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Cavity formation in high purity aluminum has been explored with the controlled addition of helium gas. Aluminum specimens containing no helium and preinjected helium concentrations of 0.1, 1.0 and 10 at.ppm were irradiated with aluminum ions at 100°C to doses of 0.5 to 26 dpa. The helium preinjection was performed at room temperature (25°C) by a controlled-thickness alpha particle source made from ^{244}Cm oxide. All of the samples containing helium exhibited cavity formation while the aluminum containing no helium showed only vacancy loops and dislocation tangles. The microstructural effects observed were very heterogeneous and scattered in the irradiated aluminum even at the highest damage levels (26 dpa). The cavities were located in the vicinity of grain boundaries but no voids were observed on the grain boundaries themselves. Cavities were observed only in aluminum containing helium.

1. INTRODUCTION

The alloys chosen for controlled thermonuclear reactors (CTR) will have to operate in a severe radiation damage environment, endure cyclic stresses and widely varying temperatures. The performance of a CTR alloy is intrinsically linked to this environment and both experimental and theoretical attempts to analyze these complex alloys are difficult. Experiments with a pure material and the controlled addition of impurities can provide a more suitable groundwork for the understanding of radiation effects. The choice of aluminum for a model material is warranted for a number of reasons. First, aluminum has been studied from many aspects and is a well-understood material both experimentally and theoretically. Second, there have been numerous studies conducted in neutron environments which provide a substantial neutron data base (for example see Refs. [1,7,8]). Third, extremely high purity aluminum can be obtained and this purity can be maintained by employing aluminum ions as irradiation particles. Finally, results from the observation of controlled injections of inert gas can lead to an understanding of the combined effects of radiation produced defects and inert gas in aluminum.

2. EXPERIMENTAL DETAILS

Three millimeter diameter discs of a high purity (1 at.ppm) aluminum stock which was used by Packan [1] and Sundquist [2], were polished and annealed at 550°C for one hour in a vacuum of less than 1 x 10^{-7} torr. Selected samples were pre-injected with helium at room temperature using an alpha emitting curium-244 source to concentrations of 0.1, 1.0 and 10 at.ppm helium. This doping was performed by Isotope Sales Division of Oak Ridge National Laboratory and a diagram of the implantation arrangement is shown in Figure 1 [3]. The curium-244 oxide is incorporated into a thin film and the emitted alpha particles have an energy distribution of 0 to 5.8 MeV. The 5.8 MeV helium has a maximum range of 27 micrometers with a constant helium concentration for more than 20 micrometers into the aluminum. These helium doped samples and samples containing no helium were irradiated with 9 MeV aluminum ions in a model EN tandem accelerator located at the University of Wisconsin. The irradiation doses ranged from 0.5 to 26 displacements per atom at an effective dose rate in the region of analysis of 7 x 10^{-4} dpa per second. The irradiation temperature was held constant at 100 degrees C. The aluminum samples were irradiated in a vacuum of less than 1.3 x 10^{-5} pascal.

The 9 MeV aluminum ions have a range of 4.5 micrometers and a corresponding peak damage at 4.25 micrometers from the front as shown in Figure 2. The samples were analyzed at a depth of 3 micrometers to avoid both surface and peak damage effects. Material removal was accomplished by electropolishing from the front surface to the required depth - indicated by

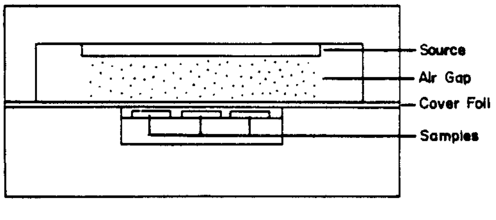


Figure 1. A schematic drawing of the helium implanting arrangement showing the source of helium atoms, a cover foil to prevent contamination and the aluminum 3 mm discs. The drawing is not to scale.

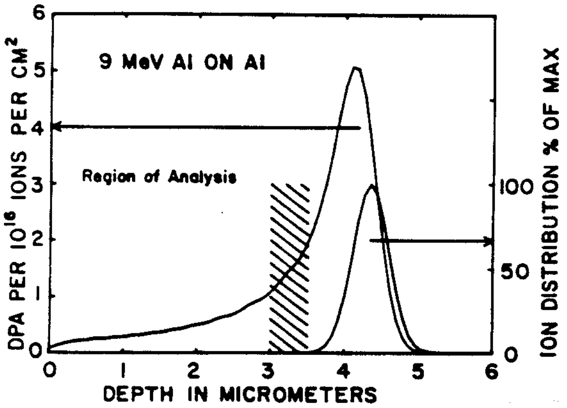


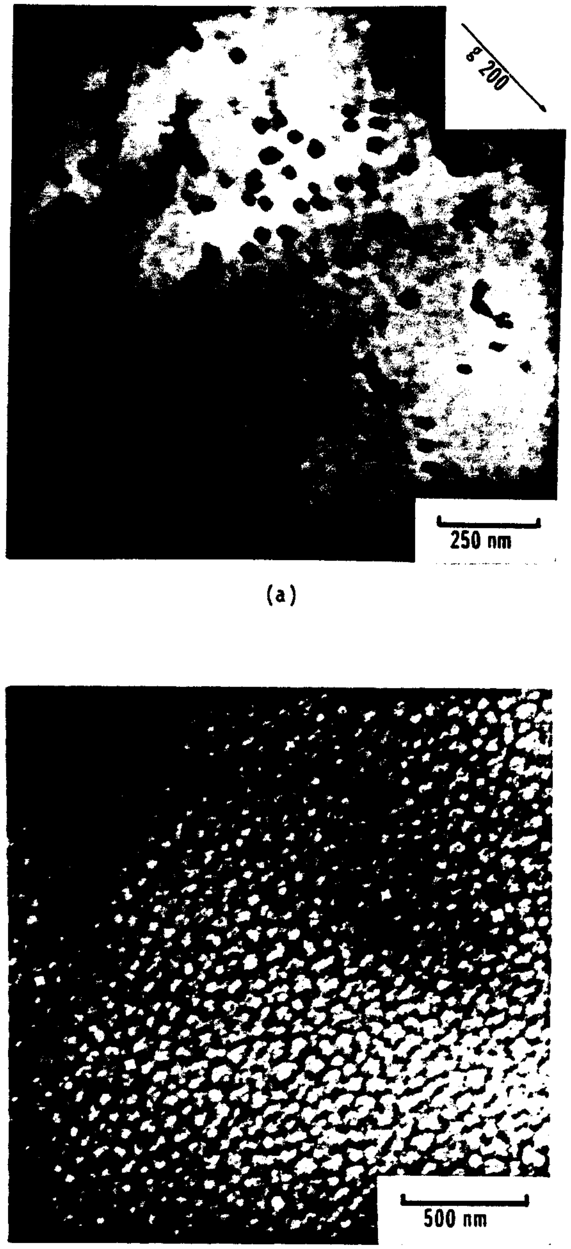
Figure 2. The calculated displacement rate curve with the distribution of deposited ions is shown. The region of examination is indicated with a typical foil thickness of 500 nm.

interference microscopy - and then back-thinning to obtain a thin foil for TEM. The displacement calculations and ranges of aluminum and helium were calculated by the Brice [4] computer code.

3. RESULTS

The most significant result of this experiment was the demonstration of the necessity for the presence of helium for cavity formation (see Table 1). Figure 3(a) shows a sample of aluminum containing no helium that has been irradiated to 6 dpa without cavity formation while Figure 3(b) illustrates void formation at 2 dpa and 0.1 at.ppm helium. It is believed that the presence of helium is required for void formation in high purity aluminum because the gas is needed to stabilize void embryos. It also appears that the high purity aluminum does not have effective traps for gas impurities. Some earlier work with irradiated aluminum has demonstrated that helium can enhance the formation of voids or cavities [5,6,7] and these results support earlier work performed at Wisconsin under different conditions [2].

The distribution of voids and dislocation loops and tangles was very heterogeneous within each sample, even up to 26 dpa (see Figures 4 and 5). This non-uniform distribution of irradiation defects presented difficulties in determining the number density of defects and in calculating the total swelling within the sample. The voids were heterogeneously distributed near grain boundaries and dislocation tangles in the interior of the grains. Since the pre-irradiation annealing temperature was so high, the very large grain size (~ 500 micrometers) allowed for only a few grain boundaries to be observed in the irradiated zone. It is believed that the absence of impurities in the aluminum



(a)

(b)

Figure 3. The effect of helium on the formation of voids in Al. a) Pure aluminum sample irradiated to 6 dpa without preinjected helium. Foil normal is (013). b) Pure aluminum sample with 0.1 at.ppm helium at 2 dpa. The foil normal is near (001) with dynamic conditions which also reveal the surface mottling along with the cavities.

Table 1. Irradiation and Void Parameters for High Purity Aluminum Irradiated at 100°C

Dose dpa	Helium Concentration at.ppm	Average Cavity Diameter nm	Average Dose Rate $x10^{-4}$ per sec	Comments
0.6	None	None	4	No voids - few loops
2	None	None	4	No voids - dislocation networks
2	None	None	15	No voids - vacancy loops
6	None	None	4	No voids - loops
0.5	1.0	24	4	Few cavities < 10^{16} cm^3, few loops
2	0.1	58	4	Voids - grain boundary nearby
11	1.0	88	16	Few voids
26	1.0	115	8	Voids along grain boundary very few inside grains

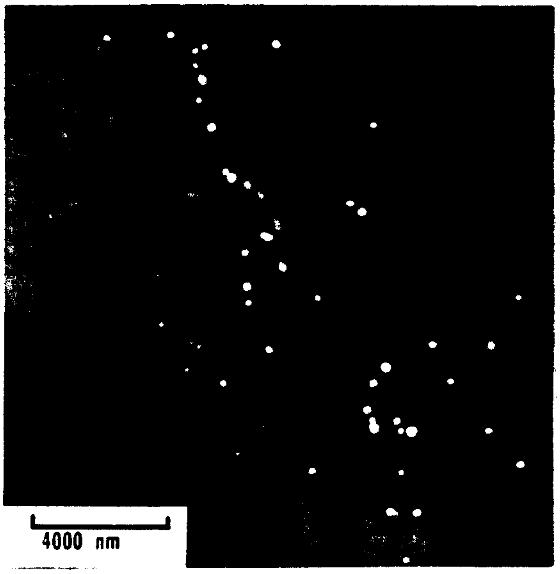


Figure 4. Voids in aluminum at 100°C and 26 dpa with 1.0 at.ppm He. The voids are associated with a grain boundary but are not bubbles on the grain boundary.

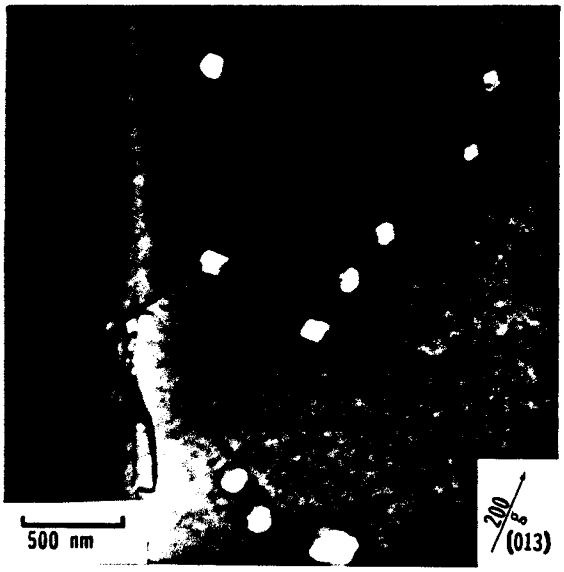


Figure 5. A typical string of voids along a dislocation line in the vicinity of a grain boundary.

prevents the trapping of helium atoms within the matrix and the damage caused by the helium injection is also not adequate to trap the helium. It is quite probable that the helium was trapped at grain boundaries and dislocation networks but only the grain boundaries can be assured to have existed prior to the irradiation. Strings of voids were observed in the vicinity of the grain boundaries and these strings of voids were connected by dislocation lines (e.g., see Figure 5).

It is possible that helium gas atoms are trapped at the grain boundaries and subsequently displaced by primary knock-on atoms. This "atmosphere" of He atoms around the grain boundaries could account for the heterogeneous distribution observed. A similar situation could exist around the dislocation tangles in the matrix, thus accounting for the appearance of voids in those regions.

The percent swelling of aluminum was not calculated because of the extreme variation in the number density of the cavities. The number density varied from essentially zero in the interior of the grain to ~ 1 x 10^{18} cavities per cubic meter within a 10 micrometer wide band that includes the grain boundary. A comparison of the void sizes with dose revealed a cube relationship with respect to the dpa dose. Average void sizes for four different fluences are listed in Table 1 and a plot of the number of vacancies, or atomic volume of the average void size, and the dose is shown in Figure 6. The number of lattice sites is given as $N = V_c/\Omega$ where $V_c = (4/3)\pi[\langle d\rangle/2]^3$ and $\Omega = a_o^3/4$ angstroms cubed and where a_o is the lattice constant for aluminum, 4.05 angstroms. The average void size seems to increase from 0.5 dpa to 25 dpa without any signs of saturation. If the reasonable assumption of a constant number density is used with the observed cavity diameter, then the swelling observed in high purity aluminum is linear with dose.

The samples of aluminum that contain no helium produced only dislocation line tangles and loops. The observed loops were analyzed to be vacancy in nature and had a Burgers vector of 1/2<110>a. The normal to the plane of the vacancy loops pointed in the <111> direction. The vacancy loops are believed to have formed by an edge dislocation climb mechanism. The absorption of interstitials by the edge dislocation causes climb and the vacancy loops could form in the extra half plane of atoms as holes by pinching off dipole segments of the climbing dislocation [8].

4. CONCLUSIONS

1) High purity aluminum containing no helium does not swell at doses of up to 6 dpa.
2) High purity aluminum containing only 0.1 at.ppm of helium develops voids at damage levels of 2 dpa. Voids are also observed in samples irradiated to damage levels of 0.5 dpa and containing helium concentrations of 1 at.ppm.
3) A heterogeneous distribution of voids and dislocations is observed in all helium doped specimens.
4) The absence of significant numbers of voids in the interior of the grains of aluminum is believed to be due to the lack of helium trapping sites in the matrix.

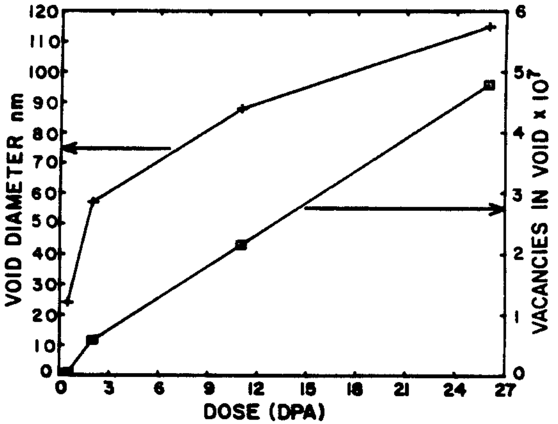


Figure 6. The void size and void volume as a function of dpa dose. The void size is indicated by the cross symbol and the void volume by an open square. The void volume was calculated by assuming a spherical cavity shape and is expressed as the number of vacant lattice sites.

ACKNOWLEDGEMENT

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