



The influence of microstructure on blistering and bubble formation by He ion irradiation in Al alloys



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HIGHLIGHTS

- In Al and Al–4Cu, He bubbles were formed, but no bubbles were observed in Al–5.6Cu–0.5Si–0.5Ge.
- Bubble formation was enhanced at incoherent matrix/precipitate interfaces in Al–4Cu.
- The bubble size was insensitive to displacement rate in pure Al.
- In Al and Al–5.6Cu–0.5Si–0.5Ge blistering was observed, which was more severe in the alloy.
- Blistering effects were enhanced by increasing the displacement rate in Al and Al–4Cu.

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ABSTRACT

The influence of microstructure and composition on the effects of ion irradiation in Al alloys was studied combining Atomic Force Microscopy, Scanning Electron Microscopy and Transmission Electron Microscopy. For this purpose, irradiation experiments with 20 keV He⁺ ions at room temperature were carried out in Al, an Al–4Cu (wt%) supersaturated solid solution, and an Al–5.6Cu–0.5Si–0.5Ge (wt.%) alloy with a very high density of precipitates, and the results were compared. In Al and Al–4Cu, He bubbles were found with an average size in between 1 nm and 2 nm that was independent of fluence. The critical fluence for bubble formation was higher in Al–4Cu than in Al. He bubbles were also observed below the critical fluence after post irradiation annealing in Al–4Cu. The incoherent interfaces between the equilibrium θ phase and the Al matrix were found to be favorable sites for the formation of He bubbles. Instead, no bubbles were observed in the precipitate rich Al–5.6Cu–0.5Si–0.5Ge alloy. In all alloys, blistering was observed, leading to surface erosion by exfoliation. The blistering effects were more severe in the Al–5.6Cu–0.5Si–0.5Ge alloy, and they were enhanced by increasing the fluence rate.

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1. Introduction

Materials in fusion reactors are subject not only to a flux of high energy neutrons, but also to the accumulation of He, that has a very low solubility in metals, through (n, α) reactions and through the decay of tritium [1]. Accumulation of He in metals is responsible for several effects that include void swelling by stabilization of vacancy clusters, formation of bubbles and surface erosion by blistering. At high irradiation temperatures, He accumulation leads to a progressive decay in the mechanical properties, causing embrittlement

[2]. For these reasons, a detailed knowledge of the influence of He incorporation in structural materials for reactors is of fundamental importance to guarantee their safe operation.

The kind of defects that He produces in materials is strongly influenced by their microstructure and composition [3]. For example, in 316 steel, He bubbles form along grain boundaries, causing loss of ductility [4]. In Oxide Dispersion Strengthened steels, which contain a fine dispersion of oxide particles within the matrix, void swelling is dramatically reduced due to the high density of interfaces that represent sinks for He atoms [5].

Although Al is not a candidate material for fusion reactors, it is a model system in which the effects of ion irradiation have been extensively studied by many authors [6–8]. Two different effects were mainly reported: large scale blistering and the formation of nanometer sized bubbles. Blister evolution was shown to be

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strongly dependent on fluence. A critical fluence is necessary to initiate the formation of blisters, and beyond such critical fluence, blisters grow, coalesce and eventually burst, leading to exfoliation. Instead, bubble size was found to be largely unaffected by fluence.

Age hardenable Al alloys exhibit a wide variety of microstructures, depending on composition and thermomechanical treatment. They are therefore adequate to study the influence of microstructure on the effects of He ion implantation. For example, a correlation between He bubbles and precipitation processes was found in Al thin foils implanted with Cu and He ions [9]. However, the effects of He ion irradiation in Al alloys have received little attention.

In this work, the influence of composition and microstructure on He bubble formation and blistering due to He ion irradiation is studied combining Atomic Force Microscopy, Scanning Electron Microscopy and Transmission Electron Microscopy. The effects in pure Al are compared with those in an Al–4Cu (wt.%) supersaturated solid solution and an Al–5.6Cu–0.5Si–0.5Ge (wt.%) alloy containing a very high density of precipitates within the Al matrix.

2. Experimental

Specimens for irradiation experiments were prepared from pure Al, Al–4Cu and Al–5.6Cu–0.5Si–1.3Ge (wt.%) alloys. The alloys were prepared using 99.999% Al, 99.99%Cu, 99.99%Si and 99.99%Ge. Master alloys of Al–Cu, Al–Si and Al–Ge were prepared by induction welding. The alloys with the final composition were prepared by arc welding from the master alloys with the appropriate additions of pure Al.

The alloys were homogenized in sealed vycor tubes with Ar gas for 48 h at 773 K followed by water quenching. In Al–4Cu, this treatment resulted in a supersaturated solid solution. In Al–5.6Cu–0.5Si–1.3Ge, a subsequent ageing treatment of 3 h at 463 K was carried out in order to produce a dense distribution of nanometer sized Si–Ge and Al₂Cu θ' precipitates [10].

Slices with a thickness of 1 mm were cut with a low speed diamond saw, from which disc shaped specimens with 3 mm diameter were extracted by spark erosion. The thickness of the discs was reduced to 0.2 mm by mechanical grinding with 600 grit emery paper. In order to eliminate the damage caused by grinding, the surface to be irradiated was electropolished using a TENUPOL 5 unit, operating with a single jet, with an electrolyte containing 8% sulfuric acid, 2% hydrofluoric acid, 5% glycerol and 85% methanol (vol%). The applied voltage was 20 V and the polishing temperature was around 243 K. A surface roughness of 0.8 nm was determined in a $1\ \mu\text{m} \times 1\ \mu\text{m}$ area with Atomic Force Microscopy, showing that the damage from mechanical grinding was effectively removed.

Ion irradiations were performed with 20 keV He ions at room temperature and normal incidence, at the 120 keV ion accelerator “Kevatrito” at Centro Atómico Bariloche. The experiments were carried out using beam current densities in the range of $1.9\text{--}3.5\ \mu\text{A cm}^{-2}$ and $5\text{--}6\ \mu\text{A cm}^{-2}$, referred in the following to as “low beam current” and “high beam current”, respectively. The displacement cross section was determined using the TRIM code [11]. The distribution of ion ranges and atomic displacements are shown in Fig. 1. From these simulations, an average displacement cross section of $\sigma_D = 5.8 \times 10^{-17}\ \text{cm}^2\text{ion}^{-1}$ was obtained within a depth of 250 nm. Using this value, a fluence of $1.72 \times 10^{16}\ \text{ions.cm}^{-2}$ corresponds to a damage of 1 displacement per atom. In the following, the number of displacements per atom (dpa) within a depth of 250 nm will be used as a measure of damage and will be referred to as “volume damage”. Irradiations were carried out to volume damages of 1 dpa, 3 dpa, 7.5 dpa and 8.5 dpa, corresponding to fluences of $1.72 \times 10^{16}\ \text{ions.cm}^{-2}$, $5.16 \times 10^{16}\ \text{ions.cm}^{-2}$, $1.29 \times 10^{17}\ \text{ions.cm}^{-2}$ and $1.46 \times 10^{17}\ \text{ions.cm}^{-2}$, respectively.

However, in the region close to the specimen surface that is typically investigated by High Resolution Transmission Electron Microscopy (HRTEM), the displacement cross section is lower than the average value obtained above. From the simulation presented in Fig. 1, an average cross section of $\sigma_{DS} = 4 \times 10^{-17}\ \text{cm}^2\text{ion}^{-1}$ for depths lower than 20 nm can be obtained. Therefore, when analyzing specimens irradiated to a given volume damage (in dpa), the number of displacements per atom in the region tested by HRTEM is 68% of the volume damage.

Surface characterization was carried out using an Autoprobe CP Atomic Force Microscope (AFM) from Park Scientific installed at the Atomic Collisions and Surface Physics Division in Centro Atómico Bariloche, using Ultralevers 0.6 μm silicon probes. Surface roughness was calculated from the topographic characterization as the root-mean-square deviation of the height distribution. An area of $2\ \mu\text{m} \times 2\ \mu\text{m}$ was analyzed in all the specimens to allow comparison. These data were complemented with Scanning Electron Microscopy (SEM) images using secondary or backscattered electrons with a FEI NanoSEM230 at the Materials Characterization Division, Centro Atómico Bariloche.

Bulk characterization was carried out with Transmission Electron Microscopy (TEM) using a LaB₆ Philips CM200 microscope equipped with an Ultratwin lens, operated at 200 kV, at the Metals Physics Division in Centro Atómico Bariloche. The electron flux for high resolution imaging was estimated using the approximate relation between exposure time and electron current given by the microscope provider. A value of $6 \times 10^{18}\ \text{electrons.cm}^{-2}\text{s}^{-1}$ was obtained. Using a displacement cross section of 100 b [12], the total displacements per atom can be kept below 0.1 dpa if care is taken not to expose the area under observation more than about 2 min before capturing images.

3. Results

3.1. Low beam current effects

3.1.1. He ion irradiation on pure Al

Fig. 2 shows the AFM (panels A to D) and SEM (panels E to H) surface images as a function of increasing damage. After 1 dpa, the AFM image (A) and its height distribution show the formation of small sized features with a surface roughness of 1.7 nm. The corresponding SEM image (E) shows the formation of blisters with a large size distribution, the smallest of which coincide with the features observed with AFM. The largest peak to valley height was about 9 nm. After 3 dpa, the surface roughness was found to increase to 2.4 nm. A height profile analysis indicates blister growth and coalescence. The SEM image shows large blisters. The smaller blisters are not observed, probably due to improper focusing or to their very low height. Linear features observed at 3 dpa are attributed to preexisting defects in the specific specimens, that is, not caused by irradiation, since they were not observed at any other fluences. After 7.5 dpa, surface roughness increased strongly to 15.4 nm due to further blister growth. The SEM image shows that large blisters have begun to fracture leading to an exfoliation process. After 8.5 dpa, a marked reduction in surface roughness was measured with AFM and large features were observed with SEM. Combining the information from both techniques, the results are attributed to a significant exfoliation process in which large blisters burst and a second generation of small blisters begins to form.

The evolution of surface roughness with irradiation time or fluence (expressed in dpa) for pure Al is shown in Fig. 3, together with the results for the Al–5.6Cu–0.5Si–1.3Ge alloy that will be presented in the following section.

Characterization of the near surface bulk (thickness of the order of 20 nm) was carried out with TEM. No contrast due to dislocations

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