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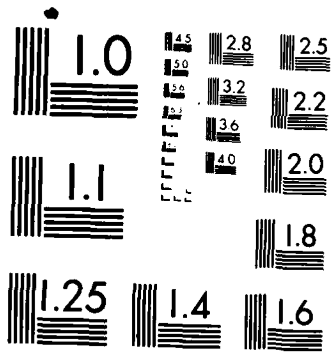

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FOREIGN TECHNOLOGY DIVISION

THE EFG GROWTH AND APPLICATION OF SMALL DIAMETER SAPPHIRE CRYSTALS WITH ROD AND TUBULAR SHAPES

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Zhang Shou-qing, Tang Lian-an, et al.

EDITED TRANSLATION

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The EFG Growth and Application of Small Diameter Sapphire Crystals with Rod and Tubular Shapes

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Abstract

Small diameter sapphire crystals with rod or tubular shapes have been grown by the EFG (Edge defined, Film-fed Growth) method. The effect of the growth technique on the perfection of a crystal has also been investigated , leading the quality of crystal grown by this method to be comparable to that by the flame fusion method. The tubular crystals have been used in making special light sources, and the growth technique of rod sapphire crystals has been applied in a factory for producing jewel bearings. The EFG method greatly simplifies the technique of processing crystals after growth , and the method of multi-rod EFG growth has also been realized. Therefore the cost of production has been lowered, achieving a better economical efficiency.

1. Introduction

Normally the crystals grown from the melt assume rod or lump shape. For application they have to be further processed to have the desired shapes. To do so, the special machining techniques of cutting and polishing are needed. Thus not only a great deal of valuable crystal material is wasted, but also the cost of labour and facilities is raised. In particular a sapphire crystal of high hardness is very difficult to work with and the material consummed in the process is severe. Therefore, it was begun early to search for a new method of crystal growth, hoping to directly grow crystals of the desired shapes. H.E.Labelle, Jr.et al[1] in 1971 reported for the first time the invention of the EFG method.

This method possesses many peculiar advantages: It can grow crystals of various special cross sections, saving the crystal material and simplifying the technological process after growth. It can realize multi-rod EFG growth, growing several crystals within a crucible at the same time. It has a high growth rate and good stability.

In the last few years, this method has made a remarkable progress in growing the silicon plates for solar batteries, the sapphire substrates used for making IC chips epitaxially, and thin plates of $LiNbO_3$ crystals used as acoustic surface wave devices and piezoelectric osillators. Some of them have been applied in practice.

In 1976 we began to investigate the growth of sapphire crystals of complex shapes by the EFG method. Of them, the work with growth of ribbon shaped crystals has been reported separately[2]. The crystal growth apparatus used for tubular and rod sapphire is the same as the one described in (2). Here we will emphasize the growth technique of tubular and rod sapphire, and the relationship between the growth parameters and the crystal quality, the mechanic, optic and electric features of the crystal, the structural perfection of crystal and the application of tubular and rod crystals to making special light sources and jewel bearings.

2. The Growth of Tubular Crystals

The single sapphire crystal possesses various advantages such as high melting point, large transparency, large tensile strength, chemical stability and corrosion-resistance against alkali-metal vapor at high temperatures. This is an excellent material for making the tubes of speciallight sources.

But it is very difficult and uneconomical to make tubes out of the crystal grown by the Czochralski or flame fusion methods. Thus it would greatly save the amount of work if one could directly grow tubular single crystals by the EFG method, providing a big advantage for making special light sources.

The die employed for growing tubular sapphire is shown in Fig.1 It consists of two Mo cylindric tubes of different diameters, which are concentrically separated by a gap ~ 0.8 mm as the capillary orifice to supply liquid crystal. According to the demands from application we have grown tubular crystals of 1 mm thick, inner diameter 1 ~ 10 mm and 150 ~ 200 mm long (Fig.2). Since the growth orientation influences the outer shape of the crystal to some extent, we chose [0001] axis as the growth direction in order to improve the roundness of the tubular crystal. While growing tubular crystals of small diameter, it requires a high accuracy of temperature control and a stability of growth mechanism. In particular during the crystal pulling and growth processes, one should take care to not allow any melt to flow into the small hole in the center of the die. Otherwise, a tubular crystal of uniform diameter will not be obtained.

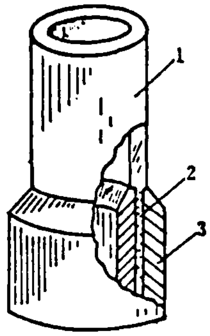

Fig.1 Die for the growth of tubular crystal

1--Tubular crystal, 2--Melt, 3--Die

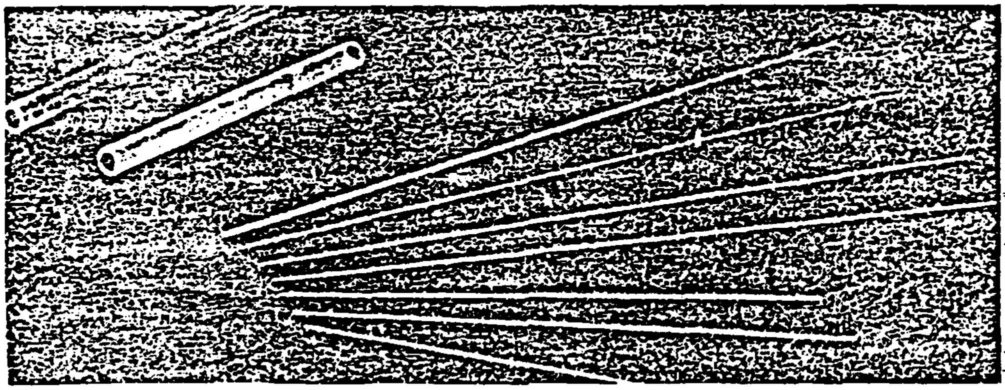

Fig.2 Rod and tubular sapphire crystal grown by EFG method 0.3x

Under observation with a microscope of high magnification power many microvoids of diameter ~ 10 μm can be seen in the early grown tubular crystals(Fig.3).

The density of sapphire crystal is 3.95 g/cm^3 and the density of its melt is only 3.05 g/cm^3. The volume shrinks by about 20% when the melt condenses into the crystal. In the case of growing sapphire crystal by the EFG method, the pulling rate is quite large. Therefore it is easy to leave many voids in the crystal due to the drastic shrinking of the melt's volume. The larger the pulling rate is, the more voids in the crystal there will be. In the worst case a cellular structure could be formed (Fig.4). Reducing the growth rate (usually ≈ 45 mm/h) could eliminate the voids located in the central region of the tubular wall, but not in the vicinity of inner and outer crystal surfaces. According to the characteristic distribution of voids, the position where voids appear corresponds to the die top(Fig.5). This is because on the liquid-solid interface corresponding to the die top the latent heat of crystallization is not easy to dissipate. Furthermore, the interface assumes a concave shape where it is easy to accumulate any impurity-like void. If the temperature of the die top were too high and the pulling rate were too large, the formation of voids would be more severe. Thus many small voids might connect to one another resulting in thin hollow tubes in the crystal.

Fig.3 Voids in EFG sapphire 50x

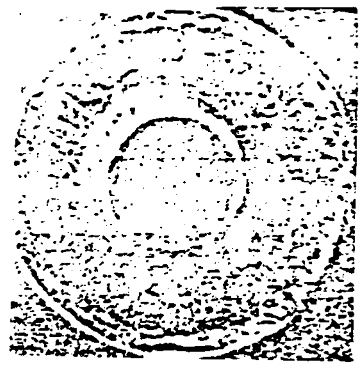

Fig.4 Cellular structure of the crystal 15x

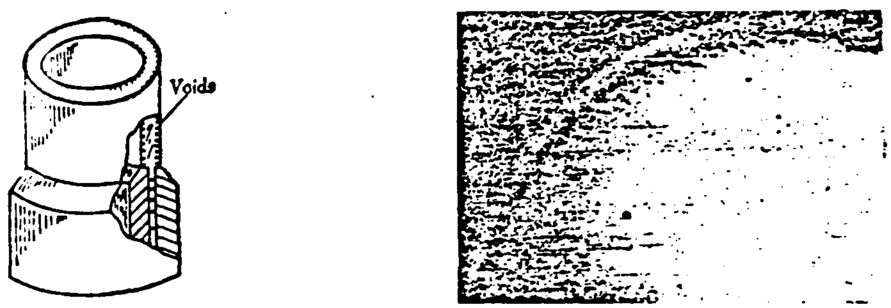

Fig.5 Correspondence of the void distribution with the die tops

A measurement of the dislocation density was conducted for a (0001) plan slice of tubular crystal. After mechanically polishing and etching in boiling phosphoric acid or molten KOH, the slice of crystal presented some regular triangularly etched pits.

The dislocation density is large in the vicinity of the tubular wall; some of them form grain boundaries(Fig.6). This might be related to the fact that there are many voids and there exists larger thermal stress in the vicinity of wall.

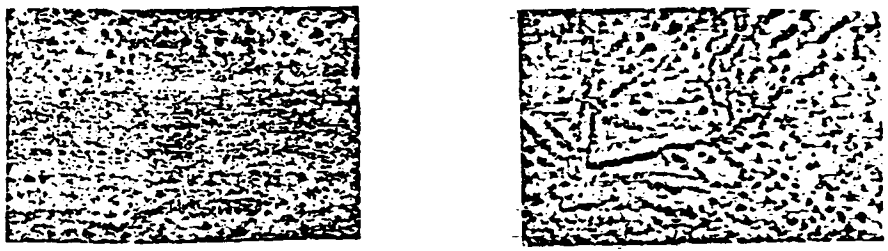

Fig.6 Dislocation in EFG sapphire 500x

For the tubular crystal grown by the EFG method, its dislocation density on the (0001) plane is usually about $3x10^5/cm^2$. As comparison, for the sapphire grown by flame fusion method the dislocation density on the corresponding plane is normally about $2x10^6/cm^2$.

Tubular sapphire crystals have been used for making Na lamps, CO_2 lasers and so on. Using a small CO_2 laser made from tubular sapphire of inner diameter 1 mm and ~100 mm long, one watt of CW output power can be obtained.

3. The Growth of Rod Crystals

A single sapphire crystal is characterized by excellent thermal conduction (its thermal conductivity is 0.06 cal/cm.s.$^\circ$C), good electric insulation and small dielectric loss, being one of the perfect materials for making the post of travelling wave tubes.

In the literature, it was reported that the EFG method was used for growing a sapphire filament of diameter 0.1 mm, which was not a perfect crystal and was dendritic. Therefore, it can only be used as a strengthening material.

In order to meet the demands for making travelling wave tubes, we employed for the first time in 1977 a die of three capillary orifices, which allows one to grow three sapphire rods of diameter 1.5 ~ 2.0 mm at the same time. The rod sapphire is straight and 250 mm long, and has a uniform diameter. Based on this experience, another die of six capillary orifices, which is easer for machining, was later designed(Fig.7).

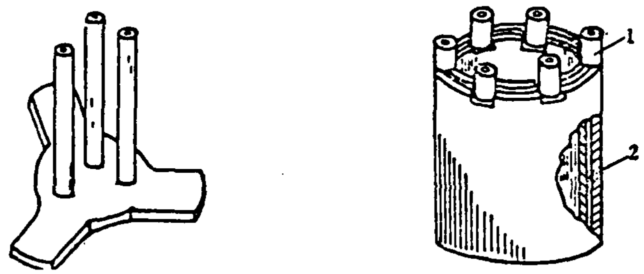

Fig.7 Schematic diagram of the dies for multi-rod growth
1--Die tops, 2--Circular melt-feeding slot

Using the rod crystal grown along [0001] direction, one could sometimes observe that its outer profile assumes a hexagonal shape. But the crystal grown along a 60° orientation often exhibits a flat chopped plane. This is because the sapphire belongs to the monoclinic system; its (0001) and { 11$\bar{2}$0 } planes are the planes of low indices. Therefore, the density of the network is so large that they easily emerge when growing slowly. The emergence of crystal planes does not affect the intrinsic quality of the crystal, but will give some trouble with processing the crystal afterwards. The experiment shows that improving the thermal insulation of the die top and adequately raising its temperature is good for preventing the flat chopped plane from appearing.

The defects often presented in the rod crystals are microvoids and subboundaries. Particularly for the rod crystal grown along [0001] axis, if the growth parameters were not properly set it would be very common to leave a large distortion stress behind in the crystal, resulting drastically in many grain boundaries. This can be the major reason that the brittleness increases and the growing orientation of crystal changes gradually. In Fig.8, a picture taken by a polarized microscope shows the crystal grains obtained from cutting a rod crystal of large brittleness. There were many grain boundaries in the rod crystal. The presence of this phenomenon could also be interpreted by what we learned about the relationship between the growing orientation of ribbon crystal and the formation of grain boundaries[2].

Fig.8 Grain boundaries in a rod crystal 15x

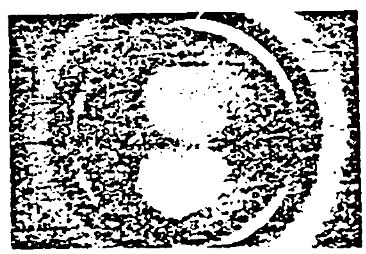

Fig.9 Isogyres of a slice cut from a rod crystal 15x

There has been success in making jewel bearings out of the rod sapphire crystal grown by the EFG method. It is required to grow the crystal along the optical axis in order to meet the demands on the roundness of jewel bearings. As mentioned previously, this is obviously a disadvantageous orientation from the point of view of growing crystals. In order to prevent creating too many grain boundaries, one should first pay attention to choose a seed crystal of high quality, increase the accuracy of temperature control and choose an appropriate temperature profile. On the other hand, one could allow the pulling direction slightly to deviate from the optical axis within certain degrees. Fig.9 is a picture of isogyres taken under an orthogonal polarized light for a slice cut from a rod crystal grown by the EFG method The isogyres are basically regular and distortion free ,which indicates pretty good structural perfection of the crystal. Since the growing direction was slightly deviated from the optical axis, the center of the black cross in the interference pattern is also slightly shifted to a side.

In the following table, the characteristic comparisons between the rod sapphire grown by the EFG method and the one by the flame fusion method are listed:

Item	Rod grown by EFG method	Rod grown by flame fusion method
Microhardness(H_V)(kg/mm^2)	2036	2038
Compressive strength(kg/cm^2)	13300~19800	3000~12700*
Bending strength(kg/cm^2)	4900~5100	2700~3400
Dielectric constant	9.5	9.4
Dielectric loss	5×10^{-3}	3×10^{-3}*
Dislocation density(0001)(cm^{-2})	3×10^{5}	2×10^{4}

• Quoted from references[3、4]

Practice shows that the rod sapphire grown by the EFG method for making jewel bearings possesses the following advantages:

(1) Because the diameter of the rod sapphire is very close to the size of the final products, it greatly simplifies the machining techniques, avoiding or simplifing the techniques such as cutting slices, cutting rods and rounding for the crystals grown by flame fusion method.

(2) Since the brittleness has been improved, the rate of product qualification has been raised from 60%-70% for the flame fusion method to [illegible]0%

(3) The utilization ratio of crystal has been remarkably increased. According to the rough estimate by the users, it could save about 70% of the crystal material if the rod crystals grown by the EFG method are employed in comparison to the boule grown by the flame fusion method.

(4) In the machining process, the consumption of electricity and other auxiliary material has also been drastically reduced.

4. Summary

The thin tubular and rod single sapphire has been grown by the EFG method. The influence of growth parameters on the perfection of the crystal has been investigated. The crystal quality given by the EFG method has achieved the comparable level given by the flame fusion method.

The tubular crystal has been used for making special light sources . A small CO_2 laser has been made succesfully.

The growth techniques for thin tubular crystals have been applied to manufacturing jewel bearings. The growth by the EFG method can simplify processing techniques of cyrstals after growing and can also save raw material, auxiliary material, and reduce energy consumption. Therefore the cost of producing bearings is substantially lowered, obtaining a remarkable economic effect.

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Abstract

Small diameter sapphire crystals with rod or tubular shapes have been grown by the EFG (Edge defined, Film-fed Growth) method. The effect of growth parameters on the perfection of the crystal has also been investigated in detail. The quality of crystals grown by this method is found to be comparable to that of flame fusion sapphire. Tubular crystals are used in making special light sources, e.g. CO_2 lasers; while rod sapphire crystals are used for producing lower cost jewel bearings.

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