

# Single-crystal fiber optics: a review

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## ABSTRACT

Single-crystal (SC) fiber optics have been grown for many years for use as passive fibers for the delivery of IR laser radiation and as active fibers useful as minirod lasers. Most of the early work on SC fiber optics involved the growth of unclad sapphire fibers for the transmission of Er:YAG laser radiation at 2.94  $\mu\text{m}$ . More recently there has been a renewed interest in rare-earth (RE) doped oxide crystal fibers for use as high power fiber lasers. By analogy with RE doped-bulk laser crystals it is expected that pure YAG and other crystalline SC fibers should be capable of transmitting extremely high laser energies. SC oxide fibers have some distinct advantages over conventional glass fibers including higher thermal conductivity and low stimulated Brillouin scattering (SBS) gain coefficients. The latter can limit the ultimate power output of glass fiber lasers. To date most of the investigators have used the technique of Laser Heated Pedestal Growth (LHPG) to grow unclad SC fibers with diameters ranging from 30 to 350  $\mu\text{m}$  and in lengths as long as 5 m. The loss for SC sapphire fibers at 2.94  $\mu\text{m}$  has been measured as low as 0.3 dB/m. In this review we discuss the technique of LHPG, the various SC fiber optics that have been grown for both active and passive applications, and methods that may be used to clad the fibers.

**Keywords:** Infrared fiber optics, single-crystal fibers, oxide crystal fibers, fiber lasers

## 1. INTRODUCTION

SC fibers have been grown since the early 1980s with most of the work concentrating on passive (pure) SC sapphire fibers.<sup>1</sup> There has also been some limited work on growing doped and undoped YAG<sup>2-4</sup> and other garnet SC fibers.<sup>5</sup> Essentially all of these fibers have been grown by the LHPG technique. In this method a CO<sub>2</sub> laser is used to melt the tip of a crystalline source rod and a fiber is pulled upward from the molten tip. The SC fibers have been unclad in the sense that there is no true fiber cladding as is commonly associated with the core/clad structures of glass fiber. The most common SC fiber studied to date has been sapphire or Al<sub>2</sub>O<sub>3</sub>. Sapphire is an insoluble, uniaxial crystal (trigonal system, hexagonal class). It is an extremely hard and robust material with a usable fiber transmission from about 0.5 to 3.2  $\mu\text{m}$ . Sapphire and other oxide crystals have outstanding physical characteristics, which rival even those of silica.<sup>6</sup> For instance, the Young's modulus of sapphire is approximately 6 times greater than silica; the melting point is over 2,000 °C; and it is extremely hard. Sapphire and other garnet crystals are very inert with thermal conductivities more than 10 times higher than silica. These properties of sapphire and similar properties for the other oxide crystals such as YAG make them good SC fiber candidates for applications from 1.5 up to 4  $\mu\text{m}$ .<sup>7</sup>

The key physical properties of sapphire, YAG, and other oxide crystal fiber candidates are given in Table 1. The properties of these oxide crystals clearly indicate the outstanding physical characteristics of these materials and how well they rival those of silica.

Table 1 – Properties of some oxide crystals used for fabrication of SC fiber optics

Material	Symbol	Structure	M <sub>p</sub> , °C	n @ 3 $\mu\text{m}$
Sapphire	Al <sub>2</sub> O <sub>3</sub>	Hexagonal – uniaxial	2040	1.709
YAG	Y <sub>3</sub> Al <sub>5</sub> O <sub>12</sub>	Garnet – cubic	1940	1.788
GGG	Gd <sub>3</sub> Ga <sub>5</sub> O <sub>12</sub>	Garnet – cubic	2098	1.915
Spinel	MgAl <sub>2</sub> O <sub>4</sub>	Octohedral – cubic	2135	1.667

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## 2. FABRICATION OF SC FIBERS

There are basically two different methods for fabricating oxide crystal fibers. One method involves the use of a seed fiber or wire to pull a fiber from a molten reservoir. In this approach a high temperature crucible, usually made of tungsten, is used to contain the melt. The SC fiber is most often pulled up from the melt as pioneered by LaBelle<sup>8</sup> or pulled down through a small hole in the crucible. The method of LaBelle and Saphikon, Inc. (now Photran) is called the edge-defined, film-fed growth (EFG) technique. In this method a single or multiple fibers are pulled upward from capillary tubes inserted into the molten crystalline material contained in the crucible. A related method is the micro-pulldown technique developed by Fukuda and his co-workers at Tohoku University in Sendai, Japan.<sup>9</sup> An alternative to these methods is a crucibleless method, called laser heated pedestal growth (LHPG). This method was first developed by Haggerty<sup>10</sup> at MIT and then further refined by a number of researchers including those at Stanford University,<sup>11</sup> Bell Labs,<sup>12</sup> Rutgers University,<sup>6</sup> University of South Florida,<sup>4</sup> Shasta Crystals, Inc.,<sup>13</sup> and others.<sup>5</sup>

The LHPG is a crucibleless technique which closely resembles the float-zone method of crystal growth.<sup>5</sup> In the float-zone method, the molten zone is freely supported between the two ends of the crystal rod. LHPG is inherently the best technique for growing high optical-quality SC fibers because the molten zone is held in place by surface tension, eliminating the need for a crucible, which could be a possible source of contamination. Furthermore, a CO<sub>2</sub> laser beam, which provides a uniform, ultra-clean heat source, is used to melt the starting rod. Unlike the EFG and micro-pulldown technique, this growth method allows only one fiber to be grown at a time. LHPG, however, has been the method used to produce the lowest loss sapphire fibers with losses approaching theoretical values at 3 μm.<sup>6</sup>

In LHPG SC fiber growth, a CO<sub>2</sub> laser beam is focused onto the tip of a source rod creating a small molten bead at the top of the oxide crystal source rod. A seed fiber is dipped into the molten region, shown schematically in Fig. 1, and slowly pulled upward forming the single-crystal fiber. The source rod, which may be single crystal or polycrystalline (ceramic), is simultaneously fed upward to replenish the supply of molten material. The shape of the

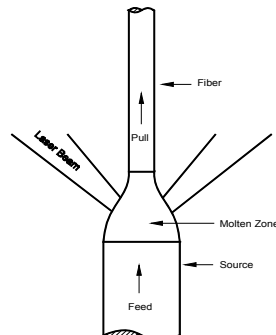


Figure 1 – Schematic of source rod, molten zone, and fiber in LHPG

molten zone is a function of the laser power, the diameter reduction, and the material being grown. In general, the length of the maximum stable zone is approximately 3 times the fiber diameter. It is much more difficult to produce SC fibers with smooth surfaces than it is for a glass fiber. This is because the viscosity of a glass is very high during drawing whereas the viscosity of crystalline material at the molten zone is very low and, thus, sensitive to any minor perturbations in the system. Since the molten region is held in place simply by surface tension, any air currents, vibrations, laser-power fluctuations, etc. will have enormous effects on the stability of growth. For this reason it is necessary to use a very stable laser source; have a small molten zone; and grow with a source-to-fiber reduction ratio of about 3:1.

The details of the LHPG technique used to grow our SC fibers have been previously published. A diagram of the key elements of the LHPG apparatus is shown in Fig. 2. In general, our LHPG system closely resembles the LHPG growth facilities at both Stanford University<sup>11</sup>, University of South Florida<sup>14</sup>, and Shasta Crystals.<sup>13</sup> Referring to Fig. 2, the key components are an amplitude stabilized, 30-W CO<sub>2</sub> laser beam, a laser micrometer for diameter control, and a reflexicon optical system. Feedback from the laser micrometer is used to stabilize the laser power to ±0.5%. Approximately 10 to 20 W of laser power is needed to melt a 1-mm-diameter source rod. The growth rate is about 1 to 2 mm/min which translates to at least 8 hours to grow one meter of fiber. The seed fiber is either an SC fiber from

the same material, another oxide crystal fiber, or a Pt wire. Normally there is a 3:1 reduction of the source rod to fiber diameter. Thus, if we start with a 1-mm source rod the fiber will be about 300- $\mu\text{m}$  in diameter. To make smaller diameter fibers we regrow the fiber and continually reduce the fiber diameter to as small as about 30  $\mu\text{m}$ . The longest length of SC fiber grown to date is a 5-m sapphire fiber. Again, most of the fibers grown have been unclad.

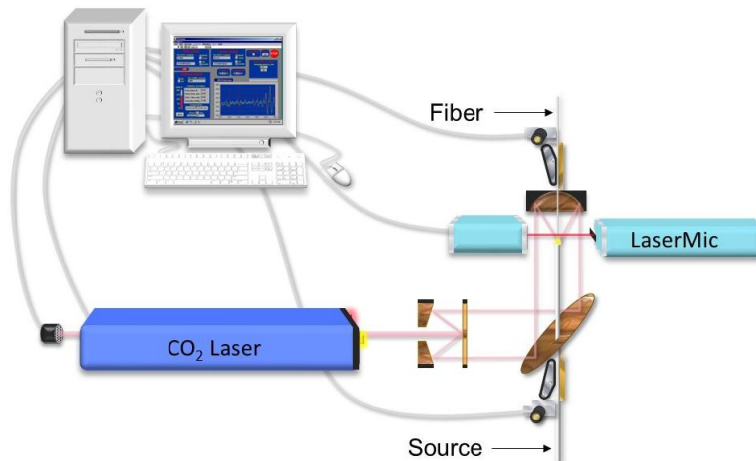


Figure 2 – Schematic diagram of LHPG apparatus showing critical components.

### 3. OPTICAL PROPERTIES OF SC OXIDE FIBERS

#### 3.1 IR absorption edge for oxide crystals

The IR absorption of most oxide crystals is well documented in the literature. In Fig. 3 we show the IR absorption edge for four different oxide crystals: sapphire, YAG, GGG, and yttria. The absorption at about 4.8  $\mu\text{m}$  in YAG is due to multiphonon absorption and, therefore, an intrinsic property of the material. Yttria also has a weak multiphonon peak at 5  $\mu\text{m}$ .<sup>15</sup> As expected the IR edge is shifted toward longer wavelengths for oxides with the heaviest ions. For example, if we extrapolate from the IR absorption data we can estimate the intrinsic absorption for a fiber made from these materials. This would give us the loss for pure sapphire, YAG, GGG, and yttria as 18, 5.5, 0.5, and 0.15 dB/m at 4  $\mu\text{m}$ , respectively. Based on these results it is clear that any fiber of a reasonable length, say greater than 1 m, would not transmit well beyond about 4  $\mu\text{m}$ . The exception would be an SC yttria fiber. The difficulty with yttria is that it has a phase transition at about 100 °C below the melting point and thus it is hard to grow a single crystal of this material. In the past we tried to grow an SC yttria fiber starting with a polycrystalline (ceramic) source rod. In all cases we obtained small cracks in the outer diameter of the fiber indicative of the effects of the phase change in this material on cooling.



Figure 3 – IR absorption edge for bulk oxide crystals. Note the good transmission of GGG and yttria at longer wavelengths.

By way of example of the losses that are achieved for SC fiber, we show the attenuation spectrum for two SC sapphire fibers in Fig. 4.<sup>6,14</sup> Several spectral features commonly found for SC fibers can be seen in this data. First, the losses in the visible region for the as-grown fiber are generally higher compared to the starting bulk crystalline material. However, this short-wavelength loss can be reduced by annealing the fiber in air at 1,000 °C for about 12 hours. The data from the University of South Florida shows how the loss at visible wavelengths can be reduced by annealing. At the longer wavelengths there may be some absorption due to OH in the 3 μm region and also some loss in the same region that has been associated with absorption by metallic impurities.<sup>6</sup>

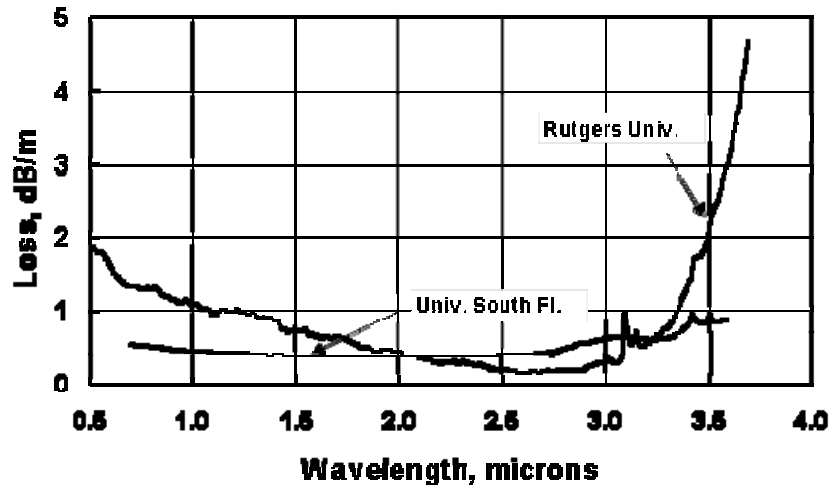


Figure 4 – Losses for two of the best SC sapphire fibers grown to date using LHPG.

### 3.2 Comparison of fiber loss for ceramic versus single-crystal source rods

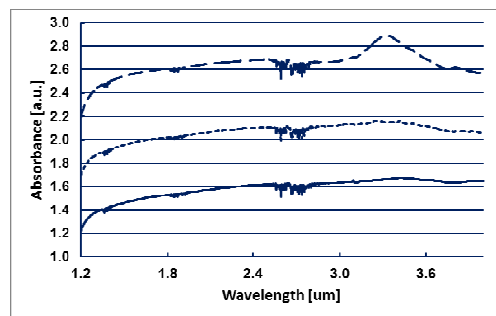


Figure 5 – Comparison of SC fiber loss for fibers grown from SC source material (solid curve), ceramic source made by the University of Michigan (dotted curve), and from CoorsTec ceramic rods (dashed curve).

One of the advantages of LHPG fiber growth is that we can begin with ceramic or polycrystalline source rods as the starting material. That is, it is often the case where it is difficult to procure SC source material in the typical lengths and diameters needed for an LHPG preform. Therefore, we can begin with the oxide powder which is then compacted, sintered, and cut into bars. The difficulty in using ceramic source material is that often the material contains a binder which is used to give the final ceramic strength. We have grown SC sapphire fibers from two ceramic  $\text{Al}_2\text{O}_3$  preforms, one a commercial alumina material from CoorsTec and the other a purer alumina ceramic made at the University of Michigan. The ceramic made by the University of Michigan group contains less binder than the CoorsTec material. In Fig. 5 we compare the losses for SC fibers grown from these two ceramic preforms and that grown from a high-quality SC source rod. The loss data indicate the relative difference in attenuation. From the data in Fig. 5 we see that the ceramic preforms have a generally higher overall loss than fiber grown from the SC

perform. In fact we would not necessarily expect good optical quality from the commercial CoorsTec material as it contains a lot of binder. Usually we see a slight yellowish coloration to the CoorsTec fiber which visibly indicates that this is not good fiber.

#### 4. CRYSTAL GROWTH ISSUES

One of the aspects of SC fibers that make them more challenging and different from glass fibers is the very fact that we are dealing with the aspects common to crystal growth instead of glassy materials which are inherently easier to draw. That is, we see the effects of crystal growth such as non-circular cross sections and faceting. Normally the SC fibers are not oriented along any particular axis unless the starting seed crystal is oriented. For non-cubic materials like sapphire, see Table 1, this could be a problem but usually orientation is of little concern. When the fiber is pulled the resultant orientation is in general random and, therefore, the resultant cross section can be nearly round or even squarish. In Fig. 6 we show an SEM photograph of the cross section of an SC YAG fiber, Fig. 6(A), and a side view, Fig. 6(B), of the same fiber which reveals the faceting that can occur. Facets are usually interpreted as crystal planes and they have been seen by others for many of the SC fibers grown. However, sometimes there is an absence of facets and we have found that there is generally less faceting for the small diameter and for sapphire fibers. The facets in Fig. 6(B) are spaced about 8  $\mu\text{m}$  apart. In a separate study of the surface morphology using AFM, we find that the peak-to-peak variation in height of the facets is about 40 nm. Obviously faceting would seem to be a deleterious effect as these features should increase the scattering losses in the fibers. One way to eliminate faceting, suggested by Ishibashi, et al.<sup>16</sup> is to orient the fiber along a specific direction. They found that they could grow facet-free  $\text{Cr}^{4+}$ :YAG fibers if the seed was oriented  $15^\circ$  from the [100] to [110] plane.

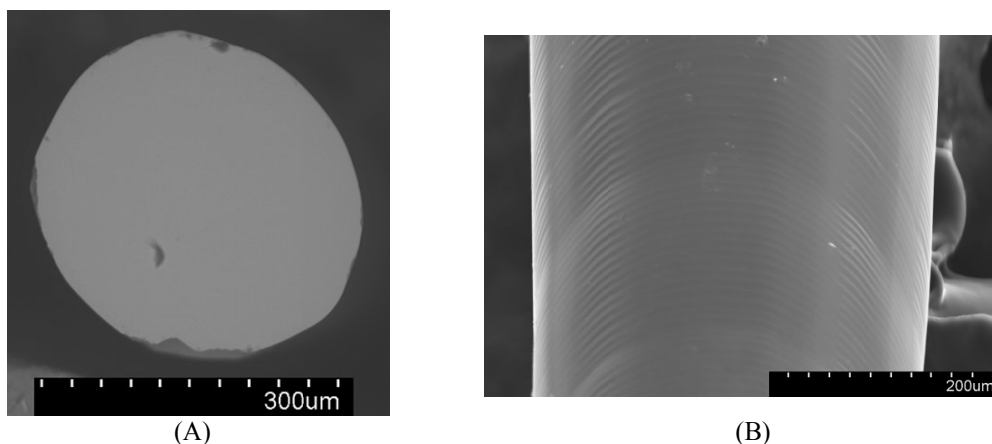


Figure 6 – SEM photographs of an SC YAG fiber showing (A) a slightly out-of-round cross section and (B) faceting.

Growing any high purity SC fibers from materials like sapphire, YAG, GGG, or yttria, presents some serious challenges both from the viewpoint of the purity of the material after fiber growth and in the growth process itself. The loss data for the two most commonly grown materials, sapphire and YAG, often show absorptions due to impurities.  $\text{OH}^-$  and some heavy metal impurities near 3 and 3.5  $\mu\text{m}$  often show up. Annealing in air has reduced the attenuation in the visible portion of the spectrum for sapphire and YAG SC fibers. In future work it will be interesting to experiment more with different atmospheres in the growth chamber to see if it is possible to reduce this impurity absorption. An interesting observation by Chang, et al.<sup>14</sup> was that growing the fibers in a He atmosphere enabled them to grow sapphire fibers at ten times the growth rate compared to the growth rate used when growing in ambient air conditions.

One of the biggest difficulties faced in the growth of these oxide crystal fibers is control of the fiber diameter. The situation is quite different for glasses because fiber is drawn from glassy materials which are in a highly viscous state. The melt zone in a crystal has a viscosity close to that of water so any vibration or irregular motion during

growth can lead to a diameter fluctuation. In our LHPG apparatus we control the laser power by virtue of a feedback signal from the laser itself. The source feed and fiber pulling motors are attached to belt drives so that the source and fiber are moved very smoothly. In addition, the source rod passes through a small bore tube to help guide the rod smoothly into the melt zone. The fiber rides in Teflon coated V-groove again to prevent sideways motion of the fiber. The motors, laser power, and laser micrometer are all controlled by a LabView program. Yet we still have diameter control issues that occur as the fiber is grown. Sometimes this occurs when the fiber moves in the tractor feed mechanism. All who work to make high quality SC fibers face these challenges but there does seem to be improvement in the mechanical as well as thermal issues involved in LHPG fiber growth.

## 5. DOPED SC FIBERS

RE-doped SC fibers have been grown for many years. By analogy with lasers made from oxide crystals it would seem like a good idea to grow fiber optics from the same well-known RE-doped crystals now used to make some of the most powerful lasers. SC fibers doped with  $\text{Nd}^{3+}$ ,  $\text{Tm}^{3+}$ , and  $\text{Er}^{3+}$  have been grown in YAG, YSGG, and YLF hosts. Most recently  $\text{Er}^{3+}$  in YAG has been grown with  $\text{Er}^{3+}$  concentrations ranging from less than 1% up to 50%. Er-doped garnets have strong emission bands in the 1.5 and 3  $\mu\text{m}$  region which makes them useful as pulsed or cw sources. Finally,  $\text{Cr}^{4+}$ :YAG fibers have been grown for potential applications in telecommunication systems as this material lases in the 1.3 to 1.6  $\mu\text{m}$  region.<sup>17</sup>

There are some distinct advantages of RE-doped SC fibers for use as a fiber laser over conventional silica fiber lasers. One of these is their higher thermal conductivity and lower SBS gain (high SBS threshold) compared to silica. For example, silica has a thermal conductivity of 1.38 W/m-K while YAG and sapphire have thermal conductivities of 9 W/m-K and 46 W/m-K, respectively. The exact SBS gain coefficient has been reported in one case to be as much as 1,000 times lower than silica. Parthasarathy, et al.<sup>18</sup> have determined that the performance limits of SC YAG fiber lasers far exceed those of silica fibers. They estimate conservatively that there is a limit of 6 kW per YAG fiber in multimode operation compared to 1.8 kW for a silica fiber laser. If the SBS gain coefficient is significantly less for YAG then the power limit for a single SC YAG fiber laser could be as high as 30 kW.

While the theory suggests that SC YAG fibers have enormous potential as fiber lasers the experimental results to date on doped YAG or other oxide crystal fibers are well below these estimates. Martial, et al.<sup>19</sup> have pumped 0.5%  $\text{Er}^{3+}$ :YAG SC fiber with a core diameter of 800  $\mu\text{m}$  with 80 W of 1532 nm power to achieve a maximum output power of 12.5 W at 1645 nm. This is the highest reported power for an Er:YAG SC fiber. Chen, et al.<sup>20</sup> also used diode pumping to produce a 1-W continuous output for Er:YAG at 3  $\mu\text{m}$ . Chang, et al.<sup>4</sup> were successful in pumping Tm:YAG, Ti:Al<sub>2</sub>O<sub>3</sub>, and other RE-doped oxide SC fibers. In most cases their work involved what could probably best be called a minirod laser as the fibers were unclad and rather short. Ishibashi, et al.<sup>16</sup> have excited  $\text{Cr}^{4+}$ :YAG SC fibers to obtain lasing at 1.5  $\mu\text{m}$  with an output power of 180 mW. In general, the application of RE-doped SC fibers for use as a powerful laser source is still some time off but the potential does exist for SC fiber lasers to be an excellent source of laser power.

## 6. CLADDING SC FIBERS

One of the great challenges for SC fibers is to properly clad the fiber in a manner analogous to that commonly used for glass fibers. The obvious problem is that SC fibers are grown from a non-viscous melt so beginning with a core/clad preform to achieve a clad fiber would seem doomed to failure as the core and clad material would mix on melting and thus the integrity of the core/clad interface would be lost. The upshot of this is that there has been very little effort made to clad these fibers. To fulfill the promising potential for these fibers, especially as fiber lasers, a cladding layer becomes increasingly important. There are basically two approaches to cladding. The first is to clad the fiber during growth and the second is to apply a cladding to the as-grown fiber. The first method would seem the Holy Grail if it can be done. Post-cladding involves growing very small (~30 to 40  $\mu\text{m}$ -diameter) pure core fiber and then somehow adding a cladding layer to make a core/clad fiber.

Post cladding was first studied as early as 1975 by Burrus and Stone<sup>21</sup> and then by Digonnet, et al.<sup>22</sup> at Stanford. Both groups clad their SC Nd:YAG fibers with glass which was chosen to closely match the thermal expansion of YAG. Burrus and Stone used a glass frit while Digonnet, et al. pulled a 41- $\mu\text{m}$  diameter Nd:YAG fiber and then inserted this fiber into an LaSF30 glass tube which was then drawn down to yield a thin cladding layer on the

Nd:YAG core fiber. The index difference,  $\Delta n$ , was measured to be about 0.048. Two groups at National Taiwan University have clad their  $\text{Cr}^{4+}$ :YAG fibers with silica in a somewhat different approach.<sup>17,23</sup> In their work they again started with a small, 68- $\mu\text{m}$  diameter doped YAG fiber which they then surrounded with a silica tube. They then melted the silica and YAG fiber combination to produce a silica/YAG mixed core and a silica clad. A somewhat related approach was taken by Dragic, et al.<sup>24</sup> They inserted an SC sapphire core into a thick wall silica tube and then drew this combination into a glass fiber. In this case the sapphire did melt and mix with the silica but they did find that there was a higher index in the center compared to the edge. Shasta Crystals attempted to clad their small diameter YAG fibers with a sol-gel glass composition but they had problems with cracking of the sol-gel films and adhesion of the sol-gel to the YAG fiber.<sup>13</sup>

It would seem that the best approach would be to fabricate a clad fiber as the fiber is being pulled. That is, somehow create an index variation in the resultant SC fiber that will act as a cladding layer. In this approach it is not necessary to repeatedly regrow core-only fiber to achieve a small diameter and then add post cladding to the fiber. One indication that it might be possible to grad the index of the fiber is past work on profiling the dopant concentration of doped oxide fibers. Chang, et al.<sup>4</sup> observed a gradient in the Nd concentration in SC YAG fibers as did Shasta Crystals.<sup>13</sup> In each case they found that there was more Nd in the center of the fiber than at the edge. Specifically, Shasta Crystals reported that there was a concentration of 3% Nd in the center which decreased to 2% at the edge of the fiber. In this work and similar observations by Chang, et al. for RE-doped YAG and Ti in sapphire fibers by Sharp's group,<sup>25</sup> the starting source material was uniformly doped. That is, there was no intentional index variation in the doped source crystal yet the resultant SC fiber did show a gradient in the RE doping. The gradient in Nd was observed to occur both radially and axially. In fact, Chang, et al.<sup>4</sup> observed that the Nd concentration varied axially from near zero at the beginning of the fiber growth to a maximum 1% concentration at the end of the fiber. What they observed was a segregation of the Nd ions in both dimensions. This seems reasonable given that the Nd ionic radius is about 10% larger than the Y ions that it replaces.

Recently, our work at Rutgers has attempted to take advantage of the movement of the RE ions in YAG to grow a core/clad SC fiber. Harrington and his group used a rod-in-tube approach to form a core/clad preform and then grew this preform into an SC fiber. Specifically, they drilled a 550- $\mu\text{m}$  hole in a single-crystal pure YAG rod (OD = 1140  $\mu\text{m}$ ) to make 1-cm long YAG tubes. Four of these YAG tubes were slipped over a 500- $\mu\text{m}$  diameter, 50% Er:YAG SC fiber. Then this 50% Er:YAG core/pureYAG rod-in-tube preform was pulled into 330- $\mu\text{m}$  diameter SC fiber. The as-grown fiber was then analyzed using fluorescence imaging methods to spatially profile the radial distribution of Er ions. Fluorescence from a 532 nm Verdi laser was measured across different diameters of the fiber. These line maps, with a resolution of about 1  $\mu\text{m}$ , indicated that indeed there was a variation in the Er ion concentration from center-to-edge across the radius of the fibers. In Fig. 7 we show the variation of  $\text{Er}^{3+}$  across the diameter of a fiber grown at 0.95 mm/min. From the data we can see that there is a higher concentration of Er ions in the center compared to the edge. At the moment we do not have a measure of how this variation translates into any variation in the refractive index but work is underway to make this measurement. What is interesting about this result is that the Er ion has about the same ionic radius as Y and Er and Er is very soluble in YAG as evidenced by the fact that we were able to use a core of 50% Er:YAG starting material.

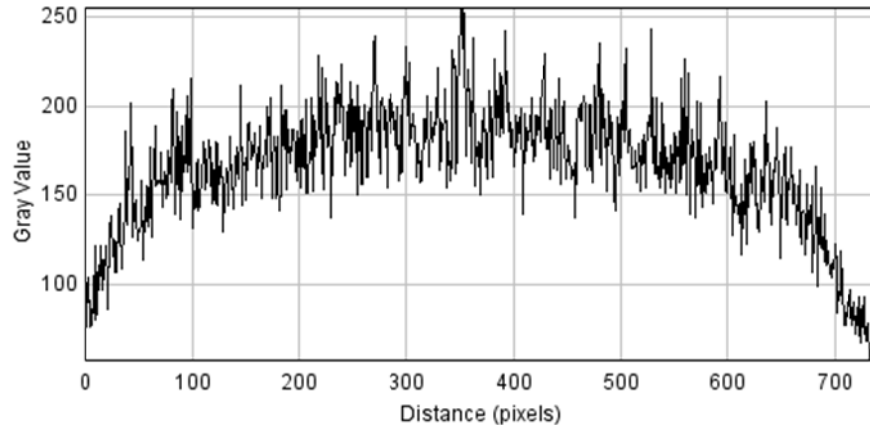


Figure 7 – Radial variation in  $\text{Er}^{3+}$  ion concentration for a 50% Er:YAG core/pure YAG clad SC fiber.

## 7. SUMMARY AND CONCLUSIONS

Oxide crystal fibers have some distinct advantages over other IR fibers in the 2 to 5  $\mu\text{m}$  range. Their extremely high  $M_p$  makes them suitable for applications involving temperatures above 1,200  $^{\circ}\text{C}$ . They are very inert and non-toxic. However, most of the SC fibers made to date lack a suitable cladding and, therefore, some applications such as SC fiber lasers have been slow in coming. Most of the emphasis to date has been on passive SC fibers for the delivery of high power laser radiation. In principle, these fibers should have very high laser damage thresholds. The losses for SC fibers are much higher than the intrinsic losses. For example, the measured loss for sapphire fibers at 2.94  $\mu\text{m}$  is about 3 times higher than the intrinsic loss. The potential for these unique fibers is high especially for use as fiber lasers. This is in large part due to their favorable physical properties like such as thermal conductivity and low SBS gain coefficients.

## 8. ACKNOWLEDGEMENTS

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