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THE EFFECT OF INTERSTITIAL GAS ATOMS ON MICROSTRUCTURAL EVOLUTION IN SELF-ION IRRADIATED NICKEL*

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Annealed foils of pure nickel were injected with He in concentrations of 100 and 600 atomic parts per million (appm) at room temperature utilizing a 700 kV electrostatic accelerator. Injections were completed at various accelerating potentials over the range 700 kV to 200 kV. This variation produced a relatively uniform concentration of interstitial gas atoms extending from a depth of approximately 1.25 μm to the surface of the sample. The injected samples were irradiated at temperatures of 525°C and 625°C with 14 MeV Ni^{3+} ions to a fluence of 1×10^{16} ions/cm^2 and prepared for transmission electron microscopy evaluation utilizing a transverse sectioning technique. Results show the formation of a void-free zone which extended from the surface to a depth of 1.25 μm. The variation of void number density, average void diameter, and swelling and a function of depth from the surface is presented for each sample. The results are compared with previous studies completed without injected gas atoms.

1. INTRODUCTION

Interstitial gas atoms introduced into the metal matrix of fusion reactor structural components via (n,p) and (n,α) transmutation reactions can greatly affect the microstructural evolution of materials in a fusion neutron environment. These gas atoms can act as nucleation sites for void formation resulting in swelling and mechanical property changes which will dictate the useful lifetime of the component. Since few intense sources of 14-MeV neutrons for materials research currently exist, electrostatic accelerators have been employed to produce displacement damage as well as inject interstitial gas atoms simultaneously.[1-7]

The purpose of this study is to address the effect of interstitial gas atoms on depth dependent cavity formation in self-ion irradiated nickel. Utilizing the transverse sectioning technique developed by Whitley,[3,4] the effect of injected He on void formation at 525°C and 625°C for 100 appm and 600 appm He content is investigated. Results show damage beyond the calculated end of range of the injected ion. Comparisons are made between previous cross-section studies with no gas preinjection and current results.

2. EXPERIMENTAL TECHNIQUE

High purity (99.995%) polycrystalline nickel foils (1 cm x 0.5 cm) were annealed for 1 hour at 1000°C in an argon atmosphere. The foils were metallographically polished, but not electropolished prior to helium implantation. A 700 kV electrostatic accelerator, described in detail elsewhere,[8] was used to inject $^4He^+$ ions at energies ranging from 200 keV to 700 keV. Initial injections at 700 kV accelerating potential were followed by injections at decreasing 100 kV increments to 200 kV. This technique provides a relatively uniform helium concentration from a depth of approximately 1.25 μm to the sample surface. Implantations were completed in a vacuum of $\sim 2.0 \times 10^{-4}$ Pa. Preinjected samples were then irradiated with 14 MeV Ni^{3+} ions to a fluence

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of 1.0 x 10^{16} ions/cm^2 at temperatures of 525°C and 625°C. Nominal vacuum during the nickel ion irradiation was ~ 1.0 x 10^{-5} Pa. Irradiated samples were prepared for TEM study using the transverse sectioning technique described in detail by Whitley.[9] The samples were examined using a JEOL 200CX transmission electron microscope and the depth distribution of voids was determined by division of the micrograph into regions of thickness 0.25 μm which are parallel to the irradiated surface. The void size was determined using a Zeiss particle analyzer and the foil thickness was determined using stereo microscopy techniques.

3. RESULTS

Figure 1 shows the typical bright field micrograph of the void microstructure produced by 14-MeV Ni^{3+} ion irradiation at 525°C to a fluence of 1 x 10^{16} ions/cm^2. This sample was preinjected with 100 appm helium at room temperature. The helium was uniformly distributed from the sample surface to a depth of 1.25 μm. The interface between the plating and the foil is noted at the left side of the figure. The incident beam direction is also shown. A void-free zone extends from the surface to a depth of approximately 1.25 μm. This denuded zone was also observed in samples irradiated to similar fluences at 625°C with 600 appm helium.

The swelling, void number density and mean void diameter as a function of depth for the 525°C irradiation are shown in Figs. 2-4, respectively. Note the decrease in the mean void diameter and void number density at a depth of 1 μm from the surface for the helium preinjected case. This corresponds with the suppression of swelling near the surface as indicated by the void-free zone shown in Fig. 1. The results of a similar irradiation by 14 MeV Ni^{3+} ions at 625 °C with 600 appm helium preinjected at room temperature are presented in Figs. 5-7. The swelling and void number density decrease at approximately 1 μm from the surface indicative of the void-free zone which was observed.

In both Figs. 2 and 5 the swelling distribution extends to approximately 3 μm from the surface. A theoretical range profile computed using Brice's[10] computer codes with theoretical LSS electronic stopping[11] for 14 MeV Ni on Ni is shown on Fig. 1. Comparison of the theoretical depth for the peak of the damage curve with the associated micrograph and the swelling curves in Figs. 2 and 5 shows that swelling occurs up to 20% beyond the theoretical end-of-range of the irradiating 14 MeV Ni^{3+} ion.

4. DISCUSSION

The formation of a void-free zone which extends to a depth of approximately 1.25 μm from the surface corresponds with the region of uniform He concentration produced by the preinjection. Previous studies of void-free zones near surfaces[16] utilizing HVEM techniques report the thickness of the void-free zone to be ~ 0.2 μm at 525°C and ~ 0.5 μm at 625°C in various stainless steels. The thickness of the void-free zone in this study suggests an effect due to the preinjection of He such as the suppression of cavity growth due to a high concentration of small clusters. These results are not entirely unexpected when one considers the dual-ion and preinjection studies completed by Agarwal et al.[1] or Packan and Farrell.[2] Agarwal et al. completed 3-MeV Ni^+ ion irradiations of Fe-20 Ni-15 Cr with 15 appm He preinjections as well as dual-ion irradiations. For the preinjected samples, there was an incubation dose of 13 dpa before the onset of swelling. Packan and Farrell carried out similar studies with 4-MeV Ni^+ ion irradiations of 316 stainless steel. Preinjection of 1400 appm He brought about drastic

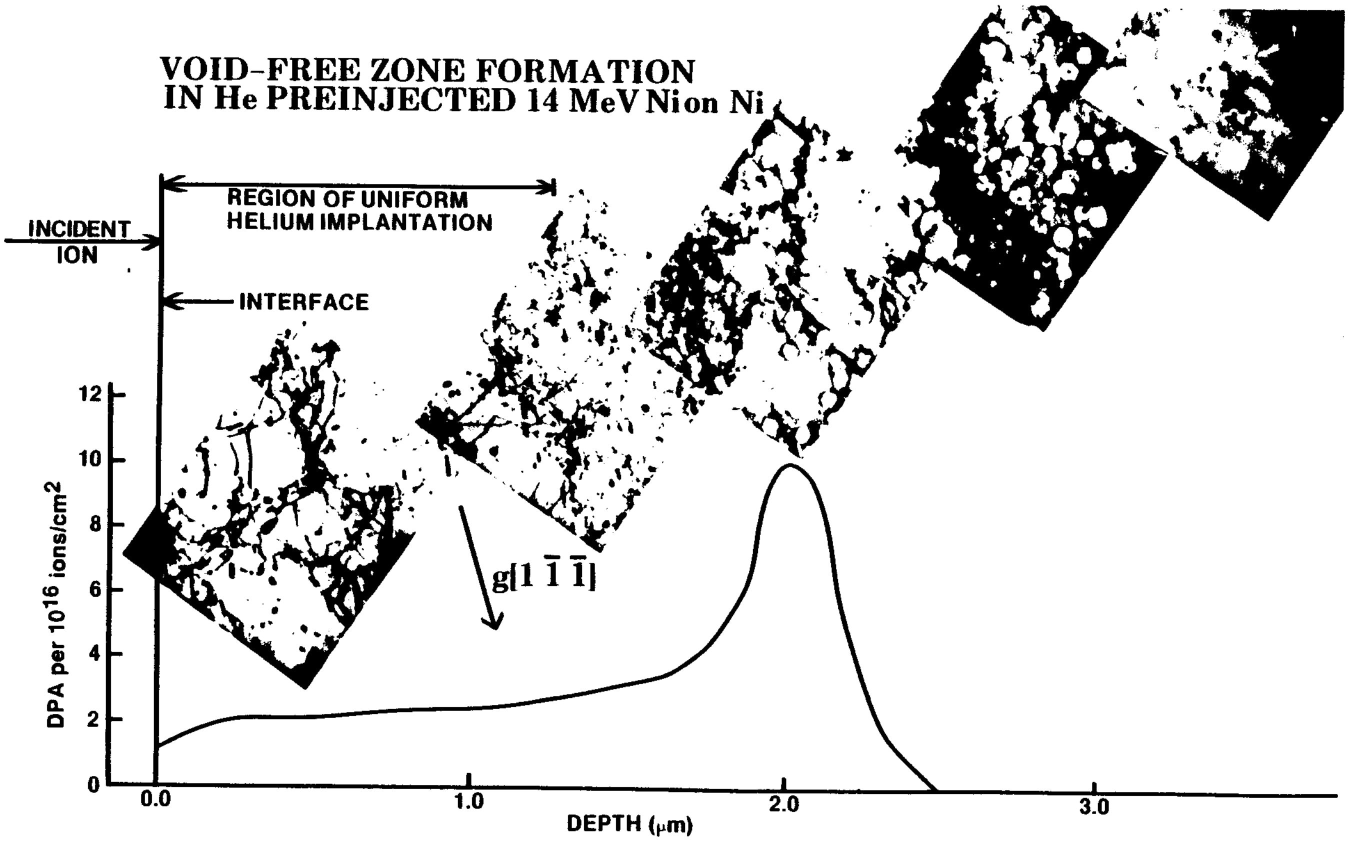

FIGURE 1
Void microstructure formation of Ni preinjected with 100 appm He at room temperature and irradiated with 14 MeV Ni^{3+} ions to a fluences of 1 x 10^{16} ions/cm^2. Note: void free zone over range of He ions and theoretical damage curve for 14 MeV Ni^{3+} ions.

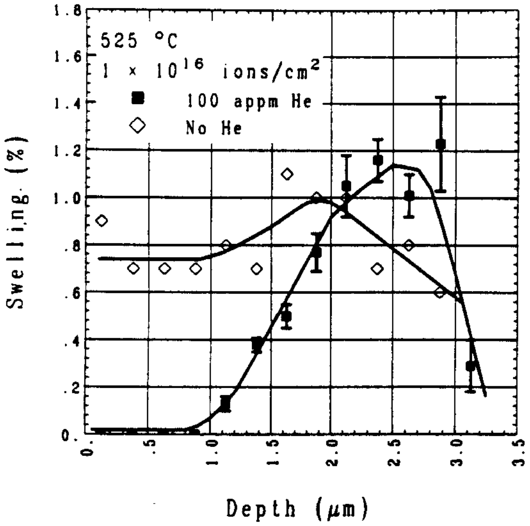

FIGURE 2
Swelling vs. depth for 14 MeV Ni^{3+} ion irradiated Ni preinjected with 100 appm He. No He data from Whitley.[3,4]

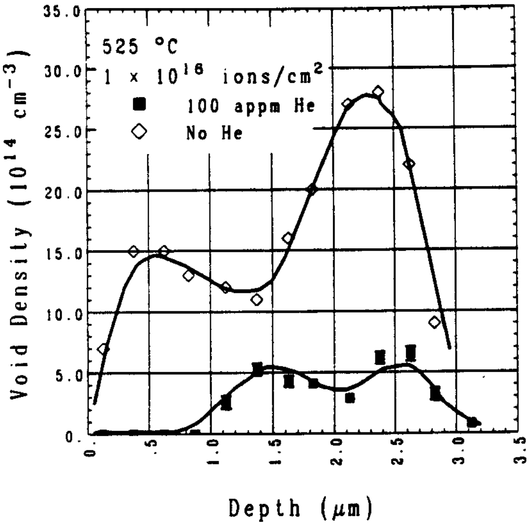

FIGURE 3
Void number density vs. depth for 14 MeV Ni^{3+} ion irradiated Ni preinjected with 100 appm He. No He data from Whitley.[3,4]

reductions in swelling due to profuse initial nucleation of cavities. The authors propose that these cavities competed with one another allowing a very limited cavity growth. This mechanism may also explain the observation of the denuded zone in this work. The high

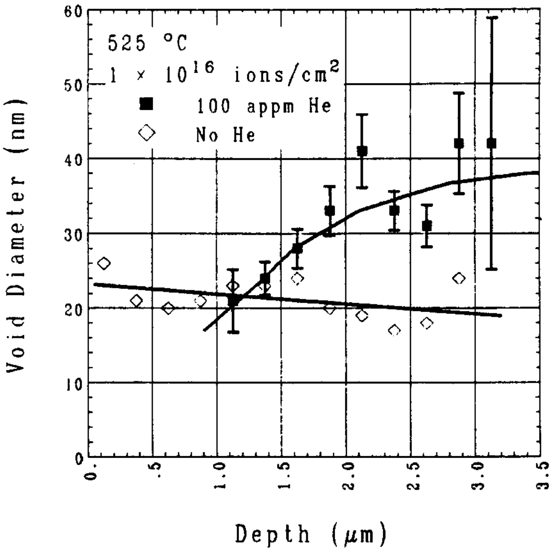

FIGURE 4
Mean void diameter vs. depth for 14 MeV Ni^{3+} ion irradiated Ni preinjected with 100 appm He. No He data from Whitley.[3,4]

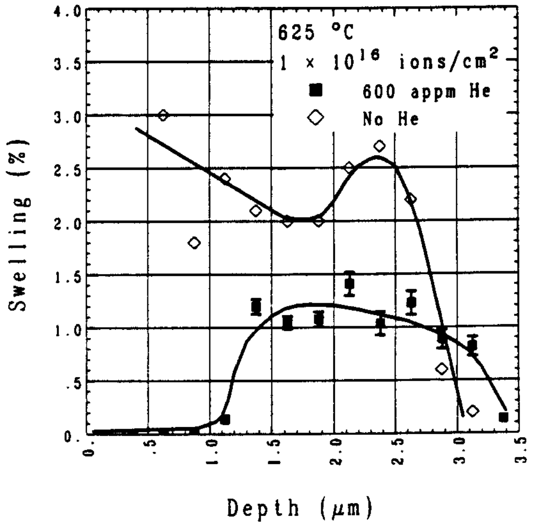

FIGURE 5
Swelling vs. depth for 14 MeV Ni^{3+} ion irradiated Ni preinjected with 600 appm He. No He data from Whitley.[3,4]

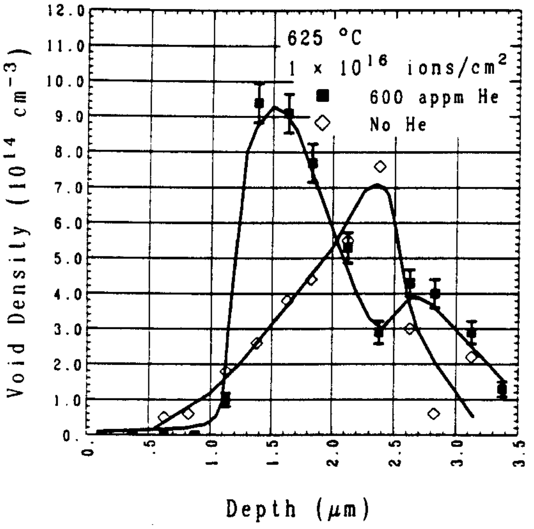

FIGURE 6
Void number density vs. depth for 14 MeV Ni^{3+} ion irradiated Ni preinjected with 600 appm He. No He data from Whitley.[3,4]

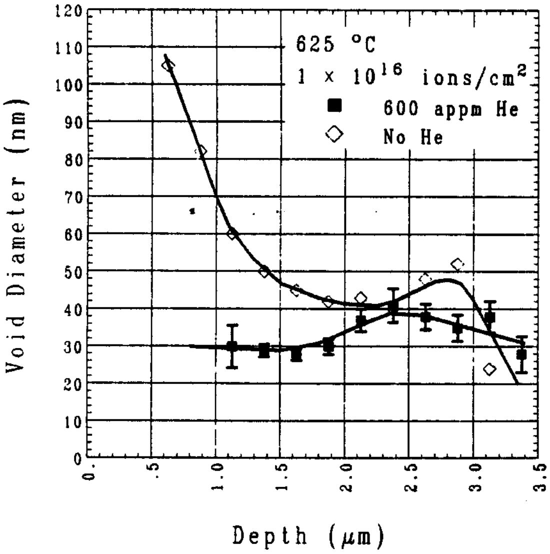

FIGURE 7
Mean void diameter vs. depth for 14 MeV Ni^{3+} ion irradiated Ni preinjected with 600 appm He. No He data from Whitley.[3,4]

initial concentrations of He (100 and 600 appm) could act as nucleation sites for submicroscopic cavities which, at the low damage levels present in the preimplanted region (< 2 dpa), had not yet grown to a size readily detectable by TEM observation.

The formation of voids well beyond the theoretical end of range of the irradiating ions observed in this study has been noted in other studies in Ni utilizing the transverse section technique. Whitley[3,4] noted void formation up to 20% beyond the theoretical end-of-range calculated using Brice's code. Fenske et al.[13-15] noted a peak in the swelling distribution at depths 8 to 15% deeper than the theoretical projected range profiles for 20 keV and 500 keV He$^+$ ion irradiations of nickel. The observations in this study, as well as those of Whitley and Fenske et al., have been attributed to inaccuracies in the electronic stopping data used to calculate range profiles as shown by Attaya[16] and Fenske.[14]

The transverse sectioning technique used in this study was also employed by Whitley[3,4] in studies of 14 MeV Ni^{3+} ion irradiated nickel. The primary difference between the Whitley procedures and this study is the method of introduction of the interstitial gas atom. Whitley demonstrated the existence of a gas effect using comparisons of thoroughly outgassed samples and samples prepared in the standard manner. Noting a drastic reduction in cavity formation and swelling in the outgassed samples, Whitley hypothesized a hydrogen interstitial effect where the hydrogen was introduced during the electropolishing stage of sample preparation. In this study the electropolishing step was omitted and interstitial gas atoms were implanted with an accelerator. This allowed for a quantification of gas content and the subsequent effects produced during irradiation. Comparison between Whitley's results and current results is, however, useful and is shown in Figs. 2-7. Whitley's data is plotted along with present data for the same irradiation conditions, where the Whitley data is designated as having no He.

A comparison of the data in Figs. 2-4 for the 525°C irradiation shows relatively good agreement between Whitley's results and this study. Whitley had comparable swelling, although in his specimens, the average void diameter was smaller and the void number density was larger than in this study. These variations are most likely due to differences in the hydrogen gas content of these samples. Figures 5-7 allow comparison for the 625°C irradiations. The variation in observed swelling for this study is most likely due to the higher gas content of the He preinjected samples, although quantitative gas content for the Whitley data is unavailable. Figure 6 shows a greater void number density for the preinjected samples while the average void diameter is smaller indicating a larger number of smaller voids which results in lower swelling.

5. CONCLUSIONS

A void-free zone corresponding to the area of preinjected He was observed. This is attributed to the formation of an extensive number of small clusters which were nucleated on He interstitials. The total damage in this region was not sufficient (< 2 dpa) to produce observable swelling. Void formation was observed up to 20% beyond the theoretical end-of-range of the irradiating 14 MeV Ni^{3+} ion. This is probably due to inaccuracies in the electronic stopping power used in the theoretical calculations. Qualitative comparisons made between this work and the work of Whitley suggest that high interstitial gas content may delay the onset of swelling due to the formation of numerous small clusters.

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