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Crystal Research and Technology

Journal of Experimental and Industrial Crystallography
Zeitschrift für experimentelle und technische Kristallographie

WILEY-VCH

Cryst. Res. Technol. **44**, No. 7, 701 – 706 (2009) / **DOI** 10.1002/crat.200900068

Analysis of the profile curves of the menisci for the sapphire capillaries and fibers growth by EFG (Stepanov) technique

S. N. Rossolenko*, V. N. Kurlov, and A. A. Asrian

Institute of Solid State Physics RAS, Chernogolovka, Moscow District, 142432, Russia

Received 4 February 2009, revised 10 April 2009, accepted 14 April 2009
Published online 4 May 2009

Key words EFG, meniscus, profile curve, sapphire.
PACS 42.70.Km, 07.05.Dz, 07.20.Hy

Profile curves of the small liquid menisci are investigated on the base of numerical solution of the Young – Laplace equation. There are considered menisci for the growth of crystal capillaries and fibers by the EFG (Stepanov) technique. Differences in the shapes of the small and "large" menisci were found.

1 Introduction

Sapphire has long recognized as a material with unique properties. Sapphire has a high refractive index and a broad transmission band spanning the ultraviolet, visible and infrared bands, a high hardness, melting point, very good thermal conductivity, biocompatibility, tensile strength and thermal shock resistance. High level of technology of growing sapphire shaped crystals and the favorable combination of excellent optical and mechanical properties of sapphire, complemented with high chemical durability make it an attractive structural material for high-technology applications [1]. Frequently it is the combination of two, or more, of its properties that make sapphire the only material available to solve complex engineering design problems.

In particular, the sapphire crystals of various cross-sections with capillary channels in their volume could be put to practical use. These crystals are applied as a basis for high-sensitivity cells for high pressure NMR spectroscopy [2], nozzles for water jet cutting and stripping by ultra high pressure, die for extrusion of polymers [3], highly efficient short-arc lamps for automotive lighting [4], miniaturized microwave gas-discharge plasma sources for optical emission spectroscopy [5], measurements of the thermo-physical properties of liquid metals, alloys, and graphite at high temperatures and pressures [6,7].

High-quality electron beam with 1 GeV energy by channeling a 40 TW peak-power laser pulse in a 3.3-cm-long gas-filled sapphire capillary discharge waveguide was received [8]. The GeV-class electron beams from these centimeter-scale structures offer unique applications. These electron accelerators are essential to synchrotron radiation facilities and free-electron lasers, and as modules for high-energy particle physics.

Also sapphire shaped crystals with capillary channels are used in oncology. Sapphire needle capillaries were developed as new laser waveguide introducer for delivery radiation in a tumour during photodynamic therapy and laser thermotherapy [9]. Smart scalpels with simultaneous incision and fluorescent diagnostics of a resected tissue immediately during surgical operation have been developed on base sapphire plates with capillary channels [10].

One of the main problems of growth of sapphire crystals with capillary channels in diameter less than 0.7 mm is the high probability of filling the channels with melt during the growth process because of

* Corresponding author: e-mail: ross@issp.ac.ru

increasing Laplace pressure in a meniscus melt with decreasing of radius of average curvature of meniscus surface (reducing of diameter of the channel) and increasing in meniscus height. The diameter of channel is significantly less than the sapphire capillary constant. In this case the growth of high quality crystal is possible only over a small range of pulling rates whilst maintaining a small height of meniscus. The state of the melting zone while pulling of these crystals is close to a condition of supercooling.

A new approach to the construction of the forming dies and to the system of automated control of the crystallization front state is required. Mathematical model of the behavior of the menisci profile curves at the various factors is required for the optimization of the automated control with use of the weight sensor by growth of the crystals with capillary channels [11]. The Investigation of the menisci profile curves and their behavior at the different growth parameters of the relatively large dimensions of the EFG (Stepanov) technique was made in [12]. Small menisci of the growth of the capillaries and fibers by the EFG (Stepanov) technique are investigated in the present paper.

2 Small internal circular menisci (capillaries)

Analysis of the profile curves of the small internal circular menisci in dependence on the various crystal radii was made for the external pressure H_d = -4 (in capillary constants). Crystal radius was changing in the range from 0.05 to 0.0488 with step -0.0002, that is for sapphire from 0.3 mm to 0.2928 mm with step -0.0012 mm. Radius of the working edges of the shaper was constant and equal to 0.0485 (in capillary constants), or 0.291 mm. Calculations was made for the positive boundary angles θ_d.

Profile curves represented in figure 1a differ from the curves shown in figure 8a represented in previous paper [12]. As it was described above, it is connected with fact that azimuthal curvature has positive sign in the case of internal meniscus and negative sign in the case of external meniscus.

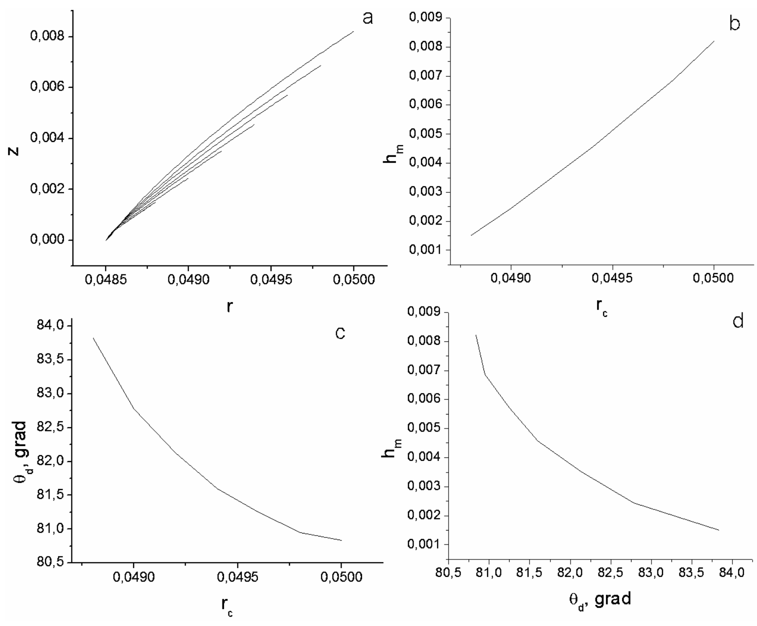

Fig. 1 a – profile curves of the small internal menisci (capillaries) at the various values of the crystal radius, b – dependence of the meniscus height on crystal radius, c – dependence of the angle θ_d on the crystal radius, d – dependence of the meniscus height on the angle θ_d. Case of the positive angles θ_d .

Besides, the weight of the small menisci is so small that the gravity factor is not significant, and it results in peculiarities in the shape of profile curves (Fig. 1a). Profile curves of the small internal menisci have convexity directed upwards because of their small weight and positivity and sufficiently large value of the azimuthal curvature. Physically it signs that by small weight of the internal menisci compressing force is sufficiently large, so it makes meniscus convex upwards. This is significant difference between small and "large" menisci – both external and internal. Second derivatives calculated for these profile curves demonstrate convexity directed upwards of these curves, because they are negative in the whole area everywhere. By the way convexity is especially significant near the edge of the shaper. Profile curves reach also small heights because of the small radii of capillaries – nearly 0.008 (in capillary constants), or 0.048 mm for sapphire (Fig. 1b). As in the case of “large” external and inner menisci their height decreases with approach of the crystal edge to the working edge of the shaper.

The relation of the maximal distance between crystal and shaper edges to the crystal radius was equivalent to one of the case of the corresponding “large” menisci. By this relation the meniscus height is very small, practically unreal in the pulling processes. For increasing of the meniscus height it is required to increase the distance between crystal and shaper edges. As calculations have shown, the “real” height of the “small” meniscus corresponds to approximately twice larger relation of the distance between crystal and shaper edges to the crystal radius in comparison with the case of the “large” menisci.

As it is shown in figure 1b, the heights of capillary menisci are very small. Behavior of this curve in this case differs from the curve behavior in the case of the “large” menisci. In the case of small menisci the curve is almost linear, with some convexity directed downwards. For the “large” menisci dependence of the menisci heights on the internal crystal radius is strongly non-linear, with convexity directed upwards.

The behavior of the dependence of the boundary angle θ_d on the crystal radius for the small menisci is similar to the corresponding dependence for the “large” menisci (Fig. 1c). But in the case of the small menisci absolute values of the angles are significantly larger than in the case of the “large” menisci. In the case of the small menisci larger angles θ_d are required, because the shape of small profile curves has convexity directed upwards differing by this from the shape of the “large” menisci.

In the case of the small menisci dependence of the meniscus height on the angle θ_d (Fig. 1d) is non-linear in difference of the case of the ”large” menisci. This is connected with presence of the convexity directed upwards of the profile curves of the small menisci. The smaller angle θ_d, the smaller this convexity of the profile curve near the working edges of the shaper (Fig. 1a), and larger height of the profile curve is required.

The crystal radius was 0.05 and shaper radius was 0.0485 in analysis of the profile curves for the various values of the external pressure. The value of the external pressure was changing from -10 to -2 with step 1. Calculations were executed for the positive angles θ_d.

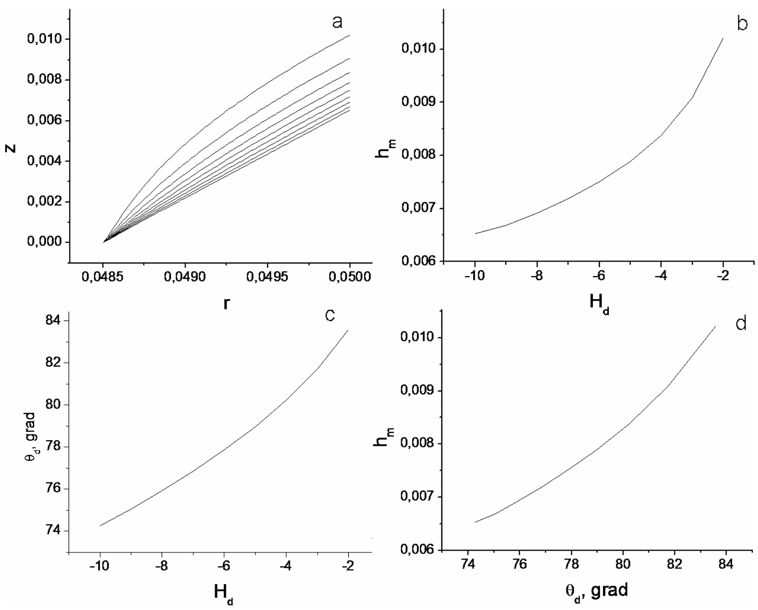

Fig. 2 a – profile curves of the small internal menisci (capillaries) for the various values of the external pressure, b – dependence of the meniscus height on the external pressure, c – dependence of the angle θ_d on the external pressure, d – dependence of the meniscus height on the angle θ_d at various values of the external pressure.

All in all, behavior of the profile curves of the small (capillary) menisci under the various values of the external pressure is similar to the behavior of the “large” menisci. As it is shown in figure 2, the meniscus height increases with decrease of the absolute value of the external pressure – faster with pressure more close to zero.

But at the relatively large absolute values of the external pressure the height of the small menisci changes weakly (Fig. 2b). This is connected with fact that with increase of the relatively large absolute value of the external pressure the force pressing meniscus to the shaper becomes relatively large, convexity directed upwards of the menisci decreases (decrease of the absolute value of the second derivative of the profile curve demonstrates this fact), influence of the vertical curvature decreases in comparison with the influence of the azimuthal curvature. Therefore, the shape of the small menisci at the large absolute values of the external pressure is close to the linear (Fig. 2a). As it is shown in figure 2b, relatively large change of the external pressure results in relatively weak change of the meniscus height in comparison with behavior of the meniscus height at the capillary radius change (Fig. 1b).

As in the case of the "large" menisci, for capillaries the angle θ_d providing fulfilling the condition of the growth angle constancy near the crystal edge grows with decrease of the absolute value of the external pressure, but in the smaller range and more linearly (Fig. 2c). The meniscus height also grows with increase of the absolute value of the angle θ_d (Fig 2d), but in the smaller ranges of the angles and heights – in comparison with case of the "large" menisci. For the constant values of the crystal and shaper radii and external pressure the calculations were made for the various values of the angle θ_d – without fulfilling of the condition of the growth angle constancy. For capillaries the real meniscus height and condition of the growth angle constancy can be achieved only at the large absolute values of the angle θ_d, as in the case of the "large" menisci.

Calculations with simultaneous changing the crystal and shaper radii were executed. Difference between these radii, i. e. the distance between the edges of the crystal and the shaper, was not changing. The value of the external pressure was equal to H_d = -4 (in capillary constants). Condition of the growth angle constancy near the crystal edge was taken into account. Dependences of the meniscus heights and the angle θ_d on the crystal and shaper radii were similar to the dependences of the "large" internal menisci by the reasons described in [12].

3 Small external circular menisci (fibers)

The analysis of the profile curves of the small external circular menisci in dependence on the various crystal radii was made for the external pressure H_d = -4 (in capillary constants). Crystal radius was changing in the range from 0.0485 to 0.0499 with step 0.0002, that is for sapphire – from 0.291 mm to 0.2994 mm with step 0.0012 mm. Radius of the working edges of the shaper was constant and equal to 0.05 (in capillary constants), or approximately 0.3 mm. Calculations was made for the negative boundary angles θ_d.

The shape of the profile curves of the small external menisci (fibers) (Fig. 3) principally differ from the shape of the small internal menisci (capillaries) (Fig. 1a). As it was described above, this can be explained by different signs of the azimuthal curvatures of the external and internal menisci.

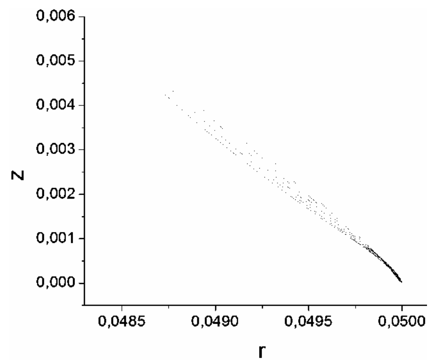

Fig. 3 Profile curves of the small external menisci (fibers) at the various values of the crystal radius. Case of negative angles θ_d .

At the same time the shape of the small external menisci (fibers) is similar to the shape of the "large" external menisci, because azimuthal curvatures have one sign for small and "large" external menisci. But deflection of the small external menisci in the middle area of the profile curve is significantly less than at "large" external menisci because of the small weights of the fiber menisci and sufficiently large factor of compressing force. Therefore, small external menisci are almost linear in their middle part of the profile curves. Second derivatives of the fiber menisci near the shaper edge are also negative. But the absolute values of these derivatives are significantly larger than in the case of the “large” menisci, i. e. convexity directed upwards of the small external menisci near the shaper edge is larger than in the case of the “large” external menisci.

The profile curves reach also small heights because of the small radii of crystal fiber: approximately, from 0.005 (in capillary constants), or 0.03 mm (for sapphire) to 0.001 (0.006 mm) in considerable range of crystal radii. It is necessary to increase the distance between crystal and shaper edges to obtain large heights of the fiber menisci. Meniscus height decreases with approach of the crystal edge to the shaper edge as in the case of the “large” menisci. The dependence of the height of the small outer menisci on crystal radius is similar to the analogous dependence of the “large” menisci with some difference, that this dependence (for fiber menisci) is more linear.

The dependence of the angle θ_d on the crystal radius for the small outer menisci is similar to the analogous dependence in the case of the “large” menisci. Difference is that the values of the angle θ_d for the small external menisci are significantly larger than these ones in the case of the “large” menisci. The dependence of the height of the small external menisci on the angle θ_d is similar to the analogous dependence of the “large” menisci.

The crystal radius was 0.0485 and shaper radius was 0.05 in analysis of the profile curves for the various values of the external pressure. The value of the external pressure was changing from -10 to 0 with step 1. Calculations were executed for the negative angles θ_d.

All in all, behavior of the profile curves of small fiber menisci at the various values of the external pressure is similar to the behavior of the “large” external menisci. Meniscus height increases with decrease of the absolute value of external pressure – faster with approach of pressure value to zero. But meniscus height changes weakly at the relatively large absolute values of the external pressure. Explanation of this fact is analogous to the explanation described above for the case of the small internal menisci (capillaries). The dependences of the angle θ_d on the external pressure and meniscus height on this angle for the small external menisci (fibers) are similar to the corresponding dependences for the case of small internal menisci (capillaries) (Fig. 2 c,d).

For the constant values of the crystal and shaper radii and external pressure the calculations were made for the various values of the angle θ_d – without fulfilling of the condition of the growth angle constancy. For the fibers the real meniscus height and condition of the growth angle constancy can be achieved only at the large absolute values of the angle θ_d, as in the case of the “large” menisci. Calculations with simultaneous changing the crystal and shaper radii were executed. Difference between these radii, i. e. the distance between the edges of the crystal and the shaper, was not changing. The value of the external pressure was equal to H_d = -4 (in capillary constants). Condition of the growth angle constancy near the crystal edge was taken into account. Dependences of the meniscus heights and the angle θ_d on the crystal and shaper radii were similar to the dependences of the “large” menisci.

4 Use of calculations of the menisci profile curves in the automated process of the growth of the sapphire capillaries and fibers

An automated control system for the growth of sapphire shaped crystals with the capillary channels in their volume and sapphire fibers with use of the weight sensor also (as for relatively large crystals) requires the use of the program observation expression. The data of this expression at each period of time are being compared with real signal of the weight sensor. Deviation of the real signal from its program one forms then a control signal of the PID (proportional-integral-differential) regulator. Oscillations of this deviation may have different characters. It can be relatively strong (under overcooling) and relatively small (under overheating) [11].

In the case of the growth of the capillaries and fibers (also in the multi – run process) it is necessary to maintain the amplitude of these oscillation in a preset sufficiently narrow range. The maintenance of this narrow range of these oscillations amplitude requires the use of the second control loop which tunes the first PID-loop by the appropriate changing the program radius of the crystal or radii of the crystals. An example of the automated growth of the capillary channel in the body of the bulk crystal is shown in figure 4. Figure 5 demonstrates sapphire fibers of 250 – 300 μm in diameter which have been grown by multi – run process (up to 100 crystals per one process).

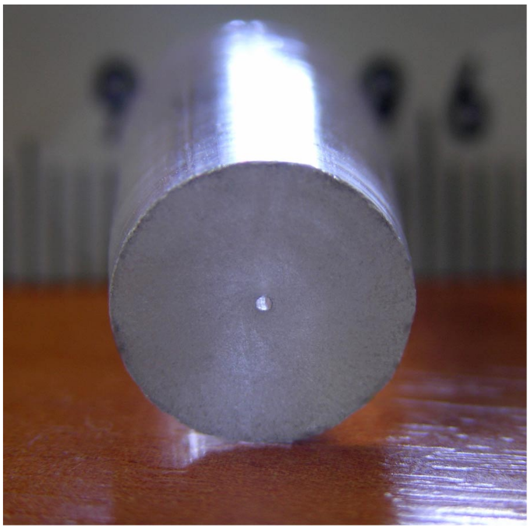

Fig. 4 Sapphire rod of 10 mm in external diameter with capillary channel of 450 μm. (Online color at www.crt-journal.org)

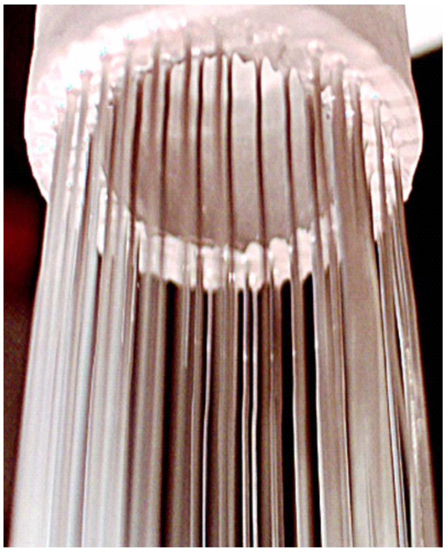

Fig. 5 Sapphire fibers of 250-300 μm in diameter grown by multi – run crystal growth process. (Online color at www.crt-journal.org)

4 Conclusions

Differences in the shape of the small and "large" menisci were found. Difference of the shape of the small menisci from the shape of the "large" menisci should be explained by the small weight of the small menisci. It is shown that profile curves of the small menisci providing the stationary growth of the capillary or fiber have significantly large absolute values of the angle θ_d than corresponding "large" menisci.

Besides, the differences in the shape of the small internal (capillaries) and small external (fibers) menisci were found. These differences should be explained by a small weight of the "small" menisci and different signs of the azimuthal curvatures of internal and external small menisci and large compressing force in the "small" menisci.

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