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Pulling growth technique towards rare earth single crystals

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Pulling growth technique serves as a popular method to grow congruent melting single crystals with multiscale sizes ranging from micrometers to centimeters. In order to obtain high quality single crystals, the crystal constituents would be arranged at the lattice sites by precisely controlling the crystal growth process. Growing interface is the position where the phase transition of crystal constituents occurs during pulling growth process. The precise control of energy at the growing interface becomes a key technique in pulling growth. In this work, we review some recent advances of pulling technique towards rare earth single crystal growth. In Czochralski pulling growth, the optimized growth parameters were designed for rare earth ions doped $Y_3Al_5O_{12}$ and Ce:$(Lu_{1-x}Y_x)_2SiO_5$ on the basis of anisotropic chemical bonding and isotropic mass transfer calculations at the growing interface. The fast growth of high quality rare earth single crystals is realized by controlling crystallization thermodynamics and kinetics in different size zones. On the other hand, the micro pulling down technique can be used for high throughput screening novel rare earth optical crystals. The growth interface control is realized by improving the crucible bottom and temperature field, which favors the growth of rare earth crystal fibers. The rare earth laser crystal fiber can serve as another kind of laser gain medium between conventional bulk single crystal and glass fiber. The future work on pulling technique might focus on the mass production of rare earth single crystals with extreme size and with the size near that of devices.

pulling growth technique, rare earth single crystals, Czochralski pulling growth, micro pulling down growth, $Y_3Al_5O_{12}$, Ce:$(Lu_{1-x}Y_x)_2SiO_5$, chemical bonding theory of single crystal growth

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1 Introduction

Phase transition is an effective way to realize the mass conversion in materials formation. Crystallization is a typical phase transition process from gas or liquid state to crystalline state, during which the crystal constituents undergo the transition from disordered arrangement to short-range and long-range ordered arrangements, and the crystalline materials are formed [1]. Crystallization process includes nucleation and crystal growth stages [2]. The crystallographic structure would be ascertained in nucleation stage and the crystal geometry and quality will be determined in crystal growth stage [3−5]. In order to obtain the single crystals, the phase transition should be driven in crystallization vessel [5]. Different growth techniques have been developed to satisfy the phase transition requirements in gas and liquid environments [6,7]. For the single crystal growth in the melt, pulling growth technique serves as a popular method to grow congruent melting single crystals. Herein, the pulling technique includes upward and downward pulling, corresponding to Czochralski (Cz) method and micro pulling down method, respectively [8,9].

Cz growth method was invented by J. Czochralski in 1917 and developed by G. K. Teal and J. B. Little in Bell Labs to grow monocrystalline Germanium. Hereafter, Cz technique has been used to grow single crystals of semiconductors (e.g.

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silicon, germanium and gallium arsenide), metals (e.g. palladium, platinum, silver, gold), salts and synthetic gemstones. In order to grow single crystals with a larger size, the crystal undergoes shoulder enlargement growth, equal-diameter growth, and tailing growth [10]. The size control method in Cz growth includes weighing the increased crystal, weighing the decreased melt, and measuring the light ring in the melt. Weighing the increased crystal becomes the most common method in controlling the diameter size of single crystal in Cz growth. The size of single crystals ranges from 10 to 500 mm, and even larger. The large single crystal will provide raw material with a large enough size for processing various devices. However, when the size of aim crystal components is too small, such as fibers, there are too many difficulties in after-processing. Micro pulling down growth technique may be used to grow single crystal fibers, which was invented by J. Ricard in 1975 and developed by Tohoku University in 1990s and Claude Bernard University Lyon 1 in 2000s [11−13]. On the basis of these efforts, the "Fibercrys" company was founded in 2003, focusing on the industrial applications of this growth technique and crystal products. As shown in Figure 1, the growth of multiscale single crystals ranging from crystal fibers to large-size single crystals can be realized by pulling technique in congruent melt.

Rare earth single crystal is defined as the crystal in which the rare earth ions can occupy a lattice site completely [14]. There are 1639 energy levels for rare earth ions in which the 4f shells are not completely filled, and the number of possible transitions between different energy levels is as high as 192177, making rare earth crystals as excellent luminescence materials [15,16]. Laser single crystals and scintillation single crystals are well known among various rare earth crystalline materials [17−20]. Rare earth doped laser crystals are widely used as gain medium in laser diode pumped lasers to achieve efficient laser output in different wavelengths. Recently, laser crystal fiber serves as another kind of laser gain medium between the conventional bulk single crystal and glass fiber. The materials composition of rare earth crystal fiber is the same to that in traditional laser crystals. Moreover, the rare earth crystal fiber also owns the geometry characteristics of glass fiber, i.e., high aspect ratio and large specific surface area. Therefore, combined with the core concept of single crystal and fiber laser, rare earth crystal fibers not only have excellent optical and thermal properties of single crystals, but also have the advantage of high optical conversion efficiency of fiber laser.

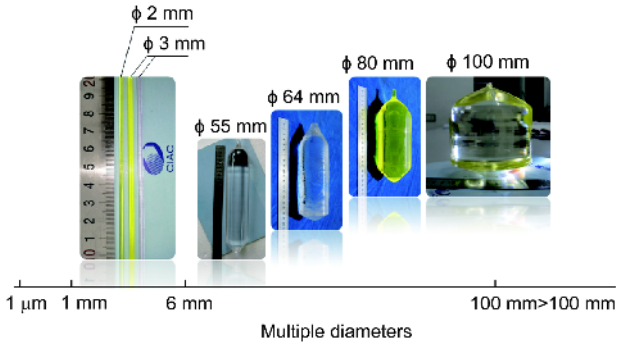

Figure 1 (Color online) Multiscale single crystals ranging from crystal fibers to large-size single crystals grown by pulling technique in congruent melt.

In this work, we review some recent advances in pulling technique towards rare earth single crystal growth. In Cz pulling growth, taking a series of rare earth laser and scintillation single crystals as examples, the application of the chemical bonding theory of single crystal growth in fast growth parameters design was summarized. Moreover, some technique progresses on rare earth crystal fibers are also reviewed, mainly focusing on the micro pulling down growth method.

2 Fast Cz growth technique towards large size rare earth single crystals

Cz growth process of rare earth single crystal includes seeding, neck shrinkage, shouldering, equal-diameter growth, and tailing stages. Before single crystal growth, high purity rare earth oxide raw materials were filled in Ir crucible. Then, Ir crucible within Cu loop was heated via intermediate frequency inductive heat power. When a thermal equilibrium was built in the melt growth system, a single-crystal seed was lowered to the melt surface. Thereafter, the rare earth single crystal grows through pulling upward the crystalline seed slowly from the melt surface (Figure 2). During Cz growth process, the single crystal growth is promoted by the phase transition at the melt/crystal interface when it was pulled out of the melt vertically. That is, the phase transition along the radial-directions drives the rare earth single crystal growth along the axial-direction (i.e., pulling direction), as shown in Figure 3. From the viewpoint of substance conservation, crystal growth rate equals to the decreased melt mass per unit time [14,21].

$$M_{\text{melt}} = 4\pi r^2 N_{\text{melt},r|R}, \tag{1}$$

where M is decreased mass of melt, $N_{\text{melt},r|R}$ is the mass flux of melt at the position of r=R, and r is the distance between crystal center and the growing interface along the radial direction.

According to the Fick's first law, eq. (1) becomes

$$M_{\text{melt}} \frac{\mathrm{d}r}{r^2} = 4\pi \frac{-CD}{1-x} \mathrm{d}x. \tag{2}$$

Boundary condition is

$$r = R, \quad x = x_R. \tag{3}$$

Then, the crystal growth rate becomes

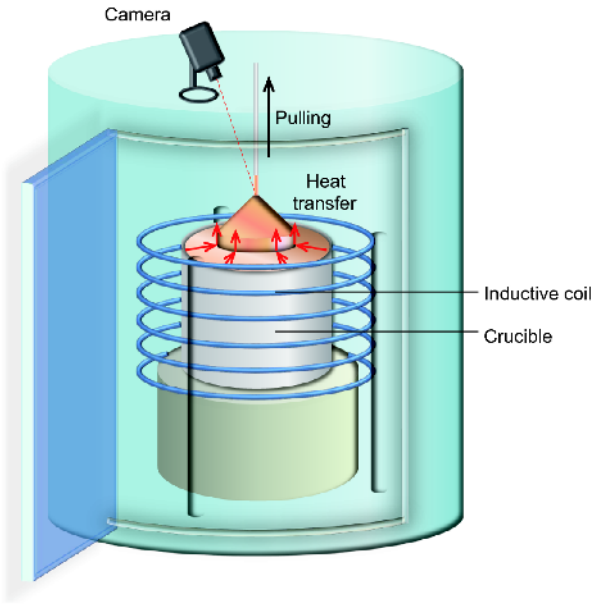

Figure 2 (Color online) Schematics of Cz growth furnace and large size single crystal growth process.

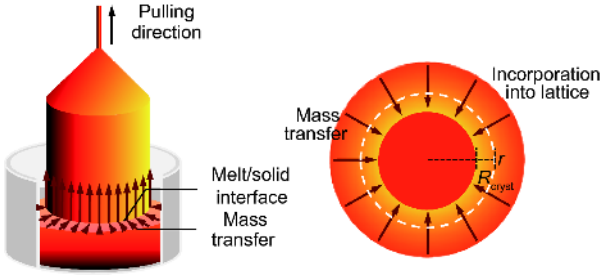

Figure 3 (Color online) Phase transition at the growing interface of rare earth single crystals in Cz system. The single crystal growth along radial directions promotes the growth along the pulling direction.

$$M_{\text{melt}} = 4\pi CDR\ln\left(\frac{1-x_R}{1-x_{\text{melt}}}\right), \tag{4}$$

where C is the concentration of crystal constituents in the melt, D is diffusion coefficient of crystal constituents in the melt, and x is the mass fraction of crystal constituents.

At the initial growth stage, temperature gradient is smaller, that is, the chemical bonding process serves as the rate-determining factor. Chemical bonding rate is much larger than diffusion rate, x_R=0. On the basis of eq. (4), it can be deduced that

$$M_{\text{melt}} = 4\pi CDR\ln\left(\frac{1}{1-x_{\text{melt}}}\right). \tag{5}$$

In such a case, the crystal growth rate depends on the anisotropic chemical bonding architectures. With increasing crystal size, the temperature gradient increases due to the shorter distance between crystal surface and the crucible. Both mass transfer and chemical bonding processes would co-determine the crystal growth rate. Boundary condition becomes

$$r = R,\ \ x = -\frac{N_{\text{melt},R}}{kC_R}. \tag{6}$$

The crystal growth rate becomes

$$M_{\text{melt}} = \frac{4\pi CDR}{1+N_{\text{melt},R}/kC_R}\ln\left(\frac{1}{1-x_{\text{melt}}}\right). \tag{7}$$

In such a case, the crystal growth rate depends on both anisotropic chemical bonding architecture and isotropic mass transfer.

In practical Cz growth, the anisotropic growth should be initially considered, which is driven by the anisotropic bonding architectures at the growing interface. It is well known that yttrium aluminum garnet crystal ($Y_3Al_5O_{12}$, YAG) is one of the most widely used laser host materials because of its favorable chemical, optical, thermal, and mechanical properties [22]. YAG single crystal can be easily doped with various rare earth ions, therefore, it acts as an excellent laser host materials. On the basis of chemical bonding theory of single crystal growth [23], the thermodynamic and dynamic morphologies of YAG can be calculated. In the calculations, the chemical bonding architectures at growing interface are important parameters, depending on the chemical bonding characteristics of crystal constituents. Starting from the orbital hybridization, recent endeavor has been made to build a relationship between outer orbitals that participate in bonding with ligands and the coordination number of central cations [24,25]. The most difference between rare earth ions and the other cations in chemical bonding is whether 4f orbitals participate in hybridization. When the 4f orbitals hybrid with the other outer 6s, and 5d orbitals to participate in the chemical bonding with ligands, the coordination number of rare earth ions is larger than 9. In YAG lattice, the rare earth ions dope Y^{3+} site, and the coordination number of Y^{3+} is 8, indicating that the 4f orbitals of rare earth ions do not participate in the chemical bonding. Therefore, there is no specific treatment for the chemical bonding calculations of rare earth ions doped YAG. The calculated results show that thermodynamic morphology of YAG prefers to expose {100}, {110}, and {111} surfaces, and the preferential pulling direction is [111]. When YAG single crystal is pulling growth along the [111] direction, the intersection of two adjacent surfaces respective perpendicular to $[0\bar{1}1]$ and $[\bar{1}01]$ owns higher energy. On the basis of lowest energy principle, the surface that is normal to $[\bar{1}\bar{1}2]$ would be exposed to reduce energy, and then the dynamic morphology of YAG single crystal is obtained. The chemical bonding architectures at solid/liquid interface along the radial directions when the YAG crystal is pulled up along [111] direction can be further confirmed.

Over the past few years, various inorganic rare earth scintillation crystals attract much interest due to their importance in nuclear and particle physics, and for medical and

high-tech industrial applications. Ce^{3+} doped $(Lu_{1-x}Y_x)_2SiO_5$ (Ce:LYSO) has been widely studied owning to its excellent scintillation properties. Similarly, the anisotropic growth of LYSO single crystal has also been investigated. The thermodynamic morphology and dynamic morphologies of LYSO were calculated on the basis of chemical bonding theory of single crystal growth. It is found that LYSO exhibits a polyhedron with {100}, {001}, {$\bar{1}$10}, and {$\bar{1}$11} surfaces thermodynamically, and the preferential pulling direction is confirmed as [010] direction. In dynamics, the high energy at the junction of two adjacent surfaces respectively perpendicular to [100], [$\bar{1}$01], [00$\bar{1}$], and [10$\bar{1}$] results in the exposure of [10$\bar{2}$] and [20$\bar{1}$] surfaces when pulling along the [010] direction. Consequently, the chemical bonding architectures at solid/melt interface along the radial directions in Cz growth of LYSO can also be confirmed.

By combining the chemical bonding theory of single crystal growth with eqs. (5) and (7), both thermodynamic and kinetic relative growth rates of rare earth crystals can be calculated. Furthermore, Cz growth parameters for rare earth single crystals were designed on the basis of the boundary conditions of anisotropic chemical bonding control and isotropic mass transfer control, including crystal-size-related pulling growth rate and rotation rate [26,27]. These growth parameters may meet the thermodynamic requirements in crystallization, favoring the construction of long-range and uniform chemical bonding architectures at the microscopic growth interface to guarantee the quality of rare earth crystals. On the other hand, the isotropic growth behavior would be encouraged at the macroscopic growth interface, which maximizes the exposure of high-index crystal planes and accelerates the growth rate. Temperature field structure was built to match the designed growth parameters, realizing the fast growth of a large series of large-size rare earth laser and scintillation single crystals. The growth rate of rare earth ions doped YAG laser crystals with the size of ϕ80 mm×240 mm is 3.0 mm/h, and the growth rate of rare earth scintillate crystals, Ce:YAG with the size of ϕ80 mm×240 mm and Ce:LYSO with the size of ϕ64 mm×220 mm, can arrive 3.5 mm/h. In the present Cz pulling technique, the fast growth of high quality rare earth single crystals is realized by controlling crystallization thermodynamics and kinetics in different size zones. Moreover, the size control in Cz growth adopts weighing the increased rare earth single crystals by the electric balance. By comparing with the designed value, the computer controls the intermediate frequency inductive heat power to modify the practical frequency.

3 Micro pulling down growth technique towards rare earth crystal fibers

During the micro pulling down growth, the crystal fiber is grown by pulling down the melt of raw materials which come out of the hole at the bottom of crucible. The scheme of micro pulling down furnace and crystal fiber is shown in Figure 4. The melt passes through the capillary-tube from the crucible, then a pending drop is formed. After the seeding process, a thin floating molten zone, the meniscus, is formed between the crucible bottom and the crystal, which would drive the the crystallization under an appropriate temperature gradient and pulling down rate. Compared with the other growth techniques, the micro pulling down technique has two main advantages. First, a crystal can be grown several times as fast as other methods and would be used for high throughput screening novel rare earth optical crystals [28]. Second, a shaped single crystal can be grown by using with a specially shaped crucible. For instance, a rectangular-column crystal can be grown by a crucible with a square-shaped die at the bottom [29]. The micro pulling down technique attracts attention for the application to mass production owning to its distinctive advantages compared with the other growth techniques.

Rare earth crystal fiber not only possesses a particular crystallographic structure but also owns a higher aspect ratio and large specific surface area like glass fibers. Once it serves as the laser gain medium, this new materials has both advantages of traditional single crystal laser and fiber laser, such as excellent optical and thermal properties of single crystal and high laser conversion efficiency. To date, the research on rare earth crystal optical fiber can be divided into two ideas. One leans from a traditional glass fiber laser, in which a doped YAG single crystal with a diameter of several tens of micrometers as the core, and the cladding structure can be formed by direct growth or after processing. Finally, a flexible single crystal fiber with a cladding layer is obtained.

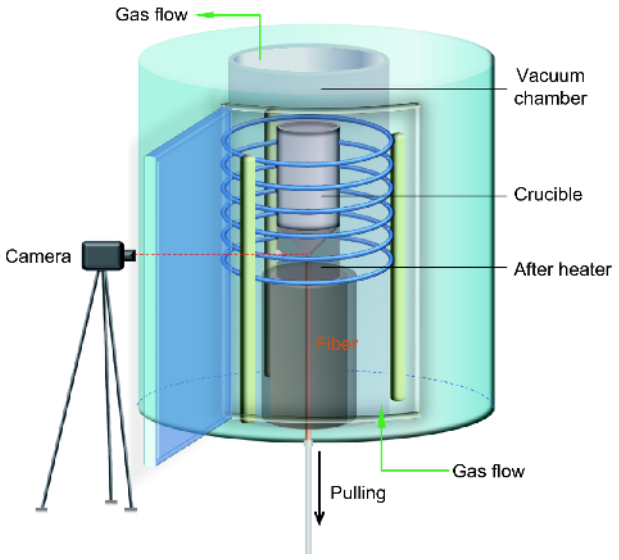

Figure 4 (Color online) Schematics of micro pulling down furnace and crystal fiber growth process.

Maxwell team in Shasta Crystals and Harrington research group in Rutgers University did a lot of work in this field [30,31]. Another idea of single crystal fiber was proposed by Delen et al. [32] in French Academy of Sciences as an intermediate transition between conventional bulk solid-state lasers and fiber lasers with typical diameters between 400 μm and 1 mm and the length of 40−60 mm. By using air as the cladding layer, multi-mode pump light is injected into the single crystal fiber and forms a waveguide. The oscillation of the laser is realized by adding a cavity mirror at both ends, ensuring the beam quality of the output laser. To date, the research of crystal optical fibers is mainly concentrated in USA, France, and Japan. Rutgers University and Shasta Crystals mainly use the laser heating pedestal method to grow single-crystal optical fibers, and realize the stable growth of rare earth doped YAG single crystal fibers by improving equipment mechanical and equal-diameter. The maximum doping concentrations of Nd^{3+}, Yb^{3+} and Er^{3+} are 4%, 10% and 50%, respectively. Moreover, the size of crystal fibers can reach several tens of micrometers in diameter and 900 mm in length. In 2016, the United States Army Research Laboratory reported the optical waveguide laser output of single crystal fiber for the first time [33]. The micro pulling down method was developed by the Fukuda laboratory in the early 1990s [13]. Then, Fibercryst SAS and Charles Fabry in France make outstanding efforts in the growth of Nd^{3+}, Yb^{3+}, Er^{3+} doped YAG single-crystal fibers by using the micro pulling down technique [34]. In 2011, Delen et al. carried out a continuous laser study using a 0.2 at% Nd doped YAG crystal fiber with the diameter of 1 mm and the length of 50 mm. The maximum output was 34 W with a pump power of 86 W, and the slope efficiency reached 53%. Delen's team [35] also realized a continuous laser output of over 250 W using a 1 at% Yb:YAG single fiber with the diameter of 1 mm and the length of 40 mm. The slope efficiency is 53%, and the optical-to-optical conversion efficiency is 44% [36].

During the micro pulling down growth process, there exist complex melt flow and heat transfer phenomena. The growth furnace is in a high-temperature environment. The convection in the melt also includes surface tension flow caused by non-equilibrium surface tension and the buoyant flow caused by gravity field and temperature gradient. Tatarchenko proposed the maximum pulling rate approximation theory and pointed out that the maximum pulling rate is the maximum value of the ratio of the meniscus height to the diameter of crystal fibers [37]. Rudolph et al. pointed out that there is an extreme value of seed crystal pulling rate on the basis of thermal equilibrium at growing interface [38]. Further combined with Tatarchenko's [37] maximum pulling rate approximation theory, YAG crystal fibers successfully grew with a fast rate of 20 mm/min. Due to the small size of the melt zone and negligible buoyant flow, the Marangoni convection plays a major role and is concentrated near the free surface. With decreasing the temperature of the crucible, decreasing the height of melt, and increasing the crystal diameter, the Marangoni convection would induces secondary convection, and the vortex center is close to the center of the melt. In recent years, non-steady state calculations have been used to explore the major influence factors in micro pulling down growth. The calculation results show that the limit of melt height is related to the crystal size and the effect of the thermal capillary flow. Moreover, the limit of crystal pulling rate depends on the temperature gradient. According to these simulation results, a technique was proposed to improve the distribution of dopant concentration in YAG by increasing the number of micro-channels and changing the entrance position [39,40].

4 Conclusions

In summary, pulling growth techniques including Cz growth and micro pulling down growth methods can be used to grow a series of rare earth single crystals with the size ranging from micrometers to centimeters. In both upward pulling and downward pulling growth processes, the growing interface serves as the key zone in which the phase transition from disordered or short-range ordered arrangement to long-range ordered arrangement drives rare earth delete single crystal growth. In order to obtain high quality rare earth crystals, the crystal constituents would be arranged at the lattice sites, and the chemical bonding between rare earth crystal constitutes plays a critical role. Starting from the chemical bonding theory of single crystal growth, the optimized growth parameters were designed for rare earth single crystals by combining with isotropic mass transfer calculations. These growth parameters promote the construction of long-range and uniform chemical bonding architectures at the microscopic growth interface to guarantee the quality of rare earth crystals, and encourage the isotropic growth at the macroscopic growth interface to accelerate the growth rate. On the other hand, the micro pulling down technique can be used for high throughput screening novel rare earth optical crystals. The growth interface control is realized by improving the crucible bottom and temperature field, which favors the growth of rare earth crystal fibers. The future work on pulling technique may focus on the mass production of rare earth single crystals with extreme sizes and with the sizes near that of devices without after-processing.

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