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Specific growing features of variable section sapphire articles by Stepanov technique

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Problems in growth of variable section corundum crystals by Stepanov technique have been considered. The structure perfection of the crystal profile variation zone has been studied as a function of the growing technology parameters. A procedure has been developed to provide variable section sapphire profiles of optical quality.

Рассмотрены проблемы получения кристаллов корунда переменного сечения методом Степанова. Исследовано структурное совершенство зоны изменения профиля кристалла от технологических параметров роста. Разработан способ выращивания сапфировых профилей переменного сечения оптического качества.

Sapphire is indispensable in numerous technological systems due to its mechanical and chemical properties. Recently, there is an increasing interest in sapphire wares shaped as crucibles of various configurations. Such articles are used as thermocouple casings, crucibles, thermostat parts. The crystal shape change during the growing results in varying thermal conditions in the crystallization zone, thus causing various defects affecting the optical properties. The dislocation density and stress level in the profile changing region increase, too.

A number of techniques is known to provide the articles including a closed volume, such as the non-capillary forming, the die rearrangement, the growing from a mold element, variation technique, and sealing [1–5]. However, none of those provides the optical quality articles. This work is aimed at the manufacturing of optical quality sapphire profiles of variable section.

The crystals were grown in a "Kristall 606" unit with inductive heating and graphite thermal assembly. The breakage of sapphire grown by the Verneuil technique with the total impurity content of max 10^{-2} mass.% was used as the raw material, the growth process was monitored using a video system. The transition zone growth speed was maintained at 15–20 mm/h in all the experiments.

The following procedure was developed to manufacture the articles with optical quality bottom. First, a crystal was grown that defined the article "walls" (a cylindrical tube in the simplest case), and then the "bottom" of desired profile, e.g., a cylindrical rod (Fig. 1), was grown onto the "wall" using the latter as a seed. The bottom end growing (sealing) was carried out using a die with one feeding channel, thus avoiding the collision of melt flows in the growth zone and formation of defect zones in the crystal [7, 8]. To provide the formation of a stable melt film, the seeding and growing were carried out at a high hydrostatic pressure of the melt in the growth zone; to that end, the maximum crucible charging was used. Various apex angles α of the bottom part were provided that define the bottom quality by varying the heater power, the thermal conditions in the crystallization zone and the pulling speed being the same. This made it possible to vary the transition length h and its profile (Fig. 2).

To study the dependence of the transition zone structure perfection on the die

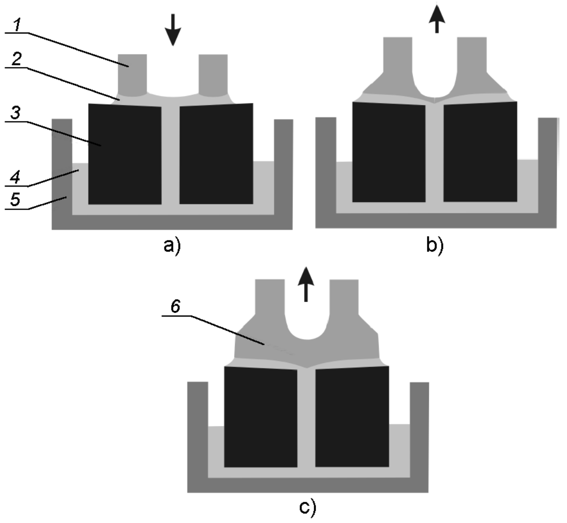

Fig.1. Sealing of a cylindrical tube: (a) seeding, (b) side growing, (c) bottom growing; *1*, tube; *2*, melt film; *3*, die; *4*, melt; *5*, crucible; *6*, bottom part of the article.

design, the tubes of the same structure perfection were sealed using the flat and concave dies (Fig. 3). All the experiments were carried out using the tubes of OD = 8 mm and ID = 5 mm and the dies of 16 mm in diameter with the 0.7 mm diameter feeding channel. The block structure and the pore density in the transition zone were studied using a MIN-8 polarization microscope and a PKR polariscope in scattered and polarized light. To that end, two parallel polished planes were made along the sample axis.

Effect of the growing angle α on the structure perfection of the bottom (Fig. 2).

α = 25°: The transition zone profile is almost flat, the transition relative length $h/\mathrm{ID}<0.3$. This is due to the high crystallization speed: the crystal radial growth speed exceeds considerably the axial one. A high defect density was observed within the transition zone (15–25 blocks with the disorientation angles of 1–3°, the pore density about 10^5 cm^{-3}, the pore size about 10 to 50 μm). The bottom is optically opaque. Cracks were often appeared in the transition zone due to the thermoelastic stresses at the block boundaries.

α = 50°: The transition zone profile is spherical with the radius 0.5ID, the transition relative length is 0.5<Ih/ID<1. The pore density in the transition zone was about 10^3 cm^{-3}, the number of blocks 3 to 5 with disorientation angles of 30′ to 40′. The bottom is optically transparent.

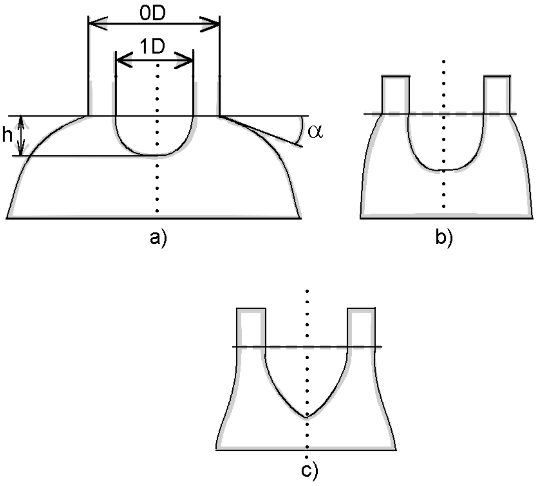

Fig.2. The tube-to-rod transition shape at various side growing angles (α~, deg): (a) 25: (b), 50; (c) 70.

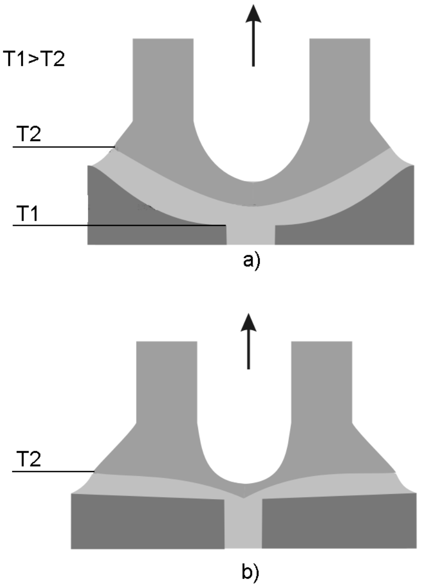

Fig.3. The tube sealing at concave (a) and flat (b) dies.

α = 70°. The transition zone profile is conical. The profile became closed at a considerable distance from the seeding point, the transition relative length is $h/\mathrm{ID}>1$. A chain of pores appeared often in the center due to the melt overheating and its increased gas saturation. The transition zone was asymmetrical.

It is seen from Table that the optimum formation conditions of the transition zone (bottom part) are provided at α = 50° and the transition zone length of 0.5ID to 1ID.

Effect of the die shaping surface on the transition zone quality.

When growing the bottom part of an article using dies with flat and concave sur-

Table. Dependence of the bottom structure perfection on the growing angle.

a	*h*/ID	Block number	Pore density, cm^{-3}
25	0.1–.30	15–20	10^5
50	0.5–1	3–5	10^3
70	1–1.5	5–10	10^4

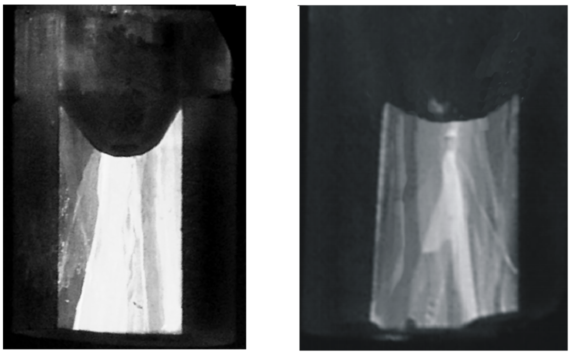
a) b)

Fig.4. The tube-to-rod transition zone grown using concave (a) and flat (b) dies as seen between crossed polaroids.

faces (Fig. 3) at the growing angle α ~ 50°, the smallest number of blocks was observed in the bottom part grown with concave shaping surface (Fig. 4). This is connected with the fact that when the bottom part is grown using the flat die, the temperature T_2 is the same over the whole plane of the crystallization front, while with the concave shaping surface, the crystal center part grows at a higher temperature T_1 (Fig. 3). With the flat die, the sealing rate is higher than with the concave one, therefore, the transition zone contains 5 to 10 blocks if the die is flat and, in contrast, only 3 to 5 blocks if the die is concave. The convex crystallization front favors also the pore forcing towards the crystal periphery and the decreased concentration thereof at the bottom center.

At α = 60° and *H*/ID = 0.6, 20 mm long crucibles have been grown with 10 mm outer diameter and optically transparent bottom. Three blocks are seen within the transition zone under polarized light (Fig. 5). The bottom part is pore-free.

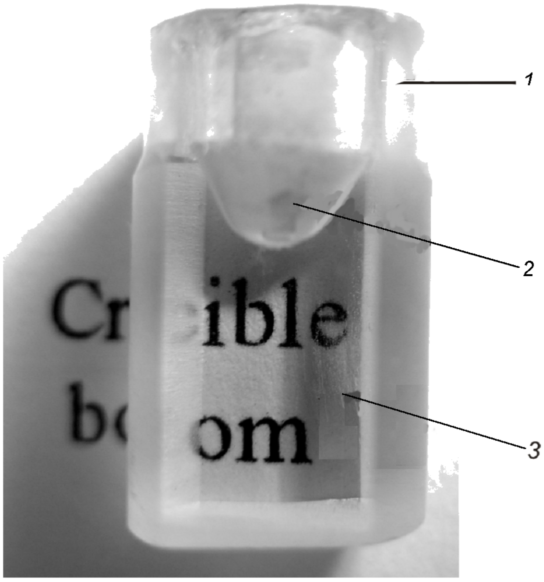

Fig.5. A 20mm long crucible seen in scattered light. Tube size: OD=10mm, ID=5mm. *1*, tube; *2*, transition zone; *3*, crucible bottom.

To conclude, the dependence of the transition zone structure perfection on the growing angle α and the die shaping surface configuration have been studied. The best crystal quality is provided at the concave shaping surface of the die and the transition zone growing angle α of 50 to 60°. Sapphire crucibles with optically transparent bottoms have been obtained.

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Особливості вирощування сапфірових виробів змінного перетину методом Степанова

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Розглянуто проблеми отримання кристалів корунду змінного перетину методом Степанова. Досліджено структурну досконалість зони зміни профілю кристала від технологічних параметрів зростання. Розроблено спосіб вирощування сапфірових профілів змінного перетину оптичної якості.