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HELIUM TRAPPING IN ALUMINUM AND SINTERED ALUMINUM POWDERS*

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ABSTRACT

The surface erosion of annealed aluminum and of sintered aluminum powder (SAP) due to blistering from implantation of 100-KeV $^4He^+$ ions at room temperature has been investigated. A substantial reduction in the blistering erosion rate in SAP was observed from that in pure annealed aluminum. In order to determine whether the observed reduction in blistering is due to enhanced helium trapping or due to helium released, the implanted helium profiles in annealed aluminum and in SAP have been studied by Rutherford backscattering. The results show that more helium is trapped in SAP than in aluminum for identical irradiation conditions. The observed reduction in erosion from helium blistering in SAP is more likely due to the dispersion of trapped helium at the large Al-Al_2O_3 interfaces and at the large grain boundaries in SAP than to helium release.

INTRODUCTION

During the operation of thermonuclear reactors the surfaces of components exposed to bombardment by energetic particles from the plasma region can be seriously eroded by radiation blistering.[1,2] In this process, particles from the plasma strike exposed surfaces with sufficient energy to be implanted in the lattice.[2,3] Near the end of their range, where damage is intense and the gas concentration is sufficiently high, gas bubbles form. Such bubbles near the surface region can grow and deform the surface skin to form blisters. Helium blistering, caused by implantation of helium projectiles, can result in serious surface erosion due to peeling of the blister skin upon rupture.

One possible way to reduce surface erosion due to helium blistering is to maintain the surfaces at a high temperature (e.g., > 900°C for Nb and V) at which some of the implanted helium is released without forming large bubbles.[3-5] However, the operating temperatures of various fusion reactor components may be limited by other design criteria. A more desirable solution would be to choose a material with a microstructure which minimizes the formation of blisters. A promising material appeared[6] to be sintered aluminum powder (SAP), which consists of an aluminum matrix strengthened by a dispersion of fine Al_2O_3 particles in concentrations ranging from 3 to 15%. It also has a very small grain size in comparison to pure annealed aluminum. SAP maintains its tensile strength and creep resistance at temperatures approaching the melting point of aluminum. A comparison of the surface erosion due to helium blistering in aluminum and sintered aluminum powder (SAP) had been made for irradiation at room temperature with 100-KeV He^+ ions to a dose of 1.0 C/cm^2. The results showed a reduction in erosion rate in SAP by more than three orders of magnitude from the erosion rate in pure aluminum.[6]

In order to determine if the observed reduction in blistering is due to enhanced helium trapping (e.g., at the trapping sites along the large Al - Al_2O_3 interface) or due to helium release, Rutherford backscattering studies of the implanted helium profile in annealed aluminum and in SAP have been conducted.

EXPERIMENTAL TECHNIQUES

The SAP used in this study was prepared at the Holifield National Laboratory and contained a nominal 10.5% Al_2O_3 (SAP895).[6] Thin discs of SAP of ∿ 60 μm thickness were cut from the billet by spark cutting. The surfaces of SAP were optically polished and then cleaned in ultrasonic baths of trichloroelthylene, acetone, distilled water and methanol, in that order. For Rutherford backscattering measurements the discs were thinned by argon-ion sputtering to a thickness of ∿ 5 μm. The polycrystalline aluminum foils used in this study were of high purity (∿ 99.99%). The samples were mechanically polished, degreased in the same four ultrasonic baths used for SAP, annealed for 2 hours at 300°C in a vacuum of $1 - 3 \times 10^{-8}$ Torr, and finally electropolished in a solution containing 617-ml H_3PO_4, 134-ml H_2SO_4, 240-ml H_2O and 156-g CrO_3 at 70°C. For Rutherford backscattering measurements, thin foils of aluminum were prepared first by electropolishing to a thickness of ∿ 20 μm and then, thinning by argon-ion sputtering to a final thickness of ∿ 3-4 μm. The targets were irradiated at room temperature with 100-keV He^+ ions from a 2-MeV Van de Graaff accelerator to a dose of 0.05 C/cm^2. The vacuum in the target chamber was maintained at ∿ 5 x 10^{-8} Torr by ion pumping during irradiation. After helium-ion irradiation Rutherford-backscattering measurements were made with a 1.5-MeV H^+ beam. The backscattered ions were energy-analyzed using solid-state surface-barrier detectors; the scattering angle was 90°. Some thick (> 75 µm) SAP and Al targets were irradiated to higher doses with 100-keV $^4He^+$ ions and were examined in a Cambridge Stereoscan S4 - 10 scanning electron microscope. Examination of polished SAP surfaces,before irradiation, at high magnifications (resolution < 200 Å) did not reveal any porosity of the surface.

RESULTS

The significant reduction of helium blistering in SAP as compared to annealed aluminum for identical irradiation conditions (100-keV He^+ ions to a dose of 1.0 C/cm^2 at room temperature) is illustrated in Figure 1. Figure 1 (a) shows an enlarged view of a portion of the irradiated area of aluminum. One can see that four exfoliated layers have been removed [layers marked 1 to 4 in Figure 1 (a)]. Figure 1 (b) shows a typical example of blisters formed on SAP. In contrast to the aluminum, where large exfoliation is observed, only a few blisters were ruptured in the case of SAP. The erosion rates estimated from the ruptured and lost skins for aluminum and SAP are 1.75 ± 0.25 and 0.001 atoms per helium ion, respectively, as reported earlier.[6]

The blister-skin thicknesses in the annealed aluminum and SAP were measured from the scanning electron micrographs to be $0.61\ \left({}^{+0.05}_{-0.04}\right)$ µm and 0.66 ± 0.02 µm, respectively. The value for aluminum, based on a number of recent observations,[7] is slightly larger than the one reported earlier,[6] but falls within the error limits quoted. The corresponding calculated projected range for 100 keV $^4He^+$ in aluminum is 0.70 µm,[8] a value which is considered to be accurate within 12%.[9] The calculated peak in the distribution of energy deposited into damage in aluminum for 100-keV $^4He^+$ ions according to Brice[8] is 0.59 µm, with an estimated accuracy of ∿ 12%. It may be noted that the measured skin-thickness values for aluminum and SAP fall between the calculated projected range and the peak in the distribution of energy deposited into damage.

Rutherford backscattering studies of implanted helium profiles in annealed aluminum and in SAP were conducted in order to determine whether the observed reduction in blistering in SAP is due to enhanced helium trapping or due to helium release. This technique has been described earlier.[10] Figures 2 (a) and 2 (b) show Rutherford-backscattering data for 1.5-MeV protons incident on 100-keV helium-ion-implanted aluminum and SAP, respectively. The

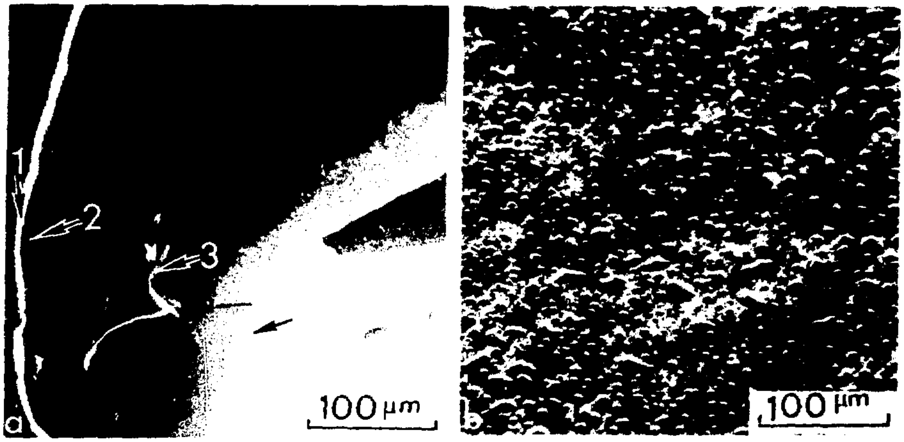

Figure 1. Scanning electron micrographs of surfaces of (a) aluminum and (b) SAP after irradiation at room temperature with 100-keV $^4He^+$ ions to a total dose of 1.0 C/cm^2.

helium-ion dose for both targets was kept at 3.15 x 10^{17} ions/cm^2, low enough to prevent helium release by blister rupture, and the implantations were done at room temperature. An examination of the irradiated surfaces by scanning electron microscopy revealed no visible blisters in either material. The backscattering spectra of 1.5 MeV protons on both aluminum [Figure 2 (a)] and SAP [Figure 2 (b)] reveal the appearence of aluminum, oxygen and helium peaks at onset energies which agree with the calculated onset energies (Figure 2) within the experimental error. One readily notices that the oxygen peak in SAP is significantly stronger than for the aluminum sample as expected. A comparison of the area under the helium peaks [see insets in Figures 2 (a) and 2 (b)] for aluminum and SAP samples reveals that a larger fraction of the implanted helium is trapped in SAP than in aluminum by about a factor of two. The distance between the onset energy and the maximum energy to the helium peak can be converted to a depth scale using known stopping cross-sections for protons in aluminum[8] as shown in the insets of Figures 2 (a) and 2 (b). For SAP the peak of the helium distribution lies at 0.68 ± 0.06 µm [Figure 2 (b)], a value which agrees closely with the calculated projected-range value of 0.70 µm for aluminum and with the observed blister skin-thickness of 0.66 ± 0.02 µm.

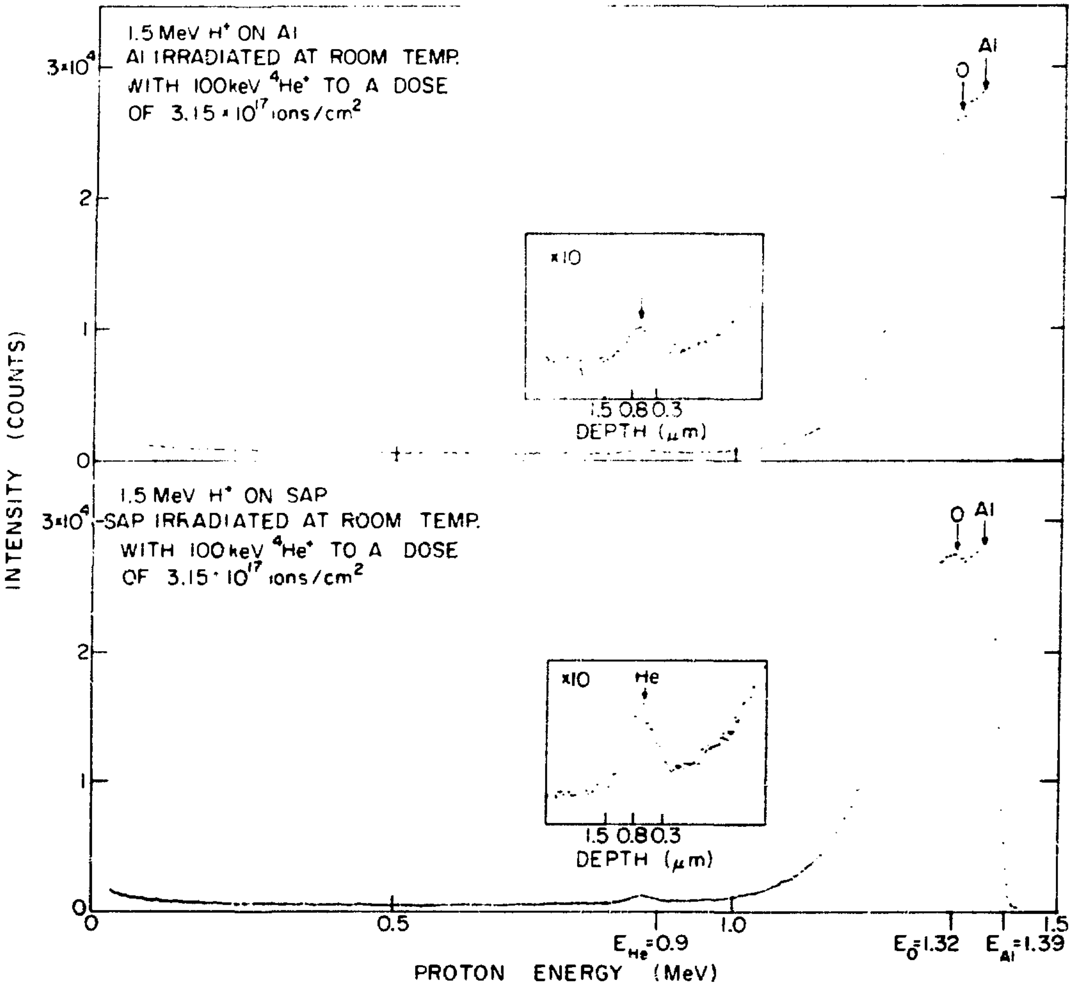

Figure 2. Multichannel analyzer output exhibiting the number of protons backscattered from (a) an aluminum foil (b) a SAP foil (both foils implanted with 100 keV $^4He^+$ at room temperature to a dose of 3.15×10^{17} ions/cm^2) as a function of energy. The incident proton energy is 1.5 MeV. The insets in (a) and (b) show a 10 x magnification of the counts observed for helium peaks. The onset energies for aluminum, oxygen and helium are indicated as E_{Al}, E_O and E_{He}, respectively.

Another set of backscattering data was obtained for a higher dose of 4.4×10^{17} helium ions/cm^2. At this dose no blisters were visible. The observed increase in the area under the helium peaks for both SAP and aluminum corresponded with $\pm$ 5% to the increase in dose. Again, at this dose the trapped helium concentration in SAP was higher by a factor of two than in aluminum.

DISCUSSION

The results presented in this paper indicate that more helium is trapped in SAP than in aluminum for identical irradiation conditions given above. In contrast to aluminum the erosion due to helium blistering in SAP is very low. These results imply that the reduction in erosion due to blistering in SAP at room temperature is not due to an enhanced helium release through the grain-boundaries (a typical average grain size in SAP is $\sim$ 0.5 μm as compared to $\sim$ 300 μm in annealed aluminum[6]). The observed reduction is more likely related to helium trapping at the Al-Al_2O_3 interfaces. In addition the large grainboundary area in SAP (which is more than 5 orders of magnitude larger then for the annealed aluminum used here) provides very effective trapping sites for helium. A dispersion of trapped helium will help to prevent helium-bubble coalescence to larger diameters and subsequent blister rupture and exfoliation. There exists other evidence for helium bubble nucleation at the Al-Al_2O_3 interface in aluminum alloys containing dispersed Al_2O_3 particles.[11] Furthermore, the fact that the yield strength of SAP ($\sim$ 35,600 psi) is much higher than for annealed aluminum ($\sim$ 1,700 psi) will help to reduce the blister rupture and exfoliation in SAP from that observed in aluminum.

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