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DISLOCATION LOOPS IN SPINEL CRYSTALS IRRADIATED SUCCESSIVELY WITH DEEP AND SHALLOW ION IMPLANTS

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ABSTRACT

This study examines the influence of microstructural defects on irradiation damage accumulation in the oxide spinel. Single crystals of the compound MgAl_2O_4 with surface normal [111] were irradiated under cryogenic temperature (100°K) either with 50 keV Ne ions (fluence $5.0 \times 10^{12}/\text{cm}^2$), 400 keV Ne ions (fluence $6.7 \times 10^{13}/\text{cm}^2$) or successively with 400 keV Ne ions followed by 50 keV Ne ions. The projected range of 50 keV Ne ions in spinel is ~50 nm ("shallow") while the projected range of 400 keV Ne ions is ~500 nm ("deep"). Transmission electron microscopy (TEM) was used to examine dislocation loops/ defect clusters formed by the implantation process. Measurements of the dislocation loop size were made using weak-beam imaging technique on cross-sectional TEM ion-implanted specimens. Defect clusters were observed in both deep and shallow implanted specimens, while dislocation loops were observed in the shallow implanted sample that was previously irradiated by 400 keV Ne ions. Cluster size was seen to increase for shallow implants in crystals irradiated with a deep implant (size ~8.5 nm) as compared to crystals treated only to a shallow implant (size ~3.1 nm).

INTRODUCTION

The damage response of many materials is controlled to a large extent by the fate of interstitials and vacancies in the residuals of cascades. At low temperatures vacancy mobility is often negligible compared to interstitial mobility. Therefore, three point defect annihilation mechanisms need to be considered [1]: (1) interstitial recombination with vacancies; (2) di-interstitial formation (i.e., nucleation of Frank loops); (3) interstitial annihilation at sinks. Sinks can be surfaces, grain boundaries, dislocations (such as Frank loops in (2)), voids, etc..

Spinel (MgAl_2O_4) is a ceramic of interest to the radiation effects community due to its surprising resistance to radiation damage. This resistance is manifested primarily in the minimal swelling of irradiated spinel, and in the lack of significant microscopic defect accumulation [2]. It has been established experimentally that the vast majority of point defects produced in single crystal spinel under irradiation annihilate by interstitial-vacancy recombination [2]. Microstructural defects produced during irradiation do not appear to be efficient sinks for interstitials. Explanations for this have been proposed in the literature [2], but the situation in spinel is not well understood.

The purpose of this study is to examine the effect that radiation-induced defects have on the evolution of subsequent radiation-induced microstructures of two spinel single crystals subjected to the same ion irradiation, but where one crystal was previously exposed to a low fluence, deep ion implant. The effect of the deep implant is to introduce a concentration of tiny microstructural defects that may serve as interstitial sinks in the subsequent irradiation experiment. If the defects play a role as sinks, we should observe a difference in the shallow implant microstructures of the two spinel crystals. If interstitial-vacancy recombination predominates, it is likely that no significant difference in the microstructures will be observed.

EXPERIMENTAL PROCEDURE

Single crystal wafers of magnesium aluminate $\text{MgO}-(\text{Al}_2\text{O}_3)_n$ with $n \sim 1$ and with [111] surface normal were obtained from Linde Division of Union Carbide. Each wafer was scribed into four pieces. Two samples were irradiated under cryogenic conditions (100°K) with 400 keV Ne ions at a fluence of $6.7 \times 10^{13}/\text{cm}^2$. One of these crystals and one unirradiated crystal were then irradiated at 100°K with 50 keV Ne ions at a fluence of $5.0 \times 10^{12}/\text{cm}^2$. The fourth sample was left unirradiated as a control sample. The projected range of 400 keV Ne ions is ~ 500 nm while the projected range of 50 keV Ne ions in spinel is ~ 50 nm, as determined using the Monte Carlo code TRIM [3]. These shall be referred to as "deep" and "shallow" implants, respectively. The fluence in the case of the deep implant was designed to produce ~ 2 target displacements per incident ion per nm at a depth of 50 nm, in conjunction with the end of the range of the shallow implant ion (determined by TRIM). This fluence was intended to produce a damage level that might introduce enough defect clusters to compete effectively with vacancies as sinks for interstitials.

TEM was performed using a Philips CM-30 analytical electron microscope operating at 300 kV, to examine the implantation microstructure of each sample. The implanted microstructure consisted of dislocation loops or small defect clusters not resolvable as loops. Dislocation loop size were measured using weak-beam imaging technique on cross-sectional TEM specimens. TEM samples were prepared by the tripod polishing technique of Benedict et al. [4].

RESULTS AND DISCUSSION

Figure 1 shows a weak-beam ($g = 440$) TEM micrograph of the deep implantation, as viewed in cross-section. Tiny defect clusters are observed across the entire implanted range, from the outer surface of the crystal to near the end of the range at ~ 500 nm. The average diameter of the defect clusters is ~ 6.2 nm with a maximum diameter of ~ 18.6 nm.

Figure 1. Weak beam ($g = 440$) image of the deep implanted sample in viewed in cross-section.

Figure 2a shows a similar weak beam ($g = 440$) micrograph viewed in cross-section from the shallow implanted sample. Again, small defect clusters are observed from the outer surface to the end of damage range at about 50 nm. The average diameter of the defect clusters is ~ 3.1 nm with a maximum diameter of ~ 7.7 nm.

Figure 2a. Weak-beam ($g = 440$) image of the shallow implanted sample viewed in cross-section.

Figure 2b shows a weak-beam ($g = 440$) micrograph viewed in cross-section from a deep plus shallow implanted sample, i.e. a sample implanted with 400 keV Ne ions, followed by a 50 keV Ne ion implantation. Defect clusters and dislocation loops are visible along the entire range of the shallow implant. The average diameter of the dislocation loops is ~ 8.5 nm with a maximum diameter of ~ 11.9 nm.

It has been determined in previous irradiation experiments using both neutrons and ions that defects resolvable as dislocation loops in spinel are faulted interstitial Frank loops with Burgers vectors of type $a/6\langle 111 \rangle$ or $a/4\langle 110 \rangle$ [2,5,6]. The later are the more stable dislocations and, in fact, $a/6\langle 111 \rangle$ loops were not observed in ion irradiated spinel crystals [6].

Results presented here indicate that the diameter of the defects in the shallow region of the deep plus shallow implanted sample (Figure 2a) are considerably larger than in the shallow-only implanted sample (Figure 2b). This result indicate that when defects are present from previous ion cascades (in this case, the deep implant ion cascades) the nucleation of the interstitial loops is suppressed during subsequent implantation (the shallow implant). The deep implant defects compete effectively with vacancies as sinks for interstitials, at least at the damage levels examined in this experiment.

In the deep plus shallow experiment, interstitials produced during the shallow implant are absorbed by deep implant interstitial loops since these appear to grow during the shallow implant. This is interesting because past studies have indicated that such loops are poor sinks for interstitials since they always incorporate a high energy stacking fault [2]. It would be interesting to perform higher fluence studies to see if these dislocation loop sinks are saturated or do eventually saturate.

Figure 2b. Weak beam ($g = 440$) image of the deep plus shallow implanted sample view in cross-section.

CONCLUSION

The effect of the deep implant is to introduce a concentration of tiny microstructural defects which may serve as interstitial sinks in the subsequent experiment. In this study, we confirmed that these defects do play a role as sinks, since we observed a difference in the shallow implant microstructures of the two spinel crystals. The apparent effectiveness of these defect clusters as sinks for interstitials may be due in part to the high concentration of defects introduced into the crystal in this experiment. In a neutron irradiation, where cascade defects are much more spatially dilute, these defects would likely not be able to compete effectively with vacancies as sinks for interstitials, except at very high neutron fluences.

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