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Abstract

This paper presents results of pedestal growth of 30 - 500 μm diameter single crystal refractory oxide fibers. Growth instabilities are discussed and an improved second generation system is described.

Introduction

The unique combination of material properties and geometry offered by single crystal optical fibers offer intriguing capabilities in a variety of optical devices. Three materials grown at Stanford University this year typify the broad range of potential applications. The large nonlinear coefficients of $LiNbO_3$ suggest a number of modulators, signal processors and parametric sources. Minature lasers made from Nd:YAG fibers[1] have been known for several years. Such fibers might also be used as in-line amplifiers in conventional glass fiber systems. Sapphire's high melting point, mechanical strength, resistance to chemical attack and favorable optical and thermal properties make it an excellent candidate for monitoring temperature or optical radiation in hostile environments and for transmission of high power laser beams.

The realization of such devices, particularly those involving nonlinear processes, requires the growth of fibers with stringently controlled optical and structural uniformity. The control of random diameter variations is one major concern. The tolerance for these perturbations depends in detail on the particular application under consideration. A reasonable target value applicable to many cases of interest is a fractional deviation of one part in a thousand.

The efficiency of a large class of nonlinear interactions is proportional to the optical intensity in the fiber. For fixed optical power the efficiency scales inversely with the square of the fiber diameter. Thus another clear goal is the minimization of fiber diameter. Based on growth experience gained during the past year, a reasonable fiber diameter is 25 microns.

The optimal technique to achieve these goals in a variety of materials is not yet clear. The methods thus far proposed fall into four categories: hot rolling, edge defined growth and its varients, Bridgman growth in capillary tubing, and miniature pedestal growth.

Polycrystalline metal halide fibers of good optical quality have been produced by the hot rolling technique.[2] However, extension of this method to higher melting point materials, and the annealing of the polycrystalline fibers to single crystal form have yet to be demonstrated.

Edge defined growth techniques have been applied successfully to a number of materials, including metal halides [3] and sapphire.[4] This technique has the advantages of producing fibers of a well defined cross-section. It is also relatively insensitive to perturbations in the growth conditions.[5] A problem that arises in both this process and the capillary Bridgman approach is the necessity of finding appropriate crucible and die materials. In addition, the small diameter capillaries used in the Bridgman technique would have to be produced with the same diameter tolerances that apply to the finished fiber, as any perturbations in the capillary are replicated in the fiber.

The fiber growth technique that we have chosen to pursue is the pedestal growth method first applied to fiber growth by Burrus and Stone.[6] The most obvious advantage of this system is the removal of the constraints imposed by crucible techniques. This flexibility is of some importance to our application, since we anticipate surveying a variety of materials. As an example we note the growth this year of calcium scandate,[7] a material not previously obtained in single crystal form. Two other advantages discussed in more detail in later sections are the potential for introducing periodic diameter variations to produce distributed Bragg mirrors and periodic poling of ferro-electric crystals to enable quasi-phasematching.[8]

The price one pays for the considerable flexibility of this growth technique is the concurrent lack of inherent stability. As will be discussed, several factors must be tightly controlled for stable growth to proceed. We have not yet achieved this goal but improvements to the present system and a second generation growth station described below are expected to yield well diameter controlled fibers suitable for device applications.

The first generation fiber growth system

Growth Station

Our first implementation of a pedestal growth station is illustrated in Fig. 1.

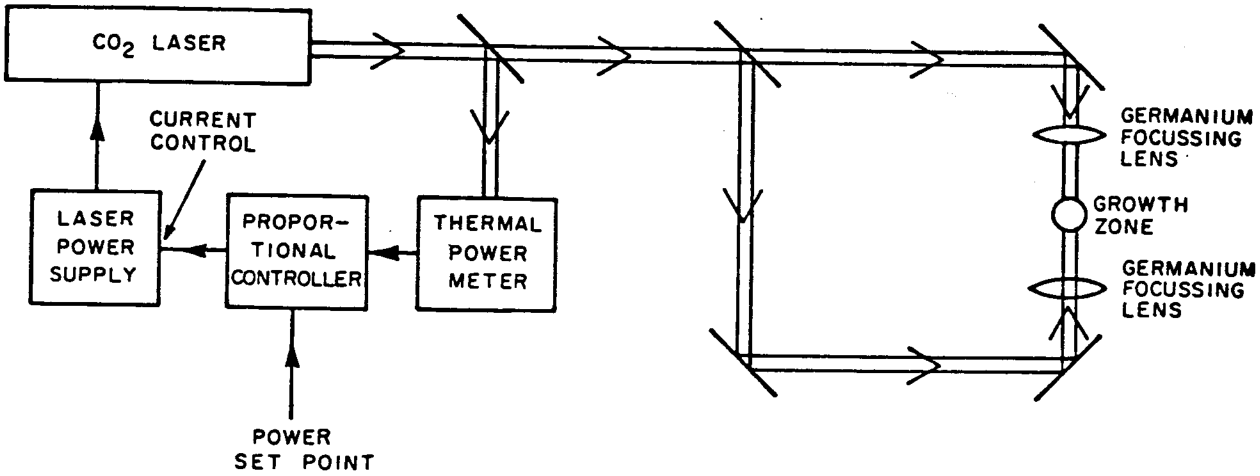

Figure 1. Block diagram of first generation growth system

The heat source is a 50 watt CO_2 laser (Coherent Model 42). The laser output power is monitored with a slow thermal power meter (time constant ~ 1 sec) and is stabilized by feedback to the tube current based on the monitor signal. A 50/50 beam splitter divides the beam into two parts which are delivered through a series of mirrors to two focussing lenses (f.ℓ. = 12.5 cm), which are arranged daimetrically opposed to one another to illuminate the melt zone with bilateral symmetry. The minimum focal spot size is 250 microns.

The source rod is fed into the melt zone and the fiber pulled from it by means of miniature three jaw chucks connected to independently driven lead screws. Motor speed control is based on a generator feedback system with approximately 1% stability. Both chucks are also provided with variable rate rotation about their axes.

Fiber growth. Growth proceeds by feeding the source rod into the focus of the laser beams until a molten bead, held in place by surface tension, forms at the rod tip as illustrated in Fig. 2. A seed crystal is dipped into the bead, then source rod feed and fiber pull motors are started simultaneously. The growth rate is somewhat material dependent, but typically lies in the range 1 - 10 mm/min, though sapphire has been grown as rapidly as 50 mm/min.

The diameter of the fiber is determined by mass conservation according to

$$d_f = (V_s/V_f)^{\frac{1}{2}} d_s \qquad (1)$$

where d_f = fiber diameter, d_s = source rod diameter, V_f = fiber pull rate and V_s = source rod pull rate.

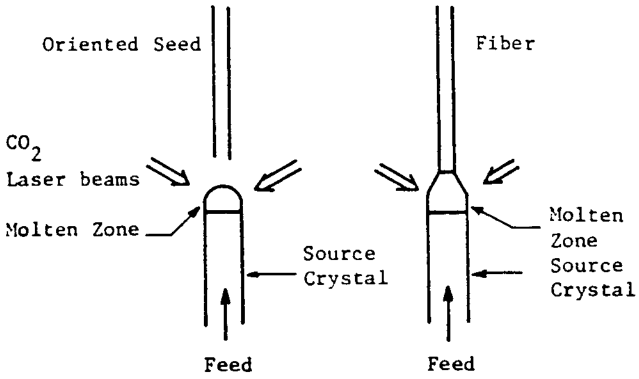

Figure 2. Schematic illustration of growth zone .

Stable growth is possible with typical diameter reduction ratios of 2 - 4 : 1. Thus, three growth cycles are generally necessary to reduce a 1 mm source rod to a 50 micron fiber. The laser power necessary to produce a stable melt zone varies from over 20 watts for a 1 mm rod of high melting point material like sapphire to several hundred milliwatts for 100 micron fibers of low melting point materials like $LiNbO_3$. This range of laser power is in agreement with a one dimensional heat flow model which shows that conduction and radiation dominate the heat loss from the melt zone, while convection, latent heat of crystallization and mass transfer make smaller contributions.

The orientation of the fiber is determined by the orientation of the seed. Among the materials successfully grown are (111), (110) and (100) Nd:YAG; (10.0), (00.1) $LiNbO_3$; (10.0), (00.1) sapphire and (100), (001) calcium scandate. The longest fibers grown thus far were 15 cm in length and 150 μm diameter (00.1) sapphire The smallest diameters obtained to date are 30 micron diameter Nd:YAG fibers.

Characterization. The Nd:YAG, sapphire and $LiNbO_3$ fibers tend to exhibit growth morphologies and cross-sections similar to those of Czolchralski grown bulk boules. An elliptical (10.0) $LiNbO_3$ fiber is shown in Fig. 3. Preliminary optical and X-ray investigations indicate that properly grown fibers are generally single crystal. Some defects, however, have been observed when growth conditions were not optimized. Large diameter Nd:YAG fibers have core defects similar to those in bulk grown boules.

Figure 3. 500 micron diameter (10.0) $LiNbO_3$ fiber showing elliptical cross-section characteristic of Czolchralski grown boules.

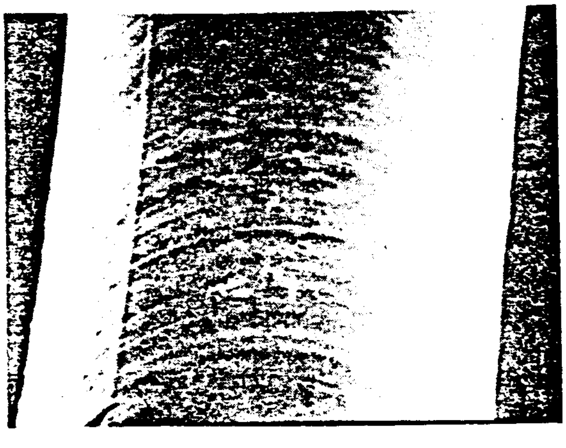

Figure 4. SEM photograph of 150 micron diameter (00.1) sapphire fiber showing diameter variations and intrinsic growth line.

An important parameter for ferro-electric fibers, like $LiNbO_3$, is the distribution of domains of spontaneous polarization. We have designed and constructed a domain orientation measurement that is based on the measurement of the pyro-electric coefficient. The measurement is capable of resolving domain distributions both along the fiber axis and across its end face with 20 micron resolution. An initial study of the domain structure of one (10.0) $LiNbO_3$ fiber shows an unusual pattern not usually observed in bulk Czolchralski crystals. The domains are layered transversely to the c-axis rather than parallel to it as is generally the case in bulk crystals. The explanation for this behavior is not yet clear though it may be related to the rapid cooling of the fiber after growth or the large surface area to volume ratio of the fiber geometry.

The geometry of the growth process is suitable for the introduction of poling electrodes immediately adjacent to the melt zone. By applying D.C. electric fields to these electrodes one could anticipate growing poled fibers. An interesting experiment would be to periodically flip the polarity of the field at a rate which, in conjunction with the pull speed, would lead to a fiber with a periodic arrangement of domains suitable for quasi-phasematching a nonlinear process.[8]

Diameter control. The major problem with the current growth system is insufficient control over fiber diameter fluctuations. Fibers produced to date have diameters varying by up to 5%.

Generally, the problem worsens as the fiber diameter decreases. Optical and scanning electron micrographs shown in Fig. 4 show that the diameter variations have a broad spectrum ranging from millimeter down to submicron periods. These deviations far exceed the tolerances discussed in the introduction. They are sufficiently large to preclude any useful measurements of the waveguide properties of the fibers. A number of components of the current fiber growth system are implicated in the diameter control problem. A primary cause of growth instability is the incomplete isolation of the growth zone from the ambient environment. The fiber is thus exposed to the perturbative influence of air currents and variations in room temperature. The problem is particularly acute in the growth of small diameter fibers. Because the support chuck moves away from the melt zone as the fiber is pulled, the tip of the fiber is less rigidly fixed as its length increases. The growth zone is thus increasingly sensitive to perturbations for small and/or long fibers. Growth stability is also degraded in small fibers by the loose focusing of the CO_2 beam, which tends to produce a melt zone that is long compared to the optimal length of roughly one source rod diameter.

Another source of fluctuations is the large diameter (1 mm) of the source rods used for the initial growth iteration. It has been necessary to rotate the rod in order to improve the thermal symmetry of the melt with the two beam focussing system. The residual thermal assymetry and any eccentricity in the rotation lead to periodic ridges on the fiber surface. It is not necessary to rotate the source rods for second and higher iterations of growth, but the perturbations introduced in the first iteration propagate through later stages.

These mechanical instabilities probably account for the lower frequency diameter variations. The higher frequency variations are probably associated with laser power fluctations outside the bandwidth of the present stabilization system. The characteristic lengths of these perturbations (down to several tenths of a micron), imply thermal fluctuations on the order of .1 - .01 seconds, consistent with the thermal time constant for the growth zone calculated from a one dimensional heat flow model. It is interesting to note that periodic diameter modulation on the same scale as these random variations would function as a distributed Bragg reflector analogous to those now used in integrated optics.

Second generation growth station

It is clear that better diameter control is necessary if the pedestal growth technique is to yield fibers suitable for device applications. Modifications to the first generation machine, including a controlled atmosphere chamber, a more flexible focussing system and wider bandwidth stabilization of the CO_2 laser are expected to yield considerably improved fibers.

Our efforts to find a more comprehensive solution to the diameter control problem have led us to the design and construction of a second generation growth machine. The new system uses the same pedestal growth technique as the first generation device, but incorporates refinements in a number of the sub-systems.

Figure 5 shows a block diagram of the system. The 20 watt waveguide CO_2 laser with active thermal stabilization is more compact and has better beam pointing stability than the conventional laser used in our original system. In addition, we designed a power stabilization system based on a temperature stabilized pyroelectric detector and resonant chopper to provide a feedback signal to the laser power supply which should achieve 1 ; 1000 output power stability over a bandwidth in excess of 1 KHz.

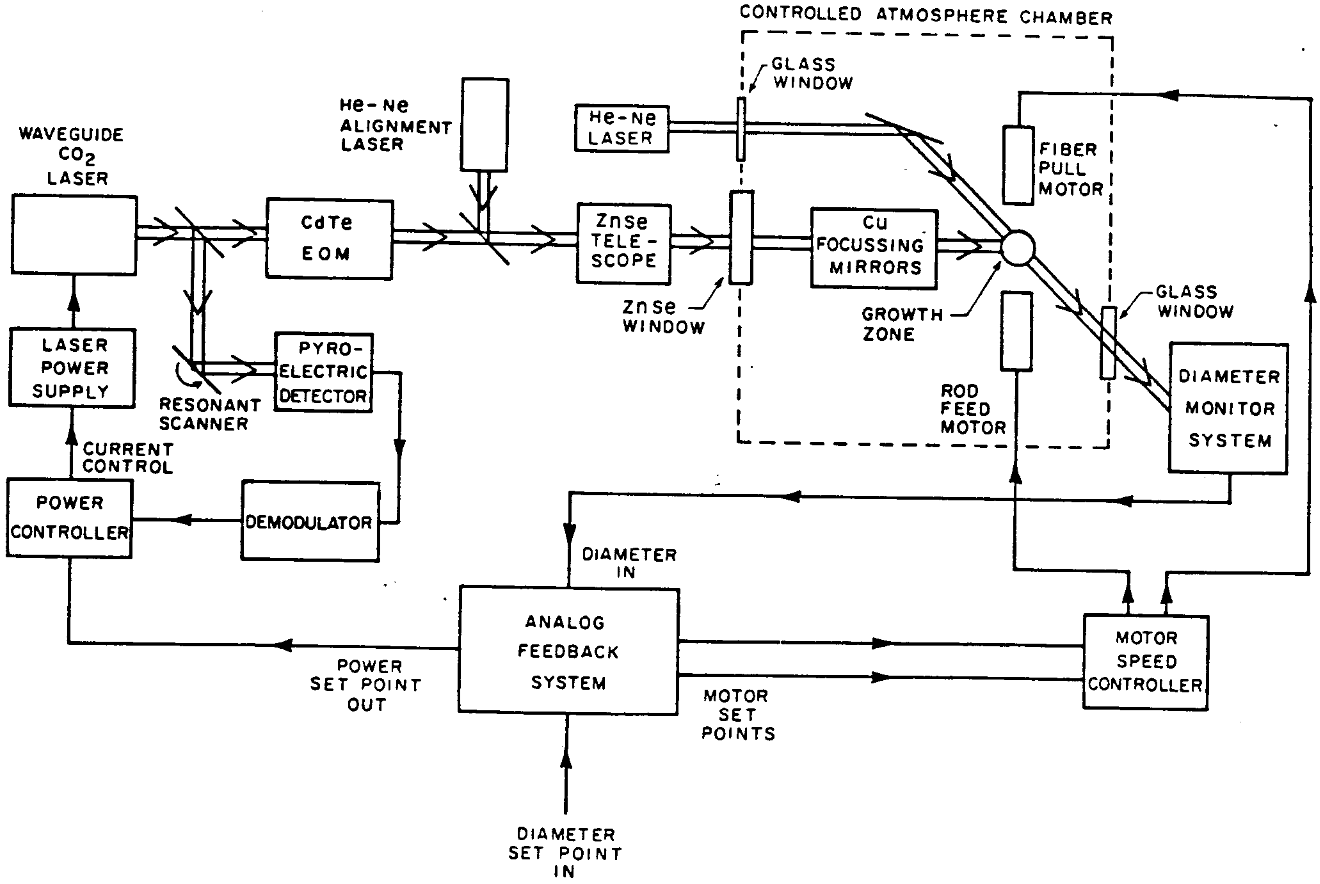

Figure 5. Block diagram of the second generation growth system. Details of the growth head are shown in Fig. 6.

We have designed a novel optical system, illustrated in Fig. 6 for focussing the laser beam onto the fiber in a 360° axial symmetric distribution. This design is a significant improvement over the two beam, rotating periscope,[10] or ellipsoidal[11] focussing systems previously used in laser drawn fiber systems. The diamond turned copper refraxicon and parabolic mirror should provide near diffraction limited f/2 focussing, yielding a minimum spot size of 30 microns. The focal spot size can be controlled by modifying the input beam divergence with the focussing telescope. This tight focussing is important for the stable growth of small diameter fibers. The symmetric irradiance will alleviate cold spots in the growth zone and eliminate the necessity of rotating the first iteration feed rod.

This focussing system is sensitive to input beam direction, so a CdTe electro-optic amplitude modulator is used to provide attenuation of the laser power without introducing beam steering.

The feed rod and fiber are translated through vacuum chuck guides with a capstan and pinch roller-design that offers several advantages over the lead screws used previously. The advantages of the present system include:

i. Maximum fiber length is no longer limited by the length of the lead screw. Thus, with a 1 cm long 1 mm diameter source rod, one could grow 16 meters of 25 micron diameter fiber.

ii. The controlled atmosphere chamber does not require mechanical feed-throughs.

iii. The DC motors driving the capstans are encoded and phaselocked to a crystal controlled oscillator, allowing stable and convenient adjustment of the growth rate.

iv. The vacuum chucks provide supports for the fiber and feed rod that are fixed with respect to the melt zone, unlike the lead screw system where the point of support moves away from the melt zone as the fiber grows.

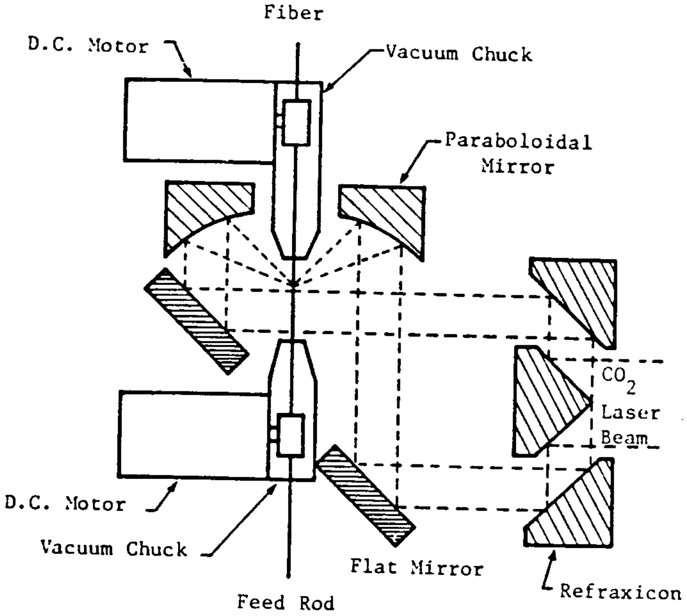

Figure 6. Detail of growth head showing CO_2 focussing optics and fiber guides.

The growth head is quite compact, fitting inside a 6 x 8 x 12" sealed box, equipped to provide oxygen, nitrogen or an inert gas environment, and isolation from ambient conditions.

While these innovations are expected to provide a substantial improvement in the open loop stability of the system, it will be useful to incorporate feedback based on an error signal derived from a measure of the deviations of the fiber diameter. No commercial diameter measurement system is capable of wide bandwidth (1 KHz) high resolution (.1 micron) monitoring in a geometry compatible with the working distance constraints imposed by the CO_2 focussing system. We have thus begun construction of an interference fringe tracking system with microprocessor based signal processing which promises resolution better than .1 micron at a measurement rate of 2 - 4 KHz. The diameter error signal will be used to control both the fiber pull speed and the laser power. It is necessary to control both these parameters as the latter is capable only of transient corrections (mass conservation dictates the steady state response) and the former is limited in its response rate.

The second generation growth machine is under construction at this time. Initial growth runs are expected by January 1982.

Conclusion

We have demonstrated the versatility of the pedestal growth technique for the growth of variety of refectory oxides single crystal fibers. Instabilities in the process have precluded the growth of useful waveguides. A second generation growth station is described which is expected to produce well diameter controlled optical fibers that will be used in device studies.

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