I6 15:10 – 15:50

Oxide single crystal growth by the Micro-Pulling-Down method

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1. Introduction

Fiber crystals have become the subject of intense study in recent years because of their remarkable characteristics [1][2][3][4][5]. Single crystalline fibers are well suited to room temperature micro lasers and nonlinear optical interactions, the efficiency of which can be greatly enhanced by the long interaction lengths and tight beam confinement. Numerous oxide fiber crystals are now under investigation for application in optoelectronics as second and higher harmonic generators, especially for conversion from red LD wavelengths into green, blue and violet regions [6][7]. Moreover, sapphire fibers show a further extraordinary characteristic - an ultrahigh tensile yield strength more than 1 GPa at 300 K at diameters of 100 - 200 μm [8]. This is due to their crystalline perfection and small dimension, which minimizes the occurrence of the defects that are responsible for the low strength of materials in bulk form. Such a property makes fibers interesting as reinforcing agents in structural components. In order to increase their mechanical stability and flexibility in high-temperature applications, several investigations have concentrated on the fiber growth of various alumina-based alloys [5][9][10][11]. In this talk, the novel fiber growth method known as Micro-Pulling-Down (μ-PD) method is introduced and several results described.

2. Micro-Pulling-Down Method

A schematic illustration of the μ-PD method used for the growth of oxide crystals with high melting point (> 1500°C) described below is given in Fig.1. The radio-frequency (RF) heating system employed in the growth process was specially designed for the fabrication of fiber crystals at high temperature (above 1800°C). The crucible was placed on an alumina pedestal in a vertical quartz tube and was heated using an RF generator. The calibrated orifices made in the crucible bottom were 0.2-0.6 mm in diameter. The crucible temperature was controlled by the power to the RF coil, which was about 90 mm long with seven windings. The crucible was charged to about 5-10 vol.% with regard to the melt. High density and high purity (99.7%) alumina ceramic was used to surround the crucible for thermal insulation. The fibers were grown in Ar atmosphere (gas flow 2 l/min) to avoid oxidation of the crucible. Visual observation of the meniscus region, solid-liquid interface and the growing crystal was made by CCD camera and monitor.

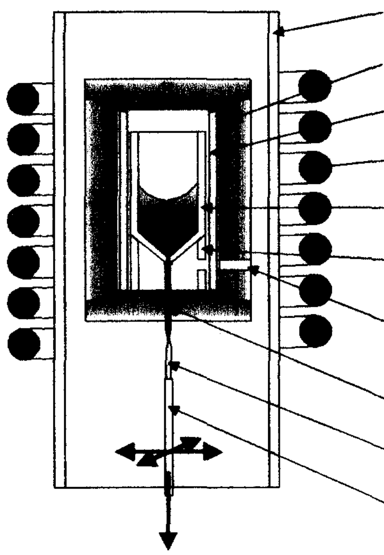

Fig.1. Schematic of the μ-PD method

3. Results and discussions

RE-doped YAG single crystal fiber

There are certain problems in the usual rod-type Nd:YAG laser. For example, because of limitations of the cooling system, the power and the quality of the mode are also limited. Furthermore, it is too difficult to produce a flexible laser system. To overcome these problems, we tried growing YAG single crystal fibers.

<111> orientation of the YAG single crystal fibers grown was confirmed by X-ray measurements. The crystal composition was studied by electron-probe microanalysis (EPMA). About 0.5 atm% of Y^{3+} host

Fig.2 Nd, Yb-doped YAG single crystal fibers

cations was substituted with Nd^{3+}. The crystals exceeded 500mm in length and were 0.7-2.0mm in diameter. A view of the Nd, Yb-doped YAG crystal fibers is given in Fig.2.

$RE_3Al_5O_{12}/Al_2O_3$ eutectic fibers

Recently, it has been found that ceramic matrix composites reinforced with sapphire phase have excellent high-temperature properties. Such high-performance materials are of interest for use in advanced aerospace structures, high efficiency gas generators, and other high-temperature applications. A view of the $RE_3Al_5O_{12}/Al_2O_3$ eutectic fibers is given in Fig.3. $Tm_3Al_5O_{12}/Al_2O_3$ showed a tensile strength of 620MPa at 1500°C in vacuum. This is the highest strength at this temperature to date.

Fig.3. $RE_3Al_5O_{12}/Al_2O_3$ eutectic fibers grown by μ-PD method

$K_3Li_2Nb_5O_{15}$ single crystal fiber

KLN has a space group of P4bm with a high second-harmonic coefficient. As KLN resists optical damage, it can be considered a potential material for devices. However, it is very difficult to obtain high enough optical quality using conventional methods like the Czochralski (Cz) or Top-Seeded Solution Growth (TSSG) methods. But in the case of the μ-PD method, because of the high growth rate, the effective segregation coefficient is ~1. Thus, we can obtain crystal with a homogeneous distribution of the cations. Fig.4 shows a photo of blue (450nm) SHG generation in a KLN single crystal fiber.

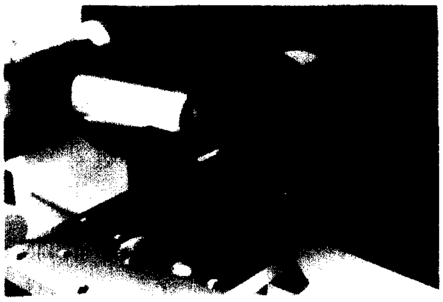

Fig.4. Blue SHG generation in a KLN single crystal fiber

$(Tb,RE)_3Al_5O_{12}$ single crystal

$Tb_3Al_5O_{12}$ (TAG) is a well-known optical material with high Verdet constant, which has technological importance for use as a Faraday effect optical isolator. But it was shown that the TAG compound melts incongruently with a primary crystallization of $TbAlO_3$ from a melt with the stoichiometry of TAG. In order to stabilize the garnet phase, we tried to dope Tm, Yb and Lu into the Tb site. This substitution was effective and we could grow those single crystals as shown in Fig.5.

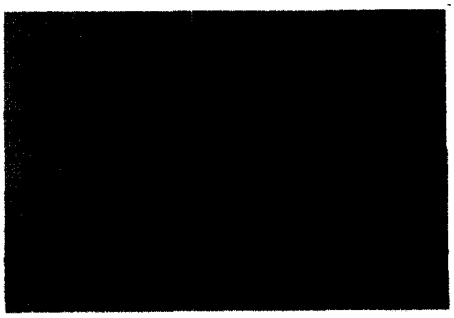

Fig.5. Photo of $(Tb,Lu)_3Al_5O_{12}$ single crystal grown by μ-PD method

4. Conclusions

The novel μ-PD fiber growth method was introduced, along with results such as RE-doped YAG for miniature lasers, $RE_3Al_5O_{12}/Al_2O_3$ eutectic fibers for high temperature structural materials, KLN as a promising non-linear optical material, and $(Tb,RE)_3Al_5O_{12}$ single crystal for application as Faraday isolator for optical communication systems.

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