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# Low-loss single-crystal sapphire optical fibers

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## Abstract

Single-crystal sapphire ($\alpha$-$A\ell_2O_3$) fibers are potentially useful in a wide variety of optical applications, particularly those involving high-power or high temperature light-guiding, over the wavelength range from 0.24 um in the ultraviolet to 4.0 um in the mid-infrared. These fibers are routinely grown at rates of up to 8 mm/min, and with diameter stability of better than 0.5% rms under feedback control. Recent measurements on unclad 150 um diameter fibers show fundamental mode scattering losses of about 0.3 dB/m in the visible and less than 0.07 dB/m at 3.39 um.

## Introduction

Single-crystal sapphire ($\alpha$-$A\ell_2O_3$) has long been recognized as a good material for optical windows, because of its wide range of high transmission (~ 0.24 - 4.0 um), its low bulk scattering, its durability and hardness, and its resistance to chemical attack. These same properties make sapphire an interesting candidate for an optical fiber material. In addition, the high melting point of sapphire fibers, ~ 2045°C, makes them potentially useful for light-guiding or thermometry in high-temperature environments, or for the transmission of high average power laser light in industrial or medical applications.

In this paper, we first briefly review the method we have used to grow optical-quality single-crystal sapphire fibers. We then summarize fiber growth results, including improved diameter stability attained through the active control of fiber diameter during growth. After a discussion of recent measurements of the scattering losses of unclad fibers at several wavelengths from the UV to the mid-IR, we conclude with a summary of the present state of development of sapphire optical fibers and suggestions for further research.

## Growth method

The process we have chosen to produce single-crystal refractory oxide fibers, known as laser-heated miniature pedestal growth,[1] is illustrated in Fig. 1. The tip of a rod of source material is heated by focused radiation from a $CO_2$ laser, forming a molten liquid droplet. An oriented seed crystal is then dipped into this droplet, and withdrawn slowly until the molten zone assumes the shape, governed by surface tension, shown in the figure. To grow a fiber, the seed pulls material out of the molten zone while fresh source material is being moved into the melt. The fiber can be grown slightly more than a factor of three smaller in diameter than the source by appropriate choice of the translation rates. This limit to the diameter reduction in a single growth step is dictated by instability of the growth process at higher diameter reduction ratios.

Several features of this process should be noted. First, materials with a high melting temperature can be grown. Second, as this is a technique using no crucibles or dies, the purity of the grown fiber may be limited only by the purity of the source material. Finally, since the material is melted and not just softened, diameter fluctuations at the freezing interface that arise from unstable heating or translation are permanently frozen into the fiber.

A block diagram of our fiber growth apparatus,[2-5] as configured for these experiments, is presented in Fig. 2. We have used this same apparatus to grow fibers of Nd:YAG and $LiNbO_3$. The controlled atmosphere chamber contains atmospheric-pressure air and is currently used in sapphire growth merely to prevent perturbation of the growth by wind currents. The copper focusing mirrors are designed to symmetrically heat the molten zone and the fiber translators are of a continuous belt-driven design which can grow unlimited lengths of fiber, and which are driven by phase-locked dc motors for accurate constant speed.

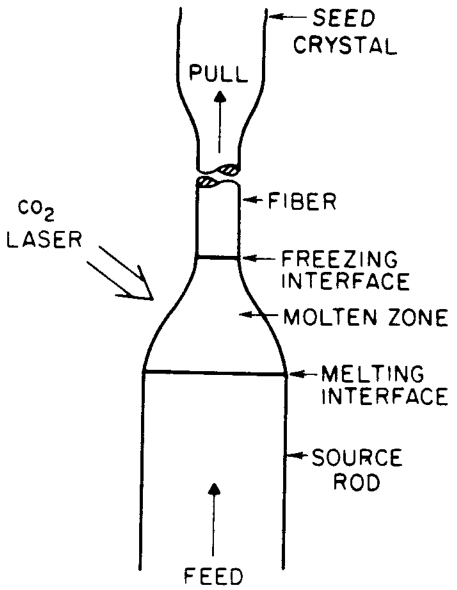

Fig. 1--The laser-heated miniature pedestal growth process

In spite of all these design preca it was determined that for some applica fibers of better diameter uniformity tha were obtained without feedback were requi To this end, we constructed a real-time,no contact diameter variation monitoring system. The fiber diameter measurement system has a diameter resolution of better than 0.01%, a working distance of 160 mm, a measurement rate of up to 1 kHz, and an axial resolution of less than 10 $\mu m$ along the fiber length. As indicated in Fig. 2, this system can be used in conjunction with a simple analog proportional controller to feed back to the pull/feed motor speed ratio in order to stabilize the diameter of a growing fiber.[7]

## Growth results

Sapphire fibers are typically grown with a diameter reduction of 3:1 from a centerless ground single crystal source rod about 500 $\mu m$ in diameter. It is possible to use these fibers as source material for further growth steps if more diameter reduction is required. Fibers can be grown with either the c or a crystallographic axis along the fiber axis. Our research fibers are typically 4.5 cm long, but both open-loop and diameter-controlled fibers have been grown as long as 20 cm. The $CO_2$ laser power needed to grow a 150 $\mu m$ sapphire fiber from a 500 $\mu m$ source rod is approximately 5 watts. Sapphire is an easy material to grow with this technique; we have grown more than 200 sapphire fibers since June of 1983.

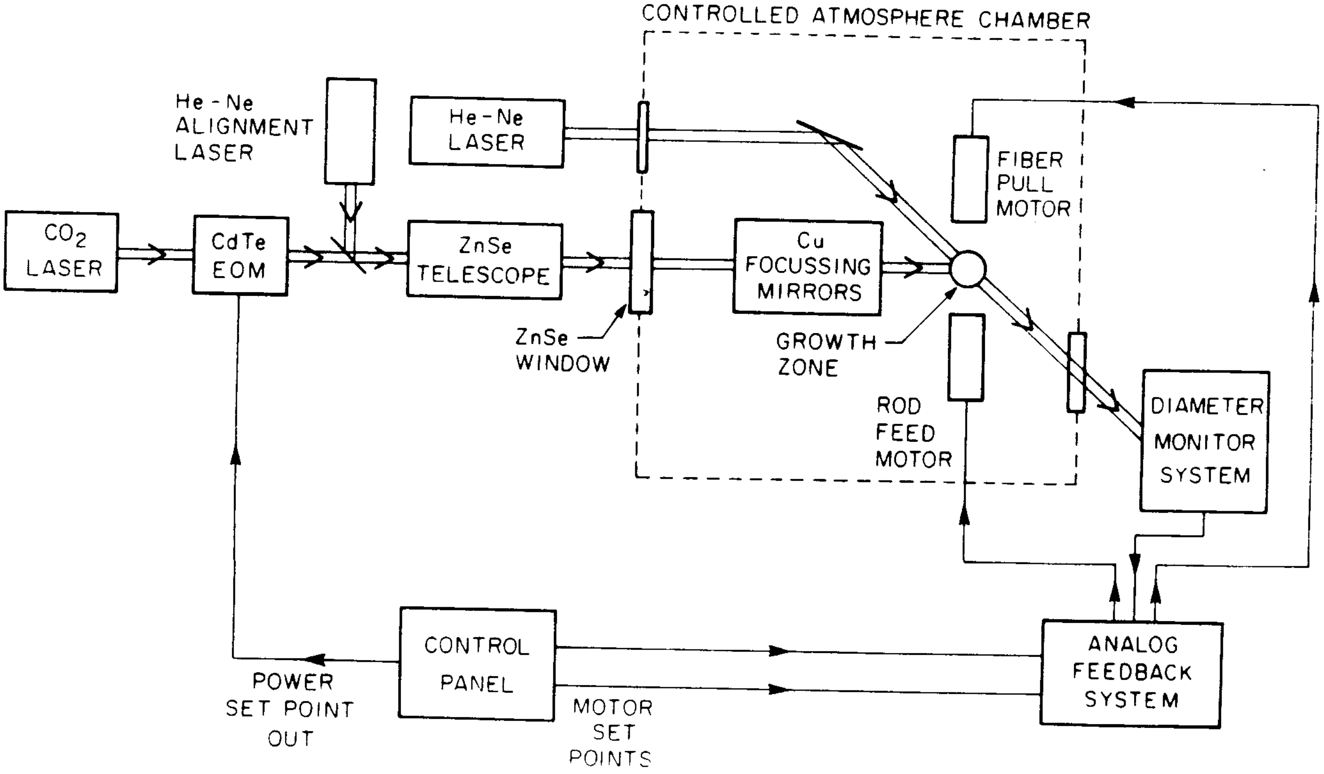

Fig. 2--Block diagram of the growth apparatus

The *cross-sectional geometry* of single crystal sapphire fibers, as shown in Fig. 3, reflects the crystal symmetry, as it does in bulk crystal growth. The cloudy areas in the centers of these fibers are regions of tiny "bubbles", or microvoids, which occur at growth speeds of over 8 mm/min (or 0.5 m/h). Nightingale[5] has proposed that microvoid formation may be the result of constitutional supercooling. If this is the case, then measures taken to steepen the temperature gradient at the freezing interface, such as growth in a helium atmosphere to increase convective heat loss from the fiber, could increase the threshold speed for microvoid formation and allow higher growth rates. The fibers used in these studies were grown at 1 mm/min, and therefore do not exhibit microvoids.

Figure 4 shows a 150 μm c-axis sapphire fiber bent into a loop 7.8 mm in diameter. Upon being released, the bent fiber immediately springs straight. This fiber sample finally broke when the loop was pulled to a diameter of ~ 5 mm. This bending radius corresponds to a maximum strain of 3%, approaching the theoretical cohesive strength of solid matter,[8] and achieving the maximum strength measured in much smaller sapphire whiskers.[9] In addition to *attesting to the crystalline* perfection and smoothness of the sapphire fiber, this experiment also suggests its inertness, since this unclad and uncoated fiber was exposed to the atmosphere for almost a year before the bending test.

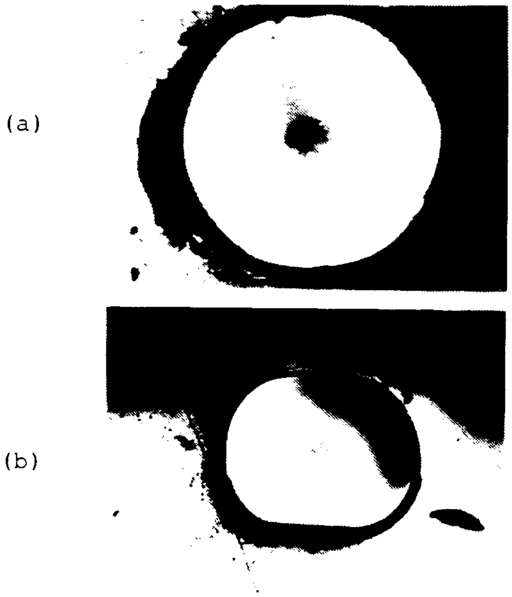

Fig. 3-- Polished cross-sections of 150 μm diameter sapphire fibers grown at 23.2 mm/min. (a) c-axis along fiber axis; (b) a-axis along fiber axis. Note microvoids (see text) near fiber centers. [From Ref.5].

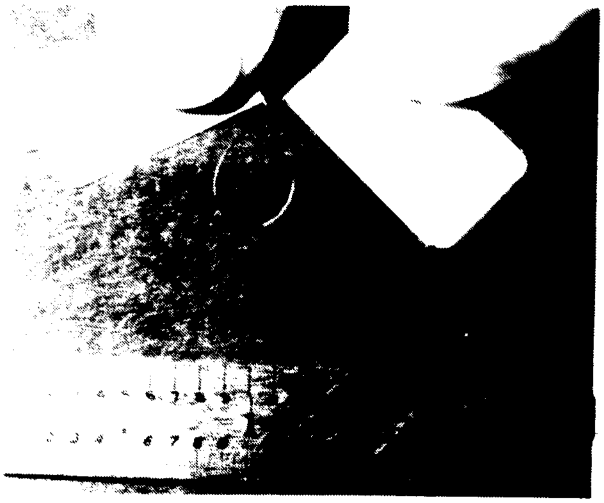

Fig. 4--Demonstration of flexibility of a 150 μm c-axis sapphire fiber. (Ruler is marked in inches).

The effect of feedback control on diameter stability[7] is illustrated in Fig. 5. This figure compares the plots of diameter vs length, obtained by the diameter monitoring system during growth, for a 1 cm section from each of two 150 μm c-axis fibers grown at 1 mm/min on the same day. Sample S-213, represented by the dashed line, exhibits the 2% rms diameter fluctuations typical of fibers grown without feedback. It is believed that the rough periodicity observed reflects a natural resonance of the pedestal growth process. Fiber S-209, represented by the dark solid line, was grown under nominally identical conditions, except that a proportional feedback gain corresponding approximately to a 44% speed change of the fiber pulling motor per 1% fiber diameter change was used. (Applying much higher feedback gain results in diameter oscillations which exponentially increase in amplitude until the fiber pinches off, terminating growth). This fiber exhibits fluctuations of under 0.2% rms amplitude, and the fluctuations appear less periodic, or at least of higher frequency. In both cases, the $CO_2$ laser power was held constant. The residual diameter noise on S-209 may largely be due to the ~ 1% fluctuations in laser power.

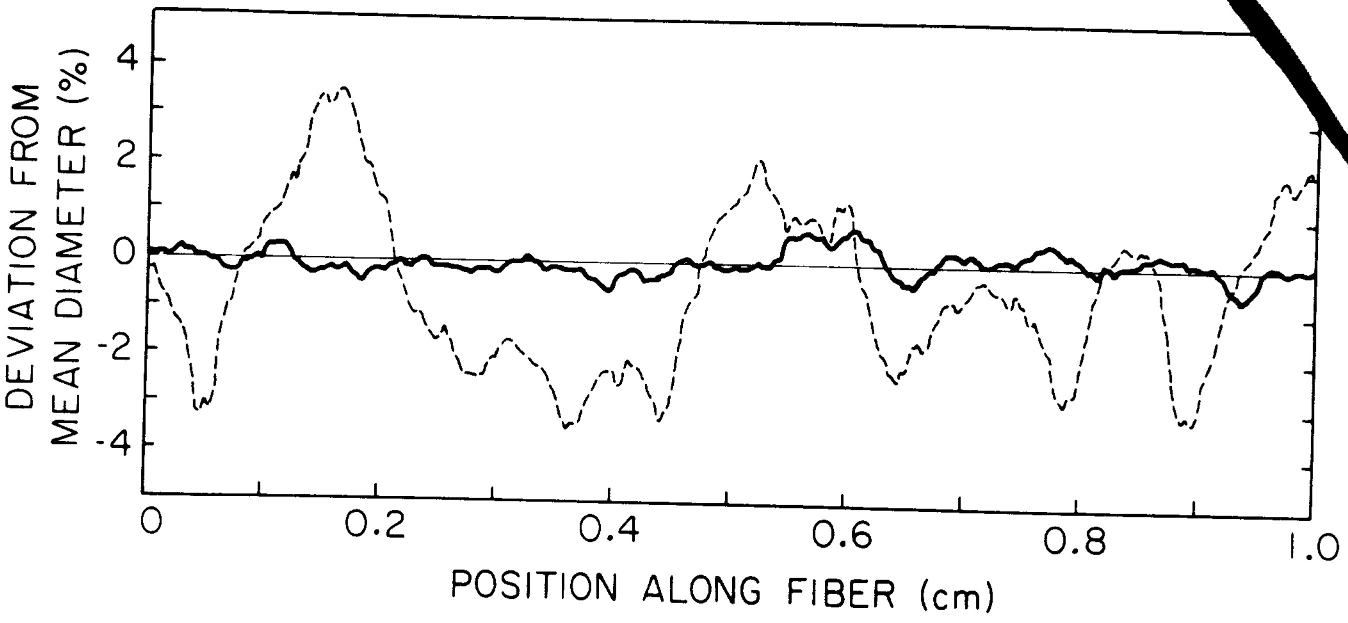

Fig. 5--Effect of feedback control on diameter stability. Dashed line shows 2% rms fluctuations on S-213, a 150 μm fiber grown without feedback; dark solid line shows tenfold improvement for S-209, grown using feedback diameter control.

Because of the unique ability of the crystal growth process to "freeze-in" diameter or composition fluctutations along an optical fiber, we have considered the possibility of making fiber structures with intentionally-modulated diameters or dopant levals. These structures may find eventual application in fiber devices with distributed Bragg filters or mirrors. The result of our first experiment along these lines[7] is shown in Fig. 6. Application of a 3-Hz sinusoidal modulation in fiber pull speed (around a mean pull rate of 1 mm/min) resulted in diameter modulation with a spatial period of about 5 μm. It is reasonable to believe that practical first-order gratings could be made by using an increased modulation frequency, a slower mean pull rate, laser power modulation, or some combination of the above.

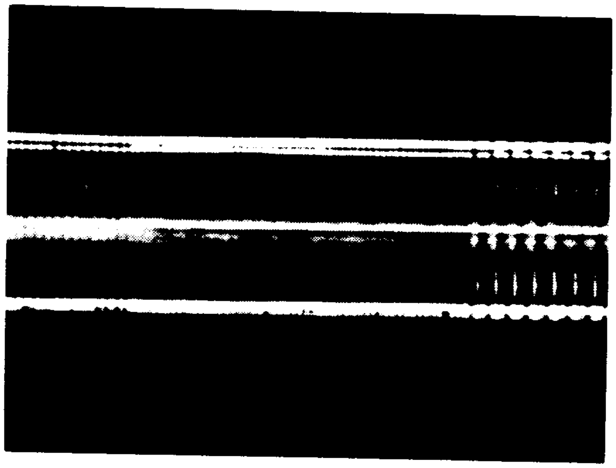

Fig. 6--Intentional periodic modulation of fiber diameter. The shortest period ripples (center) have a spacing of ~ 5 μm.

## Scattering losses

Earlier optical measurements[3] led us to the conclusion that total fiber attenuation in the visible was dominated by surface scattering resulting from diameter non-uniformity. Bulk scattering should be negligible in a single-crystal material with no grain boundaries, inclusions, or micro-voids, and a small deviation from perfect roundness should not contribute to the loss of low-order modes if the cross-sectional shape is constant along the length of the fiber. The availability of diameter. controlled fibers motivated us to make a more quantitative investigation and to extend our experiments to both shorter and longer wavelengths.

The apparatus used in our scattering loss measurements is indicated schematically in Fig. 7. The fiber under test is cleaned and threaded through a custom 25-mm diameter integrating sphere.[10] The output plug has a small hole in it to pass the fiber, yet collect small-angle forward scattering; a baffle in the sphere prevents the detector from seeing direct light from the fiber. Light from a laser source is chopped and directed at the polished input face of the fiber. Care is taken to block light which is not launched into the fiber from entering the sphere. The fiber and sphere are translated and tilted as a unit to optimize the coupling to the fundamental mode of the fiber. This optimal condition can be observed both as a minimum in the scattering signal and as a narrow farfield mode pattern on a screen at the polished output end of the fiber. The detected scattered light is measured with a lock-in amplifier. Then without disturbing the launching conditions, the sphere is translated so as to just bring the output end of the fiber into the integrating sphere, and a solid plug is placed in the output port. In this position, scattered light plus fiber throughput is measured. By taking the proper ratio, the scattering loss is obtained. The input coupling efficiency, detector sensitivity, and sphere throughput all cancel using this technique.

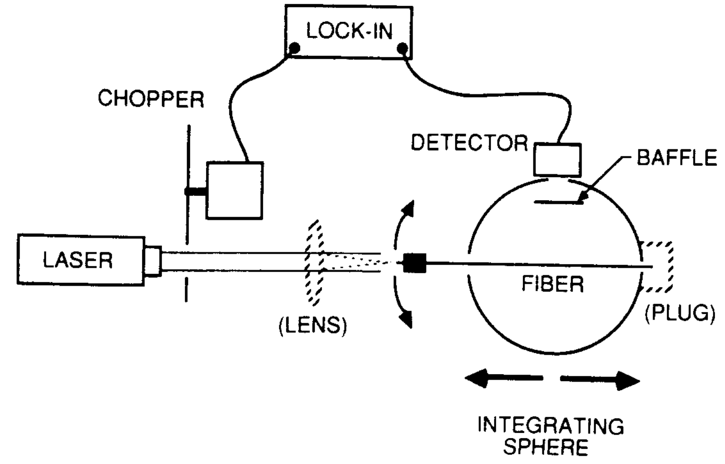

Fig. 7--Schematic diagram of the scattering loss measurement apparatus.

This measurement was performed on 150 μm c-axis samples S-209 and S-213 (the two samples of Fig.5) at the 0.325, 0.488, 0.5145, 0.6328 and 1.064 μm laser wavelengths using a barium sulfate coated sphere and silicon photodiode. The chopping frequency was ~ 440 Hz. The most consistent results were obtained with no light-launching lens, and fundamental-mode launching conditions. Since the fiber was capable of supporting many guided modes, careful adjustment of the fiber tilt was required to obtain single-mode propagation. A factor of approximately two greater loss was measured when launching multiple modes.

At the 3.392 μm wavelength, a diffuse gold coated sphere and pyroelectric detector were used for the measurement. The chopping frequency was ~ 10 Hz to optimize the detector performance. The low laser power, small scattered signal, low detector sensitivity, and high detector noise necessitated the use of a lens to increase the signal, and a lock-in time constant of 100 s. It was thus difficult to optimize the fiber tilt for single-mode launching.

The results of the scattering loss measurements are plotted in Fig. 8. The UV to near-IR losses cluster around 0.3 dB/m. The measured value at 3.392 μm of 0.07 dB/m should properly be considered an upper bound to the actual value for the reasons just stated. Many factors could explain the apparently higher scattering loss of the diameter-controlled fiber, but considering the error bars, perhaps it is best simply to say that diameter control did not lead to a significant lowering of scattering loss. The drop in loss at 0.325 μm could be due to incomplete guiding of the light in the short (4.5 cm) fiber samples.

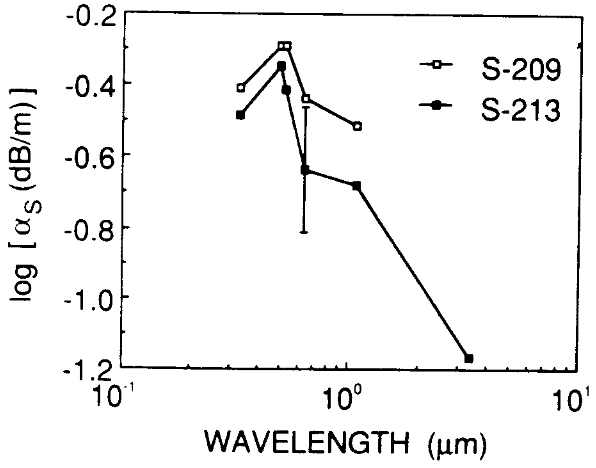

Fig. 8--Measured scattering losses vs wavelength. Closed squares are for fiber grown without feedback diameter control. Error bar applies to all points except longest wavelength point, which is an upper bound.

These scattering values are still seven orders of magnitude greater than Brillouin scattering, which is theoretically predicted to be the largest contribution to bulk scattering in single crystals.[11] We calculate that a minimum total loss of 0.08 dB/m should occur at 2.7 μm, using our interpolated scattering loss data, and assuming published values of the intrinsic bulk absorption.[11,12] This is a reasonable assumption since the only obvious extrinsic absorption found in our source material[13] using Fourier transform infrared spectroscopy was a group of narrow peaks between 3.0 and 3.1 μm, possibly attributable to $OH^-$. Better values for absorption will be obtained using a fiber calorimeter which is now under construction.

## Conclusion

Single-crystal sapphire fibers can be grown routinely, at speeds up to 8 mm/min, and with a diameter stability of better than 0.5% feedback controlled. The fibers are chemically inert, mechanically strong and transparent from the UV to the mid-IR. Measured fundamental-mode scattering losses of unclad 150 μm c-axis fibers are 0.3 dB/m in the visible and less than 0.07 dB/m at 3.39 μm. Elaborate diameter control techniques are probably not necessary for simple light-guiding. Intentional diameter modulation, however, may eventually lead to interesting fiber devices.

Future work should prove that better diameter control can lead to lower scattering losses. Higher-speed growth, fiber cladding and the use of non-crystalline source material for the growth of longer fibers are all technological issues which need to be addressed. Single-crystal sapphire fiber development has nevertheless reached the stage at which the fibers are ready for immediate use in some applications requiring their unique properties.

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