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REDUCTION OF DEFECT FLUXES USING DUAL-ION-BEAM PROCESSING

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ABSTRACT

Radiation-induced segregation (RIS) in Ni-12.7% Si and Cu-1% Au alloys was studied using Rutherford backscattering spectroscopy during He and Ne irradiation at elevated temperatures. During single ion-beam irradiation with 1.5 MeV He, strong RIS of Si toward the surface was observed in Ni-12.7% Si. Simultaneous irradiation with 400 keV Ne and 1.5 MeV He almost completely suppressed the Si segregation, even when the calculated damage production rate by Ne was only a few percent of that by He ions. A similar effect of dual-beam irradiation was observed in the Cu-1% Au alloy, i.e., the rate of near surface Au depletion was strongly reduced under simultaneous irradiation. The present result shows that dual-beam irradiation can be applied to control RIS and RED (Radiation Enhanced Diffusion) during ion beam processing.

INTRODUCTION

As shown by much work over the past two decades[1], significant nonequilibrium segregation of alloying elements has been observed in a number of alloys following irradiation at elevated temperatures. This Radiation-Induced Segregation (RIS) is caused by the interactions of radiation-produced defect fluxes with alloying elements. RIS may become a serious problem for ion beam processing at elevated temperatures, because it can cause composition changes in alloys during irradiation. In this paper, we demonstrate that simultaneous irradiation with Ne can strongly suppress the RIS by He irradiation in Ni-12.7% Si and Cu-1% Au alloys.

EXPERIMENTAL PROCEDURE

Ni-12.7% Si specimens were irradiated simultaneously with 1.5 MeV He and 400 keV Ne ions at 500 °C. During irradiation, Rutherford backscattering (RBS) spectra from the He ions were acquired simultaneously every 10 minutes. Ion currents were kept constant (100 nA through a 1 mm diameter aperture for He, and 1 nA, 0.25 nA and 0.1 nA through a 3 mm diameter aperture for Ne). Actual current densities, j , and the calculated dpa (displacements per atom) rate, K^{calc} , in the near surface region are listed in Table I. The dpa rate was calculated using the TRIM computer

code[2] and a displacement threshold energy of 33 eV. In Table I, the ratios of calculated dpa rate by Ne to that by He are also listed. The geometry for the measurements is shown in the insets of Fig. 1. In this experiment, the He ion beam was used both for defect production and for RBS analysis. For comparison, RBS spectra were also measured for a specimen pre-irradiated with Ne ions. Similar simultaneous irradiation and RBS measurements were performed in Cu-1% Au specimens at 400 °C.

TABLE I. Irradiation parameters.

Ion	I(nA)	j(nA/cm ²)	K ^{calc} (dpa/s)	K ^{calc} (Ne)/K ^{calc} (He)
1.5 MeV He	100	1.3x10 ⁴	7.6x10 ⁻⁵	-
400 keV Ne	1	14	1.9x10 ⁻⁵	0.25
	0.25	3.5	4.8x10 ⁻⁶	0.063
	0.1	1.4	1.9x10 ⁻⁶	0.025

RESULTS AND DISCUSSION

Figure 1(a) shows RBS spectra from a Ni-12.7% Si specimen before and after irradiation with 1.5 MeV He ions to a dose of $1.1 \times 10^{18}/\text{cm}^2$ at 500 °C. Reduction in the Ni RBS yield near the Ni leading edge is clearly observed after irradiation. Previous TEM and RBS studies show that this reduction is attributed to a strong RIS of Si to the surface and the precipitation of γ' -Ni₃Si[1]. The RIS is also observed as an increase in the Si yield at and just behind the Si edge. This segregation is caused by the existence of a persistent flux of defects to the surface and a preferential coupling of Si atoms to these defects. The amount of Si segregation (or the depletion of Ni) near the surface can be readily determined from the difference between the RBS spectra for irradiated and unirradiated specimens. The change in area in the spectrum near the Ni edge, or the Si edge, is directly proportional to the amount of segregation.

Figures 1(b) and 1(c) show the RBS spectra obtained after simultaneous He and Ne ion irradiation for 4 hours. RIS of Si to the surface is strongly suppressed by the simultaneous irradiation, even when the calculated dpa rate for Ne is only a few percent of that for the He ions. With increasing Ne beam current, the RIS decreases even further.

The suppression of RIS can be seen more clearly in Fig. 2. In this figure, the amount of segregation in Ni-12.7% Si is plotted as a function of He dose for only He ion irradiation, and for simultaneous He and Ne irradiation. During only He ion irradiation, the amount of segregation increases with increasing He dose. During simultaneous irradiation, the segregation rate is much smaller than that during only He irradiation, becoming nearly zero for the conditions in Fig. 1(c).

Fig. 1. RBS spectra for 1.5 MeV 4He ions incident on Ni-12.7% Si before irradiation (open circles), and after (a) 1.5 MeV He irradiation, (b) simultaneous 1.5 MeV He and 400 keV, 0.1 nA Ne irradiation, and (c) simultaneous 1.5 MeV He and 400 keV, 1 nA Ne irradiation (closed circles). Note the large drop in Ni yield near the Ni leading edge due to the segregation of Si toward the surface in (a).

Fig. 2. Amount of near-surface segregation in Ni-12.7% Si specimens during simultaneous 1.5 MeV He and 400 keV Ne irradiation for two different Ne beam currents. For comparison, segregation for the He only irradiation (open circles) is also plotted.

Fig. 3. Amount of near-surface segregation in Ni-12.7% Si specimens measured during 1.5 MeV He irradiation after pre-irradiation with 400 keV Ne (1 nA, 1 hour at 500 °C), and during simultaneous 1.5 MeV He and 400 keV, 0.25 nA Ne irradiation.

To compare the effect of simultaneous irradiation with any pre-irradiation effect on RIS, the following experiment was performed. A Ni-12.7% Si specimen was irradiated with 400 keV Ne at 500 °C to a dose of $3.2 \times 10^{14} \text{ cm}^{-2}$. The Ne beam current was 1 nA through the 3mm diameter aperture, and the irradiation time was 1 hour; the result is shown in Fig. 3. In the figure, the amount of segregation during simultaneous He and 0.25 nA Ne irradiation for 4 hours is also shown. Comparison of the two experimental curves in Fig. 3 shows clearly that the simultaneous He and Ne irradiation is far more effective than pre-irradiation with Ne for suppressing segregation. The total dose of Ne was the same in both experiments, i. e. $3.2 \times 10^{14} \text{ cm}^{-2}$.

To test the universality of the suppression effect, similar measurements were made for Cu-1% Au at 400 °C, with He and Ne currents of 100 nA through the 1mm diameter aperture, and 1 nA through the 3mm diameter aperture, respectively. In this alloy, RIS causes a depletion of Au atoms near the surface[3]. Figure 4 shows that the strong suppression effect is also observed in Cu-1% Au, although these two alloy systems have quite different defect properties.

As RIS is driven by defect fluxes to the surface, our experimental results imply that simultaneous Ne irradiation strongly reduces these defect fluxes. Ne irradiation can affect the production of freely migrating defects, and the concentration and structure of internal sinks. A reduction of the production rate of freely migrating defects or an increase in sink concentration would both reduce the defect flux. The mechanism of defect flux reduction therefore remains uncertain. In any case, simultaneous Ne irradiation suppresses RIS effectively even for very small currents, and thus may provide a useful method for controlling RIS and RED during ion beam processing.

Fig. 4. Amount of near-surface segregation in Cu-1% Au specimens during only He ion irradiation, and during simultaneous 1.5 MeV He and 400 keV, 1 nA Ne ion irradiation.

SUMMARY

Radiation induced segregation (RIS) in Ni-12.7% Si and Cu-1% Au alloys during 1.5 MeV He and 400 keV Ne ion irradiation was measured at elevated temperatures using Rutherford backscattering spectroscopy. In both alloys, RIS to the surface was strongly suppressed during simultaneous irradiation with ion beams of 1.5 MeV He and 400 keV Ne. The suppression effect of the simultaneous irradiation is much stronger than the effect of pre-irradiation.

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