Letters

Yttrium Aluminium Garnet Single Crystals: Polishing, Etching and Dislocation Distribution

An important factor which affects the operation of solid state lasers is the presence of line defects in the single crystals used for this purpose. Observations on a wide variety of host lattices for laser ions have shown that the presence of dislocations can cause considerable distortion to a plane wave of light passing through the crystal. This effect is of greatest importance in anisotropic materials such as calcium tungstate, $CaWO_4$ [1, 2], and sapphire, Al_2O_3 [3], where dislocation low angle boundaries cause a directional change in the optic axis. However, optical examination of isotropic materials such as yttrium aluminium garnet, $Y_3Al_5O_{12}$, also reveals the occurrence of strain [4] indicating the presence of dislocations in this laser host lattice. In this note, a method of revealing the dislocations as etch pits is described, together with some observed dislocation arrays.

The garnet single crystals used in this work were grown by the Czochralski pulling from the melt technique, the details being described elsewhere [5]. The crystal growth axis was parallel to a $\langle 100 \rangle$ direction and the crystals typically contained 0 to 1 at. % neodymium. {100}, {110} and {111} surfaces were exposed in these crystals using a diamond saw. $Y_3Al_5O_{12}$ is a relatively hard material (Mohs hardness 7), but the damaged layer produced by mechanical polishing is still too deep to allow meaningful etching on a surface prepared in this manner. It was found that the work-damaged layer could be removed under the conditions previously described for calcium tungstate [6], in which a featureless damage-free surface was produced by chemically polishing the sections of crystal in orthophosphoric acid at a temperature of 250° C. The polishing times were somewhat longer in the present case, taking 1 to 2 h, but a highly polished surface was obtained on the three low index surfaces examined.

Yttrium aluminium garnet is not chemically attacked by dilute or concentrated acids and acid mixtures based on HCl, HNO_3, H_2SO_4 and HF, even after prolonged heating at 70° C. Molten salts of an acidic nature such as $KHSO_3$ and $K_2S_2O_7$ cause pitting, but the whole surface is attacked, and the crystal becomes translucent in 5 to 10 secs. Basic molten salts, however, produce pits which are typical of dislocation etchants. Fig. 1 shows etch pits, square in section, which are produced by molten KOH in 30 secs on a {100} surface perpendicular to the growth direction. Fig. 2 shows the etch pit distribution on a {100} surface parallel to the growth direction. Etch pits are also produced on {110} and {111} surfaces under identical conditions;

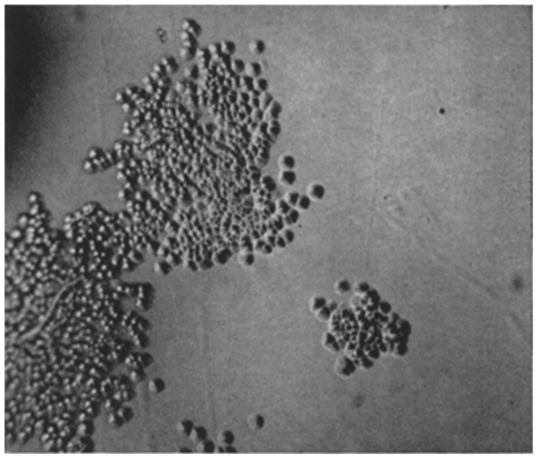

Figure 1 Randomly distributed groups of dislocation etch pits on a {100} surface perpendicular to the growth direction (X183).

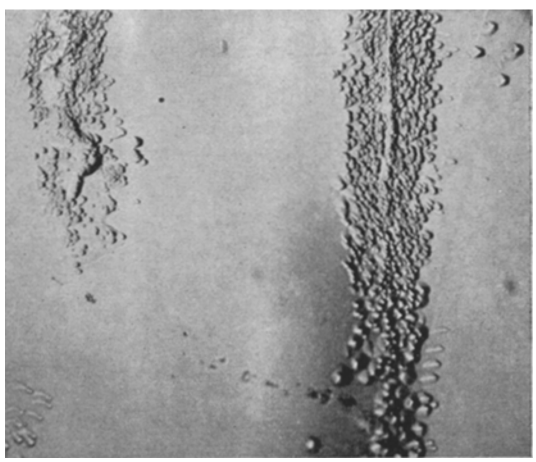

Figure 2 Bands of dislocation etch pits on a {100} surface parallel to the growth direction (X183).

in these cases the pits are triangular in section. The reagent is difficult to calibrate as a dislocation etchant by traditional means, as the garnet has no cleavage plane to allow matching of opposite surfaces, and the absence of low angle boundary T-junctions prevents calibration by measurement of the angular tilt around the point at which the boundaries intersect. However, the etch pits are considered to correspond to dislocations on the basis of the following observations:

(a) the pits line up in arrays typical of dislocation low angle boundaries as shown in fig. 2;
(b) prolonged etching produces an increase in the size of the pits but does not change their density or distribution;
(c) annealing at a temperature of 1500° C for 150 h produces a slight increase in the number of low-angle boundaries which suggest that dislocation movement has occurred.

The etch pit arrays observed in the garnet differ somewhat from those already described for other Czochralski-grown laser materials. For instance extensive arrays of low-angle boundaries are formed in both calcium fluoride and calcium tungstate and dislocations lying along slip bands are also observed [7]. In the present case, few low-angle boundaries are formed and there is no evidence of extensive slip. Crystals sectioned perpendicular to the growth axis, fig. 1, show randomly-distributed groups of dislocations. The same crystals sectioned parallel to the growth axis, fig. 2, show that these groups extend into bands in the direction of growth. These bands correspond to the strained regions described previously [4], where there is an enhanced neodymium incorporation due to the change in habit of the solid/liquid interface during growth from a planar to a cellular form. The growth striations delineating the interface shape can just be discerned in fig. 2.

Acknowledgements

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Grain Boundary "Pest" in the Intermetallic Compound NiAl

The "pest" phenomenon – the susceptibility to severe intergranular attack, which in the limit causes complete disintegration of a material – has been observed in a wide variety of intermetallic compounds, particularly silicides, beryllides and aluminides. The conditions under which it occurs are not clear. Westbrook and Wood [1] have shown that intergranular embrittlement in a wide variety of intermetallic compounds is associated with anomalous grain boundary hardening effects, resulting from the segregation of gaseous contaminants to the boundaries. Grain boundary hardening was only observed in compounds containing more than the stoichiometric proportion of the electropositive component. In a detailed study of NiGa, Seybolt and Westbrook [2] concluded that the hardening was due to the presence of dissolved oxygen, and they suggested that the hardening mechanisms might involve the lattice distortion produced by the formation of a Ga–O complex, e.g. an oxide, a sub-oxide or a cluster of oxygen atoms. Thus grain boundary hardening, which seems to be a necessary condition for the occurrence of the pest, can only be accounted for in a very tentative manner and, to date, no suggestions for a pest mechanism have been published. The purpose of this letter is to propose a mechanism for the pest which is based on some of the results of an investigation of the compound NiAl.