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### Influence of mosaicity on the fracture behavior of sapphire

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#### Abstract

The resistance to breakage of sapphire samples of different crystallographic orientations and produced by the Kyropoulos and Verneuil processes was investigated. The fracture strength was determined by using four-point bending tests, whereas the single-edge V-notch beam method was used to measure the fracture toughness. For both mechanical properties, it was found that (i) Verneuil sapphire has values up to two times higher than Kyropoulos sapphire and (ii) the data scatter is also generally higher for Verneuil sapphire. The main factor responsible for this behavior is believed to be the presence of mosaicity in Verneuil and its absence in Kyropoulos sapphire. By using the Read–Shockley model to estimate the energy of low-angle grain boundaries, together with the Griffith energy criterion for intergranular crack propagation, it is demonstrated that intergranular fracture along the mosaic block boundaries is energetically more favorable than transgranular fracture for crack-to-boundary orientations smaller than a critical deflection angle. Nevertheless, both the location of the crack in respect to the boundary, as well as the 3-D nature of these features, must be taken into consideration for a complete description of the intergranular deflection toughening mechanism.

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#### 1. Introduction

Single-crystal sapphire is the preferred material for highperformance system and component designs. This is due to its combination of favorable chemical, electrical, mechanical, optical, surface, thermal and durability properties. However, when growing a sapphire crystal, several types of defects, such as vacancies, dislocations, impurities, inclusions and mosaic blocks, can be produced and affect its functionality [1]. This aspect is particularly critical in the use of sapphire for optical applications, e.g. windows, lenses, prisms or optical fibers, as these defects may act as light absorption or scattering centers and hence compromise the optical efficiency of the devices. In addition, it is well known that for most materials, and specifically ceramics, defects have an important influence on the mechanical behavior and thus affect their tribomechanical performance during device processing—cutting, grinding, polishing, etc. Sapphire is not an exception to the rule [1]. In particular, the formation of large lateral and radial cracks may result in the generation of large chips [2], which in spite of

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increasing the process efficiency can lead to the dimensional and/or geometric tolerances of the manufactured components being exceeded.

Of all the defects that can be generated in sapphire, mosaic blocks are one of the least understood, and thus their influence on the mechanical properties of sapphire has been very little investigated [3,4]. On the one hand, a mosaic structure consists of an aggregate of low-misoriented perfect crystals (blocks) relatively smaller than its macroscopic bulk crystal, separated by dislocation-wall boundaries resembling low-angle grain boundaries. On the other hand, mosaicity corresponds to the width of the spatial distribution of misorientation angles between neighboring blocks over the entire crystal volume. The presence of mosaicity in sapphire to a smaller or larger extent depends on the production process. Two highly industrialized processes of crystal growth from the melt that produce crystals with significant differences in crystalline perfection are the Kyropoulos (KYR) and the Verneuil (VER) methods [1]. The KYR method involves crystal growth through a slow decrease of the melt temperature combined with a slow pulling of the crystal from a melt containing a seed held in a crucible. These two features result in low temperature gradients (<7-10 °C cm<sup>-1</sup>) [1], thereby improving the perfection of the crystal growth. In the crucible-less VER method, finely dispersed powder of the raw material is fed through a flame onto a  $\sim 40 \,\mu m$  thin melt film; in this case, the temperature gradients in the crystallization zone are high  $(300-1000 \text{ °C cm}^{-1})$ . As a consequence, high residual stresses (up to 100-150 MPa) may be generated in the growing crystal [1], leading to the occurrence of plastic deformation and subsequent polygonization, which result in the formation of mosaic blocks [5].

Extensive interruptions in crystal periodicity such as mosaic block boundaries may affect significantly the mechanical response of brittle materials such as sapphire, which are often assumed to deform elastically up to brittle fracture [2]. The two main mechanical properties describing the fracture behavior of a material, which is directly related to its machinability, are the fracture strength ( $\sigma_f$ ) and the fracture toughness ( $K_{Ic}$ ). Therefore, the objective of this paper is to shed some light on the influence of mosaicity on these properties. To achieve this goal,  $\sigma_f$  and  $K_{Ic}$  were determined for different crystallographic orientations of sapphire samples produced by the KYR and VER methods and their relation with the degree of material crystalline perfection was investigated.

#### 2. Experimental methods

#### 2.1. Sample preparation and orientation

Sapphire samples produced by the KYR and VER methods and cut into  $3 \text{ mm} \times 4 \text{ mm} \times 45 \text{ mm}$  bars with four different crystallographic orientations (Fig. 1) were supplied by Stettler Sapphire AG. A maximum misorientation angle of  $2^{\circ}$  with respect to the crystal growth direction was determined by performing X-ray measurements in five samples for each orientation. All the remaining faces were supplied with the same mirror-like surface polished condition, except for the extremities of the bars ( $3 \text{ mm} \times 4 \text{ mm}$  sample faces).

## 2.2. Four-point bending tests and single-edge V-notch beam method

Four-point bending tests were performed according to the norm EN 843-1 to measure the fracture strength of sapphire [6]. Fracture toughness was determined by using the singleedge V-notch beam (SEVNB) method according to the norm CEN/TS14425-5 [7]. The two types of tests were carried out in air at room temperature using a common setup (see Fig. 2a) mounted in a Zwick Z005 (Zwick GmbH, Ulm, Germany) loading machine, where the sample is supported on two (lower) bearing rollers perpendicular to its length. The bearing rollers consist of  $5.0 \pm 0.1$  mm diameter parallel cylinders capable of rolling outward on flat support surfaces. They are initially positioned  $40.0 \pm 0.5$  mm apart with respect to their centers. The two (upper) loading rollers are located at the quarter points, with an inner span of  $20 \pm 0.2$  mm and are free to roll inwards. Three of the four rollers are able to rotate to prevent any torsion during the



Fig. 1. Four different crystallographic orientations of the sapphire bar samples.

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