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Lattice disorder in alumina single crystals produced by ion bombardment

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The effect of 40 keV Kr ion bombardment of Al_2O_3 single crystals has been investigated using a Rutherford backscattering-channelling (RBC) technique. Curves of lattice disorder were found to be sigmoidal; the disorder increasing slowly up to a fluence of 1×10^{15} ions cm^{-2} and then accelerating to a saturation level at $\sim 1 \times 10^{16}$ ions cm^{-2}. These doses are about 100 times higher than comparable values found for elemental and III-V semiconductor compounds. The number of displaced atoms per incident ion estimated for an ion dose of 3×10^{14} ions cm^{-2} was found to be less than that calculated using Sigmund's equation. This difference is discussed in terms of defect recombination and re-ordering during bombardment. Measurements taken from the RBC random spectra before and after each bombardment have indicated that the stoichiometry of the alumina crystals did not alter even at the highest bombardment fluences used.

1. Introduction

There has been considerable interest in recent years in the technique of ion-bombardment doping of semiconductors in order to produce conductivity changes and consequently to fabricate electronic devices. This interest has led to a considerable number of investigations into the effect of ion bombardment on the properties of semiconductor materials including mainly, Si, Ge, GaAs and GaP and to a lesser extent a number of other III-V and II-VI semiconductor compounds[1]. However, it has recently been recognized that the technique of ion bombardment of solids has the potential to alter many of the surface and para-surface properties of a wide range of materials [2, 3]. In order to assess this potential, it is necessary to investigate the effect of ion bombardment on various materials.

In the present work, the damage produced in ion-bombarded α-Al_2O_3 has been investigated by the Rutherford backscattering-channelling technique (subsequently referred to as the RBC technique). Previous studies using X-ray diffraction [4-6], reflection electron diffraction [7], transmission electron microscopy [8, 9] and gas release measurements [10, 11] among other experimental techniques, have provided strong evidence of disordering (amorphization) of crystalline α-Al_2O_3 upon irradiation with heavy ions [7-11], fission fragments [4] and probably fast neutrons [5, 6]. The behaviour of α-Al_2O_3 upon irradiation is, therefore, apparently similar to that reported for Si, Ge [cf. 1(a)], GaAs [12, 13] and GaP [14]. Comparison of the alumina results with those for such semiconductor materials is, therefore, of interest in testing the theoretical and experimental concepts developed from the abundance of data on these materials.

2. Experimental

The specimens used in this study were in the form of optically-ground commercially pure Al_2O_3 single crystal discs, 10 mm in diameter by 1 mm thick and having *c*-axis orientation perpendicular to their surfaces. The crystals were cleaned and stabilized before each use by heating them in air at 1200°C for 4 h.

The single crystals were bombarded at room temperature using the University of Salford Isotope Separator. ^{84}Kr was injected into the specimens at an energy of 40 keV and an average beam current of 5 $\mu A\ cm^{-2}$ (i.e. a dose rate of 3×10^{13} ions $cm^{-2}\ sec^{-1}$). The ion beam from the

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isotope separator was focused to a line approximately 4 cm high by 0.2 cm wide and swept across the samples with a large amplitude sweep at 50 Hz. The instantaneous dose rate (i.e. beam current divided by the area of focused beam) is, therefore, higher than the average rate by a factor of approximately 40. The dose was varied in the range 1×10^{14} to 1×10^{16} ions cm^{-2}. Charge build-up was minimized by covering the crystals with Al foils except for a 5 mm diameter opening left in the front face.

The RBC measurements of the alumina single crystals before and after each bombardment were effected with a 2 MeV He-ion beam (with a typical current of 5 nA) from the University of Salford Van de Graaff accelerator, by recording backscattering spectra at an angle of 150° with a solid state detector and multichannel analyser system, when the beam was incident in a random direction or along an aligned ⟨0001⟩ direction. The experimental technique is well known and has been described in detail by previous authors [15].

During all the RBC measurements, the crystals were left in their aluminium foil covers, used during bombardment. This was found necessary to avoid the difficulty in measuring the He-beam current owing to the high electrical resistivity of the alumina crystals.

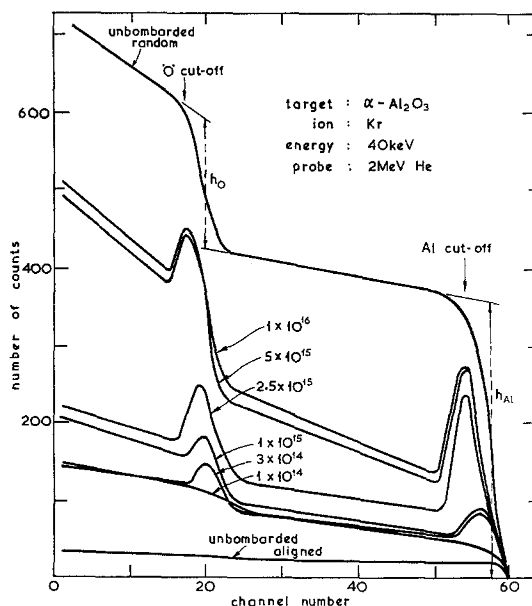

Figure 1 Random and aligned backscattering spectra of alumina single crystals bombarded at R.T. with various doses of 40 keV Kr ions. The Kr-peak (channels 100-108) omitted.

3. Results and discussion

3.1. Characteristic features of the RBC energy spectra

Fig. 1 shows typical random and aligned RBC spectra of alumina single crystals bombarded with various doses of 40 keV Kr-ions. The masses of Al and O atoms are sufficiently different that their respective contributions to the backscattered spectra result in damage peaks and cut-offs which are well separated.

It is noticed that the aligned spectrum for the unbombarded specimen has no surface peaks, which are often observed for single crystal semiconductor targets as a result of wide-angle scattering from surface atoms and the surface disorder of the crystal. The absence of the channelling peak has also been observed in the case of quartz (SiO_2) single crystals [16]. This quartz result was explained as being due to the high impurity level in the crystals used compared with most semiconductor materials for which channelling data has been obtained. This is also likely to be the case for the alumina crystals used in this study. The high impurity level might lead to a greater level of de-channelling in the aligned direction that is typical for semiconductor materials, but it would not be sufficiently high to give rise to extra features in the backscattered spectrum. This fact, together with the absence of a surface layer such as the oxide layer formed on semiconductor materials and the rather poor energy resolution of the system used, seems to be responsible for obscuring any peak which might otherwise have been evident.

As seen in Fig. 1, after bombardment to a dose of 1×10^{14} ions cm^{-2} the spectrum is considerably higher than in the unbombarded, aligned case indicating a marked increase in the de-channelling level, but again there are no peaks. It is possible, however, that at such doses small damage peaks are obscured for the same reasons outlined for the unbombarded case. In addition, although the absence of the damage peaks at 1×10^{14} ions cm^{-2} might indicate no major disorder in the crystal, the bombardment has probably produced an appreciable distortion of the crystal lattice. This distortion is the result of bombardment-enhanced lattice strain in partially ionic materials [17]. Thus for each vacancy created by bombardment in an ionic crystal there will be repulsion between its charged nearest neighbours. This repulsion-

induced lattice strain leads to a shifting of the positions of atoms adjacent to a vacancy. This accordingly, is expected to increase the low-angle scattering of the probe ions and a consequent enhancement of the de-channelling levels in the energy spectra of bombarded ionic crystals compared with the corresponding level in highly covalent materials such as Si, Ge, GaAs and GaP.

After bombardment to a dose greater than 1×10^{14} ions cm^{-2}, however, the damage peaks observed (Fig. 1) suggest that increased large-angle scattering of He-particles occurs from major disorder zones containing sufficiently large numbers of displaced atoms. As these highly damaged (amorphous) regions are created, apparently at a threshold dose of 3×10^{14} ions cm^{-2}, the characteristic damage peaks appear in the energy spectra. With increasing dose, the gradual build-up of Al and O displacements from their lattice sites increases the number of backscattered particles and, consequently, the area under each peak, until a saturation level is reached.

3.2. The amount of disorder as a function of bombardment dose

Following work on semiconductor materials, the amount of disorder introduced in the alumina crystals by bombardment has been calculated from the RBC energy spectra using two methods. In the first method the *area* under each damage peak was used as a measure of the total lattice disorder after subtracting from it (i) the area under a straight line from the front base to the rear base of the damage peak, and (ii) the area of the surface peak of the aligned unbombarded spectrum, corrected as in (i). Correction (i) is a measure of the fraction of the He-beam that becomes de-channelled in passing through the damage layer assuming a linear approximation for this de-channelled component [18]. Correction (ii) is a measure of the residual surface damage of the crystal before bombardment. However, as already pointed out, the spectrum of the aligned unbombarded specimen, as well as that for a bombardment dose of 1×10^{14} ions cm^{-2} had no surface or damage peak. In these cases, the "peaks" were assumed to extend over the same width as the peak for a dose of 3×10^{14} ions cm^{-2} in order that the same procedure could be adopted.

A second method of evaluation of disorder was used in which the *heights* of the spectra behind the peaks were measured. Since there is no lattice disorder at the rear edges of the peaks the yield is due to the scattering of ions which have been de-channelled in passage through the disordered region, the number of these ions should be proportional to the displaced atom density. The yield is, therefore, a measure of the disorder, again, after making the necessary corrections for the surface disorder in the aligned unbombarded crystals [19].

Fig. 2 shows the curves obtained for amount of disorder as a function of dose as calculated using the two methods described above. The curves have been normalized by taking the level of the disorder at a dose of 1×10^{16} ions cm^{-2} as unity so that comparison of the two methods is made easier. As shown by this figure, there is a good agreement between the two methods used for expressing the extent of disorder induced by ion bombardment.

However, it is important to point out that although the area under the Al-damage peak is a measure of Al-disorder, the height behind this peak is due to de-channelling from both Al- and O-disorder. Accordingly, any correlation between the two methods described above will depend critically on the contribution of the O damage to the de-channelling process. Thus, as shown later, the measured number of Al displacements is only one third of the number of O displacements, so that the contribution of O damage to the de-channelling may be large. This contribution will in turn depend on the character and behaviour of the damage in the O sub-lattice. If the nature of the damage in the O sub-lattice is changed, for example as a result of annealing effects during bombardment, this correlation may not be retained. Thus, in the case of binary semiconductors Bøttiger and Whitton [20] have recently suggested non-stoichiometric annealing of damage in GaP implanted at low temperature (25 K), where the damage observed in the Ga sub-lattice anneals twice as much as that in the P sub-lattice. This may partially explain the poor correlation between the yield values measured behind the damage peaks and the peak areas in hot implanted GaP, e.g. [21, 22].

It can be seen from Fig. 2 that the data points fall on sigmoidal curves which suggest the presence of a threshold dose at around 1×10^{15} ions cm^{-2}. As the dose is increased beyond this threshold, the disorder process is accelerated until a saturation level is reached at 1×10^{16} ions cm^{-2}.

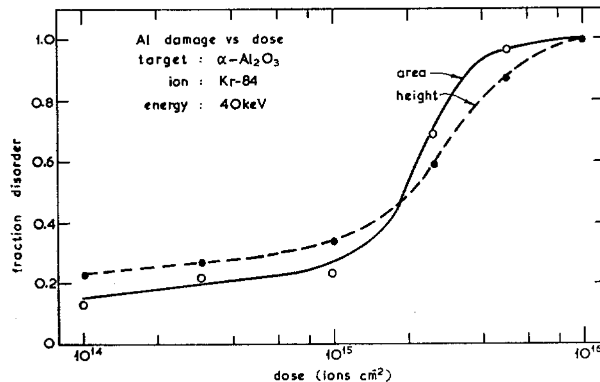

Figure 2 The dose dependence of the lattice disorder in α-Al_2O_3 for 40 keV-Kr ions at R.T., calculated using the area under the Al damage peaks and the heights behind these peaks. Curves normalized at 1×10^{16} and plotted as fraction disorder versus dose to simplify comparison.

These results are in a good agreement with electron microscopic studies on bombarded α-Al_2O_3 [7, 9] where a complete amorphous layer was observed at a dose of 1×10^{16} Kr or Xe ions cm^{-2}, injected at 40 keV. These doses, however, are a factor of 100 higher than comparable ion doses found for Si, Ge [23], GaAs and GaP [15]. One could speculate that this difference is a result of a significant damage annealing which takes places in α-Al_2O_3 at low-dose bombardments, where the defects produced are isolated and few. At least three possible sources of damage annealing could be present at this stage. The first is assumed to be *radiation annealing* of damage during its subsequent analysis in the 2 MeV-He beam. However, it has been reported previously that probe beam re-ordering has a significant effect in Si [24, 25] a slight effect in GaAs [26], but no effect in GaP [27]. It is, therefore, difficult to extrapolate a conclusion for the case of Al_2O_3 in view of the present available data*. The second possible source of annealing at low-dose bombardment could be attributed to *athermal re-ordering*. This could be markedly effective in the case of Al_2O_3 because of its crystal structure. Thus, as seen in Fig. 3, in the corundum structure of α-Al_2O_3 the arrangement of O atoms is approximately close-packed hexagonal and the Al atoms fit in the octahedral holes between the O atoms, displaced towards one apex [28]. Because of the chemical composition, only $\frac{2}{3}$ of the available holes are filled by Al atoms. It, therefore, seems likely that some Al atoms displaced by bombardment will move into the holes normally vacant in the corundum structure. This could effectively reduce the density of disorder induced by low and intermediate doses but not at high-dose bombardments where the lattice is completely disordered. Finally, a reduction in disorder at low-dose bombardment could be attributed to *thermal annealing* of damage during bombardment as a result of high mobility of defects during radiation.

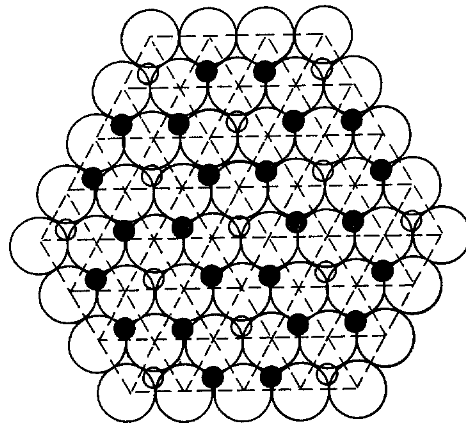

Figure 3 Arrangement of Al-ions and holes between two layers of O-ions in α-Al_2O_3. Large open circles represent underlying O-ions, small open circles represent holes and small filled circles represent Al-ions, [28].

In conclusion, one may assume that the combined effect of these annealing processes results in a slow build-up of disorder until the concentration of displaced atoms is such that the annealing process can no longer compete and

*Work is in progress to investigate the effect of beam annealing on a range of materials including Al_2O_3.

then disorder increases rapidly up to the saturation level.

The work of Jech and Kelly [11] may also be relevant to the present results. Using the technique of gas-release, these authors have investigated the progress of disordering induced in corundum single crystals injected with 10 keV ^{85}Kr as a function of ion-bombardment dose. These obtained a damage versus dose curve which has the same sigmoidal form as that obtained from the present RBC measurements (Fig. 1). The results, however, are *not* completely in agreement. The gas release measurements indicate a threshold dose and a saturation dose which were a factor of ten less than those reported in the present work. However, the differences in the bombardment conditions, particularly energy and dose rate, makes comparison of the results difficult. In addition, the damage versus dose curve of Jech and Kelly is based on a theoretical model [29] by which the fractional gas release at different doses has been converted to a fractional disorder as a function of dose. This theoretical model is based on a number of simplifying assumptions which could be partially responsible for the discrepancy between the results.

3.3. Evaluation of the number of displaced atoms

Following Marsden *et al* [30], the number of displacements per incident Kr ion can be calculated by comparing Al and O backscattered yields with the backscattered yield from the implanted Kr. The number of displacements per ion is calculated using the equation [27]

$$\frac{Y_{Al}}{Y_{Kr}} = \frac{N_{Al}}{N_{Kr}} \left(\frac{M_{Kr}}{M_{Al}}\right)^2 \left(\frac{M_{Al} + M_{He}}{M_{Kr} + M_{He}}\right)^2 \left(\frac{Z_{Al}}{Z_{Kr}}\right)^2 \frac{[\sin^4(\theta/2)(1 + \cos\theta)(1 + \gamma^2 + 2\gamma\cos\theta)]_{Al}^{3/2}}{[\sin^4(\theta/2)(1 + \cos\theta)(1 + \gamma^2 + 2\gamma\cos\theta)]_{Kr}^{3/2}} \quad (1)$$

where N = number of atoms in the damage region, M = atomic mass, Z = atomic number, θ = C.M. scattering angle, and $\gamma = M_{He}/M_{target}$. The same equation was used to determine the number of O displacements per incident Kr ion.

The calculation was carried out on the yield obtained from a crystal bombarded to a dose of 3×10^{14} ions cm^{-2}. This dose was chosen for two reasons: (a) at high doses, particularly in the saturation region, a considerable number of the implanted Kr ions may have been removed by sputtering, with the result that the area under the Kr peak is no longer proportional to the total implanted dose and it becomes impossible to compute the displacement atom density from comparison with a *known* implanted Kr concentration; (b) at high doses it is very likely that the damage regions are due to more than one bombarded ion and any measurement of the number of displacements per incident ion will be consequently reduced.

Using this technique it was estimated that the numbers of displaced Al and O atoms per incident Kr ion were 40 and 120 respectively. This gives a total number of 160 displaced atoms per ion.

Sigmund [31] has defined an *upper limit* for the number of atoms displaced per incident ion as, $N(E) = 0.42\ E/E_d$, where E is the incident ion energy in eV and E_d is the energy required to displace an atom from its lattice site. Taking an average value for E_d in Al_2O_3 as 50eV [32] Sigmund's equation gives a defect production rate for 40 keV ions of 336 displacements per ion, a value higher than that obtained experimentally.

Previous results reported for semiconductor materials, however, have shown that $N(E)$ obtained from RBC measurements are greater than those predicted by Sigmund's equation for the case of Si [30,33] and GaP [15] but *less* in the case of GaAs [15]. There is no definite explanation at present for this discrepancy though we would point out that a much greater disagreement than that obtained in our measurements has been previously reported for neutron irradiation of α-Al_2O_3 [34, 35]. It is known, however, that Sigmund's equation gives an *upper limit* for the number of displacements. It does not take into account the effect of defect recombination and re-ordering during bombardment, which may play an important role in the case of α-Al_2O_3 as already discussed in the preceding section particularly at low and intermediate doses, i.e. the dose region in which the calculation has been made. Another aspect of the Sigmund equation is the assumption of a constant displacement energy E_d which is independent of recoil direction. In crystalline targets the value of E_d is known to be highly sensitive to recoil direction and the average value may be somewhat higher than the 50 eV used in the equation.

Finally, the measured ratio of Al to O

displacements is 1:3.0 compared to the expected ratio of 1:1.5 expected if the incident ion energy is equally partitioned between the two species in a stoichiometric lattice. It should be noted that the O displaced atom density can only be determined to an accuracy of about 20% because of the large de-channelling correction. At higher incident ion fluence, the ratio of O:Al displacement changes but never falls below a value of about 2 up to the higher fluences used. We would first point out that these results cannot be attributed to change in the stoichiometric composition of the alumina crystals upon bombardment as will be discussed in the next section. However, it could be related either to the annealing processes mentioned before, or to the possibility that the energy has been shared unequally between unequal mass components or to a considerable error in calculating the number of displaced atoms from the RBC energy spectra as the damage peaks are superimposed on a large de-channelling continuum which would be mostly effective at low and intermediate doses. Experimental accuracy could be improved by double alignment techniques [24] but these were not available.

3.4. The change in stoichiometric composition upon bombardment

Previous studies have shown that some multi-components systems, such as Cu_3Au [36], GaSb [37], V_2O_5 and MoO_3 [38] can undergo a change in their chemical composition upon high-dose bombardments. The process is called preferential sputtering and it has been recently discussed, particularly in connection with oxide systems [39]. Consequently, we used the RBC technique to detect possible changes in the stoichiometry of the alumina single crystal as a function of bombardment dose. The back-scattered yields from Al and O atoms in Al_2O_3 will be proportional to their relative concentrations and will also depend on their scattering cross-sections. The ratio of Al to O yield (Y_{Al}/Y_O) can be expressed using Equation 1 with the appropriate parameters for Al and O. The calculation gives $Y_{Al}/Y_O = 2.3$ for stoichiometric Al_2O_3. Consequently, the Al and O step heights were measured on the *random spectra* for the alumina single crystal before and after each bombardment. The heights h_{Al} and h_O are measured through the steepest point of the cut-off edges (Fig. 1). The edges are rounded because of the detector resolution and the extrapolation method of producing a sharp edge enables the approximate step height to be measured [27]. The ratio of h_{Al}/h_O was found to be $2.2. \pm 0.3$, i.e. $\pm 13\%$ for all random spectra obtained. The experimental errors depend primarily on the statistics in the number of counts per channel and the resolution of the systems. This result is in a good agreement with the value obtained from the yield equation and indicates that at least for bombardment doses up to 1×10^{16} Kr ions cm^{-2}, the stoichiometry of the alumina single crystals does not change with bombardment*. Similar results have been reported previously for SiC [41] and GaP [22, 27].

4. Summary and conclusions

The main conclusions that can be drawn from these results are:

1. Rutherford backscattering-channelling techniques can be applied to investigate the effects of heavy ion bombardment of single crystals α-Al_2O_3.

2. At low doses no surface damage peaks are detected in the aligned RBC spectra but simply an increase in the level of the de-channelling continuum. This behaviour is attributed to the formation of a dislocated lattice with lattice strain in partially ionic crystals resulting in an increase in small angle scattering of the He probe beam.

3. The curves of disorder versus ion fluence are sigmoidal with a slow increase of damage up to a dose of 1×10^{15} ions cm^{-2} followed by an accelerated change up to a saturation level of 1×10^{16} ions cm^{-2}, 40 keV Kr. The total number of displaced lattice atoms (i.e. both Al and O) was calculated to be 160. This is less than the value expected from a simple application of Sigmund's equation using an average displacement energy for Al_2O_3 of 50 eV. In addition the measured ratio of Al to O displacements is found to be 1:3.0 compared to the expected ratio of 1:1.5 expected if the incident ion energy is equally partitioned between the two species in a stoichiometric lattice. Both these effects can

*It is worth pointing out that in agreement with this result no change in the electrical conductivity has been observed in α-Al_2O_3 single crystals bombarded with 40 keV Kr or Au ions at doses up to 1×10^{17} ions cm^{-2} [40] although similar bombardments have shown an increase in the order of 10^{11} or 10^{5+1} in the electrical conductivity of MoO_3 and V_2O_5 respectively as a result of preferential oxygen loss [38].

be explained by defect annealing and reordering during irradiation.

4. The disorder in the Al_2O_3 lattice was measured using both the area under the Al surface damage peak (with suitable de-channelling contributions allowed for) and also the height of the de-channelling step behind this peak. There is good agreement between these two methods although the contribution to the de-channelling behind the Al peak depends on both Al and O displacements and is possibly mainly determined by the larger amount of oxygen disorder. Any correlation between the two methods depends critically on the detailed nature and behaviour of the damage in the oxygen sub-lattice;

5. The stoichiometry of the Al_2O_3 lattice is preserved up to the highest doses used of 1×10^{16} 40 keV Kr ions cm^{-2}.

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