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Research of crack resistance of products made of leucosapphire in dependence on conditions of crystals growth by Stepanov method

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Systematic research was carried out into crack resistance on the planes {1210} of the leucosapphire disks made of the bands grown by Stepanov method in a direction of <1010> at varying technological parameters and growing modes (initial raw material grade, bands growing rate, quantity of heat-insulating shields, pressure and grades of argon inert gas).

Проведено систематическое исследование трещиностойкости на плоскости {1210} дисков из лейкосапфира, изготовленных из лент, выращенных методом Степанова в направлении <1010> при варьировании технологических параметров и режимов выращивания (марка исходного сырья, скорость выращивания лент, количество теплоизолирующих экранов, давление и марки инертного газа аргона).

1. Introduction

Fragile materials resistance to fracture is estimated with the help of destruction mechanics. When stresses at a crack (defect) top reach a critical value, a catastrophic extending of the crack — fracture begins. This situation is estimated by a value of the stress intensity factor K_{1C} (index *1* refers to the conditions of normal separation at the plane deformation); this value is also named a factor of crack resistance or ductile fracture.

Surface fracture energy of refractory compositions with an ionic type of bond is mainly determined by processes of interatomic bonds rupture due to low mobility of dislocations. Their effective surface energy γ_i exceeds a thermodynamic surface energy γ_0 not more than by 10 times. And this increase is mainly caused by geometrical factors defining increase in the effective fracture surface since γ_p — energy for microplastic deformation is practically absent. For the majority of oxide ceramic single-phase materials the value K_{1C} does not exceed ~5 MPa·m$^{1/2}$ [1].

Definition of K_{1C} by a microindentation method is convenient, and preparation of special samples with notches is not required in this case. This method allows expanding the scope of the researches of leucosapphire crack resistance which was carried out directly on the natural cleavages of the crystals grown by Stepanov method from various grades of raw materials under various growth modes, microalloying with various additives, for different single crystals planes [2]. So the crack resistance of leucosapphire K_{1C} in dependence on a crystal orientation is changing from 2.0 to 4.5 MPa·m$^{1/2}$; the greatest value is related to the basal plane; the least one is related to planes $\{\bar{1}012\}$ and $\{\bar{1}014\}$, and the plane {1210} has an intermediate value of K_{1C} [3–5]. As it was shown by radiographic researches [6], grinding and polishing of the growth

Table. Summary data on variants of growing and variable technological parameters

Variant	Brand of aluminum oxide raw material				Growth rate, mm/min				Shields quantity		Inert gas pressure, VCH or OSCH, kg/cm^2
	SPEK, Russia	Non-stand., Czech Republic	RCA, France	Pressed pellets (PRC)	0.5	0.8	1.0	1.2	1	2	+0.1
1		+				+			+		VCH
2	+					+			+		VCH
3			+			+			+		VCH
4		+				+			+		VCH
5				+		+			+		VCH
6		+				+				+	OSCH
7		+				+				+	VCH
8		+			+					+	VCH
9		+					+			+	VCH
10		+						+		+	VCH
11		+				+				+	VCH
12 with annealing		+				+				+	VCH

surface lead to occurrence of the compressive stresses from 135 up to 170 MPa within the surface layers of the leucosapphire with a depth of 14 μm.

As to the effect of internal structural crystals defects, the paper [7] describes detailed researches into influence of pores, boundary lines of blocks, dislocations, point defects on crack resistance of the sapphire grown by the Stepanov method. The crack resistance factor is falling with growing the pores density, growth dislocations, blocks disorientation angle and impurity atoms concentration; and the crack resistance factor is increasing while growing the mobile dislocations density and block structure size. In this case the boundary line of the blocks extension is not so important as the angle of their mutual disorientation. If the disorientation angles reach a value of a degree or more, the crack resistance factor sharply decreases, and the width of the area with a reduced value K_{1C} increases up to (20–60) μm.

Increase in density of inactive growth dislocations from 10^2 cm^{-2} to 10^6 cm^{-2} reduces the crack resistance factor of K_{1C} by 2 times, and increase in density of active dislocations in the near-surface layers due to mechanical treatment leads to the essential growth of the crack resistance factor already at growing their density by one order. This fact is important to be taken into account when researching the influence of the technological parameters of crystal growth on their crack resistance at choosing the finishing technological parameters, including mechanical treatment for preparation of the samples.

This paper set the task to research the crack resistance of polished leucosapphire disks of 25 mm in a diameter and 2 mm in a height depending on the technological growing parameters of the bands which the mentioned disks were made of. Dependences on the following technological parameters were studied: a grade of initial raw materials, a growth rate, shielding of the bands to be grown, a grade and pressure of the argon in which atmosphere the bands (Table) were grown.

2. Research technique

The disks were indented using a microdurometer MICROMET-2103 with the following measurement of the diagonals of the formed plastic indentation and the median cracks round it. The optimum loading on the indenter, and also an angle between the indentations diagonals direction and the crystal growth direction were chosen such as the crystal surface would be minimally damaged by secondary (near-surface) cracks formation at the maximal length of the me-

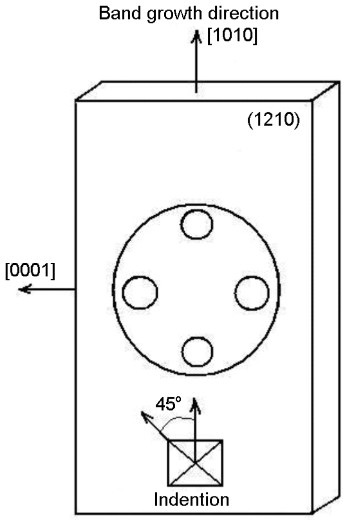

Fig. 1. Lay-out chart of crack resistance research areas on the sample with pointing the diagonals direction on indentions relative to the band growth axis direction.

dian ones; and a ration of the median cracks length c to the size of a plastic indentation d was $1.7 \le c/d \le 5.0$. Thus a loading of 500 g (5H) and the angle equal to 45° were chosen.

Measurements were carried out in four areas on each of the samples at a distance of ~5 mm from its edge (Fig. 1). Number of the measurements in each area was chosen as 12 for collecting statistical data and excluding the indentions with a large degree of damages from the final result.

The diagonals of the diamond tripod indentions and also lengths of the occurred median cracks were measured by OLYMPUS metallographic microscope using OMNIMET system for indentions collecting and processing, and making reports.

The maximal and minimal values of the investigated characteristics were excluded from the measurements results and average values were calculated for microhardness and maximal median cracks lengths in each of four indention areas in two samples of the same technological batch.

The values of the crack resistance factor were calculated by the formula:

$$K_{1c} = 0.016 \cdot \left(\frac{E}{HV}\right) \cdot P \cdot C^{-3/2} [\mathrm{MPa} \cdot \mathrm{m}^{1/2}],$$

where E — Young's modulus, 400 GPa; HV — microhardness, GPa; P — loading, 500 g; C — maximal median crack length, m.

3. Results of researches

When defining crack resistance of Al_2O_3 sapphire single crystal samples grown under

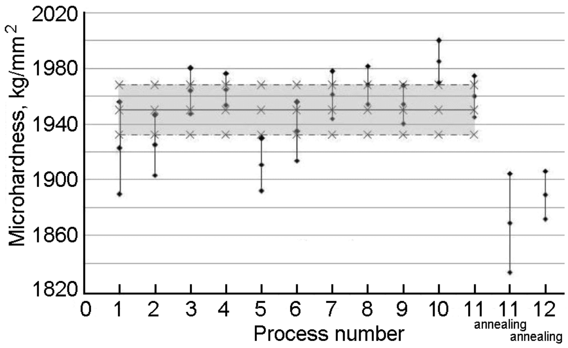

Fig. 2. Microhardness of the samples in dependence on a growing process.

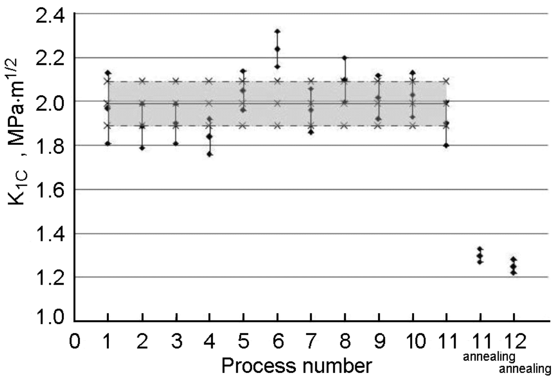

Fig. 3. Crack resistance of the samples in dependence on a growing process.

different technological modes, the special attention was paid to decrease in the measurements errors, and as consequence, to a possibility of the samples batches separation according to the technological indications. A lot of statistic data on the measurements of microhardness and median cracks lengths was collected for this purpose. As a result the maximal error in the microhardness measurement is 1.7 %, in definition of the crack resistance factor is 8.12 % for a confidence probability of $P = 0.99$. It was determined that the obtained average microhardness values of the investigated technologies variants (batches 1–11) were within the range of (1932–1968) kg/mm^2, and the average values of the crack resistance factor K_{1C} were in the range from 1.89 up to 2.09 MPa·m$^{1/2}$. The diagrams of changes in the microhardness and crack resistance of the batches samples (1–11) for a confidence probability of $P = 0.99$ are given in Fig. 2 and 3. The samples obtained under variant 10 (1925–2010) kg/mm^2 have a higher microhardness in comparison with the average microhardness value; the samples obtained under variant 5 (1873–1942) kg/mm^2 have the least microhardness in comparison with the average microhardness value. High crack resistance (2.24 MPa·m$^{1/2}$) was registered for the samples of batch 6 as regards the average crack resistance value of the samples of batches (1–11). The samples fabricated under process variant 4 have the least crack resistance — 1.84 MPa·m$^{1/2}$. Crack resistance of the samples obtained under variants 7, 9, and 10 is the closest to the average crack resistance of the samples of batches (1–11).

4. Discussion of results

Analyzing the obtained results, it should be noted that, despite a wide range of variable technological parameters of the bands growth processes, change in the crack resistance factor K_{1C} occurs in a narrow values interval of (1.84–2.24) MPa·m$^{1/2}$.

The indentation method for definition of the crack resistance factor K_{1C}, used in this paper, is surface, i.e. the median crack occurred at indenting, extends mainly in the near-surface layer.

Therefore the most probable cause of such measurements results leveling is a process of the samples preparation for testing. Irrespective of the crystal growing conditions all the samples were mechanically grinded and polished, i.e. they reached about the same structural state of the near-surface layer which was characterized by the higher dislocations density and residual compressive stresses available. This structural state rather essentially differs from the structural state of the crystal volume which is characterized by the conditions of its growing.

The results of the annealed samples research show that layer leads to essential increase ~(30–40) % in the crack resistance factor values, and as consequence, levels them. The highest values of the crack resistance factor K_{1C} of the investigated batches (1–11) refer to batch 6 which distinctive features are use of two thermoinsulating shields and excessive pressure of inert gas of OSCH grade that is equal to +0.1 kg/cm^2.

The lowest values of the crack resistance factor K_{1C} of batches (1–11) refer to batch 4 which distinctive features are use of one shield and utilizing inert gas of VCH grade.

Both these results were obtained when using raw materials of a Non-standard grade, Czech Republic, on a form-builder made of MCHVP alloy with the use of a crucible with a diameter of 120 mm and made of MCHVP alloy. It testifies to essential effect of the growing conditions on leucosapphire crack resistance, but it requires large experimental efforts for implementation of the optimal technological solutions.

5. Conclusions

Despite a wide range of variable technological parameters of bands growth processes, change of the crack resistance factor K_{1C} occurs in a narrow interval of values (1.84–2.24) MPa·m$^{1/2}$ that is caused approximately by the same structural state of the near-surface layer of the samples after mechanical treatment. With respect to an average value of the crack resistance factor K_{1C} of the samples of all the processes variants 1–11, which is equal to (1.99±0.1) MPa·m$^{1/2}$ for a confidence probability of $P = 0.99$, the samples of batch 6 have the highest crack resistance factor K_{1C} = (2.24±0.08) MPa·m$^{1/2}$ grown with a rate of 0.8 mm/min from the raw materials Non-standard, Czech Republic at an excessive pressure of +0.1 kg/cm^2 of argon of OSCH grade with using 2 shields. The samples from batch 4 have the minimal crack resistance of K_{1C} = (1.84±0.08) MPa·m$^{1/2}$; the batch was also grown from the raw materials Non-stand., Czech Republic at a rate of 0.8 mm/min and excessive pressure of +0.1 kg/cm^2, but it differs from batch 6 with using 1 shield and VCH argon grade. Considerable decrease in the crack resistance factor K_{1C} of the samples of technological batches 11_{ann}, 12_{ann} after high-temperature annealing (1800°C, 1 hour) to the values of (1.25–1.30) MPa·m$^{1/2}$ and double contraction of the values range indicate a higher degree of structural perfection of the near-surface layer of the samples owing to the structural defects of annealing and the residual compressive stresses occurred at mechanical treatment. These results, apparently, are close to the crack resistance values in the volume of unannealed samples.

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Дослідження тріщиностійкості виробів з лейкосапфіру в залежності від умов отримання кристалів методом Степанова

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Проведено систематичне дослідження тріщиностійкості на площині {1210} дисків з лейкосапфіру, виготовлених із стрічок, вирощених методом Степанова у напрямку <1010> при варіюванні технологічних параметрів і режимів вирощування (марка початкової сировини, швидкість вирощування стрічок, кількість теплоізолюючих екранів, тиск і марки інертного газу аргону).