SYMPOSIUM R

Microstructural Processes in Irradiated Materials

November 27 – 29, 2000

Chairs

Glenn (Gene) E. Lucas
Dept of CE&M and EE
Univ of California-Santa Barbara
Santa Barbara, CA 93106
805-893-4069

Lance Snead
Metals & Ceramics Div
Oak Ridge National Laboratory
Bldg 4508
Oak Ridge, TN 37830-6087
865-574-9942

Marquis A. Kirk
Matls Sci Div
Argonne Natl Lab
Argonne, IL 60439
630-252-4998

Robert Elliman
Dept of Electronic Matls Engr
Australian National Univ
RSPhysSE
Canberra, ACT, 0200
AUSTRALIA
61-2-6249-0521

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* Invited paper

SESSION R1: IRRADIATED METALS
Chairs: Gary S. Was and Helmut Trinkaus
Monday Morning, November 27, 2000
Room 310 (Hynes)

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. Birtcher, C.W. Allen, Materials Science Division, Argonne National Laboratory, IL; K. Furuya, M. Song, K. Mitsuishi, National Research Institute for Metals, Tsukuba, JAPAN.

Inert gas ions implanted into metals generally form small nanometre sized clusters. In the case of the heavy inert gases Ar, Kr and Xe, such nanoclusters 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, isotactic precipitates and to assume the cubeoctahedral shape of matrix voids. This paper reports on recent work in which solid Xe nanoclusters in Al have been subjected to 1 MeV electron irradiation in a high-voltage electron microscope. High-resolution images have been recorded on videotape 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, coalescence and the occasional apparent melting and resolidification of the Xe. Many of the changes to the nanoclusters result from random alterations 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 but is 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. Stoller, 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 that the time scale on 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. 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 A533B 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 between 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 Nicol, M.L. Jenkins, Department of Materials, University of Oxford, Oxford, UNITED KINGDOM; M.A. Kirk, Materials Science Division, Argonne National Laboratory, IL.

The matrix component of hardening of reactor steels under neutron-irradiation is due to the aggregation of irradiation-induced defects to form clusters. The exact nature of such clusters has so far eluded direct characterisation, although suggestions have included interstitial and vacancy loops; loose aggregates such as vacancy sponges; 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 0.06 dpa at 280°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 1-2nm. The matrix damage in both materials was found to consist of small (2-6 nm) dislocation loops. About 80% have Burgers vectors b = a< 100 >, and the remainder have b = a/2< 111 >. The loops in alloy 1A have a mean size d of 4.2 ± 0.3 nm, while those in 2A have d ≈ 4.5 ± 0.3 nm, whilst the number densities are about 8.5 x $10^{21}m^{-3}$ in alloy 1A, and 6.6 x $10^{21}m^{-3}$ in 2A. It can be shown 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. Dudarev, EURATOM/ UKAEA Fusion Association, Culham Science Centre, Abingdon, Oxfordshire, 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 two processes behave 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 presence of planar lattice defects in the material has a significant 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 distance z^* between the grain boundary and the maximum of the swelling profile is found to scale with the density of cavities N_0 as $z^* \sim N_0^{\beta}$, 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.H. Sencer and S.M. Bruemmer, Pacific Northwest National Laboratory, Richland, WA.

Radiation-induced displacement damage in metallic alloys can significantly modify the material microstructure and microchemistry, and in turn severely degrade structural properties. A classic example of this behavior occurs in Fe- and Ni-base austenitic stainless alloys which are extensively used for structural components in commercial light-water reactors. Yield strength can increase by as much as five times after 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 phases alter rapidly during irradiation. During neutron irradiation at 288°C, the Ni_3Nb γ'' particles present at grain 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 reprecipitate. Matrix hardening is unaffected by disappearance of the γ'', but decreases as the original γ' particles dissolve. Nano-probe compositional measurements with an analytical transmission electron microscope show that the softening coincides with compositional leveling near the particles rather than with the phase disappearances. Similar softening after initial hardening is observed in alloy 718 irradiated at low temperatures (30-60°C) in a mixed spectrum of protons and spallation neutrons. In this case, both the γ' and γ'' disappear after only 0.6 dpa. Delayed softening after the γ' and 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, Metals 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 transcends the recovery Stage V temperature regime (which corresponds to thermal dissociation of small vacancy clusters). In the present study, specimens of pure copper, CuCrZr, Type 316 stainless steel, 9Cr-2WV ferritic martensitic steel, and V-4Cr-4Ti were exposed to eight cycles of periodic temperature variations during neutron irradiation in the High Flux Isotopes Reactor 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 200/350°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. Rest and G.L. Hofman, 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. Consolidation 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/clusters 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 INDUCED BY LOW DOSE IRRADIATION. R. Schaublin, M. Victoria, Fusion Technology - Materials, Centre de Recherches en Physique des Plasmas, Ecole Polytechnique Federale 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 occuring. 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 is carefully studied in the F82H and OPTIMAX steels for doses ranging from about 0.5 dpa to 2 dpa and irradiation temperatures ranging from 310 K to 520 K. In that 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 EXPERIMENTS ON SNS TARGET MATERIALS. L.K. Mansur, T.S. Byun, K. Farrell, 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 fission and fusion reactors, yet different in detail and severity. Higher generation rates of the transmutation gases He and H caused by the GeV-range protons are part of the underlying basis of these differences. For comparison, neutrons in fusion and fission 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 transmutation products are generally also produced at higher rates in spallation target materials than in fission or fusion reactors. As part of the materials R&D program for the Spallation Neutron Source (SNS), calculations and several series of irradiations in different environments are underway to determine the irradiation response. New calculations are described that address displacement, He, H, and solid transmutant production, as well as the additional effects caused by energetic target material knock-ons. Irradiation experiments and results are described. The work is centered at 1.) the spallation sources 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 Fe (displacement-producing) beam and with He and H 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-AC05-00OR22725 with UT-Battelle, LLC.

11:30 AM R1.10
DEFORMATION MECHANISMS IN MARTENSITIC STEEL IRRADIATED IN HFIR. N. Hashimoto, S.J. Zinkle, R.L. Klueh, A.F. Rowcliffe, Oak Ridge National Laboratory, Oak Ridge, TN; K. Shiba, Japan Atomic Energy Research Institute, Ibaraki, JAPAN.

A reduced-activation martensitic steel, 9Cr-2WVTa, developed for fusion energy application was irradiated at 300 and 500°C to 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 macroscopic 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 gage 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 temperature (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-AC05-96OR22464 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
Chairs: Michael L. Jenkins and Robert G. Odette
Monday Afternoon, November 27, 2000
Room 310 (Hynes)

1:30 PM *R2.1
GRAIN BOUNDARY MODIFICATION DURING NEUTRON IRRADIATION AT INTERMEDIATE TEMPERATURES.
S.M. Bruemmer, 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

faulted interstitial loops. A distinct grain boundary denuded zone was observed at low dose that disappeared at higher doses. Interesting differences were detected in Mo-containing alloys, but could not be directly related to RIS 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-AC06-76RLO 1830 with Battelle Memorial Institute and by EPRI.

2:00 PM R2.2
IN SITU MEASUREMENTS OF NEAR-SURFACE Cr REDISTRIBUTION DURING IRRADIATION. L.E. Rehn, P.M. Baldo, L.F. 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 (IASCC), literally hundreds of measurements of Cr depletion 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 Cr redistribution at irradiation temperatures of interest to operating nuclear power reactors, i.e., $<350^{\circ}$C. Furthermore, questions also remain as to the importance of thermal and displacement-cascade processes in creating frequently observed W-shaped Cr profiles. To redress this knowledge gap, we are developing an in-situ ion-beam analysis technique for monitoring Cr redistribution in steels during irradiation. A resonant proton capture reaction (p,γ), with approximately a 1-nm depth resolution, is used to distinguish Cr from the other steel constituents. Examples of Cr segregation profiles obtained both during irradiation at reactor relevant temperatures, and as a result of various thermal treatments, will be presented. The role of various radiation-enhanced and -induced processes in creating W-shaped profiles will also be discussed. This work is supported by DOE-Office of Science under Contract W-31-109-Eng-38.

2:15 PM R2.3
DEFORMATION MECHANISMS AND MICROSTRUCTURES OF IRRADIATED AUSTENITIC STAINLESS STEELS. E.H. Lee, T.S. Byun, J.D. Hunn, L.K. Mansur, 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 fission 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 ion irradiated AISI 316LN austenitic stainless steel by employing a recently developed disk-bend test method and a transmission electron microscopy technique. Irradiation was conducted at 200°C with 360 keV 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. Jogs and stair-rod partials were produced when the leading Shockley partial interacted with a loop, and consequently the trailing partial was separated from the leading partial by jog-pinning and stair-rod 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 unfaulted by interacting with isolated or glide Shockley partials and removed from the channel bands by prismatic glide. Experimental evidence and analyses revealed 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 sponsored by the Division of Materials Sciences 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; J. Ohta, H. Kusanagi, Central Research Institute of Electric Power Industry, Tokyo, JAPAN; E.A. Kenik, Oak Ridge National Laboratory, Oak Ridge, TN.

As part of the shutdown of the EBR-II reactor, structural materials were retrieved to analyze the effect of long term irradiation on mechanical properties and microstructure. In this work, the effect of low dose rate irradiation (10-7 to 10-8 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 EBR-II irradiated at temperatures from 371-390°C to doses up to 30 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 steel, the presence of a grain boundary 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 300°C. E.P. Simonen, D.J. Edwards and S.M. 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 requirement of equal account of the two 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 directly. Comparison of microstructural evolution near grain boundaries with evolution in the grain interior indicates vacancy processes affected by a nearby dominant vacancy sink. Interstitial loop denuded 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 denuded zone edge. This indicates that long-range annihilation of vacancies is not occurring and that the partitioning of defects is on a local scale of less than tens of nm. Post-irradiation annealing offers an opportunity 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-AC06-76RLO 1830.

3:30 PM R2.6
MICROSTRUCTURAL EVOLUTION IN CHARGED PARTICLE IRRADIATED 316 SS MODIFIED TO REDUCE RADIATION DAMAGE. J. Gan, E.P. Simonen, G.W. Was and S.M. Bruemmer; Pacific Northwest National Laboratory, Richland, WA.

Microstructural evolution in austenitic stainless steels plays an important role in irradiation-assisted stress corrosion cracking (IASCC). The objective of this work is to investigate the possible effect of the addition of misfit 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 ($\sim$1wt%) as a lattice perturbation to catalyze defect recombination within the early stage of cascade formation and defect migration. Irradiations were conducted with 5 MeV Ni ions at 500C 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 to 10 dpa at 360C. Microstructures were characterized using transmission electron microscopy. While little effect was seen for Pt addition, Hf appears effectively alter the microstructural response to the irradiation, with lower number density and smaller size for Frank loops. However, the presence of precipitates (Hf and Ni enriched) in 316 Hf which were formed during heat treatment may also play an role in the microstructural evolution. This work supported by the Office of Nuclear Energy, Science & Technology, U.S. Department of Energy, under contract DE-AC06-76RLO 1830.

3:45 PM R2.7
NATURE OF THE FINE-SCALE DEFECTS AND RADIATION HARDENING IN STAINLESS STEELS NEUTRON-IRRADIATED AT 550K. Dan Edwards, 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, our knowledge of radiation effects at temperatures more appropriate for light water reactors near 573K 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 spot" damage, the latter of which is 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 focus of this work is understand the microstructural evolution in a series of 316 and 304 stainless steels irradiated at 550K from 0.7 up to 13 dpa that have been characterized by high resolution and conventional TEM. Lattice imaging, weak beam and rel rod 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 and is constant up to 13 dpa, whereas the size distribution broadens and shifts to larger average sizes as the dose increases. Other defects such as vacancy SFT's 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-AC06-76RLO 1830 and by EPRI.

4:00 PM R2.8
ISOLATION OF RADIATION-INDUCED SEGREGATION IN PROTON-IRRADIATED AUSTENITIC STAINLESS STEELS.
J.T. Busby and G.S. Was, Department of Nuclear Engineering and Radiological Sciences, University of Michigan, Ann Arbor, MI; E.A. Kenik, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN.

Microstructural effects (radiation hardening) and microcompositional effects (radiation-induced segregation (RIS)) have both been identified as potential contributors to irradiation-assisted stress corrosion cracking (IASCC). However, the role of either in IASCC is unclear. Model simulations of post-irradiation annealing indicate that 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 360°C to 1.0 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-annealed condition for anneals up to 600°C for 90 minutes. Annealing at 650°C for 45 minutes removed the irradiation-induced hardness and dislocation population entirely, while only 33% of the as-irradiated RIS remained. However, annealing at 600°C for 90 minutes completely removed the microstructural changes while leaving 83% of the RIS. The rate of removal of both dislocation loops and RIS during post-irradiation annealing 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 IASCC. This work was supported by the U. S. Department of Energy under grant #DE-FG07-99ID13768. Research at the ORNL SHaRE 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 SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities.

4:15 PM R2.9
MICROSTRUCTURE AND POST-IRRADIATION ANNEALING BEHAVIOR OF 20% COLD-WORKED 316 STAINLESS STEEL.
James I. Cole, Todd R. Allen, Argonne National Laboratory- West, Idaho Falls, ID; Hideo Kusanagi, Kenji Dohi, Joji Ohta, Central Research Institute of Electric Power Industry, Komae Research Laboratory, Tokyo, JAPAN.

Microstructural and in situ post-irradiation annealing examinations were carried out on 20% cold-worked 316 stainless steel (ss) hexagonal duct material following irradiation in the reflector region of the EBR-II reactor. Stainless steel hexagonal duct material was used to house reflector subassemblies and provide a valuable source of information on irradiation behavior of reactor structural materials at lower dose-rates (on the order of 5 x 10^{-7} dpa/sec) than previously examined. The microstructure of samples irradiated to doses of 1, 20 and 30 dpa will be detailed, while annealing behavior of samples irradiated to 1 and 30 dpa will be described. Annealing studies were performed in the temperature range of 643 K - 1073 K to examine the kinetics of radiation damage recovery as a function of annealing temperature. The initial (pre-annealed) microstructures consisted of a substantial density of irradiation induced chromium-rich precipitates which formed both on the grain boundaries and within the grain interiors. The majority of these precipitates were M_6C type carbides. Observable radiation damage was negligible in the 1 dpa samples while samples irradiated to 20 and 30 dpa possessed a population of voids and dislocation structures consisting of networks of line dislocations and faulted dislocation loops. The results will be compared to earlier studies at higher dose-rates and evaluated with regards to embrittlement of reactor core internals.

4:30 PM R2.10
ISOLATION OF THE EFFECT OF MICROSTRUCTURE ON IASCC IN PROTON-IRRADIATED AUSTENITIC STAINLESS STEELS.
B.R. Grambau, J.T. Busby, 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 (IASCC), it is necessary to separate microstructure effects (radiation hardening) from microchemistry effects (radiation-induced segregation). Under typical light water reactor (LWR) neutron-irradiation conditions (T=288°C), the microstructure is dominated by faulted dislocation loops which result in hardening and an increase in yield strength. A commercial purity 304 SS, known to be susceptible to IASCC 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 as-irradiated hardness about 40% higher than that caused by irradiation under LWR conditions to 1.0 dpa, an IASCC 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 IASCC susceptibility of the microstructures. Results of microstructure characterization, hardness measurements and IASCC susceptibility will be discussed with regard to the role of microstructure in IASCC. This work was supported by the U. S. Department of Energy under grant #DE-FG07-99ID13768. Research at the ORNL SHaRE 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 SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities.

4:45 PM R2.11
IMPLICATION OF DISORDER-INDUCED POLYMORPHOUS MELTING TO INTERGRANULAR EMBRITTLEMENT IN THE NICKEL-SULFUR SYSTEM.
J.K. Heuer[1,2], P.R. Okamoto[1], N.Q. Lam[1], and J.F. Stubbins[2]; [1]Materials Science Division, Argonne National Laboratory, Argonne, IL; [2]Department of Nuclear, Plasma, and Radiological Engineering, University of Illinois at Urbana-Champaign, Urbana, IL.

While it is known that intergranular embrittlement of Ni by S is strongly influenced by grain boundary segregation, why and how such segregation initiates fracture is still not well understood. This question has been investigated in the Ni-S system by a combination of Auger 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. The 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 segregation-induced intergranular embrittlement, like implantation-induced amorphization, may be a disorder-induced polymorphous melting process, albeit one that occurs locally at grain boundaries. Since the Ni-S phase diagram indicates that the polymorphous melting temperature for the fcc 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 polymorphously to a glass of the same composition below the glass transition temperature. The polymorphous melting surface of the Ni-S-H system provides a similar understanding of the synergistic effect of hydrogen in reducing the critical sulfur concentration for 50% intergranular fracture. A kinetic model for segregation-induced intergranular fracture consistent with the main results of this study is introduced.

SESSION R3: POSTER SESSION
Chairs: Brian D. Wirth and Tomotsugu Sawai
Monday Evening, November 27, 2000
8:00 PM
Exhibition Hall D (Hynes)

R3.1
HIGH ENERGY PROTON IRRADIATION OF PURE TITANIUM. Teresa Leguey, Claude Bailat, Nadine Baluc, Max Victoria, Swiss Federal Institute of Technology - Lausanne, Centre of Research in Plasma Physics, Fusion Technology Materials, Villigen-PSI, SWITZERLAND.

During the past ten years, a number of investigations of radiation damage effects have been performed on fcc and bcc 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 276 (2000) 114]. In order to complement such investigations, a study of the mechanical behavior and microstructural changes of irradiated hcp 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 PIREX (Proton Irradiation Experiment) facility, at the Paul Scherrer Institute, with 590 MeV protons. These irradiations were performed at ambient temperature (300-320 K) and 523 K to doses ranging between 10^{-3} and 1 dpa. The damage rate was approximately 10^{-7} dpa.s^{-1}. The as-annealed and irradiated tensile specimens were deformed at ambient temperature up to fracture. The defects associated with deformation and/or irradiation were imaged in transmission electron microscopy (TEM) by using 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 hcp structure.

R3.2
DIRECT COMPARISON BETWEEN MODELING AND EXPERIMENT: AN α-Fe ION IMPLANTATION STUDY. J. Marian, Brian D. Wirth, P. Sterne, T. Diaz de la Rubia, Lawrence Livermore National Laboratory, Livermore, CA; J.M. Perlado, E. Domínguez, D. Lodi, J. Prieto, Instituto de Fusión Nuclear, Madrid, SPAIN; M. Hernández, G. de Diego, D. Gómez-Briceño, CIEMAT, Madrid, SPAIN; R. Schäublin, CRPP-EPFL, Villigen, SWITZERLAND.

Enormous progress in computational materials science capability, improved resolution of materials science characterization tools and the emergence of techniques for simulating experimental observables based on atomistic input 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 help narrow the gap between theory and experiments. High purity, single-crystal samples have been irradiated with 150 keV Fe ions and characterized with both transmission electron microscopy (TEM) and positron annihilation 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 the observation of TEM visible defects at high dose. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48 and within the VENUS project (CSN/UNESA Coordinated Program) under contract P970530432.

R3.3
Abstract Withdrawn.

R3.4
EFFECT OF HIGH FLUENCES ON THE EMBRITTLEMENT OF LOW COPPER VVER 440 SURVEILLANCE SAMPLES. M.K. Miller, K.F. Russell, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; J. Kocik and E. Keilova, Nuclear Research Institute Rez plc, Rez near Prague, THE CZECH REPUBLIC.

An atom probe tomography microstructural characterization has been performed on low (0.06 at. %) copper surveillance samples from a VVER 440 reactor that were exposed to high fluences in order to evaluate the susceptibility of these pressure vessel steels to embrittlement during service and plant life extension. The 15Kh2MFAA base and 10KhMFT weld specimens were each examined in three conditions: unirradiated, thermally aged (10 and 5 years, respectively at 295°C) and neutron irradiated at 275°C to fluences of 1 x 10^{25} m^{-2} and 5 x 10^{24} m^{-2}, E > 0.5 MeV, respectively. The results from Charpy V-notch 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 ductile-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 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 nanovoids. Some of the spherical features may initially form through 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 Sciences and Engineering, 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 1886-N695-3W with the U.S. Department of Energy.

R3.5
THE INFLUENCE OF PKA DIRECTION ON DISPLACEMENT CASCADE EVOLUTION. Roger E. Stoller, 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 dynamic simulations. More than 300 cascades have been completed at 100K at energies between 100 eV and 100 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 [114] direction. Similar behavior was observed in [121] cascades, but to a lesser degree. Simulations that exhibit this behavior have higher than average point defect survival and higher fractions of the point defects are contained 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 HFIR. N. Hashimoto, R.L. Klueh, J.P. Robertson and A.F. Rowcliffe, Oak Ridge National Laboratory, Oak Ridge, TN.

Two ferritic/martensitic steels based on 12Cr-1MoVW and 9Cr-2VWTa were doped with ^{58}Ni to generate helium via thermal neutron reactions during irradiation in the High Flux Isotope Reactor (HFIR). Control specimens doped with ^{60}Ni, which does not lead to helium productions, were also prepared. TEM examinations showed that before irradiations the lath width of Ni-doped alloys is more narrow and dislocation density tends to be higher compared with undoped alloys. Precipitates in the alloys were mainly $M_{23}C_6$ carbides. The number density of radiation-induced cavities in the Ni-doped alloys was higher than that of undoped alloys due to higher concentration of helium, however, swelling in each alloys was < 0.01% because cavity sizes were <10 nm. Irradiation-induced a_0< 100 > and (a_0/2)< 111 > type dislocation loops were observed in all alloys; the number density and the mean diameter of a_0< 100 > type loops tends to be higher and larger than that of (a_0/2)< 111 > type loops. Also, there was a tendency for the number density of loops in the Ni-doped alloys to be higher than those in undoped alloys. Irradiation-induced precipitates were observed in all alloys, which were identified as $M_6C(\eta)$ type carbide, $\alpha\prime$ and M_2X phase. The $M_6C(\eta)$ type carbides and $\alpha\prime$ 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 is sponsored by the Office of Fusion Energy Sciences, U.S. Department

of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

R3.7
CAVITY FORMATION IN REDUCED ACTIVATION FERRITIC/ MARTENSITIC STEELS IRRADIATED WITH Fe AND He IONS. Akira Naito, Tomotsugu Sawai, Eiichi Wakai, Hiroyasu Tanigawa, Shiro Jitsukawa, Japan Atomic Energy Research Institute, Dept. of Material Science, Ibaraki, JAPAN; Akira Kohyama, Kyoto Univ., Institute of Advanced Energy, Kyoto, JAPAN.

Reduced activation ferritic/martensitic steels have been considered as the potential materials 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 F82H (Fe-8Cr-2W-0.2V-0.04Ta), JLF-1 (Fe-9Cr-2W-0.2V-0.08Ta) and HT9 (Fe-12Cr-1Mo) are ion-irradiated. 11.3 MeV Fe3 ions with and without 1.2 MeV He ions were irradiated at temperature ranging from 673 to 773 K. Displacement damage level and implanted helium level were 60 dpa and 1200 appm at peaks located at the depth of 1900 nm, respectively. A focused ion beam (FIB) processing system, FB-2000A 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 at the damage peak in specimens irradiated with dual ions. The swelling behavior and its temperature dependence are different in three steels. F82H showed the highest swelling resistance, and the largest swelling, 0.2 %, was observed at 773 K. The maximum cavity diameter in this condition reaches 20 nm. The largest swelling, 0.8 %, was observed in JLF-1 irradiated at 723 K, which contained large cavities up to 50 nm.

R3.8
POINT DEFECTS OBSERVED IN DT-NEUTRON IRRADIATED COPPER, SILVER AND GOLD AT 15°C WITH A ROTATING TARGET IN FNS JAERI. Y. Shimomura, K. Sugio, H. Ohkubo, I. Mukouda, 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 generation has 25 cm in diameter, which is smaller than previous RTNS-II in LLNL. We carried out two irradiation experiments in 1999. In these irradiations, 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^{14} to 1 x 10^{17} n/cm^2. Therefore the present experiment is complementally to the previous RTNS-II irradiation in which majority of experiments were carried out in the fluence range between 5 x 10^{18} to 1 x 10^{17} n/cm^2. By TEM observation, we observed both of interstitial cluster and vacancy cluster in Ag, Au 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 defect clusters. In 2000, four irradiation experiments are scheduled. Two are high temperature irradiation at 300°C and two are temperature-controlled irradiation at -50°C and 100°C.

R3.9
RADIATION EFFECTS ON THE PLASTICITY AND MICROSTRUCTURE OF Ti-Al-V ALLOYS CONTAINING β PHASE. T. Sawai, E. Wakai, A. Hishinuma, Japan Atomic Energy Research Insitute, Tokai-mura, JAPAN; M. Tabuchi, Hakodate National College of Technology, Hakodate, JAPAN.

Ti-Al-V intermetallic alloys containing more than 10at% 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 above 873 K. Ti-35Al-15V alloy has a microstructure mostly consisting of β phase and its elongation abruptly increases to 60% from 10% around 893 K. Transmission electron microscope (TEM) observation revealed a characteristic microstructure in deformed tensile specimens which showed high elongation. Many deformation bands were observed in deformed β grains. These bands consist of hexagonal $\alpha 2$ phase grains and β phase grains of two different orientaions. The orientation relationship of hexagonal $\alpha 2$ phase and β phase (matrix and those in deformation bands) is (101)b//(0001)h and [111]b//[1210]h. This suggests the instability of β phase and martensitic transformation during deformation. Ti-30Al-10V alloy has a microstructure containing three phases; $\alpha 2$, β and γ. Its tensile elongation is more than 6% at 673 K and 60% at 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 specimen 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.

R3.10
THE DEFECT MICROSTRUCTURE OF V-4Cr-4Ti ALLOY FOLLOWING NEUTRON IRRADIATION AT 323°C. D.T. Hoelzer, 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 reactors due to their attractive combination of good thermal conductivity and mechanical properties as well as a low 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-bonded BOR-60 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 data showed that considerable radiation hardening with an accompanying reduction in strain hardening capacity had occurred in the irradiated samples. The TEM analysis showed that these property changes occurred with the concomitant formation of defect structures that consisted of dislocation loops and Ti-enriched 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-AC05-00OR22725 with U.T.-Battelle, LLC.

R3.11
DYNAMICAL BEHAVIOR OF VOIDS IN NEUTRON-IRRADIATED COPPER AND NICKEL AT ELEVATED TEMPERATURE. I. Mukouda 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 573K to damage level between 10^{-4} to 10^{-3} dpa. Annealing temperature was 523K 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 523K annealing but SFTs were not changed. We observed 37 voids and 8 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 and 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 573K. It is concluded that void moved with dynamical structural relaxation at elevated temperature.

R3.12
THE EFFECT OF BULK COMPOSITION ON SWELLING AND RADIATION-INDUCED SEGREGATION IN AUSTENITIC ALLOYS. T.R. Allen, J.I. Cole, N.L. Dietz, Argonne National Laboratory-West, Idaho Falls, ID; Y. Wang, G.S. Was, University of Michigan, Ann Arbor, MI; E.A. Kenik, Oak Ridge National Laboratory, Oak Ridge, TN.

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 major element or impurity element concentrations can be used to reduce grain boundary chromium depletion, thus reducing susceptibility to environmental cracking. Variations in composition can also produce materials with increased resistance to void swelling. In this work, select solutes have been added to an Fe-18Cr-8Ni alloy (bulk composition corresponding to 304 stainless steel) to identify the effect of composition on microstructural and microchemical development. 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 316 stainless steel, and Fe-18Cr-8Ni-Zr 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 determine the effect of composition change on grain boundary segregation and to relate microchemical changes to swelling resistance.

R3.13
RADIATION HARDENING OF VANADIUM ALLOYS. Manabu Satou, Tohoku Univ, Dept of QSE, Sendai, JAPAN.

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 below 400C. The gaseous impurities could form Cottrell atmosphere and cause large resistance to start moving dislocations. The radiation defects could be strengthened by the impurities against moving dislocations as Orowan type obstacle. The microstructures depend on the concentration of the impurities, since the impurity could affect nucleation and growth of the radiation defect. In this paper, increasing in yield stress, changing of work-hardening and microstructures in vanadium alloys after irradiation are summarized. Radiation hardening of vanadium alloys 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. G.R. Odette, C. Cowen and D. Gragg, University of California Santa Barbara, CA, Department of Mechanical and Environmental Engineering, Santa Barbara, CA.

A number of complementary techniques are required to characterize the evolution of nanoscale features under irradiation. These typically include small angle neutron (SANS) and anomalous x-ray (ASAXS) 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 electric 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, ASAXS and ATP to achieve self-consistent mass balances of copper partitioned between precipitate and in-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 two solutes species. Temperature dependent 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 measurments in magnetic fields and use of temperature dependent TEP measurements can extend partitioning assessments to additional elements. Examples of use of using 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. M.K. Miller, K.F. Russell, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; P. Pareige, Groupe de Physique des Matériaux, UMR CNRS 6634, Faculté des Sciences et INSA de Rouen, Mont Saint Aignan, FRANCE.

An atom probe tomography study has been performed to estimate the matrix composition, and the size, composition and number density of the ultrafine copper-enriched precipitates that formed under neutron irradiation in a submerged arc weld from the HSSI fifth irradiation series (Weld 73W). The composition of this weld is Fe- 0.27 at. % Cu, 1.58% Mn, 0.57% Ni, 0.34% Mo, 0.27% Cr, 0.58% Si, 0.003% V, 0.45% C, 0.009% P, and 0.009% S. The material was examined after a typical stress relief treatment of 40 h at 607°C (U), after neutron irradiation to a fluence of 1.8 x 10^{23} n m^{-2} (E > 1 MeV) [I] at a temperature of 288°C, after irradiation and thermal annealing for 168 h at 454°C [IA], and after irradiation, thermal annealing and re-irradiation to an additional fluence of 0.8 x 10^{23} n 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 454°C. These precipitates were present after re-irradiation and some additional subnanometer diameter copper-enriched precipitates were also observed. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, 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 1886-N695-3W with the U.S. Department of Energy.

R3.16
PROTON IRRADIATION OF ZEOLITE-Y. Binxi Gu, Lumin Wang, Shixin Wang, Donggao Zhao, Rodney. C. Ewing and Victor H. Rotberg[a], Dept. of Nuclear Engineering and Radiological Sciences, [a]Michigan Ion Beam Laboratory, Univ. of Michigan, Ann Arbor, MI.

Radiation effects on zeolite-NaY have been investigated by irradiation with a 500 keV H^+ beam. Zeolite-NaY suffers structural damage under proton irradiation. The crystalline-to-amorphous transition occurs at a total dose equivalent to an ionizing energy deposition of 3×10^{10} Gy and a displacement dose of 0.01 dpa. The ion exchange capacity of the irradiated zeolite-NaY with 10 mN CsCl solution varies with the extent of the damage to the crystalline structure. After 25 hours of exchange, the Cs 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 change in the ion exchange capacity are discussed.

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

Brannerite, ideally UTi_2O_6, occurs in polyphase ceramics that are considered for plutonium immobilization. In order to investigate radiation effects caused by α of Pu, several brannerite compositions were synthesized: UTi_2O_6, $ThTi_2O_6$ and a polyphase material, which is composed of Ca-containing brannerite and pyrochlore. A 1 MeV Kr^2 irradiation was performed using IVEM-Tandem facility at Argonne National Laboratory. The transformation from the crystalline to the amorphous state was observed in situ as a function of ion dose. At room temperature, complete amorphization of UTi_2O_6 and $ThTi_2O_6$ occurred at $1.50 \pm 0.06 \times 10^{18}$ and $9.40 \pm 0.12 \times 10^{17}$ ions/m^2, respectively. The critical amorphization dose (D_c) increases with irradiation temperature. Complete amorphization of UTi_2O_6 did not occur at 973K, and the critical amorphization temperature for $ThTi_2O_6$ irradiated by 1 MeV Kr^2 is ~993K. Ca-containing brannerite is more easily amorphized than the pyrochlore phase, and the critical amorphization dose at room temperature is $1.38 \pm 0.06\times 10^{18}$ ions/m^2. The radiation resistance at room temperature increases as follows: D_c ($ThTi_2O_6$) < D_c (Ca-containing brannerite) < D_c (UTi_2O_6). The effects of composition and structure on radiation damage in brannerite and pyrochlore type ceramics are discussed.

R3.18
TOPOLOGICAL AND MOLECULAR DYNAMICS MODELING OF IRRADIATION-AMORPHIZED BETA-SiC. Xianglong Yuan, MIT, Dept of Materials Science & Engineering, Cambridge, MA; Vinay Pulim, MIT, Lab for Computer Science, Cambridge, MA; Linn W.

Hobbs, MIT, Dept of Materials Science & Engineering, Cambridge, MA.

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 Frenkel pairs and antisite defects in the crystalline-to-amorphous transition have been explored. It was found that surrounding crystalline structure plays an important role in the recovery of amorphized structures, which can evolve as quite topologically ordered but still chemically disordered. A fully recovered crystalline structure was not observed.

R3.19
MICROSTRUCTURE OF SWIFT HEAVY ION IRRADIATED SiC, Si_3N_4 AND AlN. S.J. Zinkle and J.W. Jones, Oak Ridge National Laboratory, Oak Ridge, TN; V.A. Skuratov, Joint Institute for Nuclear Research, Flerov Laboratory of Nuclear Reactions, Center of Applied Physics, Dubna, Moscow Region, RUSSIA.

Several studies have shown that swift heavy ions with electronic stopping powers >10-15 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, Si_3N_4 or AlN following 330 MeV Kr ion irradiation (dE/dx$\sim$16 keV/nm). These three materials have been irradiated with 710 MeV Bi ions at fluences of 1e12 and 1e13/cm^2 (single track regime) in order to determine if track formation can be induced at electronic stopping power values >25 keV/nm. The microstructures were examined using plan view and cross section transmission electron microscopy. The results will be compared with predictions from existing swift heavy ion irradiation models.

R3.20
MOLECULAR DYNAMIC SIMULATION OF CASCADE OVERLAP AND AMORPHIZATION IN BETA-SiC. Fei Gao, William Weber, Pacific Northwest National Laboratory, Richland, WA.

Although amorphization in SiC irradiated with ions has been investigated extensively using a wide range of techniques, the dynamics of the amorphization process are still not completely understood. Molecular dynamics (MD) has been employed to study cascade overlap and defect accumulation from a low dose level to complete amorphization in beta-SiC. A large number of 10 keV displacement cascades were randomly generated in a model crystal to achieve the amorphous state, and the corresponding dose is consistent with the experimental observations. The results show that the most 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 comparable 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 Frenkel pairs and antisite defects. The present results are discussed in terms of experimental observations and compared with models for irradiation-induced amorphization.

R3.21
THERMODYNAMIC ASPECTS OF SOLUTE MOTION IN DILUTE ALLOYS UNDER IRRADIATION. A. Caro, E. Lopasso, M. Caro, Centro Atomico Bariloche, Bariloche, ARGENTINA; M.J. Caturla, T. Diaz de la Rubia, Lawrence Livermore National Laboratory, Livermore, CA.

With the progress of computational physics, numerical simulations have contributed substantially to our understanding of the effects of irradiation on pure elements and alloy. 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 address a simple question: given an arbitrary dilute alloy, what would be the effect of ion beam irradiation at low temperature, i.e. would the zones affected by cascades be richer or poorer in solute? At least four different regimes are expected to contribute to the final result. i- In the ballistic stage, ii- In the early stages of the heat spike, when the core of the cascade is liquid, thermomigration induced by the huge thermal gradient may induce solute transport, this is the Soret effect. iii- In the re-solidification phase, the crossing of a two phase field delimited by the solidus-liquidus lines may induce compositional changes. Finally, iv- in the solid phase, the quenched core of the cascade may have a composition different from the equilibrium and solid state diffusion can help reaching it. This last regime is not considered in our study by assuming low temperature irradiation. In this paper we focus in particular in thermomigration and solidification by performing computer simulations of heat 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 and solidification may play unexpected roles and that for some materials solute motion may be significant.

R3.22
COMPUTER SIMULATION OF ENERGY DEPENDENCE 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 (PKA) corresponding to recoils with energies in the range of 0.25-150 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 melting does not occur, the defect arrangements are highly dispersed, and the tendency for defects to form clusters is much less compared to the case of metals. C Frenkel pairs are more numerous than Si Frenkel pairs and anti-site defects are part of the primary damage. The number of anti-site defects is small for energies below 1 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
ATOMISTIC MECHANISM OF NUCLEATION AND GROWTH OF VOIDS IN Cu STUDIED BY COMPUTER SIMULATION. Y. Shimomura and I. Mukouda, Faculty of Engineering, Hiroshima University, JAPAN.

We reported previously [1] an experimental result which shows that a vacancy cluster whose size in sma1ler than 300 vacancies relaxes to stacking fault tetrahedron and greater than 400 vacancies relaxes to a void in neutron-irradiated copper at 300°C. To study the detailed atomistic mechanism of nucleation and growth of sft and void in copper, we carried out the computer simulation of molecular dynamics. MD simulation was carried out with an empirical isotropic potential of embedded atom (EAM) method due to Daw and Baskes [2]. A model crystal is composed of 32000 Cu atoms. A crystal was reached at first to a thermal equilibrium at 1000 K by running MD. Then various numbers of vacancies was introduced in crystal as dispersed small clusters and the crystal was continued to relax a structure by running MD. It was found that a vacancy cluster whose size is smaller 150 vacancies relaxes to sft and that is greater than 500 vacancies are relaxes to a void at high temperature as 1000 K. We present what is the basic mechanism to lead to sft and void formation. [1] Y. Shimomura and I. Mukouda, J. Nucl.Mater. (2000) in press. [2] M.S. Daw and M.I. Baskes, Phys. Rev. B29 (1984) 6443.

R3.24
MOLECULAR DYNAMICS SIMULATIONS OF DISPLACEMENT CASCADES: ROLE OF THE INTERATOMIC POTENTIALS AND OF THE POTENTIAL HARDENING. C.S. Becquart, C. Domain and A. Legris.

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 parts of the potential 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 and potential energy transfers during the collisions have also been studied.

R3.25
KINETIC MONTE CARLO SIMULATIONS OF CASCADES IN Fe ALLOYS. C. Domain, J.C. Van Duysen, EDF-DRD, Département Etude des Matériaux, Moret sur Loing, FRANCE; C.S. Becquart,

Laboratoire de Métallurgie Physique et Génie des Matériaux, UMR 8517, Université de Lille 1, Villeneuve d'Ascq, FRANCE.

The steel vessel of PWR are embrittled by neutron irradiation. Among the solute atoms, copper play an important role in the embrittlement and different Cu rich defects have been experimentally observed to form. We have investigated by Kinetic Monte Carlo (KMC) methods on rigid lattices the evolution of the primary damage. The point defects created by the displacement cascades having very different kinetics, their evolution was tracked in two steps. In a first part, we have studied their recombination in the cascade region and the formation of interstitials clusters using "object diffusion" of interstitials and vacancies in iron. The parameters of this model were based on MD simulations with EAM potentials. The influence of impurity atoms was investigated. Data needed about Fe alloys such as the binding energies of point defects with solute atoms or impurities were evaluated or checked using first principles calculations (based on the density functional theory). In a second part, we have investigated the evolution of the primary damage and the formation of some copper objects using a simple model for the vacancy diffusion mechanism. These simulations which also rely on adapted EAM potentials show the formation of copper rich defects. Some properties of the potentials playing a key role in the above model were checked by ab initio methods. The defects resulting from the KMC simulations are subsequent to the primary damage resulting from the displacement cascades, and they exhibit similarities with the ones observed by atom probe.

R3.26
NEW INSIGHTS TO HEAVY-ION RADIATION DAMAGE IN HIGH-Z METALS. M.J. Fluss, B.D. Wirth, M. Wall, T. Diaz de la Rubia, and T.E. Felter, Lawrence Livermore National Laboratory, Livermore, CA.

Because of their radiolytic decay (e.g., Pu decays by a ~5 MeV alpha and a ~85 keV U), actinides provide a unique model system for investigating the source term for heavy-ion damage accumulation and comparing to light-ion damage from protons. Earlier self-damage studies of Pu metal and alloys have shown evidence of annealing stages commonly observed for many pure fcc metals such as Al, Ag, Au, Ni, Pb or Cu [1]. We have recently undertaken low temperature proton irradiation and isochronal annealing experiments of actinides, which are compared with in situ self-irradiation studies. These experiments, including their annealing protocols, are well described by combined molecular dynamics and kinetic Monte Carlo modeling. Through this comparison of experiments (self-damage and protons) and computations (MD and KMC) it is now clear that the collapsed vacancy and interstitial loops resulting from the U damage cascade are evident in the details of the annealing data. On comparison to MeV proton induced damage in the same actinide alloy specimens, self-damage, primarily from heavy ion recoils, exhibits a sharper stage-I, no stage-II and a "delayed" stage V. 1. S.S. Hecker and J. Martz, LA-UR-99-2751, to be published in the Proceedings of The International Conference on Ageing Studies and Lifetime Extension of Materials, Oxford, United Kingdom July 12-14, 1999 and references therein. 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.

R3.27
ATOMISTIC SIMULATION OF DISLOCATION-DEFECT INTERACTIONS IN Cu. B.D. Wirth, V.V. Bulatov, T. Diaz de la Rubia, Lawrence Livermore National Laboratory, Livermore, CA.

The mechanisms of dislocation-defect interactions are of practical importance to developing quantitative structure-property relationships, mechanistic understanding of plastic flow localization and predictive models of mechanical behavior in metals under irradiation. In Copper and other face centered cubic metals, high-energy particle irradiation produces hardening and shear localization. Post-irradiation microstructural examination in Cu reveals that irradiation has produced a high number density of nanometer sized stacking fault tetrahedra. Thus, the resultant irradiation hardening and shear localization is commonly attributed to the interaction between stacking fault tetrahedra and mobile dislocations, although the mechanism of this interaction is unknown. In this work, we present a comprehensive molecular dynamics simulation study that characterizes the interaction and fate of moving dislocations with stacking fault tetrahedra in Cu using an EAM interatomic potential. This work is intended to produce atomistic input into dislocation dynamics simulations of plastic flow localization in irradiated materials. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

R3.28
VOID LATTICE FORMATION IN METALS UNDER CASCADE DAMAGE CONDITIONS. Alexander V. Barashev, Stanislav I. Golubov, 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 one-dimensionally 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 far from equilibrium (e.g. lane formation 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 vacancies may play in establishing perfect lattices is discussed.

R3.29
STATISTICAL ANALYSIS OF DEFECT PRODUCTION DUE TO 50KEV CASCADES IN BCC-Fe. Naoki Soneda, Central Research Institute of Electric Power Industry (CRIEPI), Materials Science Dept, Tokyo, JAPAN; Shiori 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 PKA energy cascade events is very important because the average PKA 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 can not derive an overall 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 the recognition of the important role of SIA clusters in microstructure 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 inhomogeneous damage near grain boundaries, decoration of dislocations by interstitial dislocation loops, rafts 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, where 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 30 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 $< 11\bar{2}0 >$ directions. Smaller clusters ($\leq$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 drown 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 Laikhtman and Alon Hoffman, Department of Chemistry, Technion - Israel Institute of Technology, Haifa, ISRAEL.

In the present study we correlate between the secondary electron emission (SEE) properties of variously treated Xe^+ ion damaged diamond films and their bonding structure in 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 2×10^{15} cm^{-2} results in the transformation of near-surface diamond to sp^2-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 treatment does not induce desorption of chemisorbed hydrogen. Exposure of the ion bombarded films to microwave (MW) hydrogen plasma treatment for 30 minutes produces negative electron affinity diamond surface, but only partially regenerates SEE properties, retains some imperfection in the near surface atomic layers, as 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 990°C results in desorption of the surface hydrogen and formation of positive electron affinity surface leading to drastic degradation of the electron emission properties. Prolonged, up to 3 hours MW hydrogen plasma treatment of as-implanted diamond films gradually improves the crystal quality and results in further increase of SEE intensity. This treatment does not, however, substantially reduces the concentration of oxygen in the previously damaged diamond, indicating its bulk diffusion during ion bombardment.

R3.32
INFLUENCE OF PRELIMINARY THERMORADIATION TREATMENT UPON EFFICIENCY OF FORMATION AND ANNEALING OF DEFECTS IN SILICON STRUCTURES IRRADIATED BY GAMMA-RAYS. Sh. Makhkamov, 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 capacitance spectroscopy (DLTS) method in silicon p-n-structures normalized by thermoradiation treatment, which were subjected to gamma ray irradiation. To normalize electrophysical parameters of silicon p-n-structures prepared from n-type silicon with resistivity of 0.3-10 Ohm cm the neutron irradiation with fluences of $5x10^{16}$-$5x10^{17}$ cm^{-2} followed annealing at 700°C was carried out. It was shown that under certain normalization conditions the initial electrophysical parameters of 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 2-2.5 times higher than that in the non-normalized samples. As to thermostability of RD, 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.

R3.33
PHOTO STRUCTURAL REORGANIZATION IN HYDRO-GENATED AMORPHOUS SILICON (a-Si:H) LAYERS UNDER THE ACTION CONCENTRATED SOLAR RADIATION OF HIGH DENSITY. Rustam R. Kabulov, 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 [2,3]. This models do not take into account change of concentration of complexes Si-H and $Si-H_2$ bonds. For experimental check of this fact the influence of the concentrated solar radiation of high density on vibrational spectrum of Si-H and $Si-H_2$ bonds in intrinsic a-Si:H films were investigated. The researches electron paramagnetic resonance in degradated a-Si:H films have shown, that the probability of formation of the dangling bonds under the action of illumination low and makes 10^{-8} spin/quantums. Hence for supervision of changes in IR-spectrum vibrational modes it is necessery to shine a sample by light by intensity not below 10^{19}-10^{20} quantums/sm^2*s in current [5] not less than 1 hour. Vibrational spectra of a-Si:H were investigated on a technique IR-spectroscopy of the repeatedly broken complete internal reflection (RBCIR) [4] in the field of 1900-2300 sm^{-1}, where are bond stretching modes of Si-H (2000 sm^{-1}) and $Si-H_2$ (2100 sm^{-1}). The given method has allowed to investigate a-Si:H layers, which thickness is close to thickness of an i-layer in real p-i-n-a-Si:H photocells. As the photo induced degradation is accumulating process, the exposition 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 density up to 300 kW/m^2 during 2 hours has not given essential change in IR- spectrum of absorption. With increase of density of solar radiation up to 400 kW/m^2 the absorption in area of valent vibration 2000 sm^{-1} monotonously decreases and at the further increase of time of an exposition with 45 up to 120 minutes of changes in a RBCIR spectrum was not observed. At density of solar radiation 600 kW/m^2 the film on a surface of a prism completily collapses, to what the absence of peak of valent vibration in IR-spectrum RBCIR testifies. From the received experimental results follows, that under action of solar radiation the concentration of Si-H bonds decreases, while the concentration of $Si-H_2$ bonds essentially does not change. It is known, that the energy of connection of $Si-H_2$ bonds is less, than the energy of connection of Si-H bonds. Therefore it is more probable than everything, from the point of view of energy of break, owe is stronger will be destroyed $Si-H_2$ bonds. The received experimental results give the bases for deeper theoretical research of model of formation of a photo induced defects in view of power features of break of all complexes of bonds. [1]. Staebler D.L., Wronski C.R., J.Appl. Phys. 1980. V. 51. P. 3262. [2]. Adler D., Solar Cells. 1983. V.9. P. 133. [3]. Stutzman M., Jackson W.B., Tsai C.C., Appl. Phys. Lett. 1984. V.45. N.10. P.1075. [4]. Harrick N.J. 1967, Internal reflected spectroscopy, New York: Interscience Publishers.

R3.34
MICROSTRUCTURAL PROCESSES IN GAMMA-IRRADIATED AMORPHOUS CHALCOGENIDE SEMICONDUCTORS. Oleh I. Shpotyuk, Institute of Materials, Scientific Research Company "Carat", Lviv, UKRAINE; Serhiy A. Holub, Sensor Ltd., Drohobych, UKRAINE.

The influence of gamma-irradiation with doses 1 MGy - 4.4 MGy, energy of gamma-flow 1.25 MeV, power of exposure dose 20 Gy/s on the defect formation processes in bulk ternary chalcogenide glasses of As-S-Ge, Sb-S-Ge, As-Se-Ge, As-Se-Bi systems is investigated by the far IR spectrometry method (500-200 1/cm). It is shown that redistribution of chemical bonds and defects takes place after gamma-irradiation. Irradiation causes the increasing of "unregular" homopolar bonds concentration. These processes depends 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 Z=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 band gap the levels connected with radiation-induced defect centers. The dynamic and static components of radiation-induced optical effects are revealed in chalcogenide glasses. These components 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.

R3.35
NEUTRON IRRADIATION OF DIAMOND. K. Jagannadham, Dept of MS&E; K. Verghese, Nuclear Engineering, N.C. 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 Li-7 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 presented.

R3.36
CHARACTERIZATION OF PHOTO-INDUCED STRUCTURAL CHANGES IN AsSe GLASS USING X-RAY PHOTOELECTRON SPECTROSCOPY. S. Krishnaswami, J. Bloking, H. Jain, Lehigh University, Bethlehem, PA; P. Krecmer, S.R. Elliott, University of Cambridge, Cambridge, UNITED KINGDOM; M. Vlcek, University of Pardubice, Pardubice, CZECH REPUBLIC; J. Li, D.A. Drabold, Ohio University, Athens, OH.

Amorphous chalcogenides, in general, exhibit a wide variety of changes in their structure and physical properties, particularly when exposed to bandgap illumination. The photons affect the volume, amorphization/devitrification, mechanical (e.g. hardness, plasticity), rheological (e.g. viscosity), optical (e.g. darkening, birefringence, luminescence), electrical (e.g. conductivity, dielectric constant), or the chemical (e.g. etching, dissolution) properties of the glass. These

photoinduced changes are classified into three categories depending on their stability: (a) permanent changes which cannot be recovered without destroying the sample; (b) metastable changes 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 that may be used for converting an optical signal into some other signal. In this work, we have characterized the structure of amorphous arsenic selenide films and bulk samples under conditions of He-Ne laser and Na lamp light irradiation using high resolution x-ray photoelectron spectroscopy (XPS). After irradiation, we find that As/Se ratio decreases indicating that the surface of AsSe becomes irreversibly enriched with Se, and a fraction of As-As bonds transforms into As-Se bonds. In addition, there are reversible changes in the valence band, which include transformation of the lone-pair electrons on Se into new bonding states that are present only under illumination. Preliminary results also indicate an enhancement in the diffusion of the constituent atoms due to light illumination. We also are modeling these short-time photo-structural effects by excited state quantum molecular dynamics. We have proposed realistic models of a-Se, g-$GeSe_2$, and g-As_2Se_3 and fully characterized their structure, dynamics and electronic properties. We are also developing a model of a-AsSe for comparison to OME. By linking these models with the associated photo-structural response, one can have atomistic understanding of many light-induced processes in chalcogenide glasses.

R3.37
FORMATION OF ELECTRICALLY ACTIVE DEFECTS IN Cz AND Fz SILICON WAFERS BY 3 MEV PROTON IRRADIATION. Min-Doo Chun, Donghwan Kim, Joo-Youl 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 $1.0\times10^{13}cm^{-2}$ and $3.0\times10^{13}cm^{-2}$, but not in the Fz wafers even at doses as high as $2.0\times10^{15}cm^{-2}$. The 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 diffractometry 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 Cheylan 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 the luminescence intensity from the nanocrystals 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_2 layers were implanted at room temperature with 400 keV Si ions to doses in the range from $1x10^{17}cm^{-2}$ to $6x10^{17}cm^{-2}$, corresponding to peak concentrations in the range from 5 to 30 at. %. 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. Annette R. Dowd and Robert G. Elliman, Electronic Materials Engineering Department, RSPhysSE, 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 syntheising such materials is by ion-implantation and annealing, in which the implanted elements are precipitated to form small crystallites. 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 ~196°C with 1.0 MeV Ge ions to fluences in the range from 0.6 to $3x10^{17}$ $Ge.cm^{-2}$. As-implanted Ge distributions were approximately Guassian, as measured by Rutherford backscattering spectrometry, 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 -196°C with $0.6x10^{17}$ $Ge.cm^{-2}$, showed no Ge clustering. After annealing in an Ar:H2 (95%:5%) ambient at 1100°C for 60 minutes, the size distribution of the nanocrystals with much bigger nanocrystals being observed on the near-surface side of the Ge profile than might be predicted from the Ge distribution alone. This behaviour is discussed in terms beam induced nucleation of nanocrystals.

R3.40
SIZE DEPENDENCE OF DAMAGE FORMATION BY CLUSTER ION IMPACT. Takaaki Aoki, Shun-ichi Chiba, Jiro Matsuo, Gikan H. Takaoka, Kyoto Univ., Ion Beam Engineering Experimental Lab., Kyoto JAPAN.

Cluster ion irradiation shows characteristic phenomena in damage formation compared with conventional monomer 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 energy of 10keV, the cluster penetrates the surface though each atom has only 10eV. 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 substrate.
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-per-atom cluster, i.e. the size of 10 and energy of 1keV/atom, each cluster atom penetrates into the deeper region of the substrate without multiple-collision, which cause cascade-like displacements similar to monomer ions. On the other hand, at the impact of large and low energy-per-atom, such as size of 10000 with about 1eV/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 monomer 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. Litovchenko, V.P. Melnik, A.G. Rozhin, Institute of Semiconductor Physics, Ukrainian National Academy of Sciences, Kiev, UKRAINE; R.L. Politansky, Chernivtsi State University, Chernivtsy, 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 by isovalent impurities (C, Si, 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 implanted 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 FILMS AFTER POST-GROWTH LOW ENERGY ION BEAM IRRADIATION. P. Patsalas, S. Logothetidis, Aristotle Univ, Dept of Physics, Thessaloniki, GREECE.

In this work, we present the crystallization effects occurring in sputtered amorphous 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 Ellispometry (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 Tauc-Lorentz description of the dielectric function and the Bruggeman 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, sp^3/sp^2 bond ratio) of the as grown films, as they have been 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 ($\sim 2x10^{16}$ ions/cm^2) of ions with an optimum ion energy ($\sim$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 low energy IBI. The combined XRD/XRR results are considered in terms of fundamental ion-solid interaction mechanisms such as implantation damage, relocation recoils, electric charging and heterogeneous nucleation in an effort to get a deeper insight on the crystallization 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 SiO_2 IRRADIATED BY ELECTRON BEAM. Kazuya Oguri, Tsuyoshi Sunamoto, Yoshitake Nishi, Dept of Materials Science, Tokai Univ., JAPAN; Keisuke Sato, Tomio Izumi, Dept of Electronics, Tokai Univ., JAPAN.

From an 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 atoms. The sheet electron beam irradiation was homogeneously performed by an electron curtain processor. Thus, we have studied influence of aging on density of dangling bond on SiO_2 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 kHz. Spin density was calculated using a Mn^2 standard sample. The aging decreased the density of dangling bond on SiO_2. Based on rate process, the density change (X) with the aging time (t) was expressed by a following equation. $X = 1 - \exp(-kt^n)$ Furthermore, the apparent activation energy was calculated.

R3.44
Abstract Withdrawn.

R3.45
REFINEMENT OF TOPOLOGICALLY MODELED CASCADE-AMORPHIZED SILICAS USING MOLECULAR DYNAMICS SIMULATION. Xianglong Yuan, MIT, Dept of Materials Science & Engineering, Cambridge, MA; Vinay Pulim, MIT, Lab for Computer Science, Cambridge, MA; Linn W. Hobbs, MIT, Dept of Materials Science & Engineering, Cambridge, MA.

Cascade-amorphized silica structures modeled by a topological modeling technique have been refined using molecular dynamics (MD) simulation. For refined silicas generated by different cascade procedures, great similarity in total correlation function was observed. While similarly stable energetically, these different refined silicas present substantial differences in medium-range order, which are efficiently described using the concept of primitive ring content. The distinctiveness in their primitive ring statistics disappears as the MD equilibration temperature exceeds 3000 K. Strong correlation was observed between these primitive ring statistics and the first sharp diffraction peak in simulated diffraction patterns, the implications of which are highlighted in a comparison to experimental electron diffraction results for metamict quartz amorphized by different radiation types.

R3.46
IN-SITU AMORPHIZATION AND RECRYSTALLIZATION OF TITANITE. Alkiviathes Meldrum, Univ of Alberta, Dept of Physics, Edmonton, AB, CANADA.

Titanite ($CaTiSiO_5$) is a widely occurring accessory mineral that typically contains ppm-level concentrations of U Th substituting for Ca. Radiation effects in titanite are important because this phase is commonly-used for U-Pb age dating of rock formations, and it is also the main crystalline constituent of some glass-ceramic nuclear waste forms. Previous work suggested that titanite is highly susceptible to natural alpha-decay-induced amorphization. Ion irradiation experiments have so far been reported only at room temperature. Although titanite was reported to have a high cross section for ion damage, other results indicated that the amorphization dose is similar to those measured for other complex silicate minerals. In this work, the first temperature-dependent amorphization data for titanite are reported. High-purity single-crystal specimens from the Khan Mine, Namibia, generously provided by Dr. L. Heaman, were characterized by analytical electron microscopy and powder XRD. The specimens contained no detectable U Th impurities, and no evidence for pre-existing radiation damage was found. TEM specimens were then irradiated in-situ at the IVEM Facility using 800 keV Kr ions or 150 keV He ions at temperatures ranging from 30 to 973 K. Conventional bright-field and dark-field imaging and electron-diffraction techniques were used to monitor the transformation to the amorphous state. Titanite was amorphized at a relatively low dose over the entire temperature range investigated. Electron-diffraction patterns provided strong evidence that the structure of the amorphous state is different for the two types of incident ion. These results may explain recent single-crystal XRD data in favor of two distinct phases in alpha-decay-damaged natural titanite. Limited crystallization of ion-beam-amorphized titanite was observed at temperatures between 973 and 1173 K. Relatively widely dispersed precipitates, tentatively identified as a rare phase of TiO, nucleated in the amorphous matrix and grew to a maximum diameter of $\sim$30 nm. The results of these experiments are discussed in light of recent studies of alpha-decay damage in natural specimens.

SESSION R4: THEORY AND MODELING
Chairs: Louis K. Mansur and Yoshiharu Shimomura
Tuesday Morning, November 28, 2000
Room 310 (Hynes)

8:30 AM R4.1
ANALYTICAL AND MONTE CARLO STUDIES OF THE EFFECTS OF DIRECTION CHANGES ON THE DEFECT REACTION KINETICS OF 1-D MIGRATING DEFECTS. H. Trinkaus, Institute für Festkörperforschung, Jülich, GERMANY; H.L. Heinisch, Pacific Northwest National Laboratory, Richland, WA; B.N. Singh, Risø National Laboratory, Roskilde, DENMARK.

It has been recognized in recent years that in metals, clusters of self-interstitial atoms (SIAs) in the form of small glissile dislocation loops produced in displacement cascades are able to perform fast one-dimensional (1-D) diffusional glide occasionally interrupted by direction (Burgers vector) changes. The diffusion reaction kinetics of such clusters which has been shown to play a key role in cascade damage accumulation has features of 1-D and 3-D reaction kinetics depending on the mean length of 1-D diffusion segments in relation to the length scales of the microstructure. In this paper, analytical treatments and kinetic Monte Carlo simulations of these general cluster reaction kinetics are discussed and compared for random distributions of voids of varying sizes and densities. The sink strength is shown to increase monotonically when changing continuously from 1-D to 3-D reaction kinetics. The analytical treatment suggests a simple one-parametric master curve interpolating between the pure 1-D and 3-D cases which is confirmed by our MC simulations. Transversal diffusion of SIA loops by random self-climb may be included in the analytical treatment. The formation and stability of void lattices under cascade damage is discussed in terms of 1-D to 3-D cluster reaction kinetics.

8:45 AM R4.2
DEFECT CLUSTER FORMATION IN HIGH ENERGY DISPLACEMENT CASCADES IN COPPER. Yu.N. Ossetsky 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 atomistic simulation during 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 explain experimental data. For example, experiments on copper show a very effective production of stacking fault tetrahedra (SFTs) but this was not observed systematically in cascade simulation. To clarify this and related issues, we have performed extensive simulation of displacement cascades in copper using two different interatomic potentials, a short-range many-body potential (SRMBP) and a long-range pair potential (LRPP). We have studied primary knock-on-atom energy 20keV (i.e. maximum energy below that for

formation of subcascades) at temperatures 100 and 600K. Special attention was paid to cascade statistics and the accuracy of simulation in the collision stage. The former required many simulations for each temperature (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 time step. In this paper we present results showing the variety of clusters observed, e.g. SFTs, glissile and sessile interstitial clusters, and faulted and perfect interstitial dislocation loops. A number of conclusions emerge on effect of the potentials and a significant variation of the number of Frenkel pairs and clustering effects produced in different cascades under the same conditions.

9:00 AM R4.3
THE PRIMARY DAMAGE IN Fe REVISITED BY MOLECULAR DYNAMICS AND ITS BINARY COLLISION APPROXIMATION. A. Souidi, M. Hou and C.S. Becquart.

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 can be obtained 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 clusters ...) 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 time consuming. We have therefore investigated the point defect distributions subsequent to atomic collision cascades by both MD (using EAM potentials for Fe) and its BCA. MD and its BCA lead to comparable point defect predictions. The significant similarities and differences are discussed. [1] R. Stoller, to be published.

9:15 AM *R4.4
FLOW LOCALIZATION IN IRRADIATED MATERIALS: A MULTISCALE MODELING APPROACH. T. Diaz de la Rubia, H. Zbib, M. Victoria, T. Kraishi, M. Caturla, and B. Wirth, Lawrence Livermore National Laboratory, Livermore, CA.

The microstructure of a metal under irradiation evolves over a wide range of length and time scales, making radiation damage an inherently multiscale phenomena. 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 or near ambient temperature (0.2-0.3 TM, where TM is the melting temperature) 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 a 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 picosecond 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 cluster microstructure. The results of the different stages of simulation agree well with the available experimental information. In particular, it is shown that DD simulation accounts for 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
Chairs: 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. L.L. Snead, 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, electrical resistivity and recrystallization kinetics of bulk amorphous silicon carbide. Samples studied were high purity CVD SiC and 6-H alpha single crystal SiC irradiated at ~60°C to a dose of ~2.6 and 4 E 25 n/m2 (E>0.1 MeV), or ~2.6 and 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, Weilin 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 interstitial 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 10 keV Au, Si, and C primary knock-on atoms (PKAs) 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 largest clusters indicates an amorphous state. From the cluster spectra, the ratios of the cross sections for in-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, Si and C ions, respectively. The different MD results 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 also 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. This includes implantation at >600°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 led to several problems including changes in the stoichiometry and topography of the surface, as well as degradation of the electrical properties of devices (i.e., channel mobility and gate oxide integrity). 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, dynamic effects within the SiC lattice require that temperature and dose-rate be treated complimentary and, thus, optimized conjointly. Results of this study will be presented. Secondly, a more novel approach for increasing activation of implanted dopants in SiC and lowering the activation temperature will be discussed. This approach utilizes the manipulation of the ion-induced damage to enhance activation of implanted dopants. It will be shown that nearly amorphous layers containing a small amount of residual crystallinity (<10% by volume) can be recrystallized at temperatures below 900°C with little residual damage. It will be shown that recrystallization traps a high fraction, ~90%, of the implanted dopant residing within the amorphous phase (prior to annealing) onto substitutional sites within the SiC lattice.

11:15 AM R5.4
ION-IRRADIATION-INDUCED AMORPHIZATION OF CADMIUM NIOBATE PYROCHLORE. Kristen Beaty, Alkiviathes Meldrum, Univ of Alberta, Dept of Physics, Edmonton, AB, CANADA; Lynn Boatner, 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 $A_2B_2O_7$. Recent investigations have focused on the effects of chemical composition and incident ion mass and energy on the irradiation-induced amorphization of several pyrochlore compositions. $Gd_2Ti_2O_7$ has been the subject of considerable 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 Ca has been found to have a relatively minor effect on the kinetics of irradiation-induced amorphization; however, the substitution of Ti with Zr in increasing concentrations sharply increases the resistance to amorphization even at cryogenic temperatures. In the present work, we have grown large synthetic single crystals of $Cd_2Nb_2O_7$ 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 (at ambient temperature) and RBS/channeling analysis were carried out using bulk single crystals. The in-situ TEM irradiation experiments were performed using Ne or Xe ions with energies of 280 and 1200 keV, respectively. For the bulk implantation experiments, the incident ion energies were 70 keV (Ne) and 320 keV (Xe_2). The critical amorphization temperature for $Cd_2Nb_2O_7$ was found to be ~580 K (280 keV Ne) or ~700 K (1200 keV Xe_2). The dose for in-situ amorphization at room temperature is 0.22 dpa for Xe_2, but is 0.65 dpa for Ne irradiation. The intensity in the RBS-channeling results reached the random value after an ion fluence that was ~40% lower than for the in-situ TEM experiments. Effects of the ion charge state and crystallographic orientation of the specimens were investigated. The results of the in-situ irradiation and bulk implantation experiments were analyzed in light of available models for the crystalline-to-amorphous transformation and were compared to previous ion-irradiation experiments on other pyrochlore compositions.

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

The effects of ion implantation on stoichiometric magnesium aluminate spinel have been studied using analytical electron microscopy. In specimens irradiated with swift heavy ions (electron stopping power > 8 keV / nm at the specimen surface), there are three distinct regions of the irradiated zone: an amorphous region at the surface; a metastable phase; and a spinel region exhibiting cation disorder as a result of the irradiation. This third zone forms in a region where the heavy ions have slowed and the electronic stopping power is reduced. A spinel specimen irradiated with 72 MeV I ions was examined in cross section to enable characterization of these distinct regions. Atom location by channeling enhanced microanalysis (ALCHEMI) performed near the Bragg condition of the spinel 220 planes in this third zone indicates that ~30% of the Mg2 cations sit on the octahedrally coordinated sites. 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 (~15% 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). Preliminary ALCHEMI analysis of the metastable phase indicates no discrimination between the Mg2 and Al3 cations along 220 planes, which would be indicative of the random cation distribution (67% of Mg2 on the octahedral sites) in spinel. However, CBED patterns indicate that the structure of this metastable phase may have the higher symmetry Fm3m space group. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

11:45 AM R5.6
Transferred to R3.46

SESSION R6: FERRITIC AND PRESSURE VESSEL STEELS
Chairs: Roger E. Stoller and Lynn E. Rehn
Tuesday Afternoon, November 28, 2000
Room 310 (Hynes)

1:30 PM *R6.1
ATOMIC LEVEL CHARACTERIZATION OF NEUTRON IRRADIATED PRESSURE VESSEL STEELS. M.K. Miller, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; P. Pareige, Groupe de Physique des Matériaux, UMR CNRS 6634, Faculté des Sciences et INSA de Rouen, Mont Saint Aignan, FRANCE.

Atom probe tomography provides one of the most effective tools to characterize the solute distribution and precipitation that occurs in pressure vessel steels and associated model alloys during irradiation. The three-dimensional atom probe is able to experimentally determine 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 have been applied to many different types of pressure vessel steels and model alloys and have revealed and characterized several different nanostructural transformations. These radiation induced or enhanced processes lead to the formation of copper-nickel-manganese-silicon precipitates, and solute segregation to dislocations, dislocation loops, nanovoids 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. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Science and Engineering, U.S. Department of Energy, under Contract DE-AC05-000R22725 with UT-Battelle, LLC, by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U.S. Department of Energy and through the SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities. Research at the Groupe de Physique des Matériaux was sponsored by Electricté 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-Mn USING PROTON IRRADIATION.
Qingkai Yu, Gary S. Was, 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-1.0Mn, Fe-0.9Cu 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 were irradiated with 3.2 MeV protons to doses from 0.001 to 0.01 dpa at dose rates of 7 x 10-7 to 7 x 10-6 dpa/s and over a temperature range 300-360°C. Hardness was measured by Vickers indentation using a 25 g load to ensure that the plastic zone was confined to the irradiated depth. Results show that irradiation at 300°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 rate and the magnitude of hardening. The effect of dose rate and temperature are also compared to neutron results. Microstructure is analyzed using small angle x-ray scattering (SAXS) and high resolution transmission electron microscopy (HREM) and correlated to hardness 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. Gragg, 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 lifetime of components in nuclear reactor environments. The process is complicated by the large time and length scales involved. The work presented here seeks to assist 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, contributes to an understanding of the role that the in-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.% Mn alloy and unalloyed Fe to damage levels up to $1.5x10^{-2}$ 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 #W-31-109-ENG-38 and by the U.S. Nuclear Regulatory Commission Contract No. NRC-04-94-049

2:30 PM R6.4
INTERACTIONS OF FLUX, TEMPERATURE AND COMPOSITION IN MEDIATING IRRADIATION HARDENING OF PRESSURE VESSEL STEELS AND MODEL ALLOYS.
G. Robert Odette, Glenn E. Lucas and Doug Klingensmith, University of California Santa Barbara, Department of Mechanical and Environmental Engineering, Santa Barbara, CA.

A large number of metallurgical and environmental variables interact to control irradiation hardening and embrittlement in reactor pressure 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 smaller unstable matrix defects (UMD) that continuously form and dissolve with relaxation times order $3x10^5$ 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 flux thermal diffusion may also accelerate CRP evolution. At intermediate levels the flux effect has not been well established. Some test reactor studies suggested a significant effect, while this has not been observed in the surveillance database. The results of the present carefully controlled experiment show 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 $5x10^{11}$ to 10^{12} n/cm^2-s for fluence levels between about 1017 to 1019 n/cm^2 and temperatures from 270, 290 and 310°C. The alloys have systematic variations in copper, nickel and manganese content. The intermediate flux effect seems to be primarily on the copper precipitation contribution to hardening, while the contribution due to defect cluster-solute complexes seems to be relatively independent of the damage rate. The fluence needed to produce a given level of hardening varies roughly with square root flux, suggesting a recombination 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 recombination models. The microstructural basis for interpreting flux effects is also described.

2:45 PM R6.5
POSITRON ANNIHILATION SPECTROSCOPY AND SMALL ANGLE NEUTRON SCATTERING CHARACTERIZATION OF NANOSTRUCTURAL FEATURES IN IRRADIATED Fe-Cu-Mn ALLOYS. B.D. Wirth, P. Asoka-Kumar, R. Howell, Lawrence Livermore National Laboratory, Livermore, CA; G.R. Odette, University of California, Santa Barbara, Santa Barbara, CA; and P.A. Sterne, 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 both the nanometer precipitates and sub-nanometer matrix features. The PAS characterization involved measuring both the positron lifetime and the Doppler broadened spectra of positron annihilation radiation. The strong positron affinity of copper relative to iron makes copper precipitates a positron trap, as are 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 spectrum in Fe-Cu and Fe-Cu-Mn specimens thermally aged to peak hardness at 450°C and in an Fe-Cu-Mn specimen irradiated at 288°C are nearly identical to elemental Cu. Further, positron lifetime measurements clearly show the existence of a long lifetime (? 500 ps) component in Fe-Cu 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 under Contract No. W-7405-Eng-48 and the U.S. Nuclear Regulatory Commission under Contract No. NRC-04-94-049.

3:30 PM *R6.6
AN ANALYSIS OF THE STRUCTURE OF IRRADIATION INDUCED Cu-ENRICHED 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 therefore cause an increase in the 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 atom-probe microscopy (3DAP), has yielded data that question 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 re-evaluate models of hardening which are currently based on the interaction between dislocations and precipitates as opposed to ramified clusters. In order to address these issues detailed microstructural analyses using 3DAP and SANS have been performed on two high copper irradiated RPV welds, one containing low Ni and the other containing high Ni. Cu-enriched irradiation induced clusters were observed in both welds. A new algorithm is being developed to precisely identify the shape, composition and size of clusters observed in the atom-probe 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 density 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. Naoki Soneda, Kenji Dohi, Central Research Institute of Electric Power Industry (CRIEPI), Materials Science Dept, Tokyo, JAPAN; Shiori 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.

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. Barashev, Stanislav I. Golubov, David J. Bacon, The Univ of Liverpool, 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 experiences normal nucleation, growth and Ostwald ripening, a distinguishing feature of which is the bcc-9R-3R-fcc 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, however, 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 (~2-4 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 "mean-field" 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 existing experimental data on microstructure and mechanical properties of irradiated steels.

4:30 PM R6.9
ATOMISTIC SIMULATION OF VACANCY AND SELF-INTERSTITIAL DIFFUSION IN FeCu ALLOYS. J. Marian, B.D. Wirth, T. Diaz de la Rubia, Lawrence Livermore National Laboratory, Livermore, CA; J.M. Perlado, 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. High-energy displacement cascades generate 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 fate of defects and, hence, in the overall chemical and microstructural evolution under irradiation. In this work, atomistic simulations based on a many-body, alloy interatomic potential are used to model the energetics and mobility of vacancy and self-interstitial atoms in a-iron and dilute Fe-Cu alloys and to assess the effect of Cu on point defect and solute diffusion. Cu transport predominately occurs by a vacancy diffusion mechanism and the calculated Cu diffusion properties are found to be in good agreement with experimental results. Further, small vacancy-copper clusters readily form in these simulations as a result of Cu-vacancy binding and may represent precursors for Cu rich precipitate formation. Conversely, self-interstitial atoms are repelled by the Cu atoms, resulting in a 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. W-7405-Eng-48 and within the Venus Project (CSN/UNESA Coordinated Research Program) under contract P970530432.

4:45 PM R6.10
THE EFFECT OF BULK PHOSPHORUS CONTENT ON HARDENING AND INTERGRANULAR SEGREGATION AND EMBRITTLEMENT IN NEUTRON-IRRADIATED Fe-BASED ALLOYS. Yutaka Nishiyama, Japan Atomic Energy Research Institute, Ibaraki, JAPAN; Tamara E. Bloomer, U.S. Nuclear Regulatory Commission, Washington, DC; Jun Kameda, Ames Laboratory, Ames, IA and Office of Naval Research International Field Office, Tokyo, JAPAN.

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 (0.016-0.38 wt.%) on hardening and intergranular P segregation and embrittlement in Mn-doped ferritic alloys subjected to neutron irradiation (E>0.1MeV: fluence of 9.8×10^{24} n/m^2 corresponding to 0.35 dpa) or thermal annealing. It was found that increasing the bulk content of P enhanced hardening due to P rich precipitation and grain boundary P segregation in unirradiated and thermally annealed alloys. However, the magnitude of hardening and P segregation induced by the irradiation tended to diminish in alloys with high bulk P content. The mechanisms of neutron irradiation-induced P rich precipitation and P segregation are 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 and post-irradiation or thermal annealing to identify how the metallurgical variables control intergranular embrittlement. This work was supported by the USDOE, Division of Basic Energy Science, Division of Materials Science.

SESSION R7/O12: JOINT SESSION
ION BEAM SYNTHESIS OF NANOSTRUCTURES AND THIN LAYERS II
Chair: J. C. Soares
Wednesday Morning, November 29, 2000
Room 311 (Hynes)

8:30 AM *R7.1/O12.1
NANOCOMPOSITE MATERIALS FORMED BY ION IMPLANTATION: RECENT DEVELOPMENTS AND FUTURE OPPORTUNITIES. A. Meldrum, Univ of Alberta, Dept. of Physics, Edmonton, Alberta, CANADA.

Materials consisting of nanocrystalline precipitates embedded in a solid host have been utilized for over 2000 years for the formation of various types of colored glass. In "gold-ruby" glass, for example, colloidal precipitates of gold scatter light by the Mie process to produce a deep red color. More recently, materials of this type have been found to exhibit large third order optical nonlinearities. In the case of semiconductor nanoparticles, numerous novel electronic effects (i.e., blue shifting of the band gap and the presence of discrete transitions) have been found to occur. Chemical techniques in particular have been used to produce highly luminescent and monodispersed solutions of semiconductor nanoparticles, and new biological and electronic applications are being actively explored. The properties of these "nanomaterials" depend directly on the size of the particles, as well as on their crystal structure, shape, and orientation. By controlling these various parameters, it should be possible to tailor the electronic and magnetic properties of nanocomposite materials for specific applications. Ion implantation, in particular, is a highly versatile and flexible technique for forming many nanocrystal compositions embedded in a variety of selected host materials. The nanocrystals are protected from the environment and their electronic and magnetic properties can be easily measured. By tailoring the microstructure of the particles (average size, orientation, crystal structure, etc.), it should then be able to control the electronic and magnetic properties of the system, and potentially, to develop these materials for use in new kinds of sensing or information technology devices. In the work completed to date, we have demonstrated that ion implantation is a viable and versatile technique for forming embedded nanoparticles of various compositions, and that the processing parameters can be modified to tailor the microstructural properties of the composite. Obtaining narrow size distributions remains a problem; however, in new experiments we are investigating means to obtain more monodispersed nanoparticles. The implantation method, its versatility, usefulness, and flexibility, as well as its inherent problems and possible solutions will be discussed.

9:00 AM R7.2/O12.2
ION BEAM ASSISTED NUCLEATION OF NANO-CRYSTALS. D. Ila, R.L. Zimmerman, and C. Muntele, Center for Irradiation of Materials, Alabama A&M University, Normal, AL; S. Schiestel, C.A. Carosella and G.K. Hubler, Naval Research Laboratory, Washington, DC; David B. Poker and Dale K. Hensley, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

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

9:15 AM R7.3/O12.3
ION BEAM ENHANCED FORMATION AND LUMINESCENCE OF Si NANOCLUSTERS FROM a-SiO$_x$. Jung H. Shin, Se-Young Seo, and Johan Sun, Dept. of Physics, KAIST, KOREA; T.G. Kim and C.N. Whang, Dept. of Physics, Yonsei University, Seoul, KOREA; J.H. Song, Advanced Analysis Center, KIST, Seoul, KOREA.

Si nanocrystals, due to quantum confinement effects, possess optical and electrical characteristics which allow realization of Si-based devices with novel functionalities. A widely used method to prepare a dense and robust array of well-passivated Si nanocrystals is precipitating them out of SiO$_x$ (x <2). While this method has the advantage of being compatible with the standard Si processing technology, it often requires long anneals at temperatures in the excess of 1100°C, which is undesirable from the process point of view. The anneal temperature can be lowered by decreasing the value of x, but this often results in larger sized Si clusters. In this paper, we show that by ion-irradiating SiO$_x$ films prior to anneal, both the cluster formation rate and the luminescence intensity can be greatly enhanced. SiO$_x$ film with x of 0.58 was deposited by electron cyclotron resonance enhanced chemical vapor deposition of SiH_4 and O_2. Prior to anneal, some samples were implanted with 380 keV Si to a dose ranging from 5.74×10^{15}cm^{-2} to 5.74×10^{15}cm^{-2}. All films were hydrogenated after anneals to passivate defects. Ion-irradiated films already display substantial luminescence in the 900 nm region after an anneal of only 1 min at 900°C, while virtually none can be detected from the non-irradiated film. Both films luminesce with a similar spectrum when annealed for 30 min at 1000°C, but the luminescence intensity from the ion-irradiated film is several times larger. Based on the effect of the irradiation dose and the ion specie, we rule out chemical effects due to the implanted ions, and propose that irradiation damage greatly accelerates nucleation of small Si clusters from the a-SiO$_x$ matrix.

9:30 AM R7.4/O12.4
NANOSCALE LEAD-CADMIUM ALLOY INCLUSIONS IN SILICON. E. Johnson, A.S.B. Jensen, A. Johansen, V.S. Touboltsev, L.Sarholt, Niels Bohr Institute, University of Copenhagen, DENMARK; U. Dahmen, National Center for Electron Microscopy,

Lawrence Berkeley National Laboratory, Berkeley, CA.

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

SESSION R8/O13: 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/O13.1

THE LIMITATIONS AND ADVANTAGES OF USING ION BEAM SYNTHESIS TO FABRICATE PLASMONIC DEVICES.
Mark L. Brongersma, John W. Hartman, Stefan A. Maier, and Harry A. Atwater, Thomas J. Watson Laboratory of Applied Physics, California Institute of Technology, Pasadena, CA.

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

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

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

Mixed anion nitride-arsenide compound semiconductor alloys and nanostructures are promising for light emitting devices operating throughout the near infrared range. However, due to the large N-As size difference, a limited miscibility of InGaAsN on the anion sublattice is predicted. To date, only a few percent nitrogen has been incorporated into mixed anion nitride-arsenide materials synthesized by conventional epitaxial methods. Furthermore, limited studies have been performed using ion implantation. Therefore, we have investigated the synthesis of InGaAsN by N ion implantation into GaAs and InAs, using a variety of implantation and rapid thermal annealing conditions. We have characterized the nitrogen content, alloy segregation, and optical properties of the implanted structures using Rutherford backscattering spectrometry (RBS), four-circle x-ray diffraction (XRD), cross-sectional transmission electron microscopy (XTEM), and Photoluminescence (PL) spectroscopy. RBS suggests a small overall percentage of nitrogen incorporation into the implanted GaAs and InAs samples. On the other hand, XRD suggests the formation of high nitrogen content GaAsN and InAsN alloys. Since XRD is very sensitive to coherent crystallite structure, we infer that the alloyed regions are forming small nitrogen-rich clusters. This is confirmed by XTEM, which indicates the formation of precipitates with 10-20 nm diameters. Furthermore, photoluminescence spectroscopy suggests that the nitrogen implantation plus rapid thermal annealing has lowered the fundamental band gap, consistent with theoretical predictions of band gap bowing in this system. Detailed investigations of the microstructure using high resolution and analytical electron microscopy will be discussed.

11:15 AM R8.3/O13.3

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

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

11:30 AM R8.4/O13.4

DOPING OF GALLIUM NITRIDE BY ION IMPLANTATION.
E. Alves[a,b], C. Liu[a,b], A. Sequeira[a], N. Franco[a], M.F. da Silva[a,b], J.C. Soares[a,b], T. Monteiro[c], [a]Instituto Tecnológico e Nuclear, Sacavém, PORTUGAL; [b]Universidade da Lisboa, CFN, Lisboa, PORTUGAL; [c]Universidade da Aveiro, Dept. Física, Aveiro, PORTUGAL.

With the help of modern growth techniques, the quality of epitaxial GaN films continues to improve. The background electron concentration decreases to the order of 10^{16} cm^{-3}. This makes it possible to dope GaN by ion implantation. In this work, we report the structural, electrical and optical properties of ion implanted GaN. Potential acceptors such as Ca, Fe and Er were used as dopants in this study. Ion implantation was carried out at substrate temperatures of 30 and 550°C, respectively. The structure of GaN films before and after the implantation was characterized by Rutherford backscattering/channeling combined with particle induced X-ray emission, high resolution X-ray diffraction and Mössbäuer effect spectroscopy for the samples implanted with Fe. Using ion channeling the lattice site location of the dopants has been determined. The electrical and optical properties of the ion implanted GaN films have been studied by Hall effect and photoluminescence measurements. The as implanted GaN films exhibit an expanded lattice. After annealing at 1000°C, the lattice distortion does not fully recover. The Er- related luminescence near 1.54 μm is observed under below band gap excitation at liquid helium temperature. The spectra of the annealed samples consist of multiline structures with the sharpest lines found in GaN until now. Angular scans along both [0001] and [10$\bar{1}$1] directions show that the implanted dopants occupy mostly the lattice site of Ga atoms. For Er the combination of photoluminescence and ion channeling results allowed the identification of different sites for the optical active Er-related centers.

11:45 AM R8.5/O13.5

CORRELATION BETWEEN STRUCTURAL AND OPTICAL PROPERTIES OF Si NANOCRYSTALS IN SiO_2: MODEL FOR

THE VISIBLE LIGHT EMISSION. Blas Garrido, Manel Lopez, Alejandro Perez-Rodriguez, Joan Ramon Morante, EME-Departament d'Electronica, Universitat de Barcelona, Barcelona, SPAIN; Caroline Bonafos, Alain Claverie, CEMES-CNRS, Toulouse, FRANCE.

It is at large admitted that finding the correlation between the structural (average nanocrystal size) and optical properties (band-gap energies and light emission) is among the key factors to understand the emission mechanism of Si nanocrystals in SiO_2. This has not been possible up to now mainly because of the great difficulty of imaging large populations of Si nanocrystals embedded in SiO_2. We have solved this problem by developing a method in which high resolution electron microscopy (HREM) is used in conjunction with conventional TEM in dark field conditions. So, size-distribution histograms, band-gap energies and photoluminescence (PL) emission of Si nanocrystals ion beam synthesized in SiO_2 have been measured by independent and direct methods and correlated between them. The results have allowed to experimentally determine for the first time the Stokes shift between absorption and emission as a function of crystallite size. The experimental band-gap versus size correlates exceptionally well with the most accurate theoretical predictions. Moreover, the PL dependence versus size is parallel to that of band-gap energy. Consequently, the experimental Stokes shift is independent of nanocrystal size and is found to be 0.26 ± 0.03 eV. This is almost exactly twice the energy of the Si-O vibration (0.134 eV). These results suggest that the dominant emission for SiO_2 capped Si nanocrystals is a fundamental transition spatially located at the Si-SiO_2 interface with the assistance of a local Si-O vibration.

SESSION R9/O14: JOINT SESSION SEMICONDUCTORS AND ELECTRONIC MATERIALS

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

1:30 PM R9.1/O14.1

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

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

1:45 PM R9.2/O14.2

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

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

2:00 PM R9.3/O14.3

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

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

2:15 PM R9.4/O14.4

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

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

2:30 PM R9.5/O14.5

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

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

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

INVERSE OSTWALD RIPENING AND SELF-ORGANIZATION OF

NANOCLUSTERS DUE TO ION IRRADIATION. K.-H. Heinig, B. Schmidt, FZ Rossendorf, Institute of Ion Beam Physics and Materials Research, Dresden, GERMANY; M. Strobel, CNR-IMETEM, Catania, ITALY; H. Bernas, CSNSM, CNRS-IN2P3, Orsay, FRANCE.

Under ion irradiation collisional mixing competes with phase separation if the irradiated solid consists of immiscible components. If a component is a chemical compound, there is another competition between the collisional forced chemical dissociation of the compound and its thermally activated re-formation. Especially at interfaces between immiscible components, these processes far from thermodynamical equilibrium may lead to unexpected phenomena. In this contribution it will be shown how nanoclusters embedded in a matrix and the chemical composition at an interface evolve under irradiation. The inverse coarsening of clusters found almost 30 years ago by studies of reactor materials, can now be understood. The mathematical treatment of the competition between irradiation-induced detachment of atoms from clusters and their thermally activated diffusion leads to the Gibbs-Thomson relation with modified parameters. A drastical consequence results from the negative capillary length, which will be shown to be the reason for inverse Ostwald ripening. This theoretical prediction has been proven by kinetic Monte Carlo simulations and experimental studies of Au clusters in SiO_2 irradiated by MeV ions. The second unexpected phenomenon to be addressed is self-organization of nanoclusters in a δ-layer parallel to the Si/SiO_2 interface which has been found when at the interface the damage level was in the order of 1...3 dpa. It will be shown that the origin of the δ-layer is the chemical reaction of Si at the interface with free oxygen coming from the collisional forced chemical dissociation of SiO_2. Thus, a sub-stoichiometric SiO_{2-x} layer forms at the interface. During subsequent annealing this layer provides nuclei for a nanocluster δ-layer, which is of interest for non-volatile memory application.

3:45 PM R9.7/O14.7
IMPACT OF BORON, GALLIUM AND OXYGEN ON DEFECTS PRODUCTION IN SILICON. Aurangzeb Khan, Nethaji Dharmarasu, Masafumi Yamaguchi, Kenji Araki, Tuong K. Vu, Toyota Technological Institute, Nagoya, JAPAN; Tatsuo Saga, Sharp Corporation, Nara, JAPAN; Takao Abe, Shin-Etsu Handotai Co., Ltd, Gunma, JAPAN; Osamu Annzawa and Sumio Matsuda, National Space Development Agency of Japan, Tsukuba, JAPAN.

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

4:00 PM R9.8/O14.8
POST ANNEALING STUDIES OF C_{60} ION IMPLATED THIN FILMS. Nethaji Dharmarasu, Kannan L. Narayanan, Nabuaki Kojima, Yoshio Ohshita and Masafumi Yamaguchi, Toyota Technological Institute, Semiconductor Lab, Nagoya, JAPAN.

Recently ion implantation in fullerene C_{60} has attained an enormous interest for its opto-electronic device applications. We have carried out multiple energy boron ion implantation in C_{60} thin films to various doses. Physical properties like electrical, structural and optical properties were studied. The C_{60} films were prepared on quartz and silicon substrates at a substrate temperature of 150 degree celsius by molecular beam epitaxy (MBE) technique. The conductivity type of the implanted films is found to be p-type and the conductivity measurements reveal the dramatic increase in the conductivity with ion implantation. The optical gap is found to decrease due to the implantation and it could be attributed to the formation of defect levels and dopant (acceptor). FTIR results indicate the structural transformation of C_{60} to amorphous carbon phase during implantation. These results could be due to both ion implantation induced damage and doping of boron ions. To delineate the ion implantation induced damage from doping effect, the thermal annealing experiments were carried out. The implanted films subjected to thermal annealing indicate the removal of the defects caused during the implantation. Ion implantation-induced defects are found to annihilate with the annealing temperature. Electrical conductivity and optical gap are determined in the post implanted films. The observation of the systematic increase in the conductivity of the annealed films is due to the removal of the defects and the formation of defect free boron impurity acceptor. Their mechanism will be presented at the conference.

4:15 PM R9.9/O14.9
UNUSUAL CHANGE IN COLUMNAR DEFECT MORPHOLOGY IN YBCO UPON ANNEALING. Y. Yan, M. Kirk, Materials Science Division, Argonne National Laboratory, Argonne IL; A. Petrean and L. Paulius, Department of Physics, Western Michigan University, Kalamazoo, MI.

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

4:30 PM R9.10/O14.10
ION-INDUCED AMORPHOUS TRACKS IN LAYERED STRUCTURES WITH VARIOUS TYPES OF CONDUCTION. György Szenes, Eötvös Univ, Dept of General Physics, Budapest, HUNGARY.

Amorphous tracks induced by high-energy monoatomic and cluster ions are analyzed in mica (insulator), Y-Ba-Cu-O, Bi-Sr-Ca-Cu-O (HTCSs) and in GeS and MoS_2 (semiconductors) applying the author's thermal spike model. These solids have a layered structure and a strong anisotropy of the transport properties except mica, which is an insulator in any direction. The ion beams were perpendicular to the layers. Good quantitative agreement was found between the experimental R_e- S_e data (R_e - effective track radius, S_e - electronic stopping power) and the predictions of the model. Two parameters: the efficiency g and the initial Gaussian width of the thermal spike a(0) were determined from the analysis. In mica the parameters of track formation are very close to that in other covalent insulating crystals, and $0.17S_e$ is transferred to the thermal spike (g=0.17). We conclude that the layered structure does not modify the track diameter in mica. In Y-Ba-Cu-O, Bi-Sr-Ca-Cu-O, GeS and MoS_2 only about 1/3 of the excitation energy is involved in the initial logarithmic stage of track evolution (g≈0.17/3) and the derivative $dR_e^2/dlnS_e$=a^2(0)≈20.25 nm^2 is equal to that in insulators. The results are explained by the high anisotropy of the electron effective masses m^* and by the formation of a nearly isotropic electron velocity distribution after numerous elastic collisions. As m^* values are about 10-30 times higher for the wavevector component k_c normal to the layers, the energy transfer to the lattice is highly efficient and fast for about 1/3 of the excitation energy. In GeS a second stage of track evolution starts at S_e=19.4 keV/nm with a^2(0)=130 nm^2 and g=0.17. The high efficiency indicates that like in insulators, all excited electrons are involved in the energy deposition in this stage. The high value of a(0) is in agreement with the expectation of a broad thermal spike in semiconductors.

4:45 PM R9.11/O14.11
ION IMPLANTATION GENERATED NANOVOIDS IN Si AND MgO MONITORED BY HIGH RESOLUTION POSITRON BEAM ANALYSIS. S.W.H. Eijt, C.V. Falub, A. van Veen, H. Schut, P.E. Mijnarends, M.A. van Huis and A.V. Fedorov, Delft University of Technology, Interfaculty Reactor Institute, Delft, NETHERLANDS.

The formation of nanovoids in Si(100) and MgO(100) by ^{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 reactor-based variable-energy positron beam POSH. This allows a new method of monitoring thin layers containing nanovoids or defects by depth-selective high-resolution positron beam analysis. The 2D-ACAR spectra of Si with a buried layer of nanocavities reveal the presence of two additional components, the first related to para-positronium (p-Ps) formation in the nanovoids, and a second likely related to unsaturated Si-bonds at the voids' internal surface. The positronium is present in excited kinetic states with an average energy of 0.3 eV. Refilling of the cavities by means of low dose ^{3}He implantation ($1x10^{14}$ 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.