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GROWTH OF OPTICAL QUALITY SAPPHIRE SINGLE CRYSTAL FIBERS

Dieter H. Jundt, Martin. M. Fejer, and Robert. L. Byer
Applied Physics Department, Stanford University,Stanford, California 94305

ABSTRACT

Void-free sapphire single crystal fibers with diameters of 110 μm and 60 μm and lengths of over 2 m have been grown by the laser-heated pedestal growth method. The growth dynamics of the floating zone was studied and shows the features expected from a simple theoretical model. Optical losses have a minimum of 0.5 dB/m in the near infrared at 1064 nm. An absorption band centered at 400 nm results in losses of up to 20 dB/m in the visible. The fibers have potential applications in high temperature thermometry and in delivery systems for laser surgery. Absorption losses of 0.88 dB/m with a damage threshold higher than 1.2 kJ/cm^2 at 2936 nm made tissue ablation feasible with fibers several meters in length.

INTRODUCTION

Sapphire (α-Al_2O_3) single-crystal fibers are well suited for sensor applications in high temperature or chemically hostile environments,[1] as well as for use in medical power–delivery systems operating at the 2936 nm Er:YAG wavelength[2] where silica glass fibers are highly absorbing. Sapphire fibers are stronger than other mid–infrared transmitting fibers[3] and chemically inert.

GROWTH METHOD

To grow the sapphire fibers, we used a laser-heated pedestal growth apparatus.[4] The tip of a rod of source material[5] was heated by a focused CO_2 laser, forming a molten 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 was produced. 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). For growth under equilibrium conditions, the reduction ratio of source to fiber diameter is given by

$$r_f / r_m = \sqrt{v_m / v_f} \qquad (1)$$

where r_m and r_f are the source rod and fiber radii, and v_m and v_f denote the speeds of the source rod and the fiber, respectively.

The 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. A 6 cm long, 0.4 mm diameter source rod generates a 70 cm long 110 μm diameter fiber. Fibers as long as 2.5 m were grown by using a two–step reduction from a 0.8 mm diameter source rod.

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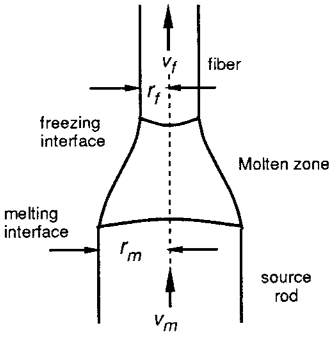

Fig. 1: The laser-heated pedestal growth process and the parameters used to describe the molten zone shape.

Diameter variations, particularly those of high spatial frequency, are the major source of scatter loss in void–free single–crystal fibers. It is thus useful to characterize the influence of perturbations to the growth system, both as an aid in optimizing the growth parameters and for the design of closed–loop diameter–controllers. Following the analysis of meniscus–controlled growth in Ref. 6, one can show that if certain thermal effects are neglected (essentially assuming that the length of the molten zone is constant), the radius of the fiber responds to perturbations as a second order system, with damping and natural frequency dependent of the dimensions of the molten zone.[7, 8] One therefore expects that the response will roll off at 12 dB/octave as the modulation frequency ω is increased. ω is defined as $2\pi/\Lambda$ where Λ is the length of a period measured along the fiber. To quantify these ideas, we define a transfer function H(ω) relating the change in the diameter of the fiber to the amplitude of a periodic perturbation.

$$H(\omega) = {}^{\delta r_f}\!\big/_{\delta v_i} \tag{2}$$

where δr_f is the variation in the fiber diameter normalized to the steady–state value, and δv_i ($i = m, f$) is the variation in the pull or feed rate normalized to the steady–state value. From the previous discussion, we expect $|H_i|$ to approach 1/2 for $\omega \to 0$, $|H_i| \sim \omega^{-2}$ for $\omega \to \infty$, and a peak in the response at a natural frequency on the order of a reciprocal molten zone length.

Measuring the transfer functions

To test these predictions, we measured the magnitude of the transfer functions by periodic modulation of the pull or feed rates. Random diameter variations of approximately 2% complicated the signal analysis, so both time and frequency domain measurements were made in order to check the internal consistency of the results.

For the frequency domain measurements, we modulated either v_m or v_f with an amplitude δv of 5-15% at fixed frequency ω for fiber lengths corresponding to about 10 periods. The magnitude of δr_f in (2) was estimated from the fourier transform of the measured[9] diameter variations. The results are shown as circles in Fig. 2.

The time domain method recorded a step-response for a system under closed–loop proportional control.[10] The setpoint of r_f was suddenly changed to a new value and the response in r_f was monitored. The error signal, i.e. the deviation of r_f from its target value, was used to control the speed of either v_m or v_f to correct for the error. A proportional gain of 10 was used. For example, if the radius of the fiber was too small by δr_f, either either v_m or v_f (depending which transfer function was investigated) was changed by ten times the amount that would be necessary to increase the equilibrium value of r_f by δr_f. The transfer function of such a closed loop system is very different from the open loop system and is given by:

$$H^{cl} = \frac{H}{1 + 10H} \tag{3}$$

where H is the open loop transfer function. Sixteen step responses were measured and averaged to further increase the signal to noise ratio. A numerical Laplace transform on the time derivative of the step response was used to obtain $H^{cl}(\omega)$. H could then be calculated from equation (3). The results for H_f and H_m are shown in Fig. 2. The agreement with the measurements done by sinusoidally modulating the speeds is very good.

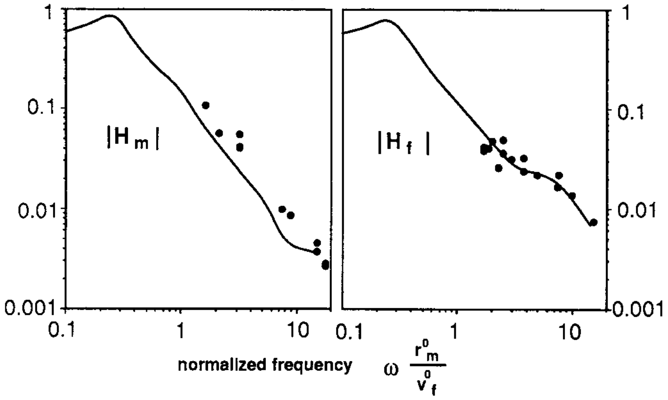

Fig. 2: Magnitude of the transfer functions H_f and H_m measured by recording a step response (solid line) and by sinusoidal modulation (circles) for a 460 μm diameter source rod, a molten zone length of 310 μm, and a diameter reduction ratio of 3.5. The period corresponding to the peaks in H_f and H_m is 5 mm.

The experimental results are in general agreement with the predicted behavior, with the correct limit for low frequencies, peaking at a frequency comparable to the reciprocal zone length, and monotonic roll–off at high frequencies. There are also significant differences. In particular, the slope in the high frequency region is smaller than the predicted ω^{-2}, and the peak response occurs at a somewhat lower frequency than the predicted natural frequency. We believe these discrepancies are due to the neglected thermal effects, which are currently under study.

We also measured the diameter of fibers grown at a constant rate for optical applications. The power spectrum of the random diameter variations peaked at the same frequency as the transfer functions, and rolled off at higher frequencies consistent with broadband input noise filtered by the transfer functions. The effect of these ~3% rms amplitude variations on the optical losses are discussed in the following section.

OPTICAL CHARACTERIZATION

The fibers grown have no inhomogeneities as verified by optical microscopy. They are unclad (core index = 1.78) and therefore highly multimode. Despite the large NA, the measured modal power distribution after propagating a laser beam through a 0.71 m long 110 μm-diam fiber has a full angle at half intensity of only 11°. This distribution was only weakly sensitive to bends in the fiber and input launching conditions.

Low propagation loss is necessary for optical applications of sapphire fibers. Two effects, scattering and absorption, contribute to the total loss.

Scatter losses

Scatter losses were measured by launching laser light into the fiber and measuring scattered radiation along the fiber with a photodiode in a 6 cm diameter integrating sphere.[11] As the scatter losses are dependent on the modal power distribution,[12] there is no unique loss figure for a given wavelength. Since the angular distribution of the output light was found to be largely insensitive to moderate fiber bending, we expect the loss measurements, taken near the output end of the 0.71 m long 110 μm-diam fiber, to be representative of those for actual applications. Results of scatter loss measurements are shown in Table I. The scatter losses for all the wavelengths measured are the same, 0.16 dB/m, within experimental accuracy. Such low losses are consistent with the predominantly low spatial frequency of the diameter variations, which do not contribute as strongly to scatter losses as would those at higher frequencies.

Absorption losses

Calorimetry was used to measure absorption losses in fibers at several laser wavelengths.[11,13] The results at six different wavelengths for a fiber grown in air are shown in Table I. A lower absorption loss of 0.88 dB/m for 2936 nm light was measured for a fiber grown in an atmosphere of pure oxygen, probably due to reduced OH incorporation. From the data in table I, it can be seen that absorption dominates scattering losses in the visible and the mid-infrared. In the near-infrared, absorption is small and comparable in magnitude to the scatter losses.

Table I. Scatter and absorption losses for a 110 μm-diam fiber grown in air. The data point in parentheses is for a fiber grown in an oxygen atmosphere.

Wavelength nm	Scatter loss dB/m	Absorption loss dB/m
457.9	0.156± 0.02	8.7 ± 0.8
488.0	0.174± 0.02	6.30± 0.09
514.5	0.156± 0.02	4.6 ± 0.15
632.8	0.130± 0.02	1.3 ± 0.2
1064	0.178± 0.03	0.28± 0.08
2936	....	1.7 ± 0.2 (0.88±0.2)

To study the absorption in the blue region of the spectrum, an arc lamp was used to illuminate one end face of the fiber. The other end of the fiber was placed inside an integrating sphere. The wavelength dependence of the output of the sphere was measured with a 1 m spectrometer and photomultiplier detector. The wavelength dependence of the throughput of the measurement system was removed by normalizing the data to the signal obtained by directly illuminating the input of the integrating sphere. The overall scale factor was set by comparison with the 633 nm data point in table I.

The resulting curve for the attenuation constant, α, in a c-axis fiber is shown in Fig. 3, along with the absorption losses measured at the laser wavelengths. There is a broad absorption band centered at 400 nm (corresponding to a photon-energy of 3.1 eV) associated with hole (or V type) centers.[3] The peak absorption is 18 dB/m (0.04 cm^{-1}).

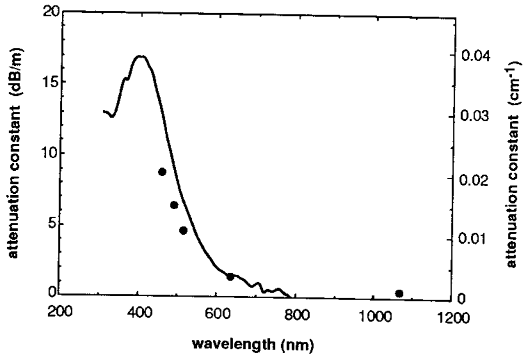

Fig. 3: Loss coefficient in 110 μm diameter fiber. The solid line depicts the attenuation constant measured with the arc lamp apparatus. Circles are absorption losses from Table I measured by laser calorimetry.

POWER DELIVERY SYSTEM

The possibility of laser surgery using sapphire fibers was investigated with an Er:YAG laser operating at 2936 nm with a pulse-duration of 110 μs and a repetition rate of 3 Hz. To establish the damage threshold, the flash lamp energy was slowly increased. No damage was observed for 130 mJ pulses falling on the 110 μm diameter input face of the fiber, corresponding to a damage threshold in excess of 1.2 kJ/cm^2 (an intensity of 11 MW/cm^2). We believe that the damage threshold could be increased by improving the polishing techniques and by annealing the polished surface.[14] Ablation of postmortem human arterial tissue was observed with 6 mJ per pulse at the output of the fiber. 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 incident fluence on the fiber would be at least seven times below the damage threshold, demonstrating the feasibility of sapphire single-crystal-fiber power delivery systems.

CONCLUSION

High quality single-crystal sapphire fibers of lengths up to 3 meters were grown. Predictions from a simple theoretical model for the dynamics of the molten zone agree with measurements. The fibers show a broad absorption band centered at 400 nm. 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 total loss of approximately 1 dB/m for fibers grown in an oxygen atmosphere.

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