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Argonne, Illinois 60439

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ION BEAM MIXING OF MARKER LAYERS IN Al AND Si

by

S. Mantl,<sup>a,b</sup> L. E. Rehn,<sup>b</sup> R. S. Averback,<sup>b</sup> and L. J. Thompson, Jr.<sup>b</sup>

<sup>a</sup>Institut für Festkörperforschung,  
Kernforschungsanlage Jülich,  
Postfach 1913, D-5170 Jülich

<sup>b</sup>Materials Science and Technology Division  
Argonne National Laboratory  
Argonne, Illinois 60439 U.S.A.

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Abstract

Ion beam mixing experiments on thin Pt, Au, and Ni markers in Al and Si have been performed at 17, 85, and 300 K. After irradiation with 300-keV Ar ions the broadening and relative shifts of the markers have been determined by RBS measurements. The marker broadenings are more pronounced in Si than in Al; in both matrices the broadenings decrease in the following order: Au, Pt, and Ni. No dependence of mixing on irradiation temperature was observed between 17 and 300 K. The shifts of the heavy Au and Pt markers relative to the Ni markers are approximately equal to the experimental accuracy. However, a shift of the Ni marker toward the surface relative to the heavier Au and Pt markers was consistently observed.

## INTRODUCTION

Irradiation of a solid with energetic ions results not only in the production of vacancies and interstitial atoms, but also in the related phenomenon of atomic mixing. Defect production in energetic displacement cascades has been studied for many years, and still, only a very limited understanding of this process has been achieved. It is not surprising, therefore, that the mechanisms underlying ion beam mixing are also difficult to elucidate. An important step in developing an understanding for mixing processes is the collection of a broad and reliable data base. Most data on ion beam mixing to date have been obtained for semiconductor systems, usually Si.<sup>(1)</sup> In this paper, the result of ion beam mixing of various markers in Al and Si are reported. These systems were studied so that a direct comparison between metallic and semiconductor systems of nearly identical atomic mass and density could be made. These two systems are also favorable to backscattering analyses of the mixing profiles. Another feature of this work is that dual markers, i.e., two different elemental markers, were employed in each sample. This procedure made it possible to measure the relative mixing of different markers in the same host under identical irradiation conditions; it also provided a simple method to measure the relative shift of the centroid of the markers in a host material.

## EXPERIMENTAL

The marker samples were prepared by sequential e<sup>-</sup>-beam vapor deposition of host and marker layers onto Si wafers or sapphire disks. The substrates were first highly polished, then lightly acid-etched, and given a final rinse in methanol. The background pressure in the cryopumped deposition chamber was  $< 1 \times 10^{-4}$  Pa during evaporation, and  $< 10^{-5}$  Pa, otherwise. The marker layers were each  $\sim 5 \text{ \AA}$  thick and were initially located at a depth of  $\sim 500 \text{ \AA}$ . Dual markers of Ni-Au and Ni-Pt were employed in both Si and Al hosts. The samples were mounted on a liquid helium flow cryostat which could be operated between  $\sim 6\text{K}$  and  $300\text{K}$ . Irradiations were performed using 300-keV Ar with the sample normals tilted  $10^\circ$  from the beam axis. Preliminary backscattering spectra were acquired using 300-keV He<sup>+</sup> immediately after each mixing irradiation at temperatures

of 10, 80, and 300K. A second 300-keV He<sup>+</sup> backscattering spectrum was acquired for each specimen after warming to room temperature; no changes in the profiles were observed due to warming. Finally, more accurate backscattering analyses were performed on the same samples using 2.0-MeV He<sup>+</sup>, it is the latter results that are reported here.

Standard procedures were employed to analyze the backscattering data.<sup>2</sup> Some of the marker yields, however, were slightly asymmetric. Therefore, the data were fit to a "joined" gaussian distribution of the form,

$$y(X) = [1 + \text{SGN}(X - X_0)] \frac{A}{2} \exp\left(-\frac{(X - X_0)^2}{2\Omega_1}\right) + \\ (1 - \text{SGN}(X - X_0)) \frac{A}{2} \exp\left(-\frac{(X - X_0)^2}{2\Omega_2}\right) \quad (1)$$

This procedure is more accurate for determining the peak position, X<sub>0</sub>, than is fitting the data to a simple gaussian distribution. The marker broadening was obtained from Ω<sub>1</sub> using the equation,

$$\Delta \Omega = (\Omega_1^2(\text{irrad}) - \Omega_1^2(\text{unirrad})) \quad (2)$$

The ratio of the variances, Ω<sub>1</sub>/Ω<sub>2</sub> showed a small tendency to increase with dose, and this effect was somewhat more pronounced for the low temperature irradiations. Assymetry in marker broadening has also been reported for Pd markers in Si, although this observation was for high, rather than low temperature irradiations.<sup>(3)</sup>

## RESULTS

The results for the marker broadenings in Al are shown in Fig. 1. Here the variances, Ω<sub>1</sub>, are plotted as functions of ion dose for the three irradiation temperatures, 18, 80, and 300 K. In all cases, the variances increase as the square root of dose, and

there is no apparent influence of specimen temperature between 18 and 300 K on the results. The lines in the figure were 'eyefit' to the data, with the requirement that they pass through the zero point. The slopes for the Au and Pt markers agree quite well with each other. The slopes for the Ni broadening obtained from the two dual marker combinations are also in good agreement with each other. However, they are ~ 25% lower than that for the Pt and Au markers. Because of the special dual marker geometry employed here, the different slopes cannot be attributed to differences in such irradiation or specimen conditions as marker depths, specimen purity, or inaccurate dose measurements.

The mixing results for the same marker combinations in Si are plotted in Fig. 2. Again, the variances are shown as a function of ion dose for the same three irradiation temperatures. As it found for the Al host, the variances increase as the square root of dose, and there is no apparent influence of temperature on the mixing. The mixing rates for the different markers in Si, however, show much larger differences than was found in Al. The Au marker spreads somewhat more rapidly than the Pt marker in Si and significantly more than the Ni marker.

The shift of the Ni marker relative to the Pt and Au markers could be measured in Al and Si to an accuracy of a  $\pm 20\text{\AA}$ . Within this experimental uncertainty, no shift was observed for Ar doses  $\leq 4.5 \times 10^{16}/\text{cm}^2$ . At  $1 \times 10^{17}/\text{cm}^2$ , however, a shift of the Ni markers toward the surface of  $\sim 20\text{-}50\text{\AA}$  relative to the Au and Pt markers was observed. The broadening of the markers at this high dose became large (fwhm  $\geq 40$  nm), causing increased uncertainty in the shift measurement. However, because a shift of the Ni marker toward the surface relative to the Au and Pt markers was found in all cases, it appears that the relative shift is real.

## DISCUSSION

The results of the marker broadening experiments in Al and Si are summarized in

Table 1. Here the quantity,  $Dt/\Phi F_D$ , is employed as a measure of mixing;

Table 1 Values of  $Dt/\Phi F_D$  ( $\text{\AA}^5/\text{eV}$ )<sup>a</sup>

	Au	Pt	Ni
Al	60	60	40
Si	130	75	45

<sup>a</sup> $F_D = 26$  and  $23 \text{ eV/\AA}$  for 300 keV  $\text{Ar}^+$  irradiation at a depth of 500 \AA in Al and Si, respectively.

$Dt = \Omega \frac{2}{1} / 2$ , and  $F_D$  is the damage energy deposition per unit length;  $F_D$  was calculated using the Monte Carlo computer simulation, TRIM.<sup>(4)</sup> The table clearly reveals that the mixing is dependent on the marker atom species as well as the host material. The influence of the specific marker is greater in Si than Al. In Si, the variation in  $Dt/\Phi F_D$  from Au to Ni is a factor of  $\sim 3$ , whereas for Al the variation is a factor of 1.5. Similar dependences of mixing on marker species have recently been reported for both Si and Al.<sup>(1,3,5,6)</sup>

The values of  $Dt/\Phi F_D$  obtained here for the three markers in Si using 300-keV  $\text{Ar}^+$  irradiations are in excellent agreement with other measurements using 300-keV  $\text{Xe}^+$  irradiation.<sup>(3,5,7)</sup> This is expected as mixing in Si has been reported to scale with  $\Phi F_D$  independent of the mass of the projectile.<sup>(3)</sup> The absolute values of  $Dt/\Phi F_D$  for the Au and Pt markers in Al, however, are more than a factor of two greater than values for the same markers observed during 300-keV Xe irradiation.<sup>(6)</sup> Although some variation in the results are expected due to uncertainties in  $F_D$  and the measurements, a factor of two seems large. One can only surmise that either (a) mixing is sensitive to specimen preparation, or (b) the projectile mass is an important parameter in ion beam mixing. In regard to option (b), we note that measurements of Sb markers in Al using 110-keV  $\text{Ar}^+$

irradiation at 80 K,<sup>(8)</sup> and In, Ag, and Sn markers in Al using 70-keV Ar<sup>+</sup> at 300 K,<sup>(9)</sup> systematically yield larger values of  $Dt/\phi F_D$ , than the values for various markers using 300-keV Xe irradiation at 80 K.<sup>(6)</sup> We note, however, that such a dependence on mass of the projectile is opposite to that observed for PtSi bilayers<sup>(10)</sup> and Pt markers in Ni.<sup>(11)</sup>

The independence of mixing to irradiation temperature below 300K is consistent with the results for several other marker systems.<sup>(1)</sup> In Al, both vacancies and interstitial atoms are mobile at room temperature, and some contribution to the mixing from thermally activated defect migration might be expected. Apparently the radiation-induced sink density at 300 K, is too high for significant radiation-enhanced diffusion to occur. Paine et al.,<sup>(8)</sup> observed that the mixing of Sb markers in Al begins to increase with temperature at  $T \sim 370$  K.

Finally, we note that in both Al and Si, the Ni markers moved toward the surface ( $\sim 50 \text{ \AA}$ ) relative to the Au and Pt markers at a dose of  $\sim 1 \times 10^{17}/\text{cm}^2$ . These are consistent with the magnitude and direction of the marker shifts of Pt in Si<sup>(7)</sup> and W in Cu.<sup>(12)</sup> For the single marker experiments, Pt and W were observed to shift away from the surface. The atomic masses of Pt and Au are greater than Ni; therefore the relative shift of Ni toward the surface is consistent with the single marker experiments. On the basis of matrix relocation theory,<sup>(13)</sup> it has been proposed that lighter markers will be relocated closer to the surface relative to heavier markers. The observed direction in all three cases, is opposite to the prediction of matrix relocation theory.

## SUMMARY

Ion beam mixing of Ni, Pt, and Au markers in Si and Al was measured at 18, 80, and 300K. For both Si and Al, the mixing is dependent on the specific marker atom. In Si, the variation in  $Dt/\phi F_D$  between Au and Ni markers is nearly a factor of three; the absolute values of  $Dt/\phi F_D$  are in good agreement with other results. The values of  $Dt/\phi F_D$  for Al in the present study using Ar<sup>+</sup> irradiation are similar to those found in other studies which employed Ar<sup>+</sup> irradiation, but they are a factor of  $\sim 2$  greater than values of  $Dt/\phi F$  reported for Xe<sup>+</sup> irradiation of Al. The shifts of the Ni marker relative to the Au and Pt markers are small; however, the Ni markers do shift toward the surface relative to the heavier Au or Pt marker.

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### Figure Captions

1. The dependence of the variance for various markers in Al as a function of dose for 300 keV Ar irradiation at different temperatures.
2. The dependence of the variance for various markers in Si as a function of dose for 300 keV Ar irradiation at different temperatures.



