LIQUID/SOLID INTERFACE PROFILE OF MELT GROWN
OXIDE CRYSTALS

II. CRYSTAL QUALITY

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Temperature gradients and rotation rates influence the quality of Czochralski grown oxide crystals. Decreasing the heat transfer from the melt level and increasing the rotation rate increases the optical homogeneity and structural perfection of the crystal, due to convex interface becoming flattened. Growth striations due to temperature fluctuations in the melt during growth are affected by the heat losses from the melt level but are practically unaffected by the rotation rate as long as a narrow ring of centrifugally streaming melt in the neighbourhood of the growing crystal is not formed. The parts of the crystals grown in the presence of this ring contain no striations.

1. INTRODUCTION

Specific defects in Czochralski grown oxide crystals depend strongly upon the liquid/solid interface profile of a growing crystal [1]. Earlier studies have shown that steep radial temperature gradients produce a conical interface causing core formation in doped crystals [2—8]. Alternatively, flattening of the isotherms to produce an almost planar interface prevents core formation entirely and the crystals have excellent optical homogeneity. In practice these conditions have been attained by rotating the crystal at relatively high speeds [1, 6]. Other defects — mainly growth striations — are influenced by the chemical composition of the melt and by temperature fluctuations or oscillations [9—12]. Constitutional supercooling causes cellular growth of the crystals [13—15].

In the previous paper of this series [16] it was shown, that there are four types of interface profiles in growing crystals. This paper presents the results of the characterization, by optical methods and X-ray topography, of oxide crystals grown with the various types of interface profiles.

2. EXPERIMENTAL

Specimens of crystals were examined by the following methods:

(a) Inspection between crossed polaroids
(b) Twyman-Green interferometry

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(c) X-ray methods. Conventional Lang topography with MoKα₁ radiation was used. The samples were 0.7 mm thick crystal slices etched in boiling 85% H₃PO₄. Dislocation densities were verified by etching studies using H₃PO₄ as an etchant [17] and by a three-crystal X-ray spectrometer using (1014) CaCO₃ crystals as monochromators (the dislocation density was calculated from the halfwidth of the reflection curve).

(d) Tyndall scattering. The crystals were immersed in monobromnaphthalene. A 200 W bulb was used as a light source. Tyndall scattering of the polished laser rods made of the crystals was measured quantitatively using a Ulbricht’s sphere with a photomultiplier.

(e) Polarized shadow projection [18] using a He–Ne laser as a light source. Striations and other optical defects were photographed at a distance of 3 m behind the crystal slice.

3. RESULTS

The four types of interface profiles (see Fig. 2 in the previous paper of this series [16]) are associated with specific defects:

(a) Optical homogeneity and structural perfection.

Interface profile of the first type are associated with highly strained structures. Rubies and sapphires grown with this type of the interface show unusual interferometer patterns (Fig. 1, Appendix I, p. 1412a). The number of the observed fringes can be substantially lowered by annealing. Such an increase of the optical homogeneity was described previously by CHARVÁT et al. [19]. A similar effect is possible in crystals containing no macroscopic structural defects, such as mosaic blocks [20]. This is also usual for the interface of the first type, because the mosaic blocks, which can originate for example at the seed/crystal transition region are oriented perpendicularly to the interface and vanish at the surface of the crystal (Fig. 2, Appendix I, p. 1412a). The dislocation density for both Al₂O₃ and YAG did not exceed 10³–10⁴ cm⁻².

Polarized shadow projection as well as inspection between crossed polaroids of sapphire and ruby revealed no obvious defects. On the other hand YAG crystals, both doped and undoped showed typical figures of the “maple leaf” form, i.e. they possessed highly strained structures. Similar patterns were shown by interferometry (Fig. 3, Appendix I, p. 1412b). Annealing had no effect in the case of YAG crystals.

The second type of interface produced typical core structures [1, 7, 8] for YAG : Nd and ruby (Fig. 4a). Shadow projection showed a slightly strained core in the case of YAG : Nd only. Undoped crystals had no core and showed practically no interference fringes. Strain facets in the core of the YAG : Nd crystals produced sometimes slightly misoriented blocks (10° to 20°), the boundaries of which were