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Growth of 1 inch $LuVO_4$ single crystals by the edge-defined film-fed-growth (EFG) technique

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Abstract In spite of their superior optical and laser properties rare-earth orthovanadate single crystals have not been adopted yet into extensive industrial applications because of crystal growth difficulties. The edge-defined film-fed-growth (EFG) technique was applied successfully for the production of such crystals. At first time 1 inch $LuVO_4$ single crystals were grown by the EFG technique using newly developed die construction of high porous iridium with the application of automatic diameter control system.

Key words Rare-earth orthovanadate, Melt, Shaped crystal growth, EFG technique

1. Introduction

At last 10~15 years the amount of reports about growth and investigation of rare-earth vanadates (mainly YVO_4 and $GdVO_4$) has been increased dramatically. It was shown that such crystals are rather perspective material for the electronics and optical applications. These crystals demonstrate some wonderful properties as large birefringence, high chemical stability and thermal conductivity, wide transparency range (400~5000 nm) [1]. Simplicity and wide range of isomorphic substitution by other rare-earth ions (like Yb and Nd) allow to believe that rare-earth vanadates would be used as high efficient laser material for micro lasers with LD pumping.

Till now there were published some articles about successful growth of rate-earth vanadates by the Czochralski technique rather small in length and unstable in shape. Shaped methods possess some important advantages in comparison with Czochralski: possibility to produce crystals with desired shape and dimensions close to the size of finished optical elements, absence of growth defects related with crystal rotation and thermal convection of the melt. Earlier, our experiments showed that EFG technique is the perspective way to produce different rare-earth vanadate single crystals with size and quality acceptable for the industrial applications [2, 3]. Moreover, this technique gives a possibility for the simultaneous production of few vanadate single crystals as well as crystal items with desired section [4]. However, in previous experiments crystal diameter was limited by 10 mm. It was estimated that during the EFG growth of crystal with the diameter more than 10 mm there are serious difficulties with the melt transport to the crystallization area and with stability of crystal shape.

In this article the possibility of growing the vanadate single crystals 1 inch in diameter using the EFG technique was investigated. $LuVO_4$ was selected as the material for such investigation. Earlier, this material was grown by the Czochralski and growth behavior similar with other vanadates was estimated [5]. At once, the melting point of that member of rare-earth vanadate family is lower in comparison with more famous YVO_4 and $GdVO_4$.

2. Experimental Procedure

It was estimated that traditional construction of Ir die couldn't provide the stable transportation of melt to the top of the die if the size of a die was over 10 mm. Moreover, there is the strong local overheating at the area of solid-liquid interface over the exit of capillary channels. This overheating is preclusive for the creation of flat solid-liquid interface. Due to these reasons the original construction of Ir die was developed for the experiments with EFG growth of large size vanadate crystals. The schematic diagram of hot zone with such die is shown on Fig. 1. The main distinctive feature of developed die is the application of high porous iridium. In that case the capillary channels for melt transportation were formed easily by pores in the volume of a die.

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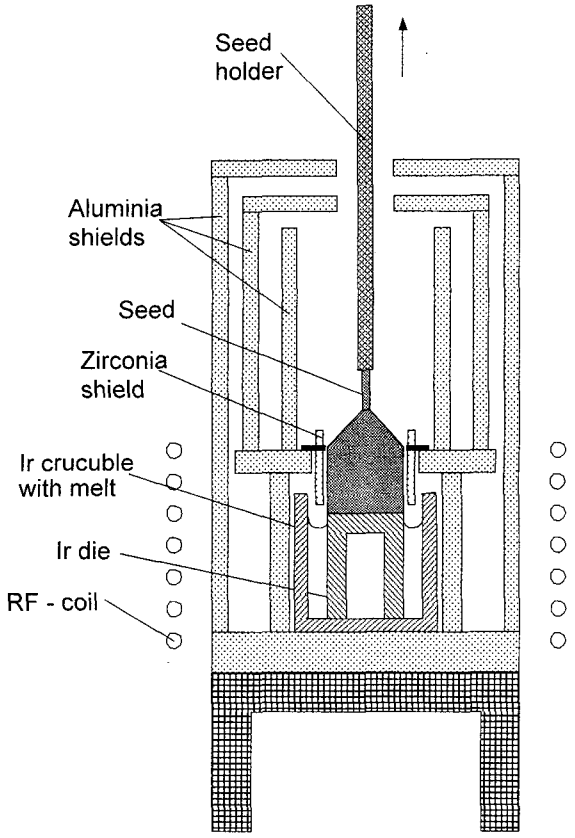

Fig. 1. Schematic diagram of the hot zone.

Also, very equable melt distribution on the surface of the die was reached. The detailed description of developed die will be published elsewhere.

The Lu_2O_3 and V_2O_5 powders of 99.99 % purity were used as raw materials. The stoichiometric composition was used as the initial charge. Before growth, the mixture of oxides was synthesized in a platinum vessel in air during 24 h at 1400°C. $LuVO_4$ crystals were grown using YVO_4 seed along the c-axis. Pulling rates in the range of 0.5~1.5 mm/h were used. The growth atmosphere was a mixture of N_2 + 1 vol% O_2. The temperature at the surface of Ir die was measured by means of an optical pyrometer. At the seeding conditions the temperature was about 1750°C.

3. Results and Discussion

Lutetium vanadate single crystals with diameter 25 mm and length up to 25 mm were successfully grown using developed technique. As-grown crystal is shown on Fig. 2. Unlike the Czochralski grown 1 inch $LuVO_4$ single crystal, EFG-grown boule was stable in shape, without strong crystal diameter fluctuations.

Fig. 2. As-grown 1 inch $LuVO_4$ single crystal.

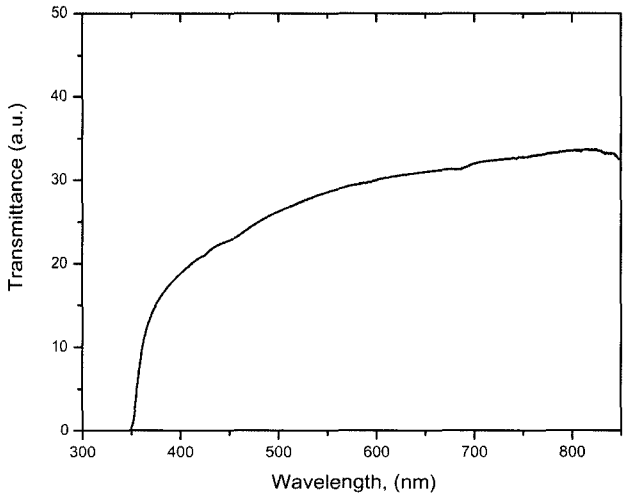

Fig. 3. Transmission spectrum of $LuVO_4$ crystal after annealing.

All crystals were transparent and dark-yellow in color due to presence of oxygen vacancies in the volume. The annealing at the air during 24 h at 1300°C decreased the intensity of the yellow color. The transmission of the crystal after annealing is shown on Fig. 3.

Usually, the EFG growth process is considered as self-stabilized without necessity of the automatic control. It was true for the EFG growth of smaller vanadate crystals. In that case it was possible to keep power constant during long time or to change it slowly with the constant rate. In opposite, at the EFG growth of

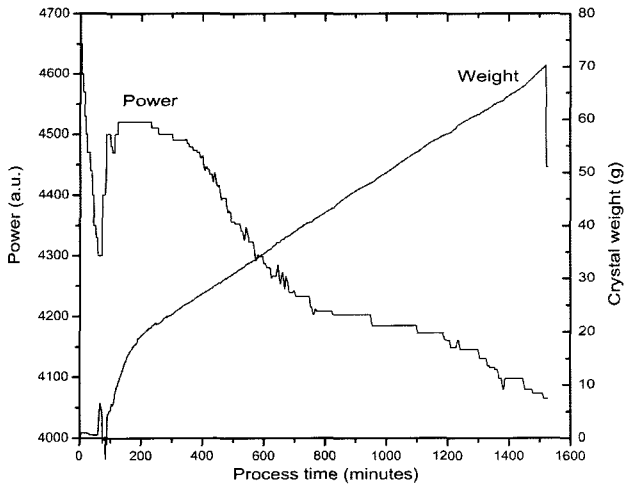

Fig. 4. Power control curve and weight sensor signal versus real time.

large size vanadate crystals the permanent and nonlinear correction of RF-generator power was needed for stable shape of the boule during entire process. Fortunately, the mass growth rate also is rather high, and it was possible to use automatic diameter control system with weight sensor for this purpose. Earlier, this system was applied successfully during Czochralski growth of vanadates with similar size [6]. Weight of growing crystal and the power of RF-generator estimated by automatic control system are shown on Fig. 4. At the application of this control system it was necessary to appreciate the constituent of surface tension force to the signal of the weight sensor. It was possible to observe that this force is rather significant on the principle that the strong weight drop after the sharp crystal separation from the die was observed (right edge of weight plot on Fig. 4). So, the contribution of surface tension was approximately 20 g at the total weight signal of 70 g.

Crystals grown during described experiments had few small cracks (mainly at the area of transition from the stage of crystal diameter expansion into the straight growth). The generation of low-angle boundaries mainly in the narrow peripheral area of the crystal was a main quality problem of all EFG grown vanadate crystals. The evident way to avoid it by maintaining of relatively high meniscus height is rather limited due to inevitable lose meniscus anchoring and shape. In contradistinction with the circular shape, small rectangular vanadate crystals were much more stable to the meniscus height increasing without lose of shape [3]. It gave a possibility to avoid the formation of defective area and to improve the quality of EFG grown vanadate crystals. It is possible to expect that it will be effective also for larger size crystals grown using the developed modification of the EFG technique.

4. Summary

It was found that the EFG technique is the effective way for the production of rare-earth vanadate single crystals. At first time 1 inch $LuVO_4$ single crystals were grown by the EFG technique using newly developed die construction of high porous iridium. Crystals up to 25 mm in length with the stable shape were grown successfully. The application of automatic control system with the use of crystal weighting was very effective for the growth of large vanadate crystals by the EFG technique, and it allowed to produce the crystals with the stable shape up to few centimeters in length.

References

[1] R.A. Fields, M. Birnbaum and C.L. Fincher, "Highly efficient Nd : YVO_4 diode laser end-pumped laser", Appl. Prys. Lett. 51 (1987) 1885.

[2] B.M. Epelbaum, K. Shimamura, K. Inaba, S. Uda, V.V. Kochurikhin, H. Mashida, Y. Terada and T. Fukuda, "Edge-defined film-fed (EFG) growth of rare-earth orthovanadates $REVO_4$ (RE=Y, Gd): approaches to attain high-quality shaped growth", Cryst. Res. Technol. 34 (1999) 301.

[3] V.V. Kochurikhin, M.A. Ivanov, W.S. Yang, S.J. Suh and D.H. Yoon, "Development of edge-defned .film-fed growth for the production of YVO_4 single crystals with various shapes", J. Cryst. Growth 229 (2001) 179.

[4] V.V. Kochurikhin, A.E. Borisova, M.A. Ivanov, V.E. Shukshin, S.N. Ushakov, S.J. Suh and D.H. Yoon, "Edge-defined film-fed growth of Yb : YVO_4 single crystals: approaches to produce a few crystals simultaneously", J. Ceram. Proc. Res. 4 (2003) 109.

[5] Y. Terada, K. Shimamura, V.V. Kochurikhin, L.V. Barashov, M.A. Ivanov and T. Fukuda, "Growth and optical properties of $ErVO_4$ and $LuVO_4$ single crystals", J. Cryst. Growth 167 (1996) 369.

[6] V.V. Kochurikhin, Y. Terada, B.M. Epelbaum, K. Shimamura and T. Fukuda, "Enhancement of the automatic diameter control system for the czochralski growth of Nd : $GdVO_4$ crystals", Cryst. Res. Technol. 33 (1998) 793.