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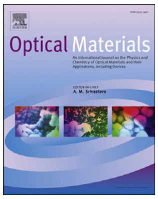

Single crystals fiber technology design. Where we are today?

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ABSTRACT

Because of its performed mechanical, physical and optical properties, today single crystal fiber shape (SCFS) can be used for a large wide of application. As a function of the needed, it can be used as active or passive element in the component. The potential of single crystal fiber is extremely high. Intensive research has been devoted to design and optimize the technology process, but the increased technological requirements require continuous improvements at all stages of the fiber design: Fiber processing (growth fiber machine), starting raw materials, crucibles, growth direction, thermal gradient, gas atmosphere, fibers polishing, dopants segregation, packaging … This is demonstrated by the successful fiber pulling from the melt of more than 1 m length of sapphire, YAG and LuAG with performed properties.

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1. Introduction

Single crystal fiber shape (SCFS) is very attractive for different technology application. It's performed properties allowed the obtainment of excellent results in different domains such optics, lasers, scintillation and mechanics. Initially, it don't exist a real equipment dedicated to pulling SCFC from the melt. In 1917, J.Czochralski [1] publish a paper on pulling up the metallic fiber from the melt and was been used for fundamental interest, since just the real structure defines its physical and chemical properties. Another experimental procedure was used to grow metallic shaped crystal from a hole around 1920 [2–4]. In 1963, A.V Stepanov [5,6], has presented the concept of shaping crystal from the melt using shaper.

But during this period the fundamental problems of single crystal fibers pulling doesn't discussed and the technological nature related to the elaboration of certain technically valuable fiber doesn't developed. The main interest was focused on the crystallography and special consideration was given to anisotropy of physical and chemical properties. Professor P. Rudolph in Ref. [7] mention that the development of optical waveguiding in 1970 has activated the research on SCFS pulling for different optical applications. Unfortunately, there aren't a real equipment designed to grow reproducible fibers with high quality. In addition during this period, the application of SCFS is limited, only few demonstration of research laboratory shown the great advantage of SCFS configuration. By using single crystal fiber for example as lasing element, it is possible to remove the heat because of temperature controlling around the fiber [7]. Initially, fiber pulling equipment was designed empirically but as the needed has grown in importance especially after the feasibility of some specific application such waveguide and laser and as the size of the fiber becomes a key factor for the application, consideration has been given to optimizing the technological SCFS processing. This has led to develop an industrial research into the fundamental of the processing together with a simulation of elements allowing the improvement of the machine stability such translation system (motor stability) and heat and mass transport. These field are successfully developed nowadays, therefore it is required to develop fiber having high performance. So, after this development some laboratory in the world have done a great effort to improve SCFS performance and to develop industrial application using SCFS as active source. Around 1980, different research program in connection to SCFS start to see the day. In USA, a great effort has been done by the teams of Prof R.S.Feigelson and Prof M.M.Fejer to design SCFS using Laser Heated Pedestal growth (LHPG) technique to pull single crystal fibers [8–14]. In 1993, Prof T. Fukuda start a big research program related to fiber crystal growth using micro-pulling down (μ-PD) technique [15–26] and from 1995, in France, Prof G. Boulon, from Lyon University, LPCML laboratory, today ILM laboratory (UMR 5306 CNRS) started a national single crystal fibers research program using LHPG technique [27–32]. It is very important to know that from 1995, Prof Fukuda Laboratory in Institute of Material Research (IMR), Tohoku university, Sendai, Japan was been the world research

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center of single crystal fiber topic. A lot of researchers from all the world visit Fukuda laboratory and they were involved in SCFS development. The different connection between the researchers in Fukuda laboratory has given a new opportunity to increase research program on fiber design. So during this period, others laboratories in Brazil, Taiwan, South Korea, Italy, Germany, Poland, Czech Republic were involved in SCFS program. Different oxide single crystals fibers materials were study and this research program is developed to search for new crystals having high sensitivity, good crystallinity and to study the spectroscopy, optics, laser, scintillation and mechanical properties. Among the materials of particular importance are oxides, garnets, silicates, eutectic as well as rare-earth alumintaes, molybdates and tungstates. During this period more than 1000 papers were published. From 2002, Dr K. Lebbou in ILM laboratory [31–45] and Prof A.Yoshikawa in Tohoku University [46–56] focused their effort on looking for specific application using SCFS design and from 2006, E.D. Bourret-Courchesne in Lawrence Berkeley National Laboratory (USA) start research program on single crystals fibers pulling by μ-PD technique [57,58]. So, from this period, SCFS research activity was oriented on the development of application especially laser and scintillations properties. Today, we feel that the SCFS community has expanded tremendously, and its needs have evolved compared to 20 years ago. As SCFS field has evolved, many publications have emerged, and still the field continues to grow and require innovation in the published paper. From 2015, SCFS grown by μ-PD start to be developed in china. Dr J. Zhong from Sun Yat-sen University, Guangzhou and Dr X.Xu from Jiangsu Normal University, Xuzhou installed μ-PD machine. Thus, in relatively few years, technology of pulling long SCFS from the melt arrived on the scene and the relentless progress is clearly observed through the different papers and books. Today, the growth of SCFS by pulling from the melt and their characterization is fully described in the classics series of books entitled “Fiber crystal growth from the melt”, “Shaped crystals” and “Crystal growth processes based on capillarity”. The significant developments made by the SCFS community are:

1 Improvement of the technology machine stability (modulation of the pulling rate)
2 Utilization of automatic mass control during the growth process even for fiber diameter less than 200 μm (performed growth software developed by Cyberstar company)
3 The control of fiber diameter including diameter decreasing (single mode fiber)
4 Multi-pulling Fiber
5 The control of dopant distribution through crucible design and continuous feeding
6 Utilization of simulation and modeling at small scale

In spite of the great effort done by the academic research laboratory and some company such Cyberstar [59] to develop the fiber growth technology to get performed fiber, the development speed is slightly insufficient which causes retardation from practical the necessities in SCFS design. In this paper, I have attempted to present SCFS technology, where we are today and the ways to improve the performance of this geometry through a huge research program including discussion with colleague's expert on SCFS design.

2. Pulling single crystal fiber shape (SCFS) from the melt

The basic of SCFS growth consist of material crystallization using a heater source. It can be radio frequency (RF) induction, resistive or laser heating source (Fig. 1). Quite often the heating source is chosen as a function of the melting temperature and melt behavior during the crystallization stage. In the case of RF induction or resistive heating source, a crucible containing a capillary die with special shape enabling the liquid to retain that geometry and by appropriate growth condition, the liquid is converted to shaped crystal (fiber, plate, square …).

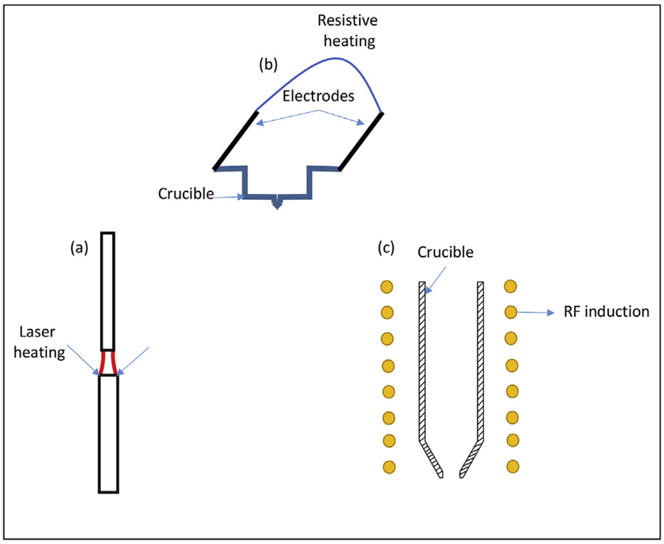

Fig. 1. Illustration of the various single crystal fibers pulling from the melt (a) LHPG technique, (b) μ-PD resistive heating and (c) μ-PD RF induction heating.

Iridium crucibles have been used for the growth of $Y_3Al_5O_{12}$ (Melting temperature around 1960 °C) and for sapphire (around 2050 °C). Because of the high temperature involved and to limit the heat flow losses, thermal insulation around the crucible is needed. This is provided by refractory materials such as alumina, magnesia and zirconia. Of course the melting temperature of the thermal insulation must exceed the fiber being grown. Quite often, materials of high melting temperature such Lu_2SiO_5 (2150 °C), necessitate the utilization of zirconia, but in the case of fiber pulling by μ-PD technique and because of the crucible size, it is possible to use alumina thermal isolation.

Because of the size of the SCFS (diameter≤1 mm), it is important to limit the area of the liquid-free surface and its perturbations. The seed crystal is positioned axially below the crucible capillary die. It is dipped into the drops at the bottom of the capillary die and if drops is not obtained or not visible, part of the seed is melted by connection to the lip of the capillary die and the melt temperature adjusted until a meniscus is supported. The pulling operation start slowly by careful adjustment of the heater power, a SCFS of the desired diameter can be grown. The quantity of fiber forming material fed per unit of time is equal to the quantity of solidified single crystal. The flow quantity Q of liquid passing through the capillary die is given by:

$$Q=(\pi R^4/8\ \mu l)\ (p_1-p_2) \tag{1}$$

Where R is the radius of the capillary die, l is the length of the capillary die, μ is the dynamic viscosity of the liquid and (p_1-p_2) is the pressure difference between the ends of the capillary die.

The whole assembly is maintained in a chamber which allows control of the gas and enables visual observation of the fiber to be grown. Fig. 2 shows a schematic illustration of Ce-doped YAG fiber single crystal pulled from a melt contained in iridium crucible heated by RF induction. The main properties of the charge container (crucible) is capability to resist to damage during growth operation and the wetting with the melt. Quite often, the degree of wetting is fixed by the value of the edge angle cosα between the

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