Reprinted from SPIE Vol. 1048—Infrared Fiber Optics

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Growth and optical properties of single-crystal sapphire fibers

D. H. Jundt, M. M. Fejer, and R. L. Byer
Applied Physics Department, Stanford University
Stanford, California 94305

ABSTRACT

Sapphire single-crystal fibers are very promising for applications in high power laser delivery systems operating in the mid-infrared, e.g. the Er:YAG line at 2.9 µm. The fibers are nontoxic and chemically resistant. Also, they are mechanically strong and can be bent to a radius of less than 10 mm without breaking. Fibers with diameters of 110 µm and lengths of up to 1 m were grown by the laser-heated pedestal growth method. The minimum loss of 0.5 dB/m was measured in the near infrared at 1064 nm. An absorption band centered at 400 nm resulted in losses of up to 20 dB/m. Absorption loss at the Er:YAG line is 0.88 dB/m for a fiber grown in an atmosphere of pure oxygen. A damage threshold higher than 1.2 kJ/cm^2 was measured for 110 µs long pulses, making tissue ablation feasible with fibers several meters long.

INTRODUCTION

Sapphire (α-Al_2O_3) has long been recognized as a good material for optical components due to its wide transparency range (0.3 µm - 4 µm) and favorable mechanical and chemical properties. These characteristics also make it a very attractive material for single-crystal fibers for energy delivery and sensor applications. Because the fibers are nontoxic and very strong, they seem ideal for use in medical delivery systems operating at the Er:YAG line at 2.94 µm where glass fibers are highly absorbing.

In this paper, we briefly review the process of growing single-crystal fibers. Typical dimension and tolerances of the fibers grown are then summarized. After discussing the scattering and absorption losses in the visible and near infrared region of the spectrum, we report absorption loss and damage threshold measurements at the Er:YAG wavelength.

GROWTH METHOD

To grow the sapphire fibers, we used a laser-heated miniature pedestal growth apparatus described in Ref. 1. The tip of a rod of source material was heated by a focused CO_2 laser, forming a molten liquid droplet. By dipping an oriented seed crystal (in our case with the z-axis along the growth axis) into the droplet, a suspended molten zone is produced with a shape governed by surface tension. The growth was then started, carefully controlling the ratio of the speeds at which the source rod is pushed into the zone and the fiber is pulled out. (See Fig. 1). The fibers grown should be of the same purity as the source-material since there was no contact of the liquid zone with crucibles or dies.

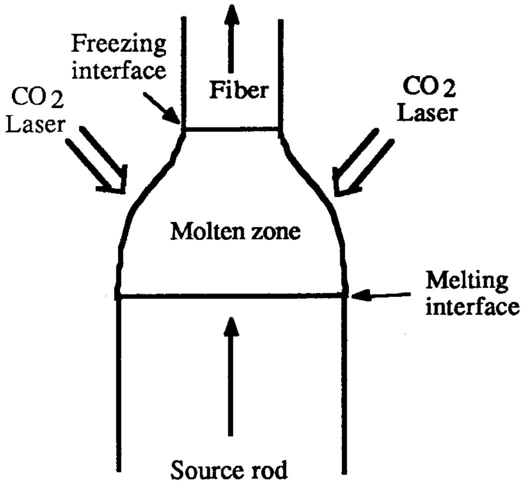

Fig. 1: The laser-heated pedestal growth process

Our sapphire fibers were typically grown in air at a speed of 4 mm per minute with a reduction ratio of source to fiber diameter of 3.5. The source rods were manufactured from material supplied by Crystal Systems Inc. and had a useful length of 6 cm. Since the mass flow into and out of the molten zone must be equal, the resulting fiber was $(3.5)^2 = 12.25$ times the source length used. The resulting fiber length typically was about 70 cm for a 6 cm source rod. Longer fibers could easily be grown by using longer source rods or using a two step reduction from a larger diameter source rod. The fiber diameter was typically 110 μm as determined by the source rod diameter and the reduction ratio.

The uniformity of the diameter of the fiber is an important factor influencing the scattering loss. An active stabilization loop was used to keep the laser power constant to within 0.5%. A diameter measurement system recorded the fiber-diameter during growth. The variations of roughly 4 % rms were caused mainly by fluctuations in the output power of the laser. The noise at spatial frequencies with a periods of less than 2 mm was small, because the molten zone damps out high frequency oscillations.

SCATTERING LOSSES

Two effects, scattering and absorption, contribute to the total loss in the sapphire fibers. As both are affected by the cleanliness of the surface of these unclad fibers, they were placed in teflon tubing for protection from environmental contaminants after cleaning them with methanol.

To measure scattering loss, a section of the fiber was placed in an integrating sphere. Laser light was launched into the fiber, and the guided power was measured by a detector at the output of the fiber. A photo-diode held in a port of the integrating sphere gave a signal proportional to the scattered power. The throughput of the integrating sphere and the response of the photo-diode were determined by directing a laser beam with known power into the sphere through one of the ports. Scattering was measured at different wavelengths ranging from the blue to the near infrared. The results are shown in Table I. The values are constant within experimental error.

ABSORPTION LOSSES

Absorption losses in fibers can be measured with high sensitivity using laser calorimetry (2). A schematic of the setup we used is shown in Fig. 2. The fiber was passed through a clear glass capillary tube that had an outer diameter of 2 mm. Scattered light thus could escape and did not contribute to the measurement. Thermo-couple junctions were arranged such that only the temperature difference between the clear and the reference tube was measured. The influence of fluctuations in the temperature of the environment was reduced by the fact that both tubes had the same thermal response times. The observed rise in temperature after turning on the laser was given by the product of the sensitivity of the apparatus, the guided power and the absorption coefficient. Sensitivity was increased by attaching several junctions to each tube and was measured with a heating wire in place of the fiber. The measured change in the thermo-couple voltage per dissipated power was 0.55 mV–m/W. The time needed after turning on the laser for the system to reach 50% of the final temperature difference was 24 s. The guided power was calculated from the measured output, taking into account the Fresnel reflection loss and loss caused by attenuation as the light travels from the point of measurement to the end of the fiber.

The results of the absorption measurements at six different wavelengths are shown in Table I. Absorption dominates scattering losses strongly in the visible and the mid-infrared. In the near-infrared, absorption is small and comparable in magnitude to the scatter losses.

Wavelength nm	Scattering loss dB/m	Wavelength nm	Absorption loss dB/m
457.9	0.156±0.02	457.9	17.4 ±0.8
488.0	0.174±0.02	488.0	6.30±0.09
514.5	0.156±0.02	514.5	4.6 ±0.15
632.8	0.130±0.02	632.8	1.3 ±0.2
1064	0.178±0.03	1064	0.28±0.08
		2936	1.7 ±0.2 (0.88±0.2)

Table I: Results of the loss measurements made for fibers grown in air. The data in parenthesis was measured for a fiber grown in oxygen.

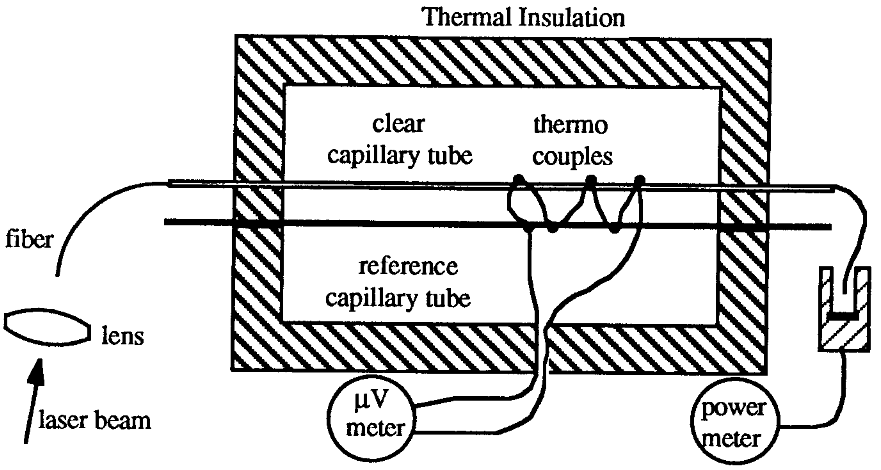

Fig. 2: Schematic diagram of the laser calorimeter.

To study the absorption in the blue region of the spectrum in greater detail, the setup shown in Fig. 3 was used. An arc lamp was imaged by a lens onto the plane of an aperture. The aperture let light pass from a small section of the arc that had a well-defined spectral emission. One end of the fiber was positioned in the aperture, the other end was placed inside an integrating sphere. The wavelength dependence of the intensity inside the sphere was measured with a 1 m spectrometer with a photo multiplier tube attached to its output slit.

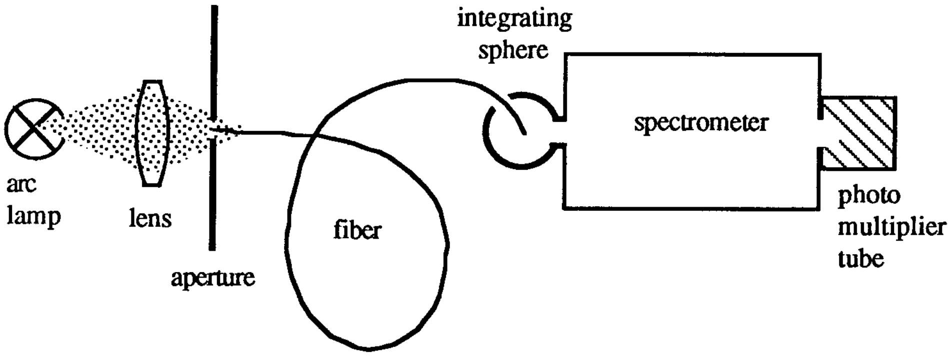

Fig 3 : Schematic diagram of setup to measure the wavelength dependence of loss.

The wavelength dependence of the apparatus was determined by removing the fiber and directing some light through the aperture onto the input port of the integrating sphere. The ratio of the data from the fiber and this calibration measurement was independent of the spectral emission of the lamp and the spectral response of the sphere, spectrometer, and photomultiplier:

$$\mathrm{Ratio} = C\left(\frac{1-n}{1+n}\right)^4 e^{-\alpha \ell}.$$

Here, n is the wavelength dependent refractive index of sapphire, ℓ is the length of the fiber, α is the power-loss coefficient (in our case dominated by absorption loss), C is a wavelength-independent, but unknown constant. Solving for α, one gets an equation with a constant offset of ln(C). The offset can be determined by matching data at a wavelength to the corresponding absorption number in Table I. The best accuracy for determining this offset was achieved using the small total loss of 0.5 dB/m in the near-infrared.

The resulting curve for α is shown in Fig. 4, along with the absorption losses measured at the laser wavelengths. There is a broad absorption band centered at 400 nm, with a peak absorption of 0.04 cm^{-1}. This band has been reported in γ-irradiated crystals and can be explained by the presence of V_{OH^-} centers (3). It would limit the use of the sapphire fibers for work in the UV and the blue-green. Annealing in a reactive atmosphere to control the H concentration might remove the color centers and reduce the absorption considerably (4).

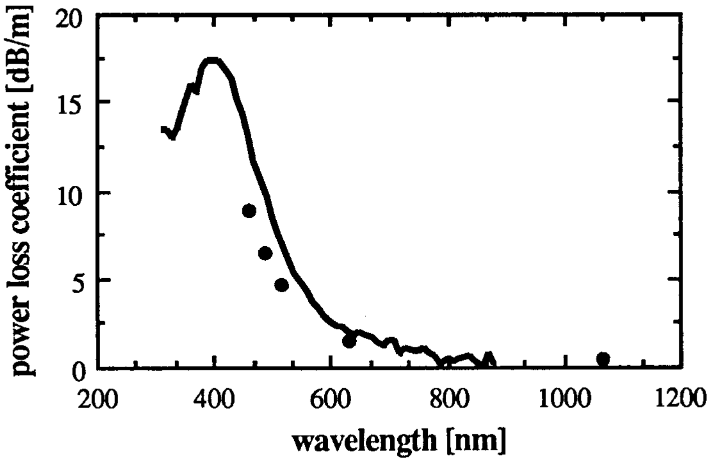

Fig. 4: The solid line is the total absorption measured with the setup of Fig. 4. Dots are absorption losses from Table I. The slight systematic discrepancy could stem from the low degree of spatial coherence of the lamp illumination at the input end of the fiber, leading to a high loss distribution of modes.

POWER DELIVERY SYSTEM

The feasibility of a laser angioplasty system operating with an Er:YAG laser and our sapphire fibers was investigated. This study was carried out at MCM Laboratories, Mountain View, CA with a Quantronix laser operating at 2.936 µm with a pulse-duration of 110 µs and a repetition rate of 3 Hz. The transverse mode-pattern could be controlled with an aperture inside the laser cavity. The beam was focussed with a 25 mm lens onto a fiber positioned a few mm behind the beam waist to uniformly illuminate the fiber end. The absorption loss was measured as described above and is shown in Table 1 for a fiber grown in air. A lower absorption loss of 0.88 dB/m was measured for a fiber grown in an atmosphere of pure oxygen.

To establish the damage threshold, the current that drives the flash lamp was gradually increased, and the fiber was periodically checked for damage. The energy icident on the input end of the fiber was estimated by recording the output power and correcting for propagation losses. No damage was observed for 130 mJ pulses hitting the 110 µm diameter fiber. A simple assumption of uniform intensity across the end face, yields a damage threshold of 1.2 kJ/cm^2. However, energy was delivered uniformly neither in space nor time: the laser was operated with multiple transverse modes, and exhibited temporal spikes which reached twice the average pulse power. Therefore, 1.2 kJ/cm^2 is merely a lower bound on the damage threshold; actual values are expected to be higher.

Sapphire fibers are strong: a bending radius of 4 mm has been demonstrated in a fiber 150 µm in diameter (5). They are also very stiff, having a Young's modulus nine times larger than that of glass(6). Assuming the same dimensions, it takes nine times as much force to bend a sapphire fiber as is required to bend a glass fiber. The force required to bend a long rod is proportional to $E\, d^4/\ell^3$ where d denotes the diameter, ℓ the length and E Young's modulus of the rod. Therefore, if the same flexibility is required for a sapphire fiber as for a glass fiber, the diameter of the crystal fiber should be reduced to 56% of that of the glass .

Ablation of postmortem human coronary tissue was demonstrated with an output energy of 6 mJ per pulse. Assuming a fiber delivery system 4 m in length with a loss coefficient of 1 dB/m, reflection losses of 8% at each end, and with an output energy of 6 mJ per pulse, the fiber input energy would be at least seven times below the damage threshold.

CONCLUSION

High quality single-crystal sapphire fibers of lengths limited only by the length of the source rod were grown. The fibers grown in air show a broad absorption band centered at 400 nm thought to be associated with V centers. Measured scattering losses were small and should be reduced still further by cladding the fibers. Total loss was dominated by absorption. Laser light at 2.94 μm was guided with a tolerable total loss of roughly 1 dB/m for fibers grown in an oxygen atmosphere. The damage threshold at this wavelength was at least 1.2 kJ/cm^2 for a 110 μs long pulse, making the system very attractive for medical applications. Power handling might further be improved by reducing hydroxyl impurities in the fiber and improved end polishing techniques.

ACKNOWLEDGMENTS

The authors wish to thank Greg Magel for helpful discussions. The help of Janet Brooks and Michel Digonnet with the Er:YAG measurements is appreciated.

This work was funded by the Air Force Office of Scientific Research, Contract AFOSR-88-0354 and by the Joint Services Electronics Program, Contract N00014-84-K-0327.

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