

# Single-crystal YAG fiber optics for the transmission of high energy laser energy

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## ABSTRACT

Single-crystal (SC) fibers have the potential of delivering extremely high laser energies. Sapphire fibers have been the most commonly studied SC fiber and the losses for sapphire fibers have been as low as 0.4 dB/m for a 300-micron core-only fiber at 3 microns. In this study we report on the growth of SC yttrium aluminum garnet,  $Y_3Al_5O_{12}$  (YAG) fibers from undoped SC source rods using the Laser Heated Pedestal Growth (LHPG) technique. The advantage of YAG over sapphire is the slight improvement in IR transmission of YAG. The IR transmission of bulk YAG has been shown to extend to 5  $\mu\text{m}$  where the absorption coefficient is 0.6  $\text{cm}^{-1}$ . The garnet family of crystals is one of the most commonly used oxide crystal hosts for lasing ions in high power solid-state lasers, with the most commercially common laser host being YAG. Thus, it is reasonable to assume that YAG fibers will have high laser damage thresholds. The optical losses for 400- $\mu\text{m}$  diameter YAG fibers have been measured to be about 3 dB/m at 2.94  $\mu\text{m}$ . The longest length of YAG fiber grown has been about 60 cm.

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

## INTRODUCTION

SC fiber optics have been grown for many years using some variation on crystal pulling.<sup>1,2</sup> The most common method used to grow SC fibers is the LHPG technique. In this method a  $\text{CO}_2$  laser is used to melt the tip of a crystalline source rod and a fiber is pulled upward from the molten tip. To date the majority of passive SC fibers grown have been made from aluminum oxide ( $\text{Al}_2\text{O}_3$ ) or sapphire. Sapphire is a uniaxial crystal in the trigonal crystal system, hexagonal class. Sapphire is inert and insoluble with a melting point of 2053  $^\circ\text{C}$ . It is an extremely robust material with a usable fiber transmission from about 0.5 to 3.2  $\mu\text{m}$ . The key physical properties of sapphire as well as 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 even those of silica. For example, sapphire has a Young's modulus approximately 6 times greater than silica; the melting point is over 2,000  $^\circ\text{C}$ ; and it is extremely hard. These properties make oxide crystal fibers almost ideal for applications not exceeding 5  $\mu\text{m}$ .

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

Material	Symbol	Structure	$M_p, ^\circ\text{C}$	$n @ 3 \mu\text{m}$
Sapphire	$\text{Al}_2\text{O}_3$	Hexagonal – uniaxial	2040	1.709
YAG	$Y_3Al_5O_{12}$	Garnet – cubic	1940	1.788
GGG	$Gd_3Ga_5O_{12}$	Garnet – cubic	2098	1.915
Spinel	$MgAl_2O_4$	Octohedral – cubic	2135	1.667

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## FABRICATION OF SC FIBERS

There are basically two methods for fabricating oxide crystal fibers. In both cases a seed fiber or wire is used to pull a fiber from a molten reservoir. In one method 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>3</sup> or pulled down through a small hole in the crucible.<sup>4</sup> The method of LaBelle and Saphikon, Inc. (now Photran) is called the edge-defined, film-fed growth (EFG) technique. The alternative is a crucibleless method, called laser heated pedestal growth (LHPG). This method was first developed by Haggerty<sup>5</sup> at MIT and then further refined by a number of researchers at Stanford University,<sup>6</sup> Bell Labs,<sup>7</sup> Rutgers University,<sup>2</sup> University of South Florida,<sup>8</sup> and others.<sup>9</sup>

The technique that we have used to grow YAG and other oxide crystal is the LHPG method. This crucibleless technique closely resembles the float-zone method of crystal growth. 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 sapphire 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 technique, this growth method allows only one fiber to be grown at a time, so commercialization is difficult. LHPG, however, is the method used to produce the lowest loss sapphire fibers with losses approaching theoretical values at 3  $\mu\text{m}$ .<sup>2</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 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, polycrystalline,

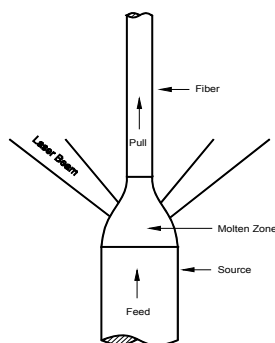


Figure 1 – Schematic of source rod, molten zone, and fiber in LHPG

sintered, or a pressed powder, is simultaneously fed upward to replenish the supply of molten material. The shape of the 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 3:1.

The details of our LHPG apparatus used to grow our SC fibers have been 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 and the University of South Florida. Referring to Fig. 2, the key components are an amplitude stabilized, 50-W CO<sub>2</sub> laser beam, a laser micrometer for diameter control, and a reflexicon optical system. 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 was either a sapphire or YAG fiber or a Pt wire. For this work the final fiber diameters were about 400  $\mu\text{m}$  when a 1-mm source

was used. Smaller diameters can be obtained by regrowing these 400  $\mu\text{m}$  fibers. The longest length of YAG fiber grown was 60 cm and all fibers were core only, i.e. unclad.

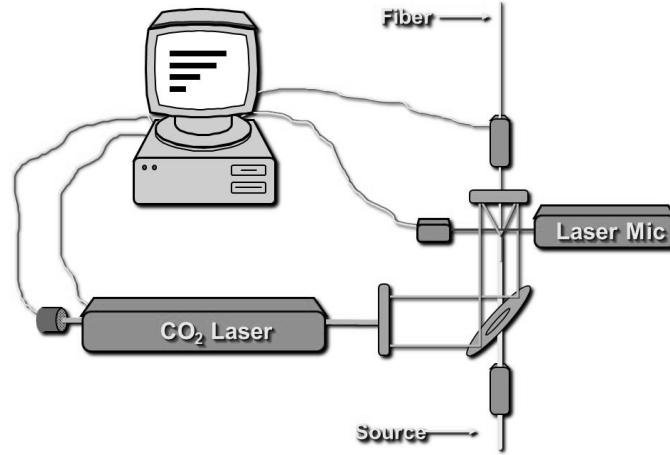


Figure 2 – Key components of the LHPG apparatus.

## OPTICAL PROPERTIES OF SC OXIDE FIBERS

### 1.1 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>10</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 the expected absorption for a fiber made from these materials, we would find that the loss for pure sapphire, YAG, GGG, and yttria is 18, 5.5, 0.5, and 0.15 dB/m at 4  $\mu\text{m}$ , respectively. Based on these results it is clear that the only viable SC fibers for use at 4  $\mu\text{m}$  from one of these four crystals would be GGG or yttria. The difficulty with yttria is that it has a phase transition at about 100  $^{\circ}\text{C}$  below the melting point and thus it is hard to grow a single crystal of this material. We have tried to grow an 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.

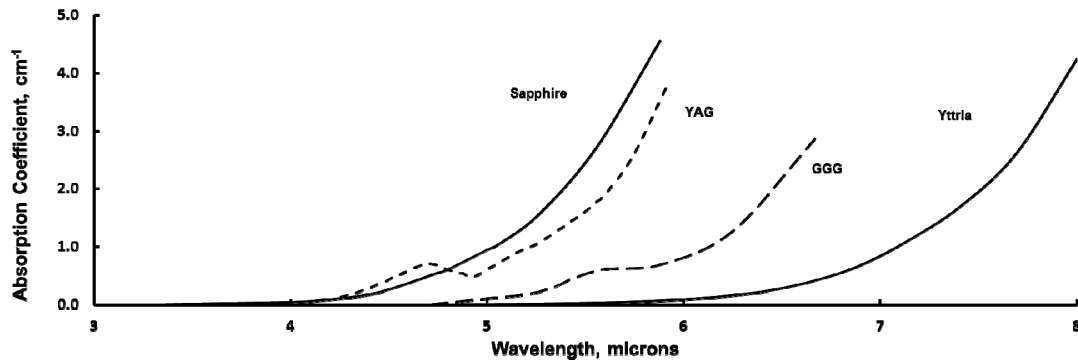


Figure 3 – IR absorption edge for bulk oxide crystals. Note the excellent transmission of GGG and yttria at longer wavelengths.

## 1.2 Loss for SC YAG fiber

The spectral loss for our YAG fiber is shown in Fig. 4. The loss measurement was taken on a Nicolet Protégé 460 FTIR spectrometer. The fiber diameter was 400  $\mu\text{m}$  and the length 5 cm. As mentioned above, the absorption near 4.8  $\mu\text{m}$  is due to multiphonon absorption. The two small peaks around 3.5  $\mu\text{m}$  are associated with OH defects. Specifically, the YAG structure in the fiber has distortions or oxygen-defects (vacancies) that give additional locations for OH producing additional hydrogen-bond stretch lengths (frequencies). The two strong absorption lines at 3.45 and 3.54  $\mu\text{m}$  are very near hydrogen-bond absorptions for the dodeca-octahedral and dodeca-tetrahedral bond lengths which are not observed for a bulk YAG crystal source rod. In addition, the absorption lines at 2.944 and 2.972  $\mu\text{m}$  in both the YAG fiber and feed crystal are well documented to be due to hydrogen-bond absorption.<sup>11,12</sup> The losses for a 35-cm length of YAG fiber measured using green and red visible lasers and an Er:YAG laser at 2.94  $\mu\text{m}$  are summarized in Table 1. These measurements were using a cutback technique to remove reflection and coupling losses. The loss of 3 dB/m at 2.94  $\mu\text{m}$  is much higher than the measured loss of 0.4 dB/m for sapphire at the same wavelength. We would not expect the loss for YAG to be this high and the work continues to improve the loss.

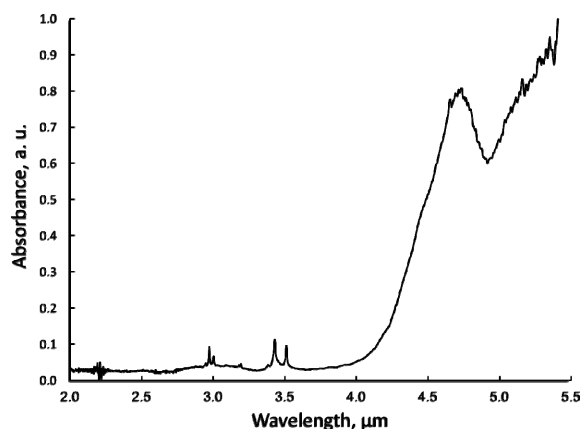


Figure 4 – Spectral loss for SC YAG fiber.

## 1.3 Loss for SC spinel fiber

Spinel fibers composed of a 50/50 mix of MgO and Al<sub>2</sub>O<sub>3</sub> were grown into fibers as long as 20 cm. The spectral response of a 400  $\mu\text{m}$  diameter by 4 cm long spinel fiber is given in Fig. 5. In the case of spinel we see strong water absorption at 3  $\mu\text{m}$  and similar OH contamination in the 3.5  $\mu\text{m}$  region as seen in Fig. 4 for YAG. Not surprisingly, the loss for the spinel fiber at 2.94  $\mu\text{m}$  is a very high 9 dB/m. The results for the losses of the 20-cm long spinel fiber using cutback methods are given along with the YAG fiber losses in Table 2.

Table 2 – Losses at some key laser wavelengths for YAG and spinel SC fiber optics.

SC Fiber Material	Laser	Wavelength	Loss, dB/m
YAG	Green	532 nm	1.9
	Red	635 nm	2.5
	Er:YAG	2.94 $\mu\text{m}$	3
Spinel	Green	532 nm	4
	Red	635 nm	9
	Er:YAG	2.94 $\mu\text{m}$	9

#### 1.4 Loss for GGG source rod

GGG is an attractive SC fiber crystal. It is cubic and, as may be seen in Fig. 3, it has good long wavelength transmission. The source bars of GGG that we obtained for fiber growth had dimensions of  $2\text{ mm} \times 2\text{ mm} \times 5\text{ cm}$  in length. The spectral loss for a GGG source bar is shown in Fig. 6. From this data we see the excellent IR transmission also displayed in the data in Fig. 3. The material seems relatively free of water absorption but again we see evidence of the OH-related defects near  $3.5\text{ }\mu\text{m}$ . So far we have not been successful in growing GGG fibers but this work continues.

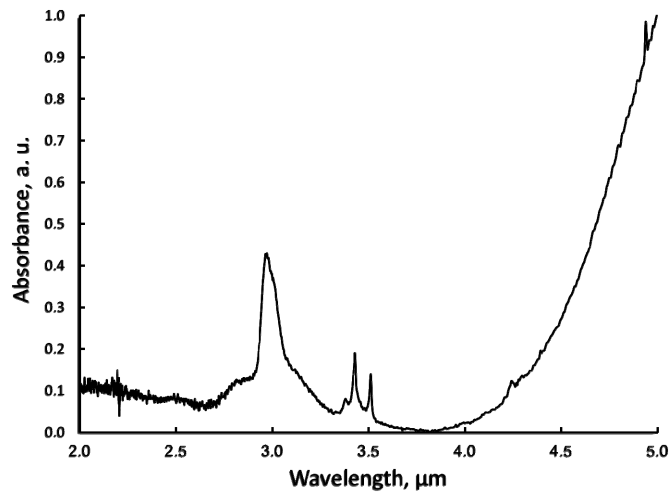


Figure 5 – Spectral loss for SC spinel fiber

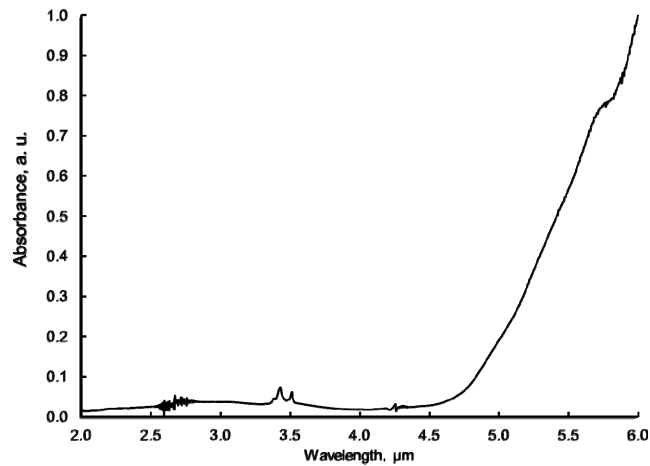


Figure 6 – Spectral loss for bulk GGG source rod.

## CHALLENGES IN THE GROWTH OF SC OXIDE FIBERS

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 data for both YAG and spinel show absorptions due to OH related defects which are not less strong in the bulk source material. These impurities and defects are present as two absorption peaks near  $3.5 \mu\text{m}$ . It is unclear just why these occur or what we might do to keep them from occurring during fiber pulling. We are beginning to anneal the fibers in air after fabrication to see if that may reduce these losses. Annealing in air has been shown to decrease the attenuation in sapphire fibers and we can hope that a similar effect occurs in YAG fibers. In general, absorption by water present in the fiber and the source rods can be a problem as evidenced by the strong water absorption in the spinel fiber at  $3 \mu\text{m}$ . This same absorption also occurs in the spinel source rod.

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 crystalline has a viscosity close to that of water so any vibration or irregular motion during grow can lead to a diameter fluctuation. In our LHPG apparatus we control the laser power by virtue of a feedback signal from the laser micrometer. 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. In Fig. 7 we show two microphotographs of the same YAG fiber. The difference is that in one case there is poor diameter control (A) leading to a diameter variation of about  $\pm 8\%$  and in the other (B) there is

excellent diameter control of about  $\pm 1\%$ . The variation in fiber diameter shown in Fig. 7 occurs as a result of some movement of the fiber or source rod that is, for some reason, not well controlled. Sometimes this occurs when the fiber goes into or out of the tractor feed mechanism or, less often, if the source or fiber slips just a small amount in their respective feed mechanisms. We are working to improve our drive controls which seem to be the most significant source of diameter fluctuations.

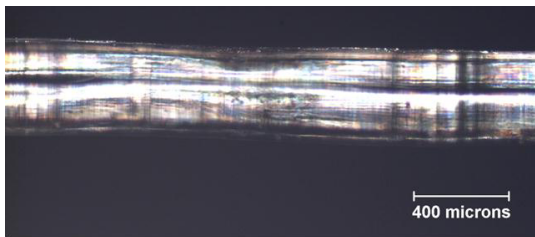


Figure 7(A) – Poor diameter control

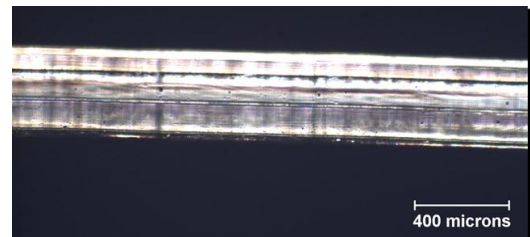


Figure 7(B) – Good diameter control

## 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. The emphasis in this work 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. This is particularly true of YAG fiber which, based on Nd:YAG lasers, should be capable of withstanding energy density produced by cw or pulsed high power lasers. The losses for our YAG and spinel SC fibers are much higher than expected. In the case of YAG we measure a loss at  $2.94 \mu\text{m}$  of about  $3 \text{ dB/m}$  compared to a calculated of about  $0.25 \text{ dB/m}$  at this Er:YAG laser wavelength. We attribute the higher measured losses to fiber diameter fluctuations and impurities. The YAG fiber that we have grown to date is not as uniform in diameter as the sapphire fiber that we have grown in the past. Work is underway to improve the diameter control and also to minimize the defects and

impurities by using purer starting material; annealing the fiber in air; and growing the fiber in a controlled atmosphere such as He or O<sub>2</sub>.

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