villigen PSI, Switzerland.

The ferritic/martensitic steels which are candidates for the first wall of the future fusion reactor are investigated in TEM. While the irradiation doses expected in this reactor are in the range of 100 dpa per year, there is still a lack of knowledge on the nature of the irradiation induced defects for the low doses at which hardening is already occurring. This hardening depends strongly on the type of interaction between the moving dislocations and the defects. The early defects, which start to appear as black dots in TEM, are expected to be either three dimensional clusters of interstitials or vacancies, or dislocation loops. The nature and size of these defects are carefully studied in the FS2H and OPTIMAX steels for doses ranging from about 0 dpa and irradiation temperatures ranging from 310 K to 520 K. In this purpose, various weak beam techniques are explored at the limit of resolution of a TEM used in diffraction mode. Results are presented here.

11:15 AM R1.9
SPALLATION RADIATION DAMAGE AS DETERMINED BY CALCULATIONS AND IRRADIATION EXPERIMENT ON SNS TARGET MATERIAL.
K.L. MacFarlane, T. S. Bristow, K. Parrell, J.D. Hunn, E.H. Lee, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; M.S. Wechsler, Department of Nuclear Engineering, North Carolina State University, Raleigh, NC.

The irradiation responses of materials in the targets of high power spallation neutron sources are qualitatively similar to behavior in fusion and fusion reactors, yet different in detail and severity. Higher generation rates of the transmission gases He and H caused by the GOX vars are part of the underlying basis of these differences. For comparison, neutrons in fusion and fusion reactors are below about 14 MeV. There are other differences as well. For example, in facilities utilizing heavy liquid metal targets, some of the high mass atoms adjacent to the structural alloy container react with the particle flux to become energetic knock-ons injected into the target container material, causing additional damage and changes in local alloy composition. High mass transmission products are generally not produced at higher target mass energy of fusion or fusion reactors. As part of the materials R&D program for the Spallation Neutron Source (SNS), calculations and several series of irradiations (which counter-environments are underway to determine the irradiation response. New calculations are described that address displacement, He, and solid transmutation production, as well as the additional effects caused by energetic target material knockons. Irradiation experiments and results are discussed in reference to 1) the Spallation source LANSCE (800 MeV) at Los Alamos National Laboratory, and SINQ (600 MeV) at the Paul Scherrer Institute in Switzerland, and 2) the Triple Ion Facility at ORNL (4 MeV), which allows separate or simultaneous irradiation of specimens with a He (displacement-producing) beam and with He and He beams to simulate some aspects of spallation radiation effects. Available results on the effects on microstructures and properties will be described.

Research sponsored by the Division of Materials Sciences, U.S. Dept. of Energy under contract DE-AC05-000R2275 with UT-Battelle, LLC.

11:30 AM R1.10
IRRADIATION MECHANISMS IN MARTENSITIC STEEL IRRADIATED IN HFIR.

A reduced-activation martensitic steel, 9Cr-3WVT, developed for fusion energy applications, was irradiated in 300 and 500°C at 5 dpa in the High Flux Isotope Reactor (HFIR). Large changes in yield strength, deformation mode, and strain hardening capacity were seen, with the magnitude of the changes dependent on irradiation temperature. The microstructural changes in mechanical properties are determined by the microstructure resulting from irradiation. In order to better determine the contributions of different microstructural features to strength and to deformation mode, transmission electron microscopy (TEM) specimens were prepared from the gauge sections of the tested (strained) flat tensile specimens and examined. Additional TEM specimens were prepared from tensile specimens irradiated at 500°C and tested at 25°C. The effect of irradiation (irradiation and test) and strain rate on the deformation microstructure will be presented. This research is sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-000R2275 with Lockheed Martin Energy Research Corp. and the Japan Atomic Energy Research Institute. This research was supported in part by an appointment to the Oak Ridge National Laboratory Postdoctoral Research Associates Program administered jointly by the Oak Ridge National Laboratory and the Oak Ridge Institute for Science and Education.

11:45 AM R1.11
Abstract Withdrawn.

SESSION R2: Austenitic Stainless Steels
Chair: Michael L. Jenkins and Robert G. Odette
Monday, November 27, 2000
Room 310 (Hyen)

1:30 PM R2.1
GRAIN BOUNDARY MODIFICATION DURING NEUTRON IRRADIATION AT INTERMEDIATE TEMPERATURES.
S.M. Brauemer, D.J. Edwards and E.P. Simonen, Pacific Northwest National Laboratory, Richland, WA.

Grain boundary compositions and near-boundary microstructures have been measured in complex Fe-Cr-Ni alloys after neutron irradiation at critical intermediate temperatures where transitions occur in defect microstructures. Radiation-induced segregation (RIS) and dislocation loop microstructures have been determined as a function of irradiation dose up to 15 dpa and at temperatures ranging from 275 to 340°C. The most significant effect on RIS was the grain boundary composition enrichment of Cr and Mo along with impurity elements before irradiation. This initial segregation was difficult to remove during subsequent irradiation and retarded the development of Cr and Mo-depleted regions. The predominant microstructural feature produced at irradiation temperatures below ~325°C was
failed interstitial loops. A distinct grain boundary damped zone was observed at low dose that disappeared at higher doses. Interesting differences were noted in the 30-40 neutron irradiation, but these differences were not directly related to BISO behavior. Radiation-induced grain boundary changes will be discussed in relation to the current understanding of irradiation-assisted SCC in light-water-reactor core components. Support was provided by the Office of Basic Energy Sciences and the Office of Nuclear Energy, U.S. Department of Energy under contract DE-AC06-76RL01830 with Battelle Memorial Institute and by EPRI.

2:00 PM R2.2 IN SITU MEASUREMENTS OF NEAR-SURFACE C and REDISTRIBUTION DURING IRRADIATION. E.L. Rege, P.M. Baldwin, E. J. Funk and A. W. McCormick, Materials Science Division, Argonne National Laboratory, Argonne, IL.

Because it has been strongly linked to Irradiation-Assisted Stress Corrosion Cracking (IASC, literally hundreds of measurements of C redistribution near grain boundaries following irradiation have previously been made using both Auger depth-profiling and analytical electron microscopy. Unfortunately, these postmortem techniques have failed to clarify the extent of C redistribution at irradiation temperatures of interest to operating nuclear power reactors, i.e., <350°C. Furthermore, questions also remain as to the importance of thermal and displacement-cascade processes in creating frequently observed W-shaped C profiles. To redress this knowledge gap, we are developing an in-situ electron-beam analysis technique for monitoring C redistribution in steels during irradiation. A resonant proton capture reaction (p,γ), with approximately a 1-μm depth resolution, is used to detect changes in the higher energy part of the C profile. Irradiation profiles obtained during irradiation at relevant temperatures, and as a result of various thermal treatments, will be presented. The roles of various radiation-enhanced and -induced processes in creating the W-shaped profiles will also be discussed. This work is supported by DOE Office of Science under Contract W-31-109Eng-38.

2:15 PM R2.3 DEFORMATION MECHANISMS AND MICROSTRUCTURES OF IRRADIATED AUSTENITIC STAINLESS STEELS. E.H. Lee, T.S. Ryan, J.D. Hinn, L.K. Meaurl, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN.

Radiation-induced degradation of mechanical properties has been the focus of attention for the steel components to be used in fusion and fusion reactors and high energy accelerators. Although the deformation mechanisms have been studied for various in-reactor or accelerator irradiated materials in the past, the understanding of detailed mechanisms is still lacking. In this work, therefore, a comprehensive study was carried out to investigate the nature of deformation microstructures for iron irradiated AISI 316LN austenitic stainless steel by employing a recently developed disk bend test method and a transmission electron microscopy technique. Irradiation was conducted to 200°C with 3 MeV He and/or 3.5 MeV Fe ions, and the irradiated specimens were strained to about 10% at room temperature by the disk-bend method. The results showed that deformation microstructures were characterized by extensive pileup dislocations on glide planes, microtwins, stacking fault fringes, and defect-reduced channel bands. These microstructures were qualitatively similar to those produced by uniaxial tensile tests. Analyses revealed that virtually all glide dislocations were Shockley partial dislocations in nature. Juggled and stacking partials were produced when the leading Shockley partial interacted with a loop, and the trailing partial was segregated from the leading partial by pileup and stacking locking. Deformation-induced microtwins and stacking fault fringes were a consequence of stacking violation between the leading and trailing partials. Defect-reduced channels were produced when Frank loops were unbalanced by interacting with isolated or glide Shockley partials and removed from the channel bands by prismatic glide. Experimental evidence and analyses indicated that the interaction between a loop and a glide dislocation occurred by a two-step reaction, first with the leading partial and then with the trailing partial. This is different from the traditional view that the interaction occurs by a one-step reaction with a perfect dislocation or with an isolated Shockley partial. Research supported by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy, under contract No. DE-AC05-00OR22725 with UT-Battelle, LLC.

2:30 PM R2.4 RADIATION-INDUCED SEGREGATION IN 316 AND 304 STAINLESS STEEL IRRADIATED AT LOW DOSE RATE. T.R. Allen, J.I. Cole, Argonne National Laboratory, West, Idaho Falls, ID. T.O. Hill, Oak Ridge National Laboratory, Oak Ridge Power Laboratory, Tokyo, JAPAN; E.A. Kenik, Oak Ridge National Laboratory, Oak Ridge, TN.

As part of the shutdown of the EBRII reactor, structural materials were retrieved to analyze the effects of long-term irradiation on mechanical properties. In this work, the effect of low dose rate irradiation (167 to 198 dpa/s) on grain boundary composition in 316 and 304 stainless steel is analyzed. Samples were taken from reflector subassemblies irradiated in the reflector region of EBRII irradiated at temperatures from 373-390°C to doses up to 10 dpa. The effect of dose, dose rate, and bulk composition on radiation-induced segregation are analyzed. In 304 stainless steel, decreasing the dose rate increases the amount of grain boundary segregation. For a dose of 20 dpa, chromium depletion and nickel enrichment are greater in 304 stainless steel than in 316 stainless steel. In 316 stainless steel, changes in grain boundary Cr and Ni concentrations occur faster than changes in iron and molybdenum concentration. In both 304 and 316 stainless steels, the presence of grain precipitate significantly changes the grain boundary compositions in the vicinity of the precipitate.

2:45 PM R2.5 THE INFLUENCE OF SUBMICROSCOPIC VACANCY CLUSTERS DURING NEUTRON IRRADIATION OF STAINLESS STEEL NEAR 350°C. E.P. Simonson, J.I. Boland, and R.F. Bruemmer, Pacific Northwest National Laboratory, Richland, WA.

Microstructural evolution necessarily requires account of equal partitioning of vacancies and interstitials into preferred components of the microstructure. At temperatures below 350°C, the documented microstructure is dominated by interstitial loops having sizes greater than 1 nm. Vacancy aggregates or annihilation mechanisms must also exist to satisfy the required equal account of interstitial components of displacement damage. Molecular dynamics simulations demonstrate that both interstitial and vacancy clusters can form directly within damage cascades. The clusters are distinct in terms of their stability and mobility but cannot be characterized.

Comparison of microstructural evolution near grain boundaries with evolution in the grain interior indicates vacancy processes affected by a nearly dominant vacancy sink. Interstitial loop damped zones are observed near irradiated grain boundaries. The initial zone width is several tens of nm but disappears after a few dpa of irradiation. The interstitial loop development is not enhanced near the demarcated zone edge. This implies that the range of radiation damage is not changing and that the partitioning of defects is a local scale of less than tens of nm. Post-irradiation annealing offers access to indirectly observe consequences of having submicroscopic vacancy sources in the matrix. During annealing, the interstitial loop size distribution responds to the competing influences of coarsening (transfer of vacancies from large interstitial loops to small interstitial loops) and dissolution (transfer of vacancies from vacancy sinks to interstitial loop sinks). Comparison of calculated changes in the interstitial loop size distributions with measured changes reveal the contribution from submicroscopic vacancy sources. This work supported by the Materials Sciences Branch, BES, U.S. Department of Energy, under Contract DE-AC06-76RL01830.

3:30 PM R2.6 MICROSTRUCTURAL EVOLUTION IN CHARGED PARTICLE IRRADIATED 316 SS MODIFIED TO THE MODIFIED RADIATION DAMAGE. J. Gou, E.P. Simonson, G.W. Warr, and R.F. Bruemmer; Pacific Northwest National Laboratory, Richland, WA.

Microstructural evolution in austenitic stainless steels plays an important role in irradiation-assisted stress corrosion cracking (IASC). The objective of this work is to investigate the effect of the addition of mixed elements in both size and mass on the evolution of irradiated microstructure in 316 SS. Alloys were modified by the addition of Pt and Hf to suppress the radiation damage. Pt and Hf were added (~1 wt%) as a hard perturbation to analyze defect recombination within the early stage of cascade formation and defect migration. Irradiations were performed with 5 MeV Ni ions at 500°C and dose rates from 4.7E-4 to 1.4E-3 dpa/s to doses up to 50 dpa or with 3.2 MeV protons at 10 dpa at 300°C. Microstructures were characterized using transmission electron microscopy. While little evidence was seen for Pt addition, Pt appears to have a microstructural response to the irradiation, with lower number density and smaller size for Pt loops. However, the presence of precipitates (Hf and Ni enriched) in 316 Hf which were formed during heat treatment may also play an important role in the microstructural evolution. This work supported by the Office of Nuclear Energy, Science & Technology, U.S. Department of Energy, under contract DE-AC05-76RL01830.

3:45 PM R2.7 NATURE OF THE FINE-SCALE DEFECTS AND RADIATION HARDENING IN STAINLESS STEELS NEUTRON-IRRADIATED AT TRIGA. Center towards, Ed Simonen and Steve Bruemmer, Pacific Northwest National Laboratory, Richland, WA.
While considerable data exists in the literature regarding radiation effects in stainless steels at temperatures pertinent to fast reactor-type environments, the lack of reactor-type environments more appropriate for light water reactors near 575K remains rather sparse by comparison. The microstructure under these conditions has been loosely described in terms of Frank loops (assumed to be faulted interstitial loops) and “black spots” (assumed to be vacancy loops). The latter are typically considered to be 4-5 nm or less in size. These “black spots” have been reported to be vacancy in nature, and some studies suggest that they are in fact vacancy-type stacking fault tetrahedra (SFT). The formation of radiation-induced void swelling in stainless steel and related high density microstructures has been of interest and red, dark-field techniques have been used to examine the defects present in the irradiated microstructure as a function of dose. The dominant defects, or most visible defects are faulted interstitial loops whose size extends down to less than 1 nm. The density of these loops is established at low doses, typically up to 13 dpa, and then the size of these loops breaks down and shifts to larger average sizes as the dose increases. Other defects such as \textit{SFTs} have been found in lower numbers. The presence of these fine-scale defects will be discussed in relation to the terminology of “black spot” damage described in the literature, as well as related to measured changes in yield strength. This work supported by the Materials Sciences Branch, BES, U.S. Department of Energy, under Contract DE-AC05-76OR27292 with UT-Battelle, LLC, and through the SHiRe Program under contract DE-AC05-76OR00003 with Oak Ridge Associated Universities.

4:00 P.M. R2.8

\textbf{ISOLATION OF RADIATION-INDUCED SEGREGATION IN PROTON-IRRADIATED AUSTENITIC STAINLESS STEELS \textit{via} AUGER ELECTRON SPECTROSCOPY}}

J.T. Bandy and G.S. Was, Department of Nuclear Engineering and Radiological Sciences, University of Michigan, Ann Arbor, M. E.A. Kenik, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN.

Microstructural effects (radiation hardening) and microcompositional effects (radiation-induced segregation \textit{RIS}) have both been identified as potential contributors to irradiation-assisted stress corrosion cracking (IASC). However, the role of either in IASC is unclear. Model simulations of post-irradiation annealing indicate that the microstructural features such as dislocation loops are removed faster than RIS. Simulations also predict that there exist time-temperature combinations that will significantly reduce the dislocation loop population while leaving the grain boundary segregation essentially unaffected. Samples of high purity and commercial purity stainless steels have been irradiated with 3.2 MeV protons at 300°C to 1.9 dpa and then annealed at temperatures between 500°C and 650°C for times varying between 45 minutes and 5 hours. Hardness, RIS and dislocation densities were measured before and after annealing. The hardness and dislocation densities decreased with increasing annealing time and temperature while the amount of RIS did not change significantly from the pre-irradiated condition for anneals up to 600°C for 90 minutes. Annealing at 650°C for 45 minutes removed the irradiation-induced hardening and dislocation population entirely, while only 33% of the irradiated RIS remained. However, annealing at 600°C for 90 minutes completely removed the microstructural changes while leaving 85% of the RIS. The rate of removal of both dislocation loop population is dependent on the time-temperature combinations, which is in agreement with model simulations and experimental data from similar studies. The separation of microstructural and microchemical effects after annealing at 600°C for 90 minutes may permit the isolation of the role of RIS in IASC. This work was supported by the U.S. Department of Energy under grant DE-FG07-99ID13788. Research at the ORNL SHiRe User Facility is partially supported by the Division of Materials Sciences and Engineering, U.S. Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC, and through the SHiRe Program under contract DE-AC05-76OR00003 with Oak Ridge Associated Universities.

4:15 P.M. R2.11

\textbf{IMPLICATION OF DISORDER-INDUCED POLYMORPHOUS MELTING TO INTERGRANULAR EMBRITTLEMENT IN THE NICKEL-SULFUR SYSTEM}}

J.K. Hester, P.R. Okamoto, N.Q. Luan, J.F. Stubbins; Materials Science Division, Argonne National Laboratory, Argonne, IL.

In a combination of Anger electron spectroscopy, slow-strain-rate tensile tests, ion-implantation, and Rutherford-backscattering spectroscopy studies, grain-boundary sulfur concentrations in a dilute Ni-S alloy were systematically varied by time-controlled annealing of specimens at 625°C. Critical sulfur concentration for 50% intergranular fracture was found to be 15.5 ± 3.4 at. % S. This is within experimental error of the sulfur concentration of 14.2 ± 3.3 at. % S found to induce 50% amorphization of single crystal nickel during implantation at liquid nitrogen temperature. This suggests that amorphous-induced intergranular embrittlement, like implantation-induced amorphization, may be a disorder-induced polymorphic melting process, albeit one that occurs locally at grain boundaries. Since the Ni-S phase diagram indicates that the polymorphic melting temperature for the primary solid solution drops rapidly to zero as the alloy composition approaches 18 at. % S, this indicates that the Ni-18 at. % S composition should melt the matrix under these conditions. The same composition below the glass transition temperature. The polymorphic melting surface of the Ni-S system provides a similar understanding of the synergetic effect of hydrogen in reducing the critical sulfur concentration for 50\% intergranular fracture. A kinetic model for segregation-induced intergranular embrittlement consistent with the main results of this study is introduced.

4:30 P.M. R2.10

\textbf{ISOLATION OF THE EFFECT OF MICROSTRUCTURE ON IASC IN PROTON-IRRADIATED AUSTENITIC STAINLESS STEELS}}

B.R. Gramling, J.T. Bandy, and G.S. Was, Department of Nuclear Engineering and Radiological Sciences, University of Michigan, Ann Arbor, E.A. Kenik, Metals and Ceramics Division, Oak Ridge National Laboratory.

Irradiation affects both the microstructure and microchemistry of materials. In order to better understand the mechanism of irradiated-assisted stress corrosion cracking (IASC), it is necessary to separate microstructural effects (radiation hardening) from microchemistry effects (radiation-induced segregation). Under typical light water reactor (LWR) neutron-irradiation conditions \textit{(Te} = 288°C\textit{)}, the microstructure is dominated by radiated dislocation loops which result in hardening and an increase in yield strength. A commercial purity 304SS, known to be susceptible to IASC under these conditions, was irradiated with 3.2 MeV protons at 50°C to a dose of 0.3 dpa. The low-temperature irradiation suppressed vacancy motion and FEG-STEM measurements showed no RIS. The microstructure was dominated by “black dots”, and a small population of faulted loops was present. Microhardness testing revealed an un irradiated hardness about 40% higher than that caused by irradiation under LWR conditions to 1.0 dpa, an IASC susceptible condition. Post-irradiation annealing was performed in a vacuum furnace for temperatures ranging from 350-500°C for times ranging from 0.5 to 36.0 hours to evolve the loop microstructure. Constant extension rate tests in normal (BWR) water chemistry at 288°C were conducted to evaluate IASC susceptibility. The results of microstructure characterization, hardness measurements and IASC susceptibility will be discussed with regard to the role of microstructure in IASC. This work was supported by the U.S. Department of Energy under grant #DE-FG02-99ID13788. Research at the ORNL SHiRe User Facility is partially supported by the Division of Materials Sciences and Engineering, U.S. Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC, and through the SHiRe Program under contract DE-AC05-76OR00003 with Oak Ridge Associated Universities.
R3.1 HIGH ENERGY PROTON IRRADIATION OF PURE TITANIUM
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During the past ten years, a number of investigations of radiation effects have been performed on fer and pure metals in the frame of international fusion programs, showing significant differences in the defect accumulation rate and the dose dependence of the hardening between these two structures [see for instance J. Nuclear Materials 270 (2000) 114]. In order to complement such investigations, a study of the mechanical behavior and microstructural changes of irradiated hip pure titanium has been undertaken. Tensile flat specimens of 8 mm in gauge length and 0.3 mm in thickness have been prepared from polycrystalline thin foils of pure titanium (99.999%) and annealed at 1023 K for 5 hours. Series of specimens were irradiated in the PHEX (Proton Irradiation Experiment) facility, at the Paul Scherrer Institute, with 580 MeV protons. These irradiations were performed under ambient (300-320 K) and 523 K to doses ranging between $10^{-3}$ and 1 dpa. The damage rate was approximately $10^{-2}$ dpa/s$^{-1}$. The annealed and irradiated tensile specimen were deformed at ambient temperature up to fracture. The defects associated with deformation and/or irradiation were investigated in transmission electron microscopy (TEM) forming the bright/dark field and weak beam techniques. After proton-irradiation to 0.1 dpa, a number of "white dot" contrasts have been observed in weak beam TEM images, that reflect the irradiation-induced formation of defect clusters. About 20% of the defect clusters have been identified as dislocation loops with an apparent mean size of 5 nm. A detailed description of the tensile mechanical behavior and defect microstructure of pure Titanium as a function of the irradiation dose is presented and the possible irradiation hardening mechanisms are discussed in terms of the corresponding hip structure.


Enormous progress in computational materials science capability, improved resolution of materials science characterization tools and the emergence of techniques for simulating experimental observations based on computer codes has occurred over the past several years and makes the direct comparison between modeling and experiment possible. As part of a collaboration among DENIM, LLNL, CIEMAT and EPFL, a custom irradiation experiment in α-Fe has been performed to test the theory and experimental validation. High purity, single-crystal samples have been irradiated with 150 keV Fe ions and characterized with both transmission electron microscopy (TEM) and post-implantation spectroscopy (PAS). Concurrently, a multiscale modeling approach involving ab-initio electronic structure calculations, molecular dynamics and kinetic Monte Carlo simulations has been used to model damage accumulation for the same irradiation conditions. The modeling predictions are used as input to simulate positron lifetimes and TEM images, for direct comparison with the experimental observations. In this work, we present results of the predicted damage accumulation and compare to the experimental observations. Small, 3-D vacancy clusters rapidly form, which are well below the TEM resolution limit, but produce a significant increase in the measured positron lifetime spectrum. However, the growth of trapped interstitial clusters leads to an observed TEM visible defects at high dose. This work is performed under the auspices of the U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48 and within the VENUS project (CNS/JUNESA Coordinated Program) under contract P07058402.

R3.3 Abstract Withdrawn.

R3.4 EFFECT OF HIGH FLUENCES ON THE EMBRITTLEMENT OF LOW COPPER VVER 440 SURVEILLANCE SAMPLES.
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An atom probe tomography microstructural characterization has been performed on low (0.06 at. %) copper surveillance samples from a VVER-440 reactor that were examined in order to evaluate the susceptibility of these pressure vessel steels to embrittlement during service and plant life extension. The 15K/3M/PAIA base and 110K/3MPT weld specimens were each examined in dry conditions unirradiated, thermally aged (10 years, respectively at 295°C) and neutron irradiated at 275°C to fluences of 1 x 10²¹ m⁻² and 5 x 10²¹ m⁻². E > 0.5 MeV, respectively. Results from Charge Yen-Note tests, and the measured ultimate and yield strengths were similar for both unirradiated and thermally aged materials. However after neutron irradiation, significant shifts (> 100°C) in the ductiles-to-brittle transition temperatures and increases in the yield and ultimate tensile strengths were found in both the base and weld materials. Three-dimensional atom probe tomography compositional measurements revealed enrichments of manganese, silicon, phosphorus, copper and carbon at dislocations in neutron irradiated materials. The solute distribution around the dislocation was not uniform and the silicon and manganese were located in different regions of the dislocation. Some spherical features were also observed which could arise from solute segregation to small dislocation loops or other small defects, such as vacancy clusters or nanocrystals. Some of the spherical features may initially form by the clustering or precipitation of copper atoms into embryos which then attract or act as a sink for the other solutes or vacancies. These solute-enriched regions can significantly impede the motion of dislocations and thereby account for the observed changes in the mechanical properties at high fluences. Research at the Oak Ridge National Laboratory SHARE User Facility was sponsored by the Division of Materials Science, Office of Science, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under inter-agency agreement DOE 1986-N065-3W with the U.S. Department of Energy.

R3.5 THE INFLUENCE OF PKA DIRECTION ON DISPLACEMENT CASCADE EVOLUTION. R.G. Sterley, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN.

An extensive database of atomic displacement cascades in iron has been developed using molecular dynamics simulations. More than 300 cascades have been completed at 100K at energies between 100 eV and 1000 keV, with fewer simulations at 600 and 900K. A systematic evaluation of the database has revealed an unexpected influence of PKA direction in simulations up to about 2 keV, with a maximum effect observed at 300 eV. A high degree of planar "channeling" was observed in cascades initiated in the [111] direction. Similar behavior was observed in [112] cascades, but to a lesser degree. Simulations that mimic this behavior have been developed and show that the survival and higher fractions of the point defects are confined in clusters. The implications of this sensitivity to PKA direction are discussed with respect to a statistical analysis of the cascade database.

R3.6 MICROSTRUCTURAL EVOLUTION OF ISOTOPICALLY TAILORED FERRITIC/MARTENSITIC STEELS IRRADIATED IN HFR. N. Wahabzadeh, R.L. Khach, J.P. Robertson and A.F. Rowcliffe, Oak Ridge National Laboratory, Oak Ridge, TN.

Two ferritic/martensitic steels based on 12Cr-1MoVW and 9Cr-3WVT were doped with $^{60}$Ni to generate helium via thermal neutron reactions during irradiation in the High Flux Isotope Reactor (HFR). Control specimens doped with $^{60}$Ni, which does not lead to helium productions, were also prepared. TEM examinations showed that before irradiations the length of $^{60}$Ni doped is more narrow and dislocation density tends to be higher compared with undoped alloys. Precipitates in the alloys were mainly $M_2\text{C}_3$ carbides. The number density of irradiation-induced carbides in the $^{60}$Ni doped alloy was higher than those in the undoped alloys due to the higher concentration of helium, however, swelling in each alloy was < 0.01% because cavity sizes were < 10 nm. Irradiation-induced $\langle n_0 \rangle < 100$ and $\langle n_0/2 \rangle < 111$ type dislocation loops were observed in all alloys, the number density and the mean type of $\langle n_0/2 \rangle$ loops tends to be higher and larger than that of $\langle n_0 \rangle < 111$ type loops. Also, there was an tendency for the number density of loops in the $^{60}$Ni doped alloys to be higher than in undoped alloys. Irradiation-induced precipitates were observed in all alloys, which were identified as $M_2\text{C}(n)$ type carbide, $\alpha$' and $M_2\text{X}$ phase. The $M_2\text{C}(n)$ type carbides and $\alpha'$ phase were formed along dislocation loops. This precipitation behavior could be involved in the radiation-induced changes in the mechanical properties of ferritic/martensitic steels. This research was sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy.
of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

p3.7 CAVITY FORMATION IN REDUCED ACTIVATION FERRITIC/MARTENSITIC STEELS IRADIATED WITH Fe AND He IONS. Akira Nakao, Takeshi Shindo, Hiroshi Hirono, Takeshi Tanigawa, Shiro Jitsukawa, Japan Atomic Energy Research Institute, Dept. of Material Science, Ibaraki, JAPAN; Akira Koyama, Kyoto Univ., Institute of Advanced Energy, Kyoto, JAPAN.

Reduced activation ferritic/martensitic steels have been considered as the potential material for the first wall of blanket (FWB) structures. Their microstructural evolution under neutron irradiation is mainly driven by the displacement damage and is also affected by the co-generation He. In this study, alloys of FS8H (Fe-8Cr, 2W-0.2V-0.04Zr), JLF-1 (Fe-8Cr, 2W-0.2V-0.08Ti) and HT9 (Fe-12Cr, 1Mo) were ion-irradiated in JRR-3M reactor with an ion flux of 1.2 MeV He+ which were irradiated at temperatures ranging from 673 to 773 K. Displacement damage level and implanted helium level were 60 dpa and 1200 ppm at peaks located at the depth of 1800 nm, respectively. A focused ion beam (FIB) processing system, FB-2008A attached with a micro sampling system was employed to prepare transmission electron microscopy (TEM) foils from irradiated specimens. No cavities were observed in specimens irradiated without He ions. On the other hand, cavities were observed in laths or the damage peak in specimens irradiated with dual ions. The swelling behavior and its temperature dependence are different in these steels. FS8H showed the highest swelling resistance, and the largest swelling, 0.2 %. No significant increase in helium density was observed in HT9. The helium density in this condition reaches 20 nm. The largest swelling, 0.8 %, was observed in JLF-1 irradiated at 728 K, which contained large cavities up to 50 nm.

p3.8 POINT DEFECTS OBSERVED IN DT-NEUTRON IRADIATED COPPER, SILVER AND GOLD AT 15°C WITH A ROTATING TARGET IN FNS JAERI. Y. Shimomura, K. Sajo, H. Okabe, I. Mikouda, Faculty of Engineering, Hiroshima University, JAPAN; C. Kutsukake, H. Takeuchi, Department of Fusion Engineering Research, JAERI, JAPAN.

We built the temperature-controlled irradiation chamber for a 14 MeV DT-neutron irradiation with a rotating target of fusion neutron source facility in FNS JAERI, Tokai, Japan. The rotating target of DT neutron generator has 25 cm in diameter, which is smaller than previous HTNSH in LLNL. We carried out two irradiation experiments in 1999. In these two experiments, specimen temperature was controlled within ±1°C during irradiation at 15°C. In these two irradiation experiments, the fluence was varied between 1 x 10^19 to 1 x 10^20 n/cm². Therefore the present experiment is complementary to the previous HTNSH irradiation in which majority of experiments were carried out in the fluence range between 5 x 10^14 to 1 x 10^17 n/cm². By TEM observation, we observed both of interstitial cluster and vacancy cluster in Au, Ag and Cu. Significant results obtained is related on the movement of vacancy clusters at room temperature. We will present how we identify the nature of these small point defects clusters. In 2000, four irradiation experiments are scheduled. Two are high temperature irradiation at 800°C and two are temperature-controlled irradiation at 50°C and 100°C.

p3.9 RADIATION EFFECTS ON THE PLASTICITY AND MICROSTRUCTURE OF Ti-ALV ALLOYS CONTAINING β PHASE. T. Sawaj E. Waki, A. Hishinuma, Japan Atomic Energy Research Institute, Tokai-mura, JAPAN; M. Tabuchi, Hirokote National College of Technology, Hakodate, JAPAN.

Ti-Al-V intermetallic alloys containing more than 10% V have been developed for high temperature and nuclear applications. β phase (ordered bcc structure) introduced by the V addition to the conventional binary Ti-Al system improves the ductility of these alloys. Elongation of these alloys increases as the test temperature increases, especially in Ti-Al-V alloy. An electron microscopy of Ti-Al-V alloy shows a microstructure mostly consisting of β phase and its elongation sharply increases to 60% from 1% around 830 K. Transmission electron microscope TEM observation revealed a characteristic microstructure of annealed tensile specimen which showed high elongation. Many deformation bands were observed in deformed β grains. These bands consist of hexagonal α2 phase grains and β phase grains of two different orientations. The orientation relationship of hexagonal α2 phase and β phase mura in deformation bands is (101)[110] / [0001][201]. This suggests the instability of β phase and martensitic transformation during deformation. Ti-30Al-1-V alloy has a microstructure containing three phases; α2, β and γ. For tensile elongation above 90% at 673 K and 873 K. Tensile specimens were neutron irradiated in Japan Research Reactor No. 3 Modified [JRR-3M] at 673 K and 873 K to the fluence of 3.5x10^25 [n/cm^2]. The specimens irradiated at 673 K was tensile tested at the same temperature, and showed almost no ductility. Specimens irradiated at 873 K were tested at the same temperature, and the elongation was about 10%. Remarkable ductility loss due to neutron irradiation is detected. Specimens irradiated at 673 K was also tested at 873 K and the elongation was only 6%, which is less than that of 873 K irradiated specimens. This also suggests the instability of β phase and phase decomposition during irradiation.

p3.10 THE DEFECT MICROSTRUCTURE OF V-4Cr-4Ti ALLOY FOLLOWING NEUTRON IRRADIATION AT 323°C. D.T. Hoelder, S.J. Zinkle, and A.F. Rowcliffe, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN.

Vanadium alloys with compositions near V-4Cr-4Ti (wt %) are candidate materials for structural applications in magnetic fusion experimental reactors due to their attractive combination of good thermal conductivity and mechanical strength and ductile-to-brittle transition temperature (DBTT) in the unirradiated condition. However, studies have shown that the tensile and impact properties of the V-4Cr-4Ti alloy are severely degraded following neutron irradiation at doses <20 dpa and temperatures <400°C. A microstructural investigation using transmission electron microscopy (TEM) was performed on the V-4Cr-4Ti alloy following neutron irradiation to a dose of ~19 dpa at a temperature of ~323°C in the Li-6B1.5O1.5 Fusion-1 experiment in order to determine the defect structures that formed under these irradiation conditions and to correlate them with the observed property changes. The tensile deformation irradiation damage was consistent with the accompanying reduction in strain hardening capacity and occurred in the irradiated samples. The TEM analysis showed that these property changes occurred with the concomitant formation of defect structures that comprised of dislocation loops and fibrillated precipitates in the irradiated thin foil samples. Results concerning the defect type, size and number density were obtained and their relation to the tensile data will be presented. Research sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-96OR2275 with U.T.-Batelle, LLC.

p3.11 DYNAMIC BEHAVIOR OF VOIDS IN NEUTRON-IRRADIATED COPPER AND NICKEL AT ELEVATED TEMPERATURE. I. Mikouda and Y. Shimomura, Faculty of Engineering, Hiroshima University, JAPAN.

We reported previously an experimental result which shows that voids can move in neutron-irradiated copper at elevated temperature [1]. To study the detailed behavior of voids, we carried out the annealing experiments and in-situ observation in neutron-irradiated copper and nickel. Neutron irradiation was carried out in the temperature controlled capsule at KUR (Kyoto University Reactor). Neutron irradiation was performed at 53K to damage level between 1 x 10^17 to 1 x 10^18 n/cm². Annealing temperature was 520K for 10, 20, 30 min sequentially for neutron-irradiated copper, after annealing TEM observation was carried out at room temperature. Experimental results show that void moved along [110] direction. Voids were moved during 337K annealing but not changed 37K and 8K voids were moved, the others vanished during annealing. The images of in-situ observation, void contrast images were recorded on VTR tape and analyzed by frame. At room temperature observation voids were static in the white circle, however at 573K in-situ observation contrast were changed like an oval and sometimes contrast was disappear. After 7 sec contrast was white circle and moved slightly and similar phenomena were observed quite frequently at 523K. It is concluded that void moved with dynamical structural relaxation at elevated temperature.


Changes in bulk composition are known to affect both radiation-induced segregation and microstructural development, including void swelling in austenitic stainless steels. Judicious choices of material or impurity element concentrations can be used to reduce grain boundary chronic defects that result in sensitivity to environmental cracking. Variations in composition can also produce materials with increased resistance to void swelling. In this work, select compositions have been added to an Fe-18Cr-8Ni alloy (bulk composition corresponding to 304 stainless steel) to identify the effect of composition on microstructure and swelling. Fe-18Cr-40Ni is analyzed to study the effect of bulk diffusivity.
increases, Fe-16Cr-13Ni to isolate the effect of major elements in improving both swelling resistance and intergranular cracking resistance in zirconium steel, and to determine the effect of an oversized element on enhancing point defect recombination. Following irradiation with high energy protons, the microstructure of irradiated samples is investigated to determine the void size distribution and dislocation microstructure. The swelling calculated from the void distribution is compared to the swelling in the Fe-18Cr-8Ni alloy to determine the effect of composition change on void swelling. For each of the alloys, grain boundary composition is also measured to evaluate the effect of grain boundary segregation and to relate microstructural changes to swelling resistance.

R3.13 RADIATION HARDENING OF VANADIUM ALLOYS.
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Vanadium alloys have been considered as a structural material for fusion reactor applications, because of their promising properties such as low induced radioactivity, high-heat-load capability and good compatibility with liquid lithium as a coolant material. The alloy development has been conducted from a viewpoint of radiation behaviors including low temperature ductility loss and high temperature helium embrittlement. Radiation hardening is an important phenomenon not only for alloy development, but also fundamental point of view. The radiation hardening can be considered in two categories, that is, resistance against starting to move dislocations and resistance against continuing to move dislocations. The hardening depends on temperature, strain rate and microstructure that may not be observable by transmission electron microscopy. Recent results indicated that level of gaseous impurities in the alloy could affect radiation hardening of the alloy very much after irradiation because gaseous impurities could form G知el atmosphere and cause large resistance to start moving dislocations. The radiation defects could be strengthened by the impurities against moving dislocations or Grown type obstacle. The microstructures depend on the concentration of the impurities, since the impurities could affect nucleation and growth of the radiation defects. In this paper, increasing in yield stress, changing of work-hardening and microstructures in vanadium alloys after irradiation are summarized. Radiation hardening and the role of the gaseous impurities are discussed from the points mentioned above.

R3.14 A NEW APPROACH TO THE USE OF ELECTRICAL RESISTIVITY IN CHARACTERIZING NANOSTRUCTURAL EVOLUTION UNDER IRRADIATION.
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A number of complementary techniques are required to characterize the evolution of nanoscale features under irradiation. These typically include small angle neutron scattering (SANS) and annular bright-field TEM (ABF-TEM) scattering, a range of high resolution and analytical electron microscopy (TEM) methods, atom probe tomography (APT), positron annihilation (PA), post-irradiation annealing (PIA) and mechanical property measurements. More recently the use of thermal electron power (TEP) has been used to track irradiation induced changes, primarily associated with solute redistribution. However, with some notable exceptions, in recent years electrical resistivity (ER) has not been used extensively to study radiation damage in general, and irradiated pressure vessel steel nanostructures in particular. Similar to TEP, ER can be used to provide information on solute redistribution and precipitation. For example, in simple binary Fe-Cu systems ER can be used along with techniques such as SANS, ABF-TEM and APT to achieve self-consistent mass balances of copper partitioned between precipitate and matrix solution sites. In alloys with more than one solute species, standard ER measurements generally cannot define which solutes have precipitated. However, based on differences in the temperature dependence of the resistivity coefficients of various elements, ER measurements over a range of temperatures can track the partitioning of solutes throughout the temperature range. Measurements also help address issues associated with details of the relation between solute distributions and resistivity, such as those associated with small clusters. Further, in principal, non-linear temperature dependence of resistivity coefficients, resistivity measurements in magnetic fields and use of temperature dependent TEP measurements can extend partitioning measurements to additional elements. Examples of use of ER on both irradiated and thermally aged Fe-Cu and Fe-Cu-Mn alloys are presented. Implications to nanostructures that form under irradiation are discussed.

R3.15 EFFECT OF ANNEALING AND RE-IRRADIATION ON THE COPPER-ENRICHED PRECIPITATES IN A NEUTRON-IRRADIATED PRESSURE VESSEL STEEL WELD. MK. Miller, K.F. Russell, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; P. Frey, Groupe de Physique des Matériaux, Université de Morbihan, France.

An atom probe tomography study has been performed to estimate the matrix composition, and the composition and number density of the ultrafine copper-enriched precipitates that formed under neutron irradiation in a submerged weld from the HSSI fifth irradiation series (Weld 72W). The composition of this weld is Fe.0.57%, Cu.1.51%, Mn.0.27%, Ni.0.54%, Sr.0.002% W.0.05% C.0.0009% P. and 0.0009% S. The material was examined after a typical stress relief treatment of 40h at 660°C (U), after neutron irradiation to a fluence of 1.9x10{19} m^-2 (E > 1 MeV) [U] at a temperature of 288°C, after irradiation and thermal annealing for 168 h at 545°C [I], and after irradiation, thermal annealing and re-irradiation to an additional fluence of 0.8x10{20} m^-2 (E > 1 MeV) [IAR]. A high number density of ultrafine copper-enriched precipitates were found to form on irradiation. These precipitates were found to coarsen and decrease in number density after the annealing treatment at 545°C. These precipitates were present after re-irradiation and some additional nanoscale diameter copper-enriched precipitates were also observed. Research at the Oak Ridge National Laboratory Shale User Facility was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Batelle, LLC and by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N065-3W with the U.S. Department of Energy.

R3.16 PROTON IRRADIATION OF ZEOLITE-Y.
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Irradiation effects on zeolite-NaY have been investigated by irradiation with a 5.0 MeV He^+ beam. Zeolite-NaY suffers structural damage under proton irradiation. The crystal-to-amorphous transition occurs at a total dose equivalent to an ionizing energy deposition of 3x10{16} Gy and a displacement dose of 0.01 dpa. The ion exchange capacity of the irradiated zeolite-NaY with 10 mCi CaCl2 solution varies with the extent of the damage. After 25 hours of exchange, the Ca concentration in the amorphous region is ~0.8 wt. % which is much lower than in the undamaged region (~20 wt. %). This result confirms that radiation-induced amorphization can cause a significant loss of ion exchange capacity. The data also suggest that the radiation damage by proton radiation in zeolite-NaY is dominated by both ionizing and displacement processes. Mechanisms related to the radiation-induced damage in the ion exchange capacity are discussed.

R3.17 ELECTRICAL IRRADIATION OF BRANNERITE-TYPE.
CERAMICS, Jie Ling, Luming Wang, Univ of Michigan, Dept of Nuclear Engineering and Radiological Sciences, Ann Arbor, MI; Greg R. Lumpkin, Australian Nuclear Science and Technology Organization, Materials Division, Menai, AUSTRALIA; Rodney C. Ewing, Univ of Michigan, Dept of Nuclear Engineering and Radiological Sciences, Ann Arbor, MI.

Brannerite, ideally UTi2O6, occurs in polynyle ceramics that are considered for plutonium immobilization. In order to investigate radiation effects caused by α of Pu, several brannerite compositions were synthesized: UTi2O6, ThTi2O6 and a polynyle material, which is composed of C Succession brannerite and pyrochlore. A 1 MeV Kr² irradiation was performed using IVE-TEM tandem facility at Argonne National Laboratory. The transformation from the crystalline to the amorphous state was observed in situ in a function of dose. At room temperature, complete amorphization of UTi2O6 and ThTi2O6 occurred at 1.5 ± 0.06 ± 0.10 × 10{4} ions/Å², respectively. The critical amorphization dose (Dc) increases with irradiation temperature. Complete amorphization of UTi2O6 did not occur at 973K, and the critical amorphization temperature for ThTi2O6 irradiated by 1 MeV Kr² is ~950 K. C Succession brannerite is more easily amorphized than the pyrochlore phase, and the critical amorphization dose at room temperature is 1.5 ± 0.06 ± 0.10 × 10{4} ions/Å². The radiation resistance to amorphization at the critical amorphization temperature decreases as follows: Dc(UTi2O6) > Dc (C Succession brannerite) > Dc (ThTi2O6). The effects of composition and structure on radiation damage in brannerite and pyrochlore type ceramics are discussed.

R3.18 TOPOLOGICAL AND DYNAMICS MODELING OF IRRADIATION-MORPHEDI BETA-SiC. Xingdong Yuan, M.MIT, Dept of Materials Science & Engineering, Cambridge, MA; Liao Hua, MIT, Lab for Computer Science, Cambridge, MA; Lin W.
An efficient computer simulation method has been developed for modeling irradiation-induced amorphization of beta-SiC by combining topological modeling and molecular dynamics (MD) simulation. Modeled structures have been compared to those derived from MD simulation only. An approach to the concept of antisite and chemical disorder in amorphous structures has been formulated and the roles played by Frankel pairs and antisite defects in the crystalline-to-amorphous transformation has been explored. The surrounding crystalline structure plays an important role in the recovery of amorphized structures, which can evolve to quite topologically ordered but still chemically disordered. A fully recovered crystalline structure was not observed.

Several studies have shown that SWAP ions with electronic stopping powers >15 keV/nm can induce phase transformations in oxide ceramics and other materials. In contrast, we have recently found that track formation did not occur in SiC, Si₃N₄ or AlN following 330 MeV Kr ion irradiation [1]. These three materials have been irradiated with 710 MeV Bi ions at fluences of 1x10²⁹ ions/cm². These results are consistent with the experimental observations. The results show that the dominant defects are single interstitials and mono- vacancies at low dose, whereas the amorphous or disordered clusters, which consist of interstitials and antisite defects, appear at intermediate dose levels. These local disordered regions play an important role in the amorphization of SiC. At higher dose, a significant proportion of antisite defects is created during continued cascade overlap, and the number of antisite defects is considerable with that of interstitials. The increase in interstitials and antisite defects with increasing dose suggests that the driving force for the crystalline-to-amorphous transition under ion irradiation in SiC is due to the accumulation of both Frankel pairs and antisite defects. The present results are discussed in terms of experimental observations and compared with models for irradiation-induced amorphization.

In the case of alloys, the process may drive the target either towards or away from equilibrium microstructures and phases. Part of the complexity comes from the competition between opposite effects: the enhanced atomic mobility due to the increased defect concentration affects the kinetics of precipitation or ordering which in turn is oppositely affected by disordering and precipitate dissolution produced by mixing. In this work we focus in particular in thermomigration and solidification by performing computer simulations of melt spikes and cascades in several systems. We determine the influence of the different regimes in the thermal phase of a collision cascade, showing that thermomigration may or may not find complex rules and that for some materials solute motion may be significant.

R3.22
COMPUTER SIMULATION OF PRIMARY DAMAGE STATES IN SiC. R. Devanathan, Indian Institute of Technology Madras, Department of Metallurgical Engineering, Chennai, INDIA; F. Gao, W.J. Weber, Pacific Northwest National Laboratory, Richland, WA.

We have performed a comprehensive molecular dynamics simulation of the primary damage states in 3C-SiC using a modified Tersoff potential. The simulations examined damage produced by Si and C primary knock-on atoms (PKAs) corresponding to recoils with energies in the range of 0.25-1.50 keV. The study also generated statistics of defect production by simulating a number of PKAs at each energy.

The results indicate that the defect production efficiency decreases with increasing PKA energy as observed previously in metals. However, the cascade lifetime is very short (less than 1 ps), localized modeling does not occur, and PKA energies are comparable to those of recoils. The cascade lifetime, and the tendency for defects to form clusters is much less compared to the case of metals. C Frankel pairs are more numerous than Si Frankel pairs and antisite defects are part of the primary damage. The number of antisite defects increases for PKA energies below 0.75 keV and increases with increasing PKA energy. These results offer insights into the primary damage states in SiC and statistics of defect production in cascades. Furthermore, the damage efficiency determined as a function of PKA energy has accurate displacement functions for both Si and C PKAs.

R3.23
ATOMIC MECHANISM OF NUCLEATION AND GROWTH OF Voids IN Cu STUDIED BY COMPUTER SIMULATION. Y. Shimomura and I. Mukoudji, Faculty of Engineering, Hiroshima University, JAPAN.

We reported previously [1] an experimental result which shows that a vacancy cluster whose size in smaller than 300 vacancies relays to stacking fault tetrahedron and greater than 400 vacancies relays to a void in neutron-irradiated copper at 300°C. To study the detailed atomistic mechanism of nucleation and growth of void and void in copper, we carried out the computer simulation of molecular dynamics. MD simulation was carried out with an empirical interatomic potential of embedded-atom method (EAM) expression [2]. A model crystal is composed of 32000 Cu atoms. A crystal was relaxed at first to a thermal equilibrium at 1000K by running MD. The various numbers of vacancies was introduced in crystal as dispersed small clusters and the crystal was continued to relax by running MD. It was found that a vacancy cluster whose size is smaller than 150 vacancies relays to a void and that greater than 500 vacancies relays to a void at high temperature as 1000K. We present which is the basic mechanism to lead to void and void formation.


R3.24

The role of interatomic potentials on the primary damage has been investigated by Molecular Dynamics simulations of displacement cascades with three different interatomic potentials dedicated to α-Fe. The primary damage caused by the neutron interaction with the matter has been found to be potential sensitive. We have investigated the equilibrium potentials of the cascade as well as the "short distance interactions" which appear to have a strong influence on the cascade morphology and defects distribution at the end of the cascade. The static properties as well as the dynamical (thermal) properties have been considered; the kinetic potential energy transfers during the collisions have also been studied.

R3.25
MONTE CARLO SIMULATIONS OF CASCADES IN Fe-Alloys. C. Domain, J.C. Van Duyzen, EDE-DIRD, Département de Matériaux, Institut du Loge, FRANCE; C.S. Bequart.
 DAMAGE CONDITIONS. Alexander V. Barashov, Stanislav I. Goloborov, The Univ of Liverpool, Dept of Engineering, Liverpool, UNITED KINGDOM.

The theory of void swelling in metals under heavy ion or neutron irradiation is generalized by involving the dependence of the collision frequency between voids and interstitially mobile clusters of self-interstitial atoms on spatial correlation between voids. It is noticed that the void lattice types observed correspond to the minimal collision frequency, a fact that has analogies in other dynamical systems for fluid-phase transitions (e.g., liquid-gas transition in pedestrian crowds, size segregation in sheared granular media). This was a driving force for employing the conventional approach developed for the description of self-organization in such cases to the problem of void lattice formation. Assuming that the mechanism for the void lattice formation is the movement of the centre of mass of the voids due to interaction with the mobile defects, the critical void radius above which the voids form a lattice is derived. The role of vacancies may play in establishing perfect lattices is discussed.

R3 29
STATISTICAL ANALYSIS OF DEFECT PRODUCTION DUE TO 50KEV CASCADES IN BCC-Fe. Nakki Sood, Central Research Institute of Electric Power Industry (CRIEPI), Materials Science Dept, Tokyo, JAPAN; Shigeru Ishino, Tokai Univ, Dept of Nuclear Engineering, Kanagawa, JAPAN; Tomas Diaz de la Rubia, Lawrence Livermore National Laboratory, Chemistry and Materials Science Directorate, Livermore, CA.

Understanding of defect production in 50keV-PAK energy cascade events is very important because the average PAK energy in iron due to fast neutron bombardment in fission reactors is about 50keV. Molecular dynamics computer simulation technique is a very powerful tool to study the cascade event, but we cannot reach a conclusion by looking at only small number of simulation results because there is large diversity in the final defect formation especially in high energy cascade results. In this study, we perform about 100 cases of MD simulations of 50keV cascade events to get better statistics to understand general trend of defect production. What we found is that we can classify the defect production into three or four groups, each of which has different tendency in defect production.

R3 30
MOBILITY OF INTERSTITIAL CLUSTERS IN HCP ZIRCONIUM. N. de Diego, Dpto. de Fisica de Materiales, Facultad de Ciencias Fisicas, Universidad Complutense, Ciudad Universitaria, Madrid, SPAIN; Yu. N. Osetsky and D.J. Bacon, Materials Science and Engineering, Department of Engineering, University of Liverpool, Liverpool, UNITED KINGDOM.

In recent years a significant attention has been given to clusters of self-interstitial atoms (SIAs) formed in displacement cascades in metals irradiated with energetic particles. The main reason for this is that recognition of the importance of SIAs to the interplay between self-structure evolution under irradiation. The successful application of the production bias model (PBM) and it’s further development in explaining of many features of radiation-induced microstructures such as homogeneous damage near grain boundaries, decoration of dislocations by interstitial dislocation loops, rafting of dislocation loops, void lattice formation, etc., has initiated extensive studies of the properties of SIA clusters in different lattices. So far these studies were concentrated on cubic lattices, e.g., FCC and BCC, whereas fundamental knowledge on structure and mobility of defect clusters and dislocation loops has been obtained. However, many of the microstructure peculiarities noted above have also been observed in irradiated hcp metals like Zr and Ti, which suggests that the properties of defect clusters and dislocation loops in hcp metals may be similar to those in cubic ones. In this work we present the first results of an atomistic study of SIA clusters in an hcp crystal. We have studied clusters of 4 to 20 SIAs over a wide temperature range using molecular dynamics and a many-body Finnis-Sinclair type interatomic potential for Zr.

The results show a qualitative similarity of the dynamic properties of clusters to those for cubic lattices. Particularly, all clusters larger than 4 SIAs exhibit thermally activated one-dimensional glide in [1120] directions. Smaller clusters (≤ 4 SIAs) exhibit behaviour peculiar to the hcp structure, however, for they can migrate two-dimensionally in the basal plane. The clusters jump frequency, activation energy and correlation factors have been estimated and difference and comparison drawn between the behaviour of SIA clusters in different structures.

R3 31
EFFECT OF ANNEALING AND MICROWAVE HYDROGEN PLASMA TREATMENT ON STRUCTURAL AND ELECTRONIC PROPERTIES OF ION IRRADIATED DIAMOND FILMS. Alexander Laidstein and Alan Hoffman, Department of Chemistry, Technion - Israel Institute of Technology, Hant, ISRAEL.

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In the present study we correlate between the secondary electron emission (SEE) properties of variously treated Xe⁺ ion damaged diamond films and the microstructures of the near surface region, as identified by near edge X-ray absorption fine structure (NEXAFS) spectroscopy and x-ray photoelectron spectroscopy. The 50 keV Xe⁺ ion bombardment of hydrogenated polycrystalline diamond films to a dose of 2 x 10¹⁴ ions/cm² results in the destruction of the surface diamond to sp² bonded amorphous carbon, increased oxygen adsorption, shift of the electron affinity from negative to positive, and strong degradation of its electron emission properties, although such changes in SEE are not determined by NEXAFS, and the concentration of oxygen remains relatively high. Subsequent annealing to 610°C produces oxygen free diamond film and somewhat increases SEE. Annealing to 900°C results in the complete recovery of the surface positive electron affinity surface leading to drastic degradation of the electron emission properties. Prolonged, up to 3 hours MW hydrogen plasma treatment of the implanted diamond films gradually improves the crystal quality and results in further increase of SEE intensity. This treatment does not moreover, however, substantially reduces the concentration of oxygen in the previously damaged diamond, indicating its bulk diffusion during ion bombardment.

### P.3.32
**INFLUENCE OF PRELIMINARY THERMORADIATION TREATMENT UPON EFFICIENCY OF FORMATION AND AMPLIFICATION OF EFFECTS IN SIMON DOTS IN CHALCOGENIDE SEMICONDUCTORS IRRADIATED BY GAMMA-RAYS**
Sh. Mshakham, N.A. Tursunov, M. Ashurov, Z.M. Khakimov, Institute of Nuclear Physics of Uzbek Academy of Sciences, Tashkent, UZBEKISTAN.

In this paper an efficiency of formation and annealing of radiation defects (RD) was studied by deep level transient spectroscopy (DLTS) method in silicon p-n-structures normalized by thermoradiation treatment, which were subjected to gamma-ray irradiation. To normalize an equilibrium parameters of silicon p-n-structures prepared from single p-type silicon with resistivity of 0.3-1.0 Ωcm on the neutron irradiation with fluences of 10¹⁰ and 10¹⁵ cm⁻² followed annealing at 700°C was carried out. It was shown that under certain conditions the initial equipartition of defects in silicon and characteristics of p-n-structures remains practically unchanged. It was revealed that the efficiency of RD formation in the normalized silicon structures was 2.29 times higher than in the non-normalized samples. So to thermoradiation, the normalization leads to shifting of annealing temperature toward high temperatures. The change of efficiency of formation and annealing temperature of RD is explained by interactions of point defects induced by gamma rays with disordered regions created by preliminary neutron irradiation and a mechanism of these interactions is discussed.

### P.3.33
**PHOTO STRUCTURAL REORGANIZATION IN HYDROGENATED AMORPHOUS SILICON (a-Si:H) LAYERS UNDER THE ACTION CONCENTRATED SOLAR RADIATION OF HIGH DENSITY**
Rustam R. Qakabov, The Physical-Technical Institute of Uzbek Academy of Sciences, Tashkent, UZBEKISTAN.

Study of processes of photo degradation [1] in a-Si:H films has allowed to improve stability of photo cells, but uniform theory explaining this process in a-Si:H layers and structures on the basis is not present [2]. This models do not take into account change of concentration of complexes Si-H and Si-H₂ bonds. For experimental check of this fact the influence of the concentrated solar radiation of high density on vibrational spectrum of Si-H and Si-H₂ bonds in intrinsic a-Si:H films was investigated. The experiment were performed using IR absorption electronic spectrometer. The undegraded a-Si:H films have shown, that the probability of formation of the dangling bonds under the action of illumination low and makes 10⁻⁶ spin/quantum. Hence for supervision of changes in IR-spectrum vibrational modes it is necessary to shine a sample by light of intensity not below 10⁻³-10⁻⁴ quantum/s²/cm² in current [3] not less than 1 hour. Vibrational spectra of a-Si:H were investigated on a technique IR-spectroscopy of the repeated broken complete internal reflection (RRCIR) [4] in the field of 1300-2800 cm⁻¹, where bond stretching modes of Si-H (2000 sm⁻¹) and Si-H₂ (2100 sm⁻¹). The given method has allowed to investigate a-Si:H layers, which thickness is close to thickness of 1-nm layer in real p-i-n a-Si:H photocells. As the photoinduced effects are accumulated during the time of samples was made for prevention of thermal heating with a break 5 minutes illumination, 2 minutes blackout. The results of experiments have shown, that the irradiation of a-Si:H films by the concentrated solar radiation of high density up to 300 kW/m² for 2 hours gives not given essential change in IR-spectrum of a-Si:H films. With increase of density of solar radiation up to 400 kW/m² the absorption in area of valent vibration 2000 sm⁻¹ monotonously decreases and at the further increase of time of exposition with 45 up to 120 minutes of changes in a RRCIR spectrum was not observed. At density of solar radiation 600 kW/m² the area on a surface of a prism completely collapses, to what the absence of peak of valent vibration in IR-spectrum of a-Si:H film is observed. From RRCIR tested the results follows, that under action of solar radiation the concentration of Si-H bonds decreases, while the concentration of Si-H₂ bonds essentially does not change. It is known, that the energy of connection of Si-H₂ bond is less than the energy of connection of Si-H bond. Therefore it is more probable than everything, from the point of view of energy of break, one is stronger Si-H₂ bond. The received experimental results give the bases for deeper theoretical research of mechanism of formation of a-Si:H layers and properties of a-Si:H films under the action of concentrated solar radiation. These processes dependents strongly on the chemical composition and stoichiometry of glasses. In stoichiometric glasses radiation-induced effects are almost linear. In the case of non-stoichiometric compositions the maximum changes are observed near the value of average coordination number Zc=2.67 which is connected by numerous investigators as the point of so-called topological phase transition. Optical transmittance spectra in visible region before and after gamma irradiation are studied with the aim to confirm obtained results. For the majority of investigated samples gamma-irradiation leads to long wave length shift of fundamental absorption edge. It is explained by the appearing in the bond gap the level connected with radiation-induced defects. The dynamic and static properties of radiation-induced optical effects are revealed in chalcohenide glasses. These properties are attached to different radiation-induced microstructural mechanisms. Observed radiation-induced phenomena are explained also in dependence on glass compactness. It is concluded that glasses which have more “free volume” are characterized by bigger static component and more compact samples have bigger dynamic component of radiation-induced effects.

### P.3.35
**NITROGEN IRRADIATION OF DIAMOND**
K. Jagners, Dept of MS&E; K. Venghese, Nuclear Engineering, N.C.S. State University, Raleigh, NC, J.E. Butler, Naval Research Laboratory, Washington, DC.

Boron-10 isotope doped diamond has been subjected to neutron irradiation to achieve Li7 dopant. Special attempts have been made to avoid amorphization or graphitization of diamond during neutron irradiation. Results of irradiation experiments for different irradiation periods in a research reactor will be presented. Characterization of the boron-10 doped diamond before and after neutron irradiation using Raman spectroscopy and secondary ion mass spectroscopy will be presented.

### P.3.36
**CHARACTERIZATION OF PHOTO-INDUCED STRUCTURAL CHANGES IN AMORPHOUS GLASS USING X-RAY PHOTOELECTRON SPECTROSCOPY**
S. Krainikwari, J. Bloking, H. Jahn, Leib University, Bethlehem, PA; P. Krecke, S.R. Elliott, University of Cambridge, Cambridge, UNITED KINGDOM; M. Veek, University of Pardubice, Pardubice, CZECH REPUBLIC; J. Li, D.A. Drabold, Ohio University, Athens, OH.

Amorphous chalcohenides, in general, exhibit a wide variety of changes in their structure and physical properties, particularly affected by bandgap irradiation. The photos are the radiation of amorphous/hardening, mechanical (e.g. hardness, plasticity), rheological (e.g. viscosity), optical (e.g. darkening, bifringence, luminescence), electrical (e.g. conductivity, dielectric constant), or the chemical (e.g. etching, dissolution) properties of the glass. These
photo-induced effects are classified into three categories depending on their stability: (a) permanent changes which cannot be recovered without doping; (b) metastable effects which can be reversed by heating the sample to the glass-transition temperature; and (c) changes which can be reversed simply by removing the light source, or exposing the specimen to appropriate light subsequently. Of particular interest are category (c) phenomena which may be used for converting an optical signal into some other signal. In this work, we have characterized the structure of amorphous Ge-Se films and bulk samples under conditions of H$_2$-Ne laser and Na & lamp light irradiation using high resolution X-ray photoelectron spectroscopy (XPS). After irradiation, we find that As$_2$Se$_3$/Si ratio decreases indicating that the surface of As$_2$Se$_3$ becomes irreversibly enriched with Se, and a fraction of As$_2$Se$_3$ transforms into As$_2$S$_3$. Additionally, there are reversible changes in the valence band, which include transformation of the long-wave polar-pairs on Si to new bonding states that are present only under illumination. Preliminary results also indicate enhancement in the diffusion of the excess atoms due to light illumination. We are also modeling these short-time photo-structural effects by exact state quantum molecular dynamics. We have proposed realistic models of As$_2$Se$_3$, g-Ge$_2$Se$_3$, and As$_2$S$_3$ and fully characterized their structure, dynamics and electronic properties. We are also developing a model of As$_2$Se$_3$ for comparison to O$_x$M$_{1-y}$. By linking these models with the associated photostructural response, one can have atomistic understanding of many light-induced processes in chalcogenide glasses.

**R3.37** FORMATION OF ELECTRICALLY ACTIVE DEFECTS IN C$_2$-P$_2$ Si AND Si$_2$ P$_2$ BY 3 MEV IRradiation. MIN-DOO ChU, DONG HWAN Kim, Joo-YOOL Huh, Division of MS&E, Korea University, Seoul, REPUBLIC OF KOREA.

Proton (3 MeV) irradiation was conducted on oxygen-rich Czochralski (Cz) and oxygen-free floating zone (FZ) boron-doped p-type Si wafers at room temperature. P$n$-junction formed in the Cz wafers when the irradiation dose reached the critical value between $1.0 \times 10^{17}$ cm$^{-2}$ and $3.0 \times 10^{17}$ cm$^{-2}$, but not in the FZ wafers even at higher doses. 2$p$-n junction formation was confirmed by the results of Hall measurements, current-voltage (I-V) measurements, spreading resistance (SR) measurements, and electron beam-induced current (EBIC) measurements. High resolution X-ray diffraction revealed a gradual broadening of (100) peak for both Cz and FZ wafers with increasing the irradiation dose, which indicates that the lattice damages induced by the proton irradiation are similar for the two wafers. The known pair of an interstitial oxygen and a silicon self-interstitial is suggested to play a key role in creating the electrical junction in the Cz wafer.

**R3.38** HYDROGEN PASSIVATION OF Si NANOCRYSTALS IN SILICA.
Stephanie Cheylin and Robert G. Elliman, Electronic Materials Engineering Department, RSPhysE, Australian National University, Canberra, AUSTRALIA.

Si crystallites can be formed in silica by high-dose ion-implantation and thermal annealing. Hydrogen passivation of these crystallites is known to increase their optical density and Si nanostructures by up to an order of magnitude. However, in this paper, it is shown that the role of the hydrogen depends on the treatment employed for nanocrystal synthesis. Fused Si plates or thermally grown SiO$_x$ layers were implanted with 100 keV Si ions to a dose in the range from $1 \times 10^{15}$ cm$^{-2}$ to $1 \times 10^{17}$ cm$^{-2}$, corresponding to peak concentrations in the range from 5 to 30 % Si. Samples were subsequently annealed at either 1000°C or 1100°C in a nitrogen or argon ambient to precipitate Si crystallites. H passivation was achieved by annealing in forming gas (5% H$_2$/95% N$_2$) and the photoluminescence studied as a function of annealing time and temperature. Important new results include the observation of a reversible red-shift and broadening in the photoluminescence emission following passivation. These results, together with luminescence lifetime data, optical absorption and transmission electron microscopy data are used to discuss the mechanism for H passivation.

**R3.39** THE SIZE DISTRIBUTION OF Ge NANOCRYSTALS IN IMPLANTED AND ANNEALED SILICA.
Amirte R. Davood and Robert G. Elliman, Electronic Materials Engineering Department, RSPhysE, Australian National University, Canberra, AUSTRALIA.

Semiconductor nanocrystals embedded in dielectric layers are of interest for novel photonic device applications based on optical nonlinearities or light emission. One means of synthesizing such materials is by ion-implantation and annealing, in which the implantation energy determines the size and density of the nanocrystals. In such cases, the size/density of crystallites is expected to reflect the implant distribution. However, our recent studies have shown that this is not necessarily the case. In this work, samples were implanted at room temperature or $\sim 190^\circ$C with 1.0 MeV Ge ions to fluences in the range from $1 \times 10^{13}$ to $3 \times 10^{14}$ cm$^{-2}$. Ge deposition was approximately Gaussian, as measured by Rutherford backscattering spectroscopy, and were essentially unchanged after annealing at 1100°C. Despite this, the size distribution of Ge nanocrystals shows a strong asymmetry, more accurately reflecting the nuclear energy deposited by the implanted ions than the initial Ge distribution. For example, transmission electron microscopy of an as-implanted sample irradiated at $-190^\circ$C with $0.6 \times 10^{15}$ cm$^{-2}$ showed no Ge clustering. After annealing in an Ar + 5% H$_2$ at 1100°C for 60 minutes, the size distribution of the nanocrystals with much bigger nanocrystals being observed on the surface. This behavior is discussed in terms of beam-induced nucleation of nanocrystals.

**R3.40** SIZE DEPENDENCE OF DAMAGE FORMATION BY CLUSTER ION IMPACT. Takashi Aski, Shin-ichi Chiba, Jiro Misuoe, Giken H. Takakura, Kyoto Univ., Ion Beam Engineering Experimental Laboratory, Kyoto JAPAN.

Cluster ion irradiation shows characteristic phenomena in damage formation compared with conventional monoenergetic ion irradiation. In order to examine the collisional process of cluster ion, molecular dynamics simulations of cluster ions impacting on solid targets were performed. When an Ar cluster consists of 1000 atoms impacts on a Si(100) substrate with the total cluster energy of 1 keV/atom, the cluster penetrates through the surface though each atom has only 1 keV. This is because the first impacting atom is accelerated by following atoms in the cluster. At the impact, large number of collisions between cluster and surface atoms occur and the cluster spreads spherically at the shallow region in the substrate. Consequently, a large crater-like damage is formed on the surface. Because of the multiple-collision and high-density energy deposition on shallow substrate region by cluster impact, substrate atoms are largely moved and amorphized damage region is formed on the surface. The radiation effect of cluster ion depends on the cluster size (number of atoms in the cluster). At the impact of small and high energy, projectile cluster, i.e. the size of 10 and energy of 1 keV/atom, each cluster atom penetrates into the deeper region of the substrate without multiple-collision, which cause cascade-like displacements similar to monoenergetic ions. On the other hand, at the impact of large and low energy-per-atom, such as size of 10000 with about 1 keV/atom, the cluster does not penetrate the surface and the surface is not damaged. These results show that cluster radiation forms characteristic damage different from that by monoenergetic ion. The density and range of displacements are controlled by choosing suitable cluster size and incident energy.

**R3.41** MODIFICATION OF THIN Si-Ge FILMS PROPERTIES BY IMPLANTATION OF ISOVALENT IMPURITIES. B.N. Romanyuk, N.I. Klyui, V. G. Liokhovenko, V. P. Melnik, A. G. Rzhanik, Institute of Semiconductor Physics, Ukrainian National Academy of Sciences, Kiev, UKRAINE, R.L. Poltavsky, Chernivtsi State University, Chernivtsi, UKRAINE.

Heterostructures based on Si-Ge alloys have attracted much interest due to their applications for advanced micro- and optoelectronics devices. The alloy properties may be modified either during deposition process or after deposition by various active treatments. A very attractive way to modify thin Si-Ge film properties is ion implantation because of possibility to implant different ions species with controllable ion energy and dose. In this work thin Si-Ge film deposited by PE-CVD or MBE methods onto Si substrates were studied. The film were implanted with 1 keV, 3 keV, 10 keV, 30 keV, 100 keV 100 keV, 300 keV Ge of different doses and energies. Effect of rapid thermal annealing on initial and implanted films was also investigated. For the film characterization ellipsometry, Raman scattering, X-ray diffraction, Auger electron spectroscopy and SIMS were used. It was shown that as a result of carbon implantation a partial relaxation of mechanical stresses in Si-Ge films due to introduction of carbon atoms into Si-Ge lattice takes place. An elevated implantation temperature allows us to maintain high structural perfection of the films. Implantation of Si or Ge ions leads to redistribution of doping impurities and at high doses results in precipitates formation. The mechanisms of the effects observed are also discussed.

**R3.42** SURFACE AND BULK MICROSTRUCTURAL MODIFICATIONS IN AMORPHOUS CARBON FIBRES AFTER ION-BEAM IRRADIATION. P. Papathanass, S. Logothetis, Aristotle Univ., Dept of Physics, Thessaloniki, GREECE.
In this work, we present the crystallization effects occurring in sputtered films of Carbon (a-C) thin films deposited on Si, with different structure and bonding, induced by post-growth low energy (0.1-8 keV) Ar⁺ ion beam irradiation (IBI). Real-time monitoring of the IBI was performed by a multi-wavelength Ellipsometer. Spectroscopic Ellipsometry (SE) provides information on the a-C film microstructure through its correlation with the film’s optical properties. Ex-situ SE was also used to study the structural and bonding modifications of the amorphous matrix after IBI in terms of the Dielectric Tensor Effective Medium Theory. The a-C films after IBI have the form of an amorphous matrix with embedded crystalline regions. X-ray diffraction (XRD) and Electron Microscopy measurements identified the crystalline phases of carbon (e.g. graphite and diamond) and SiC. XRD was used to quantify the relative content, grain size and distribution of each particular crystalline phase that depend strongly on the microstructural characteristics (density, void content, size, shape) of the a-C bond structure. Growth mechanisms were studied by X-ray Reflectivity (XRR) and SE. We also study in detail the effects of ion energy and fluence on the crystallization process. It was found that low fluence (~2x10¹⁰ ions/cm²) of ions with an optimum ion energy (~1.5 keV) promoted the diamond formation. Finally, XRR discriminated the IBI induced surface and bulk effects by studying the density and the a-C surface and a-C/Si interface structure. Surface smoothing was found more prominent for lower ion energy IBI. The combined XRD/XRR results are considered in terms of fundamental ion-solid interaction mechanisms such as implantation damage, relaxation recrystallization and microstructural evolution induced by the growth mechanism of a-C. Appropriate modeling was employed to investigate the validity of fundamental ion-solid interaction mechanisms.

R3.43 INFLUENCE OF AGING ON DENSITY OF DANGLING BOND ON SiO₂ IRRADIATED BY ELECTRON BEAM. Kanyua Oguri, Tsyoshi Sumimoto, Yoshikane Nishi, Dept of Materials Science, Tokai Univ., Japan; Keyaka Sató, Toshihiko Iizumi, Dept of Electronics, Tokai Univ., Japan.

From a materials engineering point of view, it is important to know the surface condition of materials. Sheet electron beam irradiation (SEBI) generates the dangling bond without the residual impurity zones. The sheet electron beam irradiation was homogeneously performed by an electron beam. Thus, we have studied influence of aging on density of dangling bond on SiO₂ irradiated by electron beam. The density of dangling bond was measured by electron spin resonance. The microwave frequency range used in the ESR analysis was the X-band. The field modulation was 100 Hz. Spin density was calculated using a N² standard sample. The aging decreased the density of dangling bond on SiO₂. Based on rate process, the density change [X] with the aging time [t] was expressed by a following equation. X = 1 - exp (-kt). Furthermore, the apparent activation energy was calculated.

R3.44 Abstract Withdrawn.

R3.45 REFINEMENT OF TOPOLOGICALLY MODELED CASCADE-AMORPHIZED SILICAS USING MOLECULAR DYNAMICS SIMULATION: Role of Diffusion – M. H. H. Timmis, Institute for Festkörperforschung, Julich, Germany; H. Heinrich, Pacific Northwest National Laboratory, Richland, WA; B.N. Singh, NSF National Laboratory, Roskilde, Denmark.

It has been recognized in recent years that in real cases, clusters of self-interstitial atoms (SIAs) in the form of small glass droplets produced in displacement cascades are able to form fast one-dimensional (1-D) and 3-D reaction kinetics depending on the mean length of the defect diffusivity in relation to the defect lifetimes. The present work is to show the occurrence of monotonously when changing continuously from 1-D to 3-D reaction kinetics in some of these clusters. The analytical treatment suggests a simple one-parameter master curve interpolating between the pure 1-D and 3-D cases which is confirmed by the 1D MC simulations. The displacement diffusivity of SI loops by random self-climb may be included in the analytical treatment. The formation and stability of void loops under cascade damage is discussed in terms of 1-D to 3-D reaction kinetics.

8:45 AM R4.2 DIRECT CLUSTER FORMATION IN HIGH ENERGY DISPLACEMENT CASCADES IN COPPER. Yu.N. Osetsky and D.J. Bacon, Materials Science and Engineering, Department of Engineering, University of Liverpool, Liverpool, United Kingdom.

Primary radiation damage in displacement cascades in metals has been studied extensively by atomic simulation during the last decade. Nevertheless, there is a clear lack in the variety of defect types observed in cascade simulation which, in many cases, makes it difficult to understand experimental data. For example, experiments on copper show a very effective production of stacking fault tetrahedra (SFTs) but this was not observed experimentally in cascade simulation. To clarify this and related issues, we have performed extensive simulation of displacement cascades, using various interatomic potentials, including short-range potentials, and many-body potentials (SRMP) which have been studied primary knock-on atom energy 20keV (i.e. minimum energy below that for
formation of subcascades) at temperatures 100 and 600K. Special attention was paid to cascade simulation and the accuracy of simulation in the collision stage. The form of the simulated flux showed significant differences in temperatures (typically more than 30 events) whereas the latter involved a modification of the simulation method when a hot region is treated with high accuracy by applying a smaller step size. In this paper we present new results showing the variety of clusters observed, e.g. SFTs, grain and sessile interstitial clusters, and faulted and perfect interstitial dislocation loops. A number of conclusions emerge on effect of the potentials and a significant number of dislocation faults as well as Frenkel pairs are produced in different cascades under the same conditions.

9:00 AM *R4.3 THE PRIMARY DAMAGE IN Fe-R: REVISED BY MOLECULAR DYNAMICS AND ITS BINARY COLLISION APPROXIMATION. A. Pouridian, M. Hou and C.S. Beecroft.

Molecular Dynamics (MD) is a very powerful tool to study the displacement cascades initiated by the neutrons when they interact with matter and thus evaluate the primary damage. The mean number of point defects created with a fair standard error with a reasonable number of cascade simulations (10 to 20 [1]), however other cascades characteristics (spatial distribution, size and amount of defect cluster) display a huge variability and may need to be studied using faster methods such as Binary Collision Approximation (BCA) which is several orders of magnitude less consuming. We have therefore investigated the point defect distributions subsequent to atomic collision cascades by both MD (using EAM potentials in Fe) and BCA and its BCA lead to comparable point defect predictions. The simple differences and differences are discussed. [1] R. Stoller, to be published.


The microstructure of a metal under irradiation evolves over a wide range of length and time scales, making radiation damage an inherently multiscale phenomenon. The degradation of the mechanical properties of the material is a well-established consequence of this evolution. The main characteristics of this phenomena as evidenced in tensile tests performed in pure metals irradiated and tested at near room temperature (RT) are: (i) A sharp increase of the yield stress as compared to that of the unirradiated material, (ii) The appearance of a yield point followed by a yield region at constant average stress and (iii) A very strong flow localization in so-called "dislocation channels", which leads to loss of ductility and premature failure. Multiscale computing modeling has been used to establish the defect microstructure induced by the irradiation and its interaction with dislocations. At the atomic and microstructural scales, the defect production arising from the unfolding of the displacement cascade is determined with molecular dynamics (MD). The evolution of this initial defect population is then followed with kinetic Monte Carlo (KMC) techniques over diffusion distances and timescales. Finally, 3-D dislocation dynamics (DD) is used to study the interaction between dislocations and the resulting defect microstructure. The results of the different stages of simulation agree well with the available experimental information. In particular, the KMC simulations allow to study DD simulations of the different characteristics of the tensile response of the irradiated material and the scaling of the dislocation channeling. This work was performed in part under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract W-7405-Eng-48.

SESSION R5 IRRADIATED CERAMICS
Chair: Yuri N. Osetsky and Steven J. Zinkle Tuesday Morning, November 28, 2000 Room 310 (Hynes)

10:15 AM R5.1 AMORPHIZATION AND RECRYSTALLIZATION OF NEUTRON IRRADIATED SILICON CARBIDE. E.L. Snesal, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN.

It has been previously shown that it is possible to amorphize silicon carbide under fast neutron irradiation thereby allowing measurement of bulk thermomechanical properties. This paper extends this work by providing further information on the transition microstructure, thermal conductivity, and mechanical relaxation kinetics of bulk amorphous silicon carbide. Samples studied were high purity CVD Sic and 6H alpha single crystal SiC irradiated at ~60°C to a dose of ~2.6 and 4 E5 n/cm2 (E>0.1 MeV), or ~2.6 and 4.4 dpa.

10:30 AM R5.2 DEFECT BEHAVIOR AND EFFECTS OF ION MASS ON PRIMARY DAMAGE STATES IN SILICON CARBIDE. William J. Weber, Fei Gao, Weimin Jiang, Pacific Northwest National Laboratory, Richland, WA; Ram Devanathan, Dept. of Metallurgical Engineering, Indian Institute of Technology Madras, Chennai, INDIA.

Defect behavior and primary damage states in SiC have been investigated using ab initio calculations, molecular dynamics (MD) simulations and ion-channeling experiments. Density functional theory with a local density approximation has been used to determine the stable configurations that form in SiC, and the results are in agreement with those predicted by MD simulations using a newly refined Tersoff potential. The effects of cascade energy, up to 50 keV, on primary damage state have been studied but are detailed in another presentation. MD simulations for 1 keV Au, Si, and C projectiles have also been carried out. Both Si and C PKAs produce highly dispersed Frenkel pairs and only a few small interstitial clusters, with the largest cluster containing 4 interstitials. However, Au PKAs produce both highly dispersed Frenkel pairs and large disordered clusters. Analysis of the pair correlation function and long-range order parameter in the primary clusters indicates an amorphous state. From the cluster spectra, the ratio of the cross sections for n-cascade amorphization and interstitial production have been determined for Au, Si, and C, respectively. The MD cross section ratios are in excellent agreement with the ratios determined by a fit of a theoretical amorphization model to experimental ion-channeling data for SiC irradiated with Au and C ions, respectively. The different MD methods for Au, Si, and C PKAs provide atomic-level insights into the observed thermal annealing behavior in SiC irradiated to low doses with these ions. The ratio of C to Si displacements measured at very low damage levels is almost consistent with results of MD simulations. The excellent agreement between the MD simulations and experimental results validates the model potential and confirms the atomic-level interpretation of damage accumulation and annealing processes in SiC. Additional ongoing activities will be discussed.

10:45 AM R5.3 DOPANT IMPLANTATION AND ACTIVATION IN SiC. O.W. Holland, Oak Ridge National Laboratory, Solid State Division, Oak Ridge, TN.

Activation of implanted dopants in SiC will be discussed. Conventional wisdom states that the best electrical results (i.e., carrier concentrations and mobility) are achieved by processing at the highest possible temperatures. These includes implantation into 6H SiC at ~100°C followed by furnace annealing at temperatures as high as 1750°C. Despite such aggressive and extreme processing, implantation suffers because of poor dopant activation, typically ranging between 2%–50% with p-type dopants represented in the lower portion of this range and n-types in the upper. Additionally, high-temperature processing can lead to several problems including changes in the stoichiometry and topography of the surface, as well as degradation of the electrical devices (i.e., channel mobility and gate integration). Two approaches for resolving these problems in 4H SiC will be discussed. The first consists of a traditional implantation scheme optimized over a wider range of parameters than normally studied. In particular, the different dopants and DD simulating the different characteristics of the tensile response of the irradiated material and the scaling of the dislocation channeling. This work was performed in part under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract W-7405-Eng-48.

11:15 AM R5.4 ION-BASED AMORPHIZATION AND RECRYSTALLIZATION OF CADMIUM NIOBATE PYROCHLORIE. Kristen Benz, Arkivolvics Meldrum, University of Alberta, Dept of Physics, Edmonton, CAN; John B. Bonner, Woody White, Oak Ridge Nat’l Lab, Solid State Div, Oak Ridge, TN.

Pyrochlores represent a large class of compounds with the general chemical formula A2B2O7. Recent investigations have focused on the effects of chemical composition and incident ion mass and energy on the irradiation-induced amorphization of several pyrochlores, including Gd2Ti2O7. This study was prompted by the possibility study since it is one of the three main actinide-bearing phases of SYNROC, a polyphase ceramic waste form proposed for the disposition of...
high level nuclear waste. Replacement of the Gd with other lanthanide elements or Cs has been found to have a relatively minor effect on the kinetics of irradiation, but it has produced anomalous differences in the substitution of Ti with Zr in increasing concentrations sharply decreases the resistance to amorphization even at cryogenic temperatures. In the present work, we have grown large synthetic single crystals of Cd$_2$Te and investigated the irradiation-induced transformation to the amorphous state. In situ temperature-dependent ion-irradiation experiments were performed directly in a transmission electron microscope. Additionally, ion-implantation experiments were performed by the Siemens solid state science laboratory, Oak Ridge National Laboratory, U.S. Department of Energy, under contract DE-ACO5-000R22775 with UT-Battelle, LLC, by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under interagency agreement DOE 1866-N653SW with the U.S. Department of Energy and through the Shaire Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities. Research at the Groupe de Physique des Matériaux was sponsored by Electricité de France (Centre de recherche des Renardières).

11:30 AM R5.5
ANALYTICAL ELECTRON MICROSCOPY STUDY OF ION IRRADIATED SPINEL
D.M. Anderson, D.T. Hoeger, S.J. Zinkle, Oak Ridge National Laboratory, Oak Ridge, TN.

The effects of ion implantation on stoichiometric magnesium aluminum silicate spinel were studied using a SEM equipped with an EDS analysis. In specimens irradiated with swift heavy ions (electron stopping power > 8 keV/nm at the specimen surface), there are three distinct regions of the irradated zone: an amorphous region at the surface; a metastable phase; and a spinel phase exhibiting radiation damage as a result of the irradiation. This third zone forms in a region where the heavy ions have slowed and the electronic stopping power has been reduced. A spinel irradiated with 72 MeV I ions was examined in cross section to enable characterization of these distinct regions. Atom lociation by channeling enhanced microanalysis (ALCHEMI) performed near the Bragg condition of the spinel 220 planes in this zone indicates that ~30% of the Mg cations sit on anion sites at the irradiated depth. This level of disorder is similar to that measured in spinel irradiated with 2.4 MeV Mg ions, with lower stopping powers, and is substantially larger than the degree of disorder in unirradiated synthetic spinel (~13% of the Mg on the octahedral sites). The metastable phase that forms in the second zone of the swift heavy ion irradiated specimen has been characterized by ALCHEMI and convergent beam electron diffraction (CBED).

11:45 AM R5.6
SESSION R6 FERRITIC AND PRESSURE VESSEL STEELS
Chair: Roger E. Stoller and Lynn E. Rehn

1:30 PM *R6.1
ATOMIC LEVEL CHARACTERIZATION OF NEUTRON IRRADIATED FERRITIC PRESSURE VESSEL STEELS
M.K. Miller, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; P. Periaux, Groupe de Physique des Matériaux, UMFR CNRS 6854, Faculté des Sciences et INSA de Rouen, Mont Saint Aignan, FRANCE.

Atom probe tomography provides one of the most effective tools to characterize the cation distribution and precipitation that occurs in pressure vessels associated with temperature and pressure drops during irradiation. The three-dimensional atom probe is able to experimentally attain the spatial coordinates and the elemental identities of the atoms with near atomic resolution so that their distribution within small volumes of the specimen can be reconstructed and analyzed. This technique together with conventional atom probe field ion microscopy has been applied to many different types of pressure vessel steels and model alloys. An overview of the findings will be presented, including several microstructural transformations. These radiation induced or enhanced processes lead to the formation of copper-nickel-manganese-silicon precipitates, and sublimate segregation to dislocations, dislocation loops, micronoids and boundaries. A review and a comparison of the radiation induced phase transformations and segregation in Western, Russian and French reactor pressure vessel steels will be presented.

Researchers at the Oak Ridge National Laboratory's Shaire User Facility will present developments in materials science. The Shaire User Facility, U.S. Department of Energy, under contract DE-AC05-000R22775 with with UT-Battelle, LLC, by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under interagency agreement DOE 1866-N653SW with the U.S. Department of Energy and through the Shaire Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities. Research at the Group of Physics of Materials was sponsored by Electricité de France (Centre de recherche des Renardières).

2:00 PM R6.2
HARDENING AND MICROSTRUCTURE OF MODEL ALLOYS OF Fe, Fe-Cu AND Fe-Cu,3Cr USING PROTON IRRADIATION
Qingkg Yiu, Gary S. Wilk, Lumin Wang, University of Michigan, Dept. of Nuclear Engineering and Radiological Sciences, Ann Arbor, MI; Robert Odette, University of California at Santa Barbara, Department of Mechanical and Environmental Engineering, Santa Barbara, CA; Dale Alexander, Argonne National Laboratory, Argonne, IL.

Model reactor pressure vessel alloys of compositions Fe-0.9Cr-1.0Mo, Fe-0.9Cr-3.0Co and Fe were irradiated with protons to study the hardening process and to determine if proton irradiation is effective in causing hardening and microstructure evolution similar to that produced by neutron irradiation. Samples 3 mm thick were irradiated to fluences of around 10^17 p/measurement. The hardness was measured using Vickers indentation using a 25 g load to ensure that the elastic zone was confined to the irradiated depth. Results show that irradiation up to 300,360°C and a fixed dose rate resulted in a dose dependence on hardness that closely follows that resulting from neutron irradiation in both the hardening effect and the magnitude of hardness increase. The effect of dose rate and temperature are also compared to neutron results. Microcracks are analyzed using small angle x-ray scattering (SAXS) and high-resolution transmission electron microscopy (HREM) and correlated to hardening and neutron-induced microstructure.

2:15 PM R6.3
THE ROLE OF PRIMARY DAMAGE STATE IN RADIATION EMBRITTLEMENT OF PRESSURE VESSEL ALLOYS
Dale E. Alexander, L.E. Rehn, Materials Science Division, Argonne National Laboratory, Argonne, IL; G.R. Odette, G.E. Lucas, D. Klingensmith, D. Gregg, Department of Mechanical and Environmental Engineering, University of California-Santa Barbara, Santa Barbara, CA.

The ability to predict radiation hardening (embrittlement) is critical for assessing the integrity and service life of components in high temperature reactor environments. The process is complicated by the large time and length scales involved. The work presented here seeks to maintain modeling efforts in an important element of this process by providing experimental insight into the effects of the primary damage state (i.e., the nascent spatial distribution of radiation produced defects) on hardening. Comparative experiments have been performed examining tensile property behavior in 10 MeV electron and test reactor neutron-irradiated alloys of interest to reactor pressure vessel embrittlement. Changes resulting from neutron irradiation, in which nascent point defect clusters form primarily in dense cascades, with electron irradiation, where cascade formation is minimized, contribute to an understanding of the role that the nascent cascade point defect clusters have on the mechanisms of embrittlement. Irradiations were performed at 300°C on Fe-0.9 wt% Cu-1.0 wt% Mo alloy and unalloyed Fe to damage levels up to 5x10^17 dpa. Despite the significant differences in primary damage state, similar embrittlement trends with increasing radiation damage were observed for electrons and neutrons in both the alloys when compared on a dpa basis.

This work was supported in part by the U.S. Department of Energy, Office of Science, under contract DE-AC05-00OR22725 with UT-Battelle, LLC, by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under interagency agreement DOE 1866-N653SW with the U.S. Department of Energy and through the Shaire Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities. Research at the Group of Physics of Materials was sponsored by Electricité de France (Centre de recherche des Renardières).
A large number of metallurgical and environmental variables interact to control irradiation and embrittlement in reactor vessel steels. Hardening is due to three classes of features forming under irradiation: copper-rich precipitates (CRPs); stable matrix features (SMFs) that are associated with defect solute cluster complexes, and mixed unstable matrix defects (UMD) that continuously form and dissolve with relaxation times order $3 \times 10^5$ s. At very high flux levels the UMD act as sinks and delay CRP evolution but also add a flux-dependent increment to hardening. At very low fluxes they may also contribute an additional increment. At intermediate levels the flux effect has not been well established. Some proposed test reactor studies suggested a significant effect, while this has not been observed in test reactor irradiation data. The present carefully controlled experiment shows a systematic effect of decreasing flux in both steels and model alloys manifested as shifts in hardening curves to lower fluence. The flux ranged from about $5 \times 10^3$ to $10^4$ n/cm$^2$/s for fluences between about 7 to $10^9$ n/cm$^2$ and temperatures from 270, 280, and 310°C. The flux dependence can vary in copper, nickel and manganese content. The intermediate flux effect seems to be primarily on the copper precipitation mechanism. Evidence is presented that the flux effect in this regime is due to trapping of vacancies by solutes such as manganese and nickel. The corresponding effect of irradiation temperature on the flux dependence is compared to solute trapping enhanced precipitation models. The microstructural basis for interpreting flux effects is also described.

**2:45 PM R6.5**

**FORMATION ANDAnnihilation SCATTERING CHARACTERIZATION OF NONSTRUCTURAL FEATURES IN IRRADIATED Fe-Cu-Mn ALLOYS. B.D. Wirth, P. Asokan-Kumar, R. Howell, Lawrence Livermore National Laboratory, Livermore, CA; G.R. Odette, University of California, Santa Barbara, Santa Barbara, CA; and P.A. Stern, Lawrence Livermore National Laboratory, Livermore, CA.**

Radiation embrittlement of nuclear reactor pressure vessel steels results from a high number density of nanometer sized copper-manganese-nickel rich precipitates and sub-nanometer matrix features. However, outstanding questions exist regarding the composition of the precipitates and the atomic character and composition of the matrix features which impact embrittlement predictions of these steels at higher irradiation exposure. In this work, we present results of positron annihilation spectroscopy (PAS) and small angle neutron scattering (SANS) characterization of irradiated and thermally aged binary and ternary Fe-Cu-Mn alloys. These complementary techniques provide insight into the composition and character of the nanometer precipitates and sub-nanometer matrix features. The PAS characterizations are performed using both Doppler broadening measurements and the Doppler broadened spectra of positron annihilation radiation. The strong positron affinity of copper relative to iron makes copper precipitation a dominant trap for vacancy defects. A recently developed coincidence, two-detector setup improves the signal-to-noise ratio and allows measurement of the high momentum region of the Doppler broadened spectra, providing elemental sensitivity. The measured Doppler broadened spectra in Fe-Cu and Fe-Cu-Mn specimens differ from previous work to peak hydrogen in Fe-Cu-Mn specimens irradiated at 288°C are nearly identical to elemental Cu. Further, positron lifetime measurements clearly show the existence of a long lifetime (7.50 ps) component in Fe-Cu-Mn specimens irradiated at 288°C, which exhibited strong scattering from copper precipitates in SANS measurements. These results indicate the existence of 3D vacancy clusters that may be complexed with Cu. The interplay between the matrix features and copper rich precipitates in positron measurements is discussed. This work is performed under the auspices of the U.S. Department of Energy and Lawrence Livermore National Laboratory. Joint work with R. Howell and P.A. Stern.

**3:30 PM R6.6**

**AN ANALYSIS OF THE STRUCTURE OF IRRADIATION INDUCED COPPER RICH CLUSTERS IN LOW AND HIGH NICKEL REACTOR PRESSURE VESSEL STEELS. Jonathan M. Hyde, Colin A. English, AEA Technology plc, Harwell, Didcot, Oxon, UNITED KINGDOM; David Ellis and Timothy J. Williams, Rolls-Royce plc; Derby, UNITED KINGDOM.**

Extensive research has shown that nanometre scale features are precipitated in high Cu reactor pressure vessel (RPV) steels during irradiation. These clusters or precipitates act as barriers to dislocation movement and may be one cause of increased material hardness and a shift in the ductile to brittle transition temperature. Conventionally, features are assumed to be spherical and contain Cu, Mn, Ni and Si. However, more recent work, enabled by dramatic advances in 3D nano-computer graphics and electron microscopy, has raised the question of these assumptions. In particular, recent data suggests that the clusters are not perfectly spherical, but have a complex ramified morphology and contain a significant quantity of Fe. These are important issues that need to be resolved. In particular, if the interpretation of the atom probe data is correct then, previously published small angle neutron scattering (SANS) data needs to be re-evaluated using more realistic feature compositions. Furthermore, it will also be necessary to develop new models of irradiation hardening which are currently based on the interaction between dislocations and precipitates as opposed to ramified clusters. In order to address these issues we are developing a new simulation tool using DARTM. We have performed on two high copper irradiated RPV steels, one containing low Ni and the other containing high Ni. Cu-enriched irradiation induced clusters were observed in both steels. A new algorithm is being developed to precisely identify the shape, composition and size of the nano-scale data. Representative irradiation induced clusters from each weld will then be analysed to determine the extent of ramification and whether or not Cu is more strongly associated with the core of the clusters than elements such as Mn or Ni. The mean radius of gyration of each cluster will provide a direct comparison with the results of the SANS experiments. Finally, the number of features will be estimated from the SANS data by using the compositional information from the 3DAP observations and making some assumptions concerning the magnetic properties of the clusters.

**4:00 PM R6.7**

**KINETIC MONTE CARLO SIMULATION OF Cu clustering IN Fe-Cu ALLOY UNDER IRRADIATION. Nicki Sondi, Kenji Dohi, Central Research Institute of Electric Power Industry (CRIEPI), Materials Science Dept, Tokyo, JAPAN; Shigeru Ishino, Tokai Univ, Dept of Nuclear Engineering, Koriyama, JAPAN; Tomas Diaz de la Rubia, Lawrence Livermore National Laboratory, Chemistry and Materials Science Directorate, Livermore, CA.**

Recent Atom Probe observations show that copper enriched clusters formed in Fe-Cu alloy during neutron irradiation have very different morphology from those formed during thermal ageing. The primary difference is that the clusters formed during irradiation contain a large number of Fe atoms. This difference should be due to the effect of displacement cascade events where not only point defects but also their clusters are formed. In this paper, we present kinetic Monte Carlo computer simulation results in order to investigate the role of the cascade events in Cu clustering. We show that vacancy flow towards lattice defect clusters can promote Cu clustering around the defect clusters.

**4:15 PM R6.8**

**MODELLING OF COPPER PRECIPITATION IN Fe-Cu ALLOYS UNDER IRRADIATION. Alexander V. Barashche, Stanislav I. Golovch, David J. Brown, The Microscopy (3DAP), Dept of Engineering, Liverpool, UNITED KINGDOM.**

Precipitation of copper-rich clusters is a major cause of in-service hardening of reactor pressure vessel steels and has attracted much attention. Experimental studies of microstructural changes in alloys under various conditions have revealed similarities and differences. It has been established that under ageing the precipitate ensemble exists in normal mid-energy and 50°C and 250°C growth, with overlapping, connecting, and merging features which is the bcc-ufB-ufc transformations which the precipitates undergo during growth. The main effect of electron irradiation is believed to be enhancement of the diffusion of copper and hence acceleration of the kinetics. In the case of neutron irradiation, there are two aspects that are not clear. First, at temperatures less than about 300°C, the precipitate sizes are observed to be very small (3-4 nm), i.e. the coarsening rate is very low. Second, the clusters of vacancies and self-interstitial atoms created in cascades accumulate in the matrix but their influence on the precipitation kinetics is still not well understood. In this paper we present results of computer simulations based on the "mesh" approach for describing microstructural evolution. The main aim is to elucidate possible reasons lying behind the differences in evolution of precipitates under neutron and electron irradiation by comparing results of the model with experimental measurements and microstructure and mechanical properties of irradiated steels.

**4:30 PM R6.9**

**ATOMICISTIC SIMULATION OF VACCENCY AND SELF-INTERSTELLAR DIFFUSION IN Fe-Cu ALLOYS. J. Marín, B.D. Wirth, T. Díaz de la Rubia, Lawrence Livermore National Laboratory, Livermore, CA; J.M. Perdau, Instituto de Fusión Nuclear, Madrid, SPAIN.**

Neutron hardening and embrittlement of pressure vessel steels is due
to a high density of nm scale features, including copper-manganese-nickel rich precipitates which form as a result of radiation enhanced diffusion. Irradiation procedures can also produce large numbers of both isolated point defects and clusters of vacancies and interstitials. Point defect mobility, as well as defect cluster stability and mobility, play key roles in solute transport and the subsequent formation of complex precipitation. Point defects themselves can be used to measure the energetic and mobility of vacancy and interstitial atoms in silicon and other materials. Few experimental studies have been performed to measure point defect and solute diffusion. Cu transport predominantly occurs by a vacancy diffusion mechanism. Small vacancy clusters form as a result of Cu-vacancy binding and may represent precursors for Cu rich precipitation. Conversely, small interstitial clusters are repelled by the Cu atoms, resulting in decreased activation energy for self-interstitial migration.

This work is performed under the auspices of the U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. 89-147-Eng-48 and within the Venus Project (CON/UNE) Coordinating Research Program) under contract P-87/031543Z.

4:45 PM R6.16

Neutron irradiation-induced P segregation at grain boundaries has become one of the important topics in assessing safety of ageing nuclear reactor pressure vessel steels. This paper describes the effect of bulk P contents (0.018 wt. %) on irradiation and intergranular P segregation and embrittlemnent in Fe-based alloys subjected to neutron irradiation. Correlation analysis was performed to study the effect of irradiation on P and Cr segregation in neutron-irradiated and thermally annealed alloys. The magnitude of hardening and P segregation induced by the irradiation tended to diminish in alloys with high bulk P contents. The mechanism of neutron irradiation-induced P enrichment and P segregation was discussed in light of dynamic interaction between point defects and P in the grain matrix and near-grain boundaries. In addition, the correlation of ductile-brittle transition temperature (DBTT) with the hardness, segregated P, S and C is presented in various model Fe alloys subjected to neutron irradiation, or thermally annealed to identify how the mechanical variables control intergranular hardening and embrittlement. This work was supported by the USDOE, Division of Basic Energy Science, Division of Materials Science.

SESSION R7/012 JOINT SESSION
ION BEAM SYNTHESIS OF NANOSTRUCTURES AND THIN LAYERS II
Chair: J. S. Cazes
Wednesday Morning, November 29, 2000
Room 311 (Hynes)

8:30 AM *R7.1/012.1
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 colored red. More recently, materials of this type have been found to exhibit large third order optical nonlinearities. In the case of semiconductor nanocomposites, numerous novel electronic effects (i.e., blue shifting of the band gap and the presence of discrete transitions in the spectrum) have been observed. In particular, these new materials 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 “nanoengineered” systems depend directly on the size of the particles, as well as on their crystal structure, shape, and orientation. By controlling these 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 nanocrystallite composites 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 be possible to manipulate 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 versatile tool for forming nanocomposite materials of various compositions, and that the processing parameters can be modified to tailor the microstructural properties of the composites. 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 R7.2/012.2
ION BEAM ASSISTED NUCLEATION OF NANO-CRYSTALS. D. Lu, R. L. Zimmerman, and C. Mantele, Center for Irradiation of Materials, Alabama A&M University, Normal, AL; S. Schiestl, C.A. Cronnell 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 nanocrystals. 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 nanocrystals. In our recent work, we have used the energy deposited due to the electronic excitation by post-implantation irradiation to induce the nucleation of nanocrystals. This process was used to reduce the threshold implantation dose by at least two orders of magnitude. In this paper, we are presenting a similar technique, post-irradiation electronic excitation, to films produced by low-energy ion beam assisted deposition (IBAD). As-damaged Eu-silica films (co-deposited at various concentrations and temperatures) are grown in the presence of a 50 eV Ar ion beam, then post-irradiated. The resulting Au-nanocrystals were characterized 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-AC05-94OR22464 with Lockheed Martin Energy Research Corp.

9:15 AM R7.3/012.3
ION BEAM ENHANCED FORMATION AND LUMINESCENCE OF Si NANOCUSTERS FROM a-SiO2. Jung H. Shin, So-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-dispersed Si nanocrystals is to ion-beam precipitating them out of SiOx (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 mean 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 ion-irradiating SiOx films prior to anneal, both the cluster formation rate and the luminescence intensity can be greatly enhanced. SiOx films 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 irradiated with 380 eV Si ions to a fluence ranging from 5.4 x 1016 to 2.4 x 1017 ions/cm2. The films were hydrogenated after anneals to passivate defects. Ion-irradiated films already display substantial luminescence in the 800 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 propose chemical effects due to the ion beam, ion beam for evidence that irradiation damage greatly accelerates nucleation of small Si clusters from the a-SiOx matrix.
Lawrence Berkeley National Laboratory, Berkeley, CA.

Cadmium rich nanocaps lead-cadmium alloy inclusions have been made in silicon by sequential ion implantation and subsequently analyzed by transmission electron microscopy (TEM) and Rutherford backscattering/channeling 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 cubo-octahedral 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 metal. The voids tend to be hexagonal in orientation 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 energy considerations.

RBS experiments show that melting of the inclusions takes place in a twostage process. The first stage is due to eutectic melting at a temperature around 160°C leading to a curved liquid/glassy 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. Shrinkage of the inclusions into a two-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 R8/013: 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 *R8.1 /013.1

THE LIMITATIONS AND ADVANTAGES OF USING ION BEAM SYNTHESIS TO FABRICATE PLASMONIC DEVICES.
Mark L. Brongersma, IBM, Almaden Research Center, San Jose, CA, 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 modulating light must have dimensions comparable to the wavelength of light. Recently however, it was theoretically shown that this problem can be circumvented by “plasmonic” devices, in which a specific part of electromagnetic energy is trapped in subwavelength metallic nanostructures (the “non-radiative” part of the energy). This transport relies on the coupled near-field electrodynamic interaction between metal particles that set up a plasmon mode. 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 radii < 100λ), 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, 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 *R8.2 /013.2

SYNTHESIS OF [InGa]AAs NANOSTRUCTURES BY ION IMPLANTATION.
S. Cherk, X. Weng, S. Kumar, and R.E. Goldman, Dept of Materials Science and Engineering, V.H. Rothberg, Dept of Nuclear Engineering and Radiological Sciences, S. Krishna and P.K. Bhattacharya, Dept of Electrical Engineering and Computer Science; J. Spasok and A. Francis, Dept of Chemistry; A. Daniel and R. Cherk, Dept of Physics, University of Michigan, Ann Arbor, MI.

Mixed ion nitride-arsenide compound semiconductor alloys and nanostructures are promising for light emitting devices operating throughout the ultraviolet to infrared range. However, due to the large NAs difference, a limited miscibility of InGaNAs on the arsenic sublattice is predicted. To date, only a few percent nitrogen has been incorporated into mixed ion nitride-arsenide materials synthesized by conventional epitaxial methods. Furthermore, limited studies have been performed using ion implantation. Therefore, we have investigated the synthesis of InGaNAs by N ion implantation into GaNAs 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. XRD suggests formation of high nitrogen content GaNAs and InAsN alloys. Since XRD is very sensitive to coherent crystalline structure, we infer that the implanted regions are amorphous. 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 prediction-gap lowering.

Detailed investigations of the microstructure using high resolution and analytical electron microscopy will be discussed.

11:15 AM R8.3 /013.3

SYNTHESIS OF HgMnSe-V2- alloys by N ION IMPLANTATION.

We have successfully synthesized dilute HgMnSe-V2- alloys (with x=0.001-0.005) by nitrogen implantation followed by appropriate post implantation annealing treatments. For GaN, optical investigations show that the fundamental band gap energy decreases with increasing N implantation dose in a manner similar to that commonly observed in InGaAs- 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 site decreases at higher annealing temperatures. As inferred from the magnitude of the band gap shift, typically only 15% 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 or Ga) element is co-implanted with N in GaN. The co-implantation is believed to enhance N substitution on the As sites by creating a more “favorable” environment rich region. Results on the synthesis of dilute nitriles of other III-V materials such as Al,Ga1-xN,As1-x (x=0.01-0.15), GaN0.15P0.85, InN0.15P0.85. Using N ion implantation will also be discussed.

11:30 AM R8.4 /013.4

DOPING OF GALIUM NITRITE BY ION IMPLANTATION.

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 10^5 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 C, Fe and Er were used as dopants in this study. Ion implantation was carried out at substrate temperatures of 300 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 spectroscopy. The samples were implanted with Fe. Using ion channeling the accurate site location of the dopants is determined by X- ray and optical measurements. The electrical and optical properties of the ion implanted GaN films have been studied by Hall effect and photoluminescence measurements.

The implanted GaN films exhibit an expanded lattice. After annealing at 1000°C, the lattice distortion does not fully recover. The X- ray reduced luminescence near 1.54 µm is observed under below band gap excitation at liquid helium temperature. The spectra of the annealed samples consist of multipe lines with the sharpest lines appearing in GaN until now. Strong lines belonging both to [01T] and [10T] 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 centered defects.

11:45 AM R8.5 /013.5

CORRELATION BETWEEN STRUCTURAL AND OPTICAL PROPERTIES OF Si NANOCTH. IN SO 2 MODEL FOR
THE VISIBILITY LIGHT EMISSION. Elia Garabai, Mikel Lopez, Alejandro Perez-Rodriguez, Jesus Ramon Mateo, EAE-Department of Electronics, Universidad de Barcelona, Barcelona, SPAIN; Caroline Bonafos, Alain Claverie, CEMES-CNRS, Toulouse, FRANCE.

It is now widely 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. Size distribution histogram, band-gap energies and photoluminescence (PL) emission of Si nanocrystals ion bombarded 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 maximum and emission maximum 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 R9/04: JOINT SESSION SEMICONDUCTORS AND ELECTRONIC MATERIALS

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

1:30 P.M. R9/1/04.1 MEDIAN-RANGE ORDER IN ION-IMPLANTED AMORPHOUS SILICON AND DAMAGE MODEL. J.Y. Cheng, Univ. of Illinois, Dept. of Materials Science Engineering, Urbana, IL; Martin Gibson, Argonne National Lab, Materials Science Div, Argonne, IL.

We have measured medium-range order in ion-implanted amorphous silicon, based on small angle X-ray scattering. Low energy ion implantation leads to a highly topologically ordered crystalline 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 P.M. R9/2/04.2 DAMAGE PROCESSES IN GaN UNDER ION BOMBARDMENT. S.O. Kucheyev, J.S. Williams, C. Jagdish, The Australian National University, Dept of Electronic Materials Engineering, Research School of Physical Sciences and Engineering, Canberra, AUSTRALIA; J. Zou, The University of Queensland, 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/d 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 defects associated with displacement of planar defects. Planar defect sites are characterized in detail on 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.

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. Only then can the required process control for advanced device applications. Secondary ion mass spectrometry (SIMS) is ordinarily employed for this purpose. However, in the present studies, implantation profiles have been determined by direct imaging of B using high resolution transmission electron microscopy. The as-implanted B impurity profile is determined with theoretical simulations. The effects of thermal annealing on the results of SIMS measurements are discussed. Changes in the B clustering and clustering that occur after amending of the implanted layers are also described.

2:15 P.M. R9/4/04.4 CRYSTALIZATION OF ISOLATED AMORPHOUS ZONES IN SEMICONDUCTOR MATERIALS. E.P. Hellier, M. Robertson, University of Illinois, Dept of Materials Science and Engineering, Urbana, IL; Igor Jenicek, Josef Stefan Institute, Ljubljana, SLOVENIA.

Crystalization 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+ ion implantation. Significant thermal growth 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 180 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 523 nm green laser (223 eV) caused crystallization in Si at a rate comparable to a thermal anneal of 523 K and caused minimal crystallization in GaP (Eq. = 2.26 eV). The electron and photon irradiation results are consistent with the model that crystalization is controlled by defects (dangling bonds and kinks) created by electronic excitation at the amorphous-crystalline interface.

2:30 P.M. R9/5/04.5 INVESTIGATION OF IRRADIATION DAMAGE IN SILICON DIOXIDE POLYMORPHS USING CATHODOLUMINESCENCE MICROANALYSIS. Marian A. Stevens Kalloch, Faculty of Science, University of Technology, Sydney, NSW, AUSTRALIA.

Cathodoluminescence (CL) is the luminescence 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, and information about structural and distribution of defects in wide band gap materials (i.e., materials with low electron conductivity). CL microanalysis allows the in situ monitoring of electron irradiation induced damage as well as the post irradiation examination of damage induced by energetic irradiation. CL microanalysis complements the assay 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 specimens. 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 recombination 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 P.M. R9/6/04.6 INVERSE DISTILLATION HEATING AND SELF-ORGANIZATION OF
NANOCLASTERS DUE TO ION IRRADIATION. K.-H. Heinig, B. Schmidt, F. Z. Roosendorf, Institute of Ion Beam Physics and Materials Research, Darmstadt, and R. M. Nemanich, University of California, Santa Barbara, CA, ITALY. The Beam of free and solid targets of amorphous materials. If a component is a chemical compound, there is another competition between the collisional and chemical dissociation of the compound and its thermal activated re-formation. Especially at interfaces between immiscible components, these processes far from thermodynamic 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 consequences of reduced sticking coefficients 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 Gilbert-Thomson relation with modified parameters. A dramatic consequence result 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 selforganization of nanoclusters in a δ-layer parallel to the Si/SiO2 interface which has been found when using the interface damage level 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 substoichiometric SiO2-δ 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 R0.07/014.7 IMPACT OF BORON, GALLIUM AND OXYGEN ON DEFECTS PRODUCTION IN SILICON. Aminreza Khan, Nethaji Dhamarruppu, Masanori Yamaguchi, Kenji Araki, Tatsuo K. Ku, Toyota Technological Institute. Nagoya, JAPAN. Sharp Corporation, Nara, JAPAN; Takeo Aki, Shin-Etsu Handotai Co. Ltd, Gunma, JAPAN; Osamu Amanzawa and Sumio Masuda, 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 complexes. An approach to avoid the deleterious effects of the boron-oxygen complex is to use different dopants, such as Ga, which shows less light degradation in Si Solar cells. In order to clarify the potential of Ga doped Si solar cell 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 x 10¹⁴ to 5 x 10¹⁶ 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 irradiation. The interesting new feature of our results is that the gallium appears to strongly suppress the radiation induced defects, especially hole level Eₗ, 0.36 eV, which is thought to act as a recombination center, considerably the damage level of electron level Eₑ, 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 microstructural annealing recovery of the photovoltaic parameters of both gallium and 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 R0.08/014.8 POST ANNEALING STUDIES OF C60 ION IMPLANTED THIN FILMS. Nethaji Dhamarruppu, Kuman L. Narayanan, Nambik Kojima, Yoshinori Nakamura, Toyota Technological Institute, Semiconductor Lab., Nagoya, JAPAN.

Recently, ion implantation in fullerene C60 has attained an enormous interest for electronic device applications. We have studied multi energy boron ion implantation in C60 thin films to various devices. Physical properties like electrical, structural and optical properties were studied. The C60 films were prepared on quartz and silicon substrates using a pulsed deposition of 150 degree in a 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 increase due to the implantation and it could be attributed to the formation of defect level 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 dopant 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.
The formation of nanovoids in Si(100) and MgO(100) by $^3$He 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 recombined variable-energy positron beam POSB. 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 unannihilated 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 $^3$He implantation ($1 \times 10^{14}$ 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.