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RADIATION-INDUCED SEGREGATION IN LIGHT-ION BOMBARDED Ni-8% Si*

N. H. Packan[†], L. Heatherly[†], W. Kesternich[#], and H. Schroeder[#][†]Metals and Ceramics Division, Oak Ridge National Laboratory,
P. O. Box X, Oak Ridge, TN 37831[#]Institute for Solid State Research, Jülich Nuclear Research
Facility, Jülich, Federal Republic of Germany

Radiation-induced segregation (RIS) is an important area of current research because of potential detrimental effects including embrittlement. In this work, a quantitative assessment is made of radiation-induced silicon segregation to internal grain boundaries compared with that to the external surfaces. Tensile specimens of Ni-8 at. % Si have been irradiated at 475°C (750 K) with either 7 MeV protons or 28 MeV alpha particles to damage levels of 0.1 to 0.3 dpa. Postirradiation tensile testing at 20, 475, or 700°C produces ductile failures with elongations >15%. However specimens which had implantation of 250–1000 at. ppm He at 700°C (975 K) prior to irradiation did fracture intergranularly when pulled at 700°C inside a scanning Auger microprobe, thereby exposing grain boundary surfaces for depth profile analysis. After correction for preferential sputtering effects, RIS of silicon to outer surfaces is found to give rise to a strongly-enriched surface layer 20–30 nm thick followed by a shallow depletion zone for the next ~200 nm, in agreement with previous studies in the literature. By contrast, silicon segregation to fracture-exposed grain faces varies considerably from face to face but generally declines more

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gradually with little or no depletion zone. Observations of silicon profiles and Ni_3Si formation at grain boundaries obtained by analytical TEM generally confirm the Auger results.

INTRODUCTION

Radiation-Induced Segregation (RIS) is an active research area because RIS processes have the potential of undermining the performance of structural alloys exposed to radiation. Studies in neutron-irradiated austenitic superalloys, for example, have found marked ductility reductions which were attributed to intergranular fracture that may have been promoted by RIS-generated layers of grain boundary precipitate [1,2]. Fundamental investigations of RIS phenomena have focused on simpler systems such as silicon in nickel. Silicon is an element of great interest because it has a major influence on void swelling [3] and also has a strong tendency to segregate during irradiation. It appears to couple readily to the interstitial flux and migrate to appropriate sinks such as dislocation loops, internal interfaces and the external surface [4,5]. Numerous studies have been conducted using secondary ion mass spectrometry [5], Auger electron spectroscopy [6,7] or Rutherford backscattering spectroscopy [8] techniques to follow the evolution of silicon segregation at the charged-particle-bombarded surface of Ni-Si alloys. What has not yet been explored is the correlation between segregation to the external surface with RIS profiles at internal grain boundaries, the latter having great relevance to possible deterioration of materials properties such as

irradiation-induced embrittlement or intergranular corrosion. Such a comparison is the subject of this investigation.

EXPERIMENTAL DETAILS

The material employed in this experiment was a nickel-8 atomic per cent silicon (about 4 wt % Si) alloy. This composition lies well below the solubility boundary (about 10.5 at. % Si) at room temperature. The components, 99.99% pure Ni and 99.999% Si, were obtained from Materials Research Corp. and the alloy was made by arc-melting and remelting several times. Rolling, with intermediate anneals of 15 min. at 800°C (1075 K) in argon, yielded sheet with a final thickness of 0.055 to 0.060 mm. Flat tensile specimens were spark cut to a shape consisting of a 2 × 8 mm straight gage section leading, through 1 mm radii fillets, into square 8 × 8 mm grip portions with central 2.7 mm diam. holes. The final anneal before bombardment was carried out in a 10⁻⁴ Pa vacuum. Annealing conditions of 800°C for about 7 min. were chosen as a standard treatment after various trials seeking recrystallization with minimum grain growth. Nevertheless the as-bombarded specimens contained grains ranging in size from small (<10 μm, often in clusters) to large, 40 to 80 μm.

Bombardments were carried out at the compact cyclotron facility of the Institute for Solid State Research (IFF) of the Jülich Nuclear Research Center in Germany. Details of the damage chamber and bombardment procedures have been published previously [9,10]. Control of specimen temperature was accomplished with the use of five pairs of helium jets whose

flow rates are individually controlled. This permitted maintaining the desired bombardment temperature ($475 \pm 5^\circ\text{C}$) over the entire gage length, as determined by an infrared pyrometer that could scan the specimen in both the width and length dimensions. Specimens were held with a small tensile stress, about 25 MPa, to keep them straight and the bombarded area was 2×10 mm.

Damage was created by beams of either 7 MeV protons or 28 MeV alpha particles which were normally caused to stop throughout the specimen thickness by means of a degrader wheel. Damage production rates of about 3×10^{-6} dpa/s (3×10^{14} ions/cm².s) for the H⁺ ions and 5.5×10^{-6} dpa/s (2×10^{13} ions/cm².s) for the He⁺⁺ ion beams resulted in the attainment of doses of 0.1 to 0.3 dpa for bombardment times from 7.5 to 28 h. The alpha particle bombardments inevitably introduced helium as well as damage. For example, the irradiation of Specimen No. 2 with a 5 μA beam of 28 MeV alphas generated (per hour) about 140 at. ppm He together with 0.02 dpa damage.

The irradiation temperature in all cases was 475°C (750 K). This temperature is thought to be approximately the peak temperature for RIS in this alloy and these damage rates, based upon the observations of Okamoto [11] that maximum RIS occurs about 40° below the peak swelling temperature for pure nickel.

The first few postirradiation fractures of either alpha or proton bombarded specimens exposed no intergranular surface area to study. Some specimens were therefore given an elevated temperature (700°C , 975 K)

preimplantation of from 250 to 1000 at. ppm helium before irradiation. These specimens were fractured after irradiation inside an ultrahigh vacuum chamber. Direct ohmic heating with alternating electrical current was used to rapidly heat them to the desired temperature of 700–750°C before fracturing. One portion of each specimen was then transferred under UHV conditions ($<10^{-7}$ Pa) to a Physical Electronics Industries model 590 Scanning Auger Microprobe (SAM) for surface analysis and depth profiling.

The Auger microprobe consisted of a cylindrical mirror analyzer incorporating a coaxial electron gun with a minimum beam size of 200 nm. The sputtering operations for depth profiling were carried out using a Kratos Minibeam II differentially-pumped grazing incidence ion gun that was capable of being rastered. A small beam (<100 μm diam) was rastered over a large area and the Auger analysis was simultaneously conducted on a small spot, typically <30 μm wide, near the center of the sputtered region. The sputtering rate for this system was standardized by sputtering through a layer of Ta_2O_5 of known thickness and assuming that the rate for Ni-8% Si is similar. An independent measurement of the sputter rate was carried out by observing dimensional changes of one of the fracture surfaces of a Ni-8% Si specimen. A portion of fracture surface was sputtered long enough to remove approximately 10 μm of material. By comparing the SEM images from before and after the sputtering, the depth of material removed was determined and this value agreed well with the value obtained from the Ta_2O_5 film calibration. In order to get semiquantitative values for the silicon concentration, the 92 eV silicon and the 848 eV nickel Auger peaks

were employed, with sensitivity factors of 0.28 and 0.27, respectively, as provided by the manufacturer of the scanning Auger microprobe.

RESULTS

Tensile tests of a number of specimens were carried out at either room temperature or 450°C (725 K) and the results are presented in detail elsewhere [12]. A summary of the irradiation conditions and the mechanical property data is given in Table 1. For the proton-bombarded specimens there were minor changes in the fracture stress or elongation values compared to the unirradiated control specimens, which were quite ductile. For specimens damaged by alpha particles there was a further increase in the fracture stress and about a 50% drop in elongation. Still, fractures were essentially ductile. One of these chisel-edged ductile fractures is seen in Fig. 1. Only the specimens which were intentionally embrittled by 700°C helium implantation and fractured at ~700°C exposed interior grain faces.

In order to obtain quantitative composition profiles using Auger Electron Spectroscopy, sputter depth profiling was conducted on control material to look for possible preferential sputtering effects. The profiles in Fig. 2 come from a portion of the unexposed grip region of proton-bombarded specimen No. 3. One curve shows the silicon profile inward from the external surface after first sputtering 10 s (removal rate of 0.09 nm/s) to remove the initial oxide layer. The second curve pertains to an area within a deep scratch that was administered inside the Auger apparatus to expose bulk material. Its very first data point exhibits the

expected "true" bulk concentration value of 8% silicon, but thereafter the process of preferential sputtering rapidly establishes (for both profiles) an indicated steady-state value of 14–16 at. % Si. Moreover, an initial analysis (without sputtering) of some sputtered ejecta on a nearby region yielded a value of 8 at. % silicon, indicating that the true concentration of material actually removed by sputtering was 8%. Based on these findings, in the profiles of bombarded material to follow, a band between 14 and 16 at. % Si should be regarded as actually representing the initial 8% silicon concentration for sputtered depths >5 nm.

The depth variation of silicon below the outer surfaces of two ion bombarded specimens is presented in curves A, B, and C in Fig. 3. The A and B curves pertain to locations quite close to the fracture surface and therefore, like it, represent material that briefly reached 700–750°C during the in-situ fracture. Since these were external surfaces on air-exposed samples it was necessary to remove the surface layers of oxide and contamination. In these two cases these layers were about 12–13 nm thick, consisting primarily of carbon and silicon oxides, which required 90–100 s of sputtering to remove. The C curve, by contrast, is from the same proton-bombarded specimen but a different portion which was not pulled at ~700°C and thus remained at room temperature since the end of bombardment. Shorter preliminary sputtering, 60 s or about 8.5 nm, was applied before the collection of these data points. Other than the C data yielding an initial silicon concentration about 50% higher than the A and B curves, the overall shape of each of these external surface RIS profiles is the same:

a strong peak in the first 10–20 nm followed by a broad, shallow Si-depleted region for the 20–200 nm depth range. Both the shape and the depth values are similar to previously reported studies in Ni-Si alloys [6–8] all of which likewise pertained to the external surface regions of ion-bombarded specimens.

Also shown in Fig. 3 are depth profiles for three exposed grain boundary faces from the fracture surface of a proton-irradiated specimen. The grains, which were simultaneously sputtered, are identified on the fracture micrograph, Fig. 4. The depth profile sites were never close to the specimens' outer surfaces. There clearly are considerable grain-to-grain variations in the levels of silicon at a given depth. Such variations were the rule whenever a number of grains were profiled. A collection of six grains farther to the right of those in Fig. 4 exhibited similar variations. In Fig. 5 two interior grain profiles (D and F from Fig. 3) for proton-bombarded specimen No. 3 are compared with typical profiles from specimen No. 7 that was alpha-bombarded. Both have 0.3 dpa damage and again there is notable grain-to-grain spread. A common characteristic for all the interior grain face profiles, however, was that they lacked the depletion zone exhibited by all the exterior profiles (compare curves D, E, F with A, B, C in Fig. 3).

Several attempts were made to try to map the silicon signal on portions of individual grain faces (during a profile, at about 100 nm deep). There were indications of Si-rich and poor spots (sizes $>2\ \mu\text{m}$) deviating as much as 3 at. % above or below the average values which were about 19 at. %

(indicated) at that time, but local topography variations make these spot determinations unsure.

Another way of observing silicon enrichment is the imaging of the ordered Ni_3Si phase using transmission electron microscopy (TEM). Formation of the gamma prime (Ni_3Si) phase was recorded on about twenty different grain boundaries by dark field TEM [13]. One example is shown in Fig. 6 for a grain boundary in specimen No. 5 (proton irradiated to 0.13 dpa). A superlattice reflection from the Ni_3Si phase of the upper grain in Fig. 6 was used for this image which reveals gamma prime phase at the dislocation loops in the upper grain and at the grain boundary. The grain boundary precipitation sometimes gives the impression of being a single layer with antiphase boundaries and in other cases seems to consist of discrete small particles. A noteworthy point, however, in this and the other grain boundaries observed is that the precipitate layer never formed symmetrically with respect to the boundary but developed only on one side, except for coherent twin boundaries where Ni_3Si precipitate formation did develop symmetrically on both sides of the boundaries. Sometimes the precipitate layer was seen to switch over from one side of a grain boundary to the other.

Analysis of the silicon concentration near grain boundaries has also been carried out on some of these specimens using quantitative Energy Dispersive X-ray Spectroscopy (EDXS) in a transmission electron microscope [13]. Sequential EDXS spectra traversing such boundaries revealed strongly-enriched regions (to about 18 at. % Si) about 20 to 30 nm wide which is

about the average width of the precipitate bands seen in dark field TEM. Beyond the precipitate layer the Si concentration dropped sharply to a constant value of ~8%. This behavior, including the absence of any significant depletion zone, is quite consistent with our Auger observations for internal grain boundaries, although the Auger profiles depict a more gradual decay of the Si concentration (beyond about 30 nm from the boundary) compared to the EDXS data. The reason(s) for the latter discrepancy between SAM and EDXS results may be partially understood by considering the characteristics of the two techniques. TEM/EDXS offers an accurate report from a chosen but extremely small volume. By comparison even the SAM capability of depth profiling a ~30 μm diam spot on a given grain face is practically "macro" scale — such inhomogeneities as grain face topography and place-to-place variations in the thickness of the Ni_3Si precipitate layer (or even its absence, as when it switches sides of the boundary) will be averaged-over in the Auger analysis. Any sputtering rate differences due to such inhomogeneities could further stretch out the depth range from which enhanced silicon is being seen.

DISCUSSION

In contrast to heavy-ion irradiations [5-8] in which radiation effects are confined to a thin (<1 μm) layer just beneath the bombarded surface, these light-ion bombardments have offered the first opportunity to assess the effects of radiation-induced segregation in a quasi-bulk environment and to obtain high-depth-resolution RIS profiles from interior

grain boundaries. In spite of substantial silicon segregation that did occur to all grain boundaries investigated, no embrittlement resulted. Recourse therefore had to be made to helium embrittlement to probe the specimen interiors. The question arises whether the brief time (roughly half a minute) at $\sim 700^{\circ}\text{C}$ for fracture may have caused any significant relaxation of the RIS profiles. For answer one may compare the outer surface profiles of Fig. 3 which show (other than a markedly higher near-surface Si level for the unheated material, curve C) a close similarity for all three curves (A, B, and C). There is essentially no inward spreading of the A and B profiles — hence no signs of segregation relaxation by thermal diffusion. The lower near-surface Si values for the A and B profiles may reflect the likelihood that these sharply-heated materials gave up silicon to their 50%-thicker surface layers which have had to be removed. Corroboration of this picture of the surface region has been provided by a TEM foil of specimen No. 3 back-thinned to the surface which exhibited a 10 to 20 nm thick amorphous layer composed only of silicon (plus EDXS-undetectable oxygen, presumably). The A and B curves may therefore be underestimates of the true near-surface Si concentration, but all three profiles are equivalent after ~ 5 nm.

Since depth-profile Auger analyses could only be conducted on helium-embrittled specimens, the question of whether helium itself altered the development of silicon segregation or precipitation must be referred to the TEM investigations. From those there appeared to be no systematic differences in the appearances or widths of Ni_3Si grain boundary layers for

proton versus alpha-particle-bombarded material. Where there were large grain boundary cavities in the high-temperature helium preimplanted specimens, these were observed to have surrounding shells or coatings of gamma-prime precipitate but the boundary layers otherwise also looked the same.

There is a disagreement between this study and those of Rehn and Okamoto [6,7,11] regarding whether there is any significant influence of preferential sputtering upon the depth profiles of nickel-silicon alloys. Rehn et al. observed essentially no preferential sputtering when they sputtered unbombarded control material (Ni-1% Si), but as in all of their examinations the surface probed was an external surface which initially had to be briefly sputtered to remove oxide and carbonaceous contamination. Their control profile (Fig. 6 in Ref. 6) could not include a zero value (no sputtering). Our control analysis was on material that was scratched in vacuum and thereby measured the silicon concentration of the bulk of an 8 at. % Si specimen. The effects of preferential sputtering were evident when this specimen was then sputtered and the surface concentration quickly reached values in the 14-16 at. % range. It is this indicated silicon level — the 14-16% band — which must be used to evaluate radiation-induced segregation behavior. Our control calibration applies only to our sputtering conditions and to compositions in the vicinity of 8 at. % Si; we plan but have not yet carried out tests on in-situ scratched or broken Ni_3Si (~24% Si).

The persistent variation of profiles from grain to grain is a noteworthy observation; such variability has not been characteristic of

previous outer-surface RIS determinations. Some of this may be ascribed to sputtering differences in differently-oriented grains, although care was taken to select grains appropriately facing the sputter gun and Auger analyzer. A stronger factor was probably the observed tendency of Ni_3Si precipitation (and presumably RIS generally) to develop asymmetrically at grain boundaries. Upon grain boundary separation at fracture, some grains may have been exposed with full-thickness RIS layers while their former neighbors might exhibit relatively little segregated material. Indications of patchy RIS development on a given grain boundary, suggested both by Auger silicon mapping and the TEM observations of gamma prime layers switching sides, may contribute a yet-finer-scale variability. Finally, and of most fundamental interest, the sink strength for interstitials (and silicon) may intrinsically depend upon the type of boundary, presumably increasing as one progresses from twins through low angle and high angle grain boundaries to a maximum for the outer surface. Supporting evidence is provided by the observations of differing widths and concentration maxima in the TEM-dark field and TEM-EDXS studies [13]. As one example, the gamma prime layers were consistently narrower for twin boundaries; they are evidently less effective sinks as a class compared to grain boundaries. The outer surfaces, on the other hand, may have been such strong sinks for silicon (some fraction of it probably sputtered off with the preliminary contamination layer removal) that they gave rise to the zones of depleted silicon observed adjacent to them (but not seen next to the internal boundaries).

SUMMARY

Tensile specimens 60 μm thick of Ni-8 at. % Si have been bombarded at 475°C to doses of 0.1 to 0.3 dpa with either 7 MeV proton or 28 MeV alpha particle beams. Deliberate embrittlement by high temperature (700°C) preimplantation of helium was required to produce intergranular fracture. Depth profile sputtering and analysis in a Scanning Auger Microprobe was then used to study radiation-induced segregation of silicon both at the external surfaces and at internal interfaces. The external surfaces exhibited a strongly silicon-enriched zone for the first 10–20 nm followed by a broad (~ 200 nm), shallow silicon-depleted region. Segregation of silicon to grain boundaries varied from interface to interface and possibly from region to region on a given interface. In general, however, depth profiles of silicon content with distance from internal boundaries showed no noticeable depletion zone and a more gradual fall-off compared to the profiles from external surfaces. The variations of RIS among boundaries and with type of interface probably reflect, at least in part, intrinsic differences in sink efficiency.

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FIGURE CAPTIONS

Fig. 1. Ductile fracture exhibited by a specimen (No. 9) bombarded at 475°C with 7 MeV protons to 0.1 dpa and tested at 450°C.

Fig. 2. Profiles of the silicon concentration inward from the external surface of unbombarded Ni-8 at. % Si material illustrating immediate development of an elevated value due to preferential sputtering of nickel.

Fig. 3. Silicon depth profiles for the external surfaces of proton (specimen No. 3) and alpha (specimen No. 7) bombarded specimens compared with profiles from several internal grain boundaries of the proton-bombarded specimen.

Fig. 4. View of the intergranular fracture surface of proton-irradiated specimen No. 3 showing the location and appearance of grain boundary regions 5b, 6, and 6b whose silicon depth profiles are seen in Figs. 3 and 5.

Fig. 5. Comparison of the silicon depth profiles for grains in a proton-bombarded (No. 3) and an alpha-bombarded specimen (No. 7).

Fig. 6. Grain boundary layer of discrete gamma prime (Ni_3Si) precipitates in a proton-irradiated specimen as revealed by dark field transmission electron microscopy.

Table 1

Spec. No.	Pre-impl. He (ppm)	Dam. Bomb. 475°C dpa	He (ppm)	Postirr. Mech. Props.				Other Studies
				Test Temp (°C)	yld (MPa)	uts (MPa)	elong (%)	
CONTROLS								
6	--	--	--	20	133	432	39	
8	--	--	--	20	133	452	35	
10	--	--	--	450	99	334	25	
BOMBARDED by 28 MeV ALPHAS								
2	--	0.2	1500	20	328	561	17	TEM
7	260	0.3	2300	20	336	611	18	Fract.in SAM;TEM
11	1000	0.15	1100	--				TEM
12	1000	0.3	2300	450	270	397	2.5	TEM
BOMBARDED by 7 MeV PROTONS								
3	525	0.3	--	--				Fract.in SAM
4	--	0.13	--	20	198	504	28	Fract.in SAM (ductile)
5	--	0.13	--	--				TEM
9	--	0.1	--	450	151	391	24

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