ION-BEAM MIXING IN SILICON AND GERMANIUM AT LOW TEMPERATURES

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ABSTRACT

Ion-beam mixing of thin marker layers in amorphous silicon and germanium was studied using irradiations with Xe ions at temperatures of 34K and 77K. The marker species, ion energies and doses were: in silicon, markers of Ge and Pt irradiated with 200-keV Xe up to 2.7×10^{16} ions cm^{-2} ; and in germanium, markers of Al and Si bombarded with 295-keV Xe up to 1.63×10^{16} ions cm^{-2} . In silicon, Pt markers were found to broaden at about the same rate at 34K and 77K; and the rate of broadening was similar to that found by other workers when expressed as an efficiency of mixing, i.e., when dependence on ion dose and deposited energy was factored out. However, a Ge marker irradiated at 34K did not broaden from its original thickness. In germanium, markers of both Al and Si were mixed by irradiation at 34K, but at 77K only the Al marker broadened; the Si marker did not. The broadening of the markers is ascribed to ballistic mixing, while the cases where no broadening occurred are explicable if diffusion by a defect mechanism transported displaced marker atoms back to traps near their original sites.

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INTRODUCTION

Transport of atoms set in motion by collisions with moving atoms or ions in an irradiated solid can be called "ballistic mixing". Studies (1-3) of the broadening of thin marker layers which have been ion bombarded show that the mixing can be characterised by a diffusion coefficient (as proposed by Andersen (4)) which has the expected linear dependence on ion flux and deposited energy, i.e. is proportional to damage rate. The magnitude of the diffusion coefficient is considerably larger than that predicted by Andersen, however. Subsequent theoretical treatments by Matteson (5,6) and Sigmund and Gras-Marti (7-9) disagree in their conclusions as to the magnitude of the diffusion coefficient. Matteson's estimates agree to within a factor of two with experiment for reasonable values of the displacement energy E_d (which is a free parameter in his theory). His calculations are based on the theory of random flights, using power law cross sections to compute the recoil ranges, while the energy spectrum of moving atoms follows the usual low energy $1/E^2$ law. However Sigmund and Gras Marti's calculation (9) of an ostensibly similar quantity (the contribution of low-energy recoils: the so-called "cascade mixing") yields a rate of increase of the variance of a thin marker which is about an order-of-magnitude smaller than experimentally measured values. When the contribution of rare high energy recoils is included by Sigmund and Gras-Marti, the rate of increase of variance becomes much larger, but arises mainly from an extended tail formed on the marker distribution. This tail is not included in most experimental measurements of the variance, which are typically obtained by fitting a Gaussian to the marker profile.

The identification of the mechanism of broadening of marker layers as ballistic mixing mainly rests on the absence of any temperature dependence of the diffusion coefficient when irradiations are performed at sufficiently low temperature (2,10), and on the linear dependence on damage rate already mentioned. An additional contribution of defect-mediated diffusion has been noted at higher temperatures, generally near or above room temperature in silicon (2,3). Where it occurs, defect-mediated motion can yield diffusion coefficients larger than those due to ballistic mixing, and allows the formation of crystalline phases which would be absent if ballistic diffusion were the only operative transport mechanism. Unfortunately, observation of a temperature-independent diffusion coefficient is not sufficient to guarantee that ballistic mixing is the dominant mechanism, because under certain conditions diffusion by interstitialcy mechanisms can, in principle, also be temperature independent (11). It is therefore quite difficult to rule out a point defect contribution, except by irradiating at such low temperatures that interstitials are immobile.

In the present study ion beam mixing of thin marker layers in Si and Ge was investigated using RBS. By comparing mixing rates of different solutes at 34K and 77K it was hoped that defect-mediated diffusion could be detected. The anomalous behaviour of some of the solutes leads us to conclude that such effects were indeed present, even when irradiation was performed at 34K (case of Ge in Si). The contribution of defect-mediated diffusion was found to depend on the solute species, being dominant in some cases, but apparently absent in others. In the latter case the

rate of mixing was consistent with that found at higher temperatures by other workers, and attributed by them to ballistic mixing.

EXPERIMENTAL

Thin ($10 \text{ \AA}$) marker layers were incorporated into thick films of evaporated Si and Ge. All the targets were prepared by evaporation from electron-beam heated crucibles onto cleaned polished Si 25 mm discs held at 200°C . The deposition rate of Si and Ge was $30 \text{ \AA s}^{-1}$ and $4 \text{ \AA s}^{-1}$ respectively. The Si was deposited at the higher rate to reduce the amount of impurities, especially oxygen, incorporated into the target. The pressure during evaporation was less than 2×10^{-7} torr. Before depositing the target films, "deep markers" $10 \text{ \AA}$ thick were evaporated onto the Si substrates. The deep markers were Ge (Si targets) or Pt (Ge targets), and were included to allow marker shifts to be measured; this information is not included in the present paper, however. After evaporation of the deep markers, thick layers of Si ($1700 \text{ \AA}$) or Ge ($2500 \text{ \AA}$) were deposited. Then the marker layers were evaporated on. Either Ge or Pt layers were incorporated into the Si targets, and Si or Al markers were used in the Ge targets. Lastly, a $200 \text{ \AA}$ cap of the target material was deposited on top of the markers. The final target structure therefore consisted of a thin marker layer buried $200 \text{ \AA}$ beneath the surface of an evaporated target layer, which itself covered a thin "deep marker" on top of the Si substrate. In many of the RBS spectra obtained from these targets thin layers of oxygen were detected at the depth of the marker positions. Presumably these accumulated during the pauses in the evaporation sequence which

were associated with the setting-up of the apparatus to deposit the marker layers.

Targets were irradiated at a nominal 77K (at IBM Yorktown Heights) or at 34.5K (at Solid State Division of Oak Ridge National Laboratory). Si targets were bombarded with 200 keV Xe ions, and the Ge targets with 295 keV Xe. Doses lay in the range 5.2×10^{15} to 2.7×10^{16} ions cm^{-2} of 200 keV Xe, and from 3.3×10^{15} to 1.63×10^{16} ions cm^{-2} of 295 keV Xe.

In the 77K irradiations done at IBM the Si wafers carrying the target layers were mounted on a copper block cooled by liquid nitrogen, and were thermally bonded to it by heat transfer compound. The temperature of the block was monitored during the irradiation and did not rise by more than 2K. The total ion beam current did not exceed $0.5 \mu\text{A}$. For the irradiation done at ORNL the specimens were mounted on a cooled copper block by tabs and 'Cry-con' grease. The block temperature was $34.5 \pm 0.5\text{K}$ during irradiation. The ion beam currents were between 1 and $2 \mu\text{A cm}^{-2}$, giving about 1 watt of total beam heating. In all the irradiations the ion dose of was measured by charge integration, and subsequently checked by RBS analysis in selected cases. The two methods agreed to within 2%.

The broadening of the marker layers was measured by RBS using 2.3 MeV He ions at a scattering angle of 170° . The angle between the beam and the target normal was 70° , except in the case of the Ge markers in Si, where it was decreased to 45° in order to increase the separation in energy between the Ge peak and that of the implanted Xe. The resolution of the detector system was

≈ 20 keV. Typical data are shown in figures 1-3.

ANALYSIS OF DATA

The variance in depth of the marker layers was obtained from the data in order to extract the effective diffusion coefficient during the irradiation. First, to determine the variance in energy of marker peaks such as those shown in figures 1-3, background was subtracted by least-squares fitting a suitable polynomial (usually a quadratic). Next, after subtraction of the background, non-negative channel contents were used to compute the first three central moments of the data in the region of the peak. The moments were used to construct a skewed Gaussian fit to the data (using the Edgeworth expansion described by Gibbons et al. (12)) and the quality of the fit was assessed subjectively. In all cases at least two different fits were compared, and the variances of the acceptable fits averaged.

We found it necessary to use skewed Gaussians to achieve satisfactory fits. The profiles of many of the irradiated marker layers were significantly skewed, as can be clearly seen in fig. 3, and the skewness appeared to increase with increasing bombardment dose. The skewness of the unirradiated markers was negligibly small, their profiles being essentially Gaussian. It proved difficult to extract consistent values for the skewness. Fits which appeared to be equally good, within the statistical scatter of the data, yielded widely differing values of skewness. Therefore, only values of variance, σ^2 , are reported here.

To complete the analysis of the experimental data the variances

in energy of the marker profiles were converted to variances in length using the near-surface approximation (13) and the He stopping powers of Ziegler and Chu (14).

RESULTS

Measured energy variances of the marker peaks are plotted as a function of Xe dose in figs 4a (markers in Si) and 4b (markers in Ge). The following features of the data should be noted:

Si target.

Pt marker layers broadened under irradiation at both 34K and 77K, and their variance is proportional to the dose. The initial distribution of the marker layer of Ge was much wider than that of the layer of Pt, probably because of thermal diffusion of the Ge atoms during the evaporations at 200°C, while the Pt was prevented from diffusing by the formation of Pt silicide. Moreover, the Ge layer did not broaden appreciably under irradiation at 34K or, as indicated by preliminary data, at 77K. This behaviour is thought to be a consequence of thermally activated diffusion of Ge atoms during the irradiation, as discussed below.

Ge target.

At 34K both Al and Si marker layers broadened under irradiation, and showed the expected linear dependence of variance on ion dose. However, at 77K the Si marker did

not broaden, although the Al markers did. The variance of the Si marker remained at its original value, while the rate of increase of the Al marker appeared to be the same at 34K and at 77K. It should be noted that while the 34K data was taken from only one set of irradiated samples, the 77K data was verified by repeated runs on samples prepared at different times.

As already noted, the atomic transport due to collisions with low-energy recoils can be described by a diffusion coefficient, given by (1)

$$D = (0.42/6) (F_D/N) \phi \langle r^2 \rangle / E_d,$$

where F_D is the energy deposited per unit length at the depth of interest, N is the atomic density of the target, ϕ the ion flux, $\langle r^2 \rangle$ the effective mean-square displacement of marker atoms per displacement event, and E_d the displacement energy. The diffusion coefficient is effectively determined by the two latter parameters for a given bombarding ion flux. We therefore define a figure of merit, Q , for the efficiency of mixing:

$$Q = \langle r^2 \rangle / E_d.$$

The choice of Q as a parameter to describe the data allows the comparison of results obtained with different bombarding ion species or energies.

To determine Q from the data it is necessary to know the deposited energy F_D . We have calculated F_D with moments obtained

by interpolation from Winterbon's tables (15), using an Edgeworth expansion (12) to construct the damage-depth profile. The values obtained are shown in Table I. Also shown in the table are derived values of the mixing efficiency, Q , for the data obtained in this work, for some early results on Ge markers in Si and Si markers in Ge (16), and for the Sb marker data of Matteson et al. (2). We have selected the latter as being representative of their work (the data on Ge markers in Si in ref. 2 cannot be used for this purpose, since it is averaged over several depths in the target at which the values of F_D are different).

DISCUSSION

Examination of Table I shows that the efficiencies of mixing, Q , deduced from the broadening of Pt markers in Si in the present work agrees in magnitude with efficiencies deduced from the data of Matteson et al. (2) for Sb markers in Si. Values of Q lie in the range 50 to 61 $\text{\AA}^2/\text{eV}$. Given the experimental uncertainties, this constitutes good agreement. For the Ge targets, the efficiencies of mixing of Al and Si at 34K, and of Al at 77K, are of similar magnitude to those measured in the Si targets. Given the limited data available, it is not possible to say at present whether the value of Q determined for 34K mixing in Ge is significantly different from the values measured in Si.

There appears to be no difference in the rates of mixing in Si and Ge, although denser sub-cascades are produced in the latter by the Xe ion bombardment. One view of dense cascades in Ge is that they are essentially molten for a short period (17,18). The rapid liquid phase diffusion which occurs in semiconductors might

then give rise to extra atomic motion. An essentially equivalent view of the cooling stage of a collision cascade, based on recent modelling with molecular-dynamics codes (19), is that vacancy motion takes place in the few picoseconds during which energy diffuses out of the cascade volume. This movement is thought to be responsible for the collapse of cascades in metals to form vacancy loops (20), and for the recombination of many of the vacancies and interstitials produced in the cascade. The vacancy movement might also give rise to atomic movement which could be measured as extra atomic mixing.

However, the degree of atomic mixing observed in the Ge target was not significantly greater than that in the Si target. This may be due to the fact that the sub-cascades caused by Ge primary recoils were very small. Calculation shows that half the displacements produced by the 295 keV Xe beam occur in sub-cascades where the energy of the Ge primary recoil was less than 3 keV. According to Sigmund's estimates (21) the energy density in a 3 keV cascade in Ge is 7.8 eV atom^{-1} , but the time for which heat is trapped in the cascade is only comparable to the slowing-down time of the projectile, some 10^{-13} s . Even with a liquid-phase diffusion coefficient of $10^{-4} \text{ cm}^2 \text{ s}^{-1}$, this implies a diffusion length within the cascade volume of only $0.3 \text{ \AA}$, which is much less than that due to ballistic mixing.

Given that the mixing efficiency in Si and Ge is similar, it seems reasonable to assume that a common mixing mechanism is operative in both cases, and that this is ballistic mixing, although the discrepancies between experiment and some calculations (9) of the rate of mixing need to be resolved. The

discrepancy may arise because of the existence of mechanisms of atomic transport in addition to pure ballistic mixing. One such possibility, suggested by Corbett (22) is that continual reconstruction of damaged regions takes place as new defects are introduced into regions which are already heavily damaged.

The noticeable skewness of the irradiated Pt and Al marker peaks (see fig. 1 (top) and fig. 3) indicates that some other process is occurring in addition to random diffusion-like transport thought to be characteristic of low-energy recoils. Two effects might be responsible. The first is any contribution of medium energy recoils to the ballistic mixing, because these retain some preferred momentum in the direction of the incident ion. Secondly, the non-uniformity of the deposited energy F_D may contribute to a skewing of the profile. All the markers were at a depth at which F_D is increasing with increasing distance from the surface, so that the effective diffusion coefficient due to ballistic mixing was larger on the deeper side of the marker layer than on the shallower side.

What of the peaks that did not broaden under irradiation? We believe that this singular behaviour is due to defect mediated diffusion under irradiation. Briefly, the model proposed is as follows:

1. Marker atoms are displaced from their original sites by collisions with moving atoms.
2. The marker atoms can diffuse because of the presence of mobile point defects in the irradiated host material.

3. Migration ceases when the marker atoms become trapped. We postulate that in our targets the highest trap density occurs at the interface between the original marker layer and the Si (or Ge) host, possibly associated with oxygen atoms in the interface.

We will consider the above stages in more detail.

The first step in the process leading to a non-broadened marker is the displacement of the marker atoms from their original sites. Every atom in a heavily bombarded target is displaced many times. This number is not, of course the same for every component of the target, because the charge and mass of each component determines its share of the energy deposited in the target by the incident ion beam, but displaced the target atoms must be.

The fact that the Si marker in Ge broadens at 34K indicates that ballistic mixing operates in the Si, and therefore the lack of broadening at 77K must be due to the additional operation of another process, for the rate of ballistic mixing is, by the nature of the process, expected to be essentially temperature-independent. We do not have similar data for Ge markers in Si, for even at 34K the Ge marker did not broaden appreciably under bombardment. However, we assume that broadening would take place at a sufficiently low irradiation temperature.

The second stage of the postulated process to explain the non-broadening of certain marker layers is the diffusion of marker atoms by interaction with mobile point defects. Both Si and Ge

targets are deposited as amorphous material and it is known that ion bombardment does not affect the degree of amorphisation (23). It is also known from EPR studies (24) that the character of defects in amorphous silicon produced by irradiation of single crystals and that prepared by deposition are similar. Little is known about the mobility of point defects in amorphised semiconductors. However since one expects point defect mobility to be primarily determined by short range order, which is largely preserved in amorphous Si and Ge, we may expect that the evidence for defect motion in crystalline material at low temperatures will serve as a guide for the present case.

Discussion of solute motion in irradiated semiconductors is complicated by two effects. First, there is the dependence of a defect's mobility on its charge state, and hence on impurity content through the Fermi level. Secondly, there is the well-attested existence of radiation-induced diffusion of defects at low temperature. Irradiation affects the migration of point defects in semiconductors via a number of mechanisms which can have the effect of rendering mobile an otherwise immobile defect. Bourgoin and Corbett (25) summarise the consequences as follows: under irradiation, interstitial motion is observed in silicon even at 0.5K, while in n-type germanium interstitial motion begins at 65K, which can be reduced to 27K by photon illumination. This difference between Si and Ge is consistent with our results, which indicate mobility under irradiation in Si at 34K, but not in Ge.

Although the migration of irradiation produced point-defects during irradiation can induce mobility as large or greater than

that due to ballistic mixing (11), not all solute atoms are expected to be equally mobile in a given concentration of defects. Our Ge results (fig. 4b) show that an Al marker broadened at 77K while a Si marker did not. This can be due to the Si atoms being mobile via a defect-mediated mechanism while the Al atoms are not. Similar considerations apply to the observation that Pt markers in Si broadened while Ge markers did not. The anomalous behaviour of Si in Ge, and Ge in Si, is not unexpected, given their chemical similarity.

Mobile solute atoms will diffuse until they reach a trap, if traps are present. In the present experiment the highest density of traps was evidently at the original marker position where contaminants were noted in the RBS spectrum. Thus, although during irradiation solute will be dynamically distributed between traps in the bulk of the target and traps on the interface, the latter appear to dominate the post-irradiation solute distribution. From the narrowness of the Si distribution in Ge after irradiation, it is apparent that the trap distribution is broadening at a rate much less than the Al distribution. This may be because the trapping atoms are themselves mobile and can be re-trapped on their own trap sites.

It is impossible to be sure why other workers (2,16) have found marker peaks of Ge in Si and Si in Ge broadening under irradiation, whereas in the present experiments they were found not to broaden. However, one suggestion as to the reason can be put forward. In the model used to explain the present data, the major solute trap site is on the original interface, and therefore diffusing solute atoms are only re-deposited on that

plane. If, however, the trap density were high and uniform, then solute atoms would diffuse a distance determined by the trap density. If the trap density were very high, then diffusion lengths would be determined essentially by ballistic mixing. Therefore, one explanation of the discrepancy between the present data and that of earlier workers is that their material contained a much higher concentration of trap sites than ours did. If this is correct, then for certain species the measured diffusion coefficient will depend on the composition and, perhaps, the structure of the targets. It follows that a certain amount of irreproducibility of data is to be expected.

CONCLUSIONS

1. The broadening of marker layers in Xe irradiated Si and Ge can be affected by point-defect-mediated diffusion even at low irradiation temperatures. In Ge at 34K marker layers of Al and Si increased in variance, but at 77K the variance of Si layers was not appreciably increased by irradiation, although Al layers broadened at the same rate as at 34K. In Si targets, the rate of increase of the variance of Pt marker layers bombarded at 34K and 77K was found to be consistent with earlier measurements, but a Ge marker irradiated at 34K preserved its original thickness. These findings can be explained if, in the cases where markers did not broaden, solute diffusion by a defect mechanism could carry displaced marker atoms back to trap sites near their original positions.

2. Where marker peaks did broaden, the rate of increase of their

variance, expressed as a mixing efficiency, was consistent with other measurements and was similar for the Si and Ge targets. This implies that the mechanism was ballistic mixing in both cases.

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TABLE 1

Values of the mixing efficiency, Q .

Target	Marker	Temp. K	Ion	Energy keV	Depth Å	F_{D_0} eV/Å	$\frac{Q}{\text{Å}^2/\text{eV}}$	Ref.
Si	Pt	34, 77	Xe	200	200	109	53	this
Si	Ge	34	Xe	200	200	109	0	this
Si	Ge	77	Xe	300	200	84	82	16
Si	Sb	77	Ar	110	200	23	53	2
Si	Sb	77	Kr	220	200	48	61	2
Si	Sb	77	Xe	300	200	84	50	2
Ge	Al, Si	34	Xe	295	200	205	55	this
Ge	Si	77	Xe	295	200	205	≈ 5	this
Ge	Si	77	Xe	300	400	265	72	16

FIGURE CAPTIONS

1. RBS spectra of Ge and Pt markers in Si irradiated with 200 keV Xe ions at 34K. (a) Ge marker as deposited (dotted line) and after implantation of 5×10^{15} ions cm^{-2} (solid line), showing the lack of broadening of this marker species. The target was tilted at 45° to the beam during analysis. (b) Pt marker for doses of 0, 0.52 and 2.2×10^{16} ions cm^{-2} , showing the progressive broadening of the marker layer. The analysis angle was 70° . The "deep marker" layers can also be seen.
2. RBS spectra of a Si marker layer in Ge at zero dose and after irradiation at 34K with 3.3×10^{15} cm^{-2} of 295 keV Xe ions.
3. RBS spectra of an Al layer in Ge for zero dose and after irradiation at 77K with 8×10^{15} cm^{-2} of 295 keV Xe ions. Note the pronounced skewness of the irradiated distribution.
4. Plots of the square of the equivalent full width at half maximum ($\text{FWHM} = 2.35\sigma$) of marker peaks as a function of bombarding ion fluence. (a) Ge and Pt markers in Si. (b) Al and Si markers in Ge. The lines shown are least-squares fits to the points representing the marker layers which broadened under irradiation. The slopes of the lines were used to construct Table I.

4a

Broadening of markers in Ge