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Crack-arresting compression layers produced by ion implantation

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Abstract

Compression layers were produced in aluminium oxide and magnesium oxide samples using high energy ion implantation. In the experiments, the samples were implanted with 3.0 MeV H^+ ions to a fluence of 3.3×10^{17} cm^{-2} over the area of $\sim$3.0 cm^2. The lattice expansion and compressive stress distribution with depth was measured using high resolution Raman microscopy. The implantation-induced compression layers were demonstrated to represent effective barriers for arresting the propagating cracks. Major factors that govern the crack closure, in particular the effect of ion energy and state of stress on the localization and efficiency of the implantation-induced compression layers, are discussed.

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Keywords: Crack arrest; Compressive stress; Ion implantation

1. Introduction

Ion implantation is known to produce lattice expansion in a number of ceramic materials which is attributed to injection of ions, production of point defects, structural change, charge accumulation and other factors [1,2]. Such expansion is constrained by the bulk of a sample, thus resulting in compressive stresses in the implanted layer. The compressive stress is beneficial for suppressing crack propagation in particular in brittle ceramic materials easily fractured under dynamic loading. The efficiency of crack arrest depends on the distribution and magnitude of the implantation-induced compressive stress as well as the tensile stress that causes the crack propagation. The high-energy low-mass ions have a long ion range and thus produce the compressive stress layer relatively deep below the surface. It is expected to be particularly efficient in crack arresting in cases where the applied tensile stress reduces from the surface to the sample interior.

This work aims at investigating the distribution of the implantation-induced compressive stress within the implanted layer using a high resolution Raman probe and analysing its interaction with cracks propagating under thermal shock.The efficiency of the compressive stress barrier in arresting propagating cracks is explored.

2. Experiment

The $(11\bar{2}0)$ and $(0\,0\,1)$ faces of sapphire and magnesium oxide single crystals, respectively, are uniformly implanted with 3.0 MeV H^+ ions to a fluence of 3.3×10^{17} cm^{-2} at room temperature. The samples used are $\sim$10 mm thick with the implanted surface area of $\sim 15 \times 30$ mm^2. The ion range under these conditions is 50 μm for sapphire and 60 μm for magnesium oxide crystals as determined by SRIM calculations and verified by optical microscopy. Following implantation, the cross sectional planes of sapphire are prepared using a diamond saw and analysed by Raman technique using a Renishaw machine at a laser wavelength of 325 nm. Using a 100× objective and a confocal aperture of about 100 μm, the focused laser beam illuminates a spot on the sample surface

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of 1 μm in diameter, as described in previous work [2]. The technique allows the measurement of the Raman peak shift, strain and stress distribution on the micron scale. The relationship between the Raman shift and strain components in trigonal crystals has been established previously [3]. The distribution of implantation-induced stress with depth in the implanted layer is determined using the stress–strain relations for trigonal symmetry [4].

Following implantation, the samples have been exposed to thermal shock produced by plasma pulse of ~10 μs duration. Only half of the crystal surface is implantated to compare the fracture response of implanted and unimplanted regions subjected to similar thermal shock conditions. In these experiments, the surface peak temperature is determined by measuring the size of fragments, bounded by cracks, and the gap between them as described elsewhere [5]. The crack penetration produced by thermal shock is analysed on the cross sectional plane in both implanted and unimplanted regions.

3. Results and discussion

Direct evidence of crack arrest is demonstrated in the optical photographs presented in Fig. 1(a) for MgO and in (b) for sapphire. The photographs are taken from cross sections made perpendicular to the ion implanted faces of the crystals. Both photographs show that the thermal shock-initiated cracks are effectively stopped within the implanted layer. Fig. 1(a) indicates that in the unimplanted region of the MgO crystal, cracks propagate to the depth of ~360 μm and deeper, while in the adjacent implanted region where the thermal shock intensity is the same they are arrested within the ion range of 60 μm.

The colour centres generated by the ion beam in the sapphire crystal make the end of ion range clearly visible in Fig. 1(b) at 50 μm below the surface. The cracks propagating from the surface reach a depth of ~40 μm below the surface and change their direction of propagation there. The fracture pattern indicates that beyond the 40 μm range the implantation-induced compressive stress barrier closes the cracks while within the range a system of cracks relieve the thermal shock-induced tensile stresses.

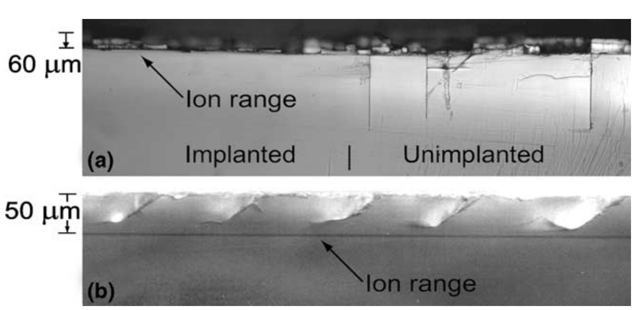

Fig. 1. (a) Fracture pattern profile of MgO crystal in the implanted (left) and unimplanted (right) regions and (b) fracture pattern profile in the implanted sapphire crystal.

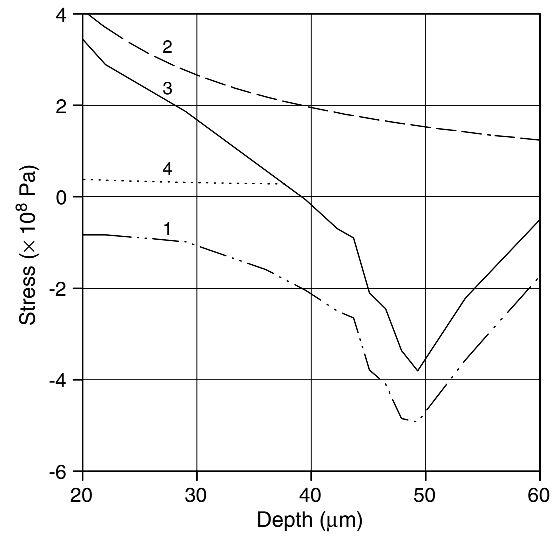

Fig. 2. Distribution of stress with depth in the implanted sapphire, 1 – implantation-induced stress, 2 – thermal stress, 3 – net stress, 4 – stress required for crack propagation.

A quantitative description of the crack arresting process in sapphire crystals is presented in Fig. 2. The implantation-induced compressive stress as determined by the Raman high-resolution method is presented by curve 1. It reaches a maximum value of ~0.5 GPa at the end of the ion range at 50 μm below the surface. According to SRIM data, the ion concentration and lattice damage drop abruptly to zero beyond the end of ion range. However, the Raman frequency shift and Raman luminescence intensity data indicate that the lattice damage is still substantial beyond 50 μm. Such an effect is most likely to be attributable to the ion "channeling tail". As a result, according to curve 1 in Fig. 2, the compressive stress barrier extends up to ~65 μm and has a full width at half maximum of ~14 μm.

The cracks originate at the surface and propagate into the crystal interior under the thermal shock-induced tensile stress. The latter is calculated using the stress–temperature diagram relevant for pulse heating [6] and presented by curve 2 in Fig. 2 for sapphire. The tensile stress distribution corresponds to experimental conditions under which the plasma pulse heating provides the peak surface temperature reaching the melting point of 2000 °C for sapphire. The superposition of the implantation-induced compressive stress and thermal shock-induced tensile stress fields results in the net stress distribution presented by curve 3 in Fig. 2.

The stress σ_g required for crack propagation, which is estimated for sapphire according to a fracture mechanics expression [7], is presented by curve 4 in Fig. 2. The cracks are arrested when the stress required for their growth is equal to or greater than the net tensile stress, $\sigma_g \geqslant \sigma$. From Fig. 2, the crack arrest takes places at the depth of ~38 μm, which is consistent with data on the optical photograph in Fig. 1(b).

According to Fig. 2, a further reduction in the range of crack propagation can be achieved by reducing the ion beam energy. In this case, the compressive stress barrier and hence the range of crack propagation are shifted towards the surface. Assuming the maximum of the compressive stress distribution remains unchanged, the analysis based on data in Fig. 2 yields that the depth of crack propagation can be reduced to ∼19 μm in sapphire.

4. Conclusion

Compressive stress layers are produced in sapphire and magnesium oxide crystals using 3.0 MeV H^+ ion implantation. The layers are observed to represent effective crack-arresting barriers. The implantation-induced compressive stress distribution and its effect on crack closure are considered. The analysis is used to evaluate the range of crack propagation and the efficiency with which cracks are arrested in the implanted magnesium oxide and sapphire crystals subjected thermal shock loading.

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