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# FINAL REPORT

"GROWTH OF SAPPHIRE FIBERS WITH A CO LASER"

GRANT NO.: N00014-92-J-1864

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By



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The objective of this program was to explore ways to increase the growth rate of optical quality sapphire fibers produced by the Laser Heated Pedestal Growth (LHPG) technique. Prior to this program, the maximum rate at which this could be accomplished was 3-4 mm/min. When the latter rate was exceeded, the transmission of the fiber rapidly deteriorated. The cause of the degradation was thought to be the increasing disturbance by convection currents in the molten zone. To reduce the severity of the latter, we proposed to examine the use of a different wavelength of the laser heat source and the modification of the spatial profile of the laser beam to heat the molten zone more uniformly.

In the standard implementation of LHPG a $CO_2$ laser with output in the 9-10$\mu$m region is used. This radiation is absorbed in a very thin surface layer (~10$\mu$m) of the molten zone. The subsequent redistribution of thermal energy within the molten zone is believed to be one of the causes of the convection. We therefore proposed to use a CO laser with output in the 5-6$\mu$m region as the heat source, with the notion that the deeper penetration at the shorter wavelength would lead to more uniform heating of the molten zone. This in turn would reduce the extent of convection. To this end, a power stabilized CO laser was constructed. Unfortunately, in spite of great efforts the best stability we could achieve was about 2-3% (compared to 0.5% for the $CO_2$ laser which we have been using). As a result, we were unable to obtain any high optical quality sapphire fiber with the CO laser as the heat source.

The other approach which we tried was to vary the spatial profile of the $CO_2$ laser beam to produce a more uniform heating pattern on the molten zone. In the conventional LHPG apparatus a reflaxicon arrangement is used to transform the Gaussian beam to an annular beam. While this spatial profile allows one to work with very small diameter (~30μm) feed rods, for the typical growth of sapphire fibers with ~300μm feeds its irradiation is far too localized. To spread out the $CO_2$ laser beam more evenly on the molten zone, we used a graded reflector to transform the Gaussian beam into a high order super-Gaussian beam. While we found that the modified beam did help to stabilize the growth process at high speeds, it did not significantly improve the maximum speed for growing optical quality sapphire fibers. However, we discovered that when He was used as the growth atmosphere instead of the usual air, it was possible to increase the maximum speed to 2 cm/min, which amounts to a 6-fold improvement. The details of our findings can be found in the attached preprint of a paper which has been submitted to the Review of Scientific Instruments. We have also filed for a patent on our new process.

The 6-fold increase in the growth rate of optical quality sapphire fibers which we have demonstrated under this program represents a major step towards the commercialization of this technology. The main problem which remains to be addressed is the practical and effective cladding of these fibers.

# APPENDIX

# A Novel Implementation of Laser Heated Pedestal Growth for the Rapid Drawing of Sapphire Fibers

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**ABSTRACT**

A new version of Laser Heated Pedestal Growth system is described which is amenable for the rapid growth of single crystal fibers. When He is used as the growth atmosphere, high optical quality sapphire fibers can be produced at rates as high as 2 cm/min with this apparatus.

Optical quality sapphire fibers have been produced by a number of groups in recent years.[1-3] These fibers were all produced by the Laser Heated Pedestal Growth (LHPG) technique in which a $CO_2$ laser is used as the heat source.[4-5] Up to now the highest reported growth rate of optical quality sapphire fibers has been 3-4 mm/min. When the growth rate exceeded this limit, the transmission of the fiber rapidly deteriorated. We report here a new way of implementing LHPG which has enabled the growth of high optical quality sapphire fibers at speeds as high as 20 mm/min. The key features of our system are the inclusion of beam shaping optics, the reliance on self-centering force in the molten zone, and the use of He as the growth atmosphere.

The growth of sapphire fiber by the LHPG method is illustrated in Fig. 1. The beam from a $CO_2$ laser is brought to focus on the tip of a feed sapphire rod. An oriented seed is then lowered into the molten zone. Once the seed is joined onto the feed rod through the molten zone, they are translated upwards by respective pulling and pushing mechanisms. Under stable growth conditions the diameter reduction is given by the square root of the ratio between the pulling and pushing speeds. It has been found in the past that there is a maximum drawing speed beyond which an opaque core begins to appear in the fiber.[6] We show here that this maximum speed can be greatly increased by tailoring the distribution of laser radiation on and immediately above the molten zone and by carrying out the growth in a He atmosphere.

A schematic of our sapphire fiber drawing apparatus is shown in Fig. 2. The collimated $CO_2$ laser beam is incident on a Gaussian reflector which transforms it into a top-hat shaped beam. The latter is reflected off a 45° mirror and then focused by a paraboloidal mirror onto the tip of the feed rod. Both the feed rod and the seed fiber are translated by roller pairs which consist of a rubber covered cylinder and a stainless steel cylinder with a V-groove in the middle. The feed rod is guided by a tight-fitting capillary tube to position it at the focus of the laser beam. The seed fiber is initially roughly positioned by moving the entire top roller box. It only needs to make contact with the molten zone when lowered towards the feed rod. Once contact is made, surface tension automatically centers the seed with respect to the feed rod. The entire growth apparatus can be evacuated by a mechanical pump and filled with the desired growth atmosphere.

The LHPG technique has gone through several stages of modification already. In the version just prior to the one reported here a reflaxicon was used to transform the Gaussian $CO_2$ laser beam into a ring-shaped beam.[4] That arrangement results in a very tight focusing on the shoulder of the molten zone. In contrast, with our Gaussian reflector approach, the laser radiation is not only more evenly spread out over the molten zone, but also impinges on a short length of the newly grown fiber. The optimum beam profile would presumably depend on the kind of crystal fiber one wishes to grow. In the case of sapphire we also found that leaving out the Gaussian reflector made the growth process much less stable.

C-axis sapphire fibers measuring 100$\mu m$ in diameter were drawn from 300$\mu m$ feed rods at the rate of 20 mm/min in a He atmosphere at different pressures. The fibers were annealed at 1200°C for 24 hours afterwards. They were then cut to 1 m lengths and polished at both ends. Transmission measurements were made using lasers at 543 nm, 633 nm, 840 nm, and 1.15$\mu m$. After correction for Fresnel losses the attenuations are given in Fig. 3. It is seen that there appears to be an optimum He pressure under the indicated experimental conditions, although we were unable to conclusively prove it as the available laser power did not permit growth at He pressures greater than 20 torr. The higher loss at shorter wavelengths is believed to be associated with one of the V-centers in $Al_2O_3$.[7]

When either air, $N_2$ or Ar was used as the growth atmosphere at high drawing rates, the resulting fibers were found to have significantly poorer transmission. The difference is believed to be caused by the entrapment of the ambient gas by convection currents present in the molten zone. The problem is less serious for He because once entrapped it can more readily diffuse out to the surface again. This would also explain why increasing the He pressure from 15 torr to 20 torr reduced the transmission of the fiber. Clearly, for any given growth atmosphere the entrapment problem becomes worse as the pressure is increased. At very low pressures material loss through evaporation becomes important. Since evaporation leaves behind excess oxygen,[8] it can again lead to microvoids in the grown fiber. Thus, there is an optimum pressure as the data in Fig. 3 indicate.

We have not been able to explore fiber growth at rates faster than 20 mm/min as we were limited by the power of our $CO_2$ laser. We have also yet to experiment with different profiles of the Gaussian reflector. The exact distribution of the laser beam in the vicinity of the molten zone may be important in minimizing the magnitude of the convection currents. Such optimization may well lead to further improvements in the growth rate of optical quality sapphire fibers.

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Figure Captions:

Fig. 1 Schematic of fiber growth by the LHPG technique.

Fig. 2 Schematic of the LHPG apparatus. Parts indicated are: 1. Incident $CO_2$ laser beam 2. Gaussian reflector 3. ZnSe window 4. Turning mirror 5. Paraboloidal focusing mirror 6. Capillary guide 7. Feed rod 8. Grown fiber 9. Roller pair 10. Translation stage assembly 11. Gas inlet 12. Gas outlet.

Fig. 3 Attenuation at four laser wavelengths for fibers grown at different He pressures. All fibers were 1 m long, 100$\mu$m in diameter, and grown at 2 cm/min. The data for 15 torr He are averaged values for three fibers. All other data are for single fibers.

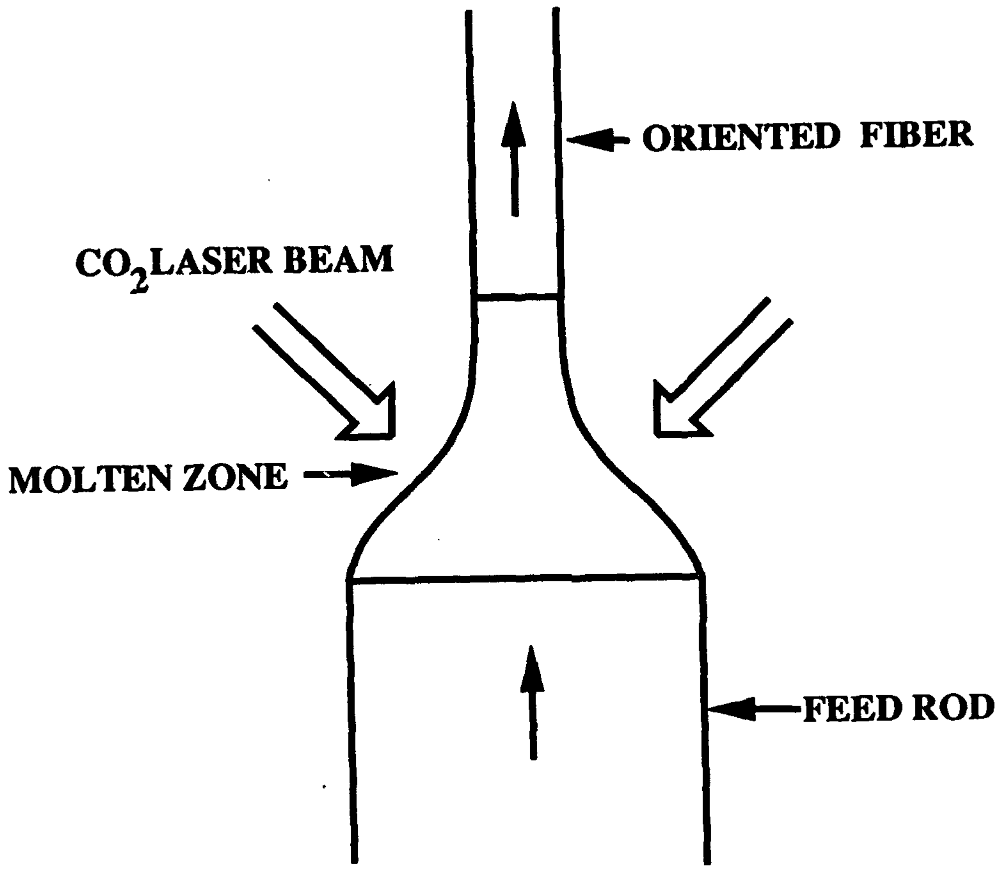


FIGURE 1

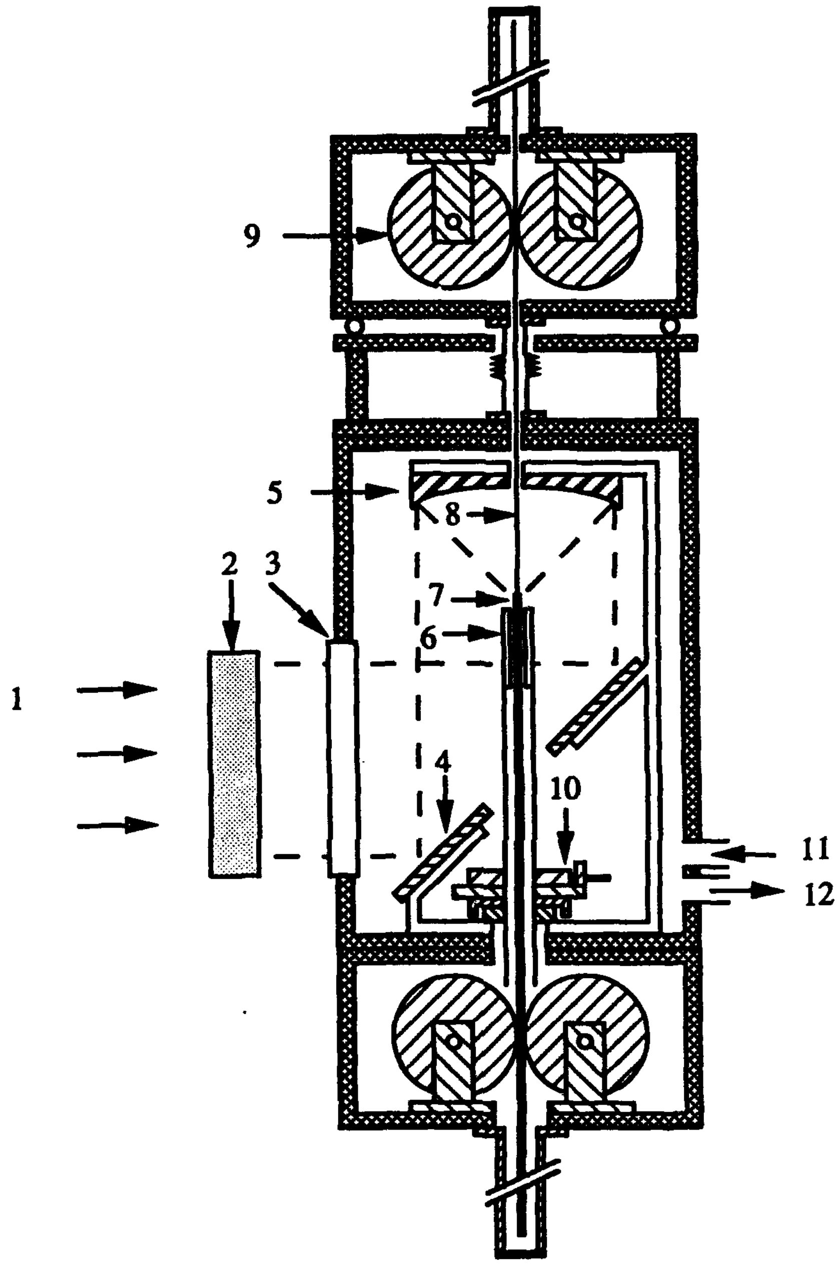


FIGURE 2

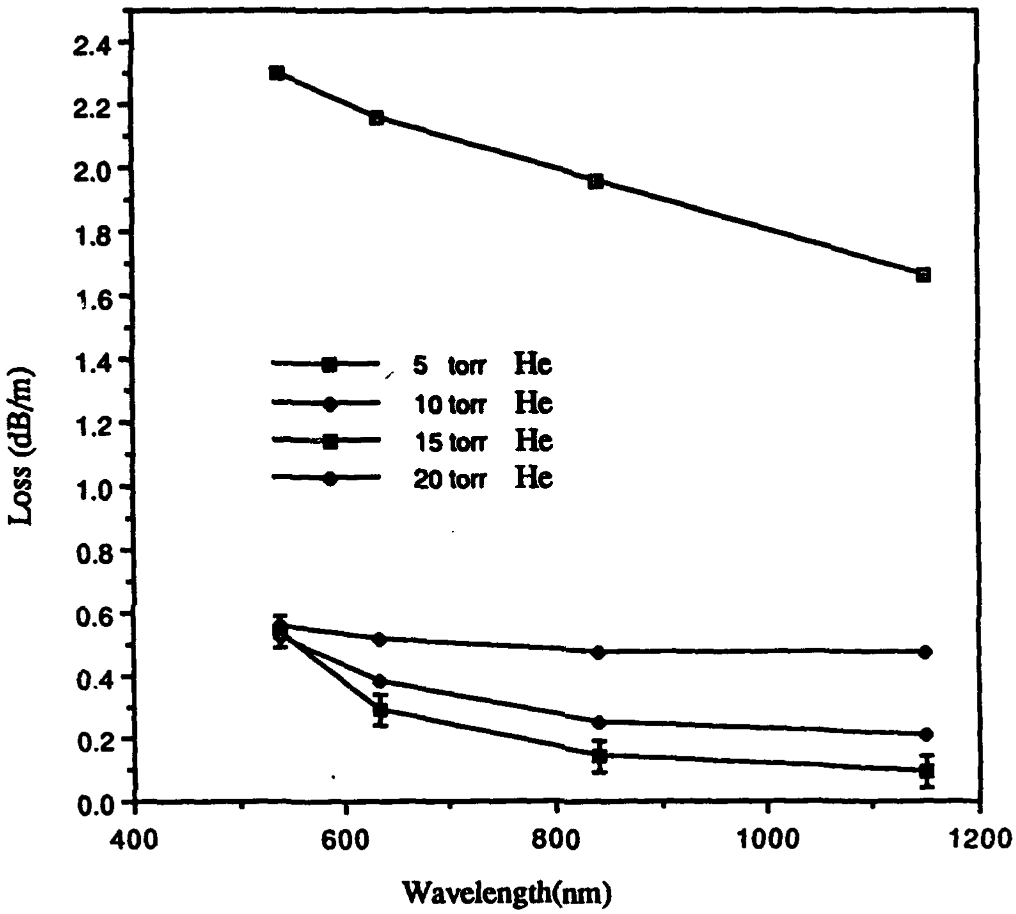
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
Loss (dB/m)
5 torr He
10 torr He
15 torr He
20 torr He
400
600
800
1000
1200
Wavelength(nm)

FIGURE 3

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**ACKNOWLEDGMENT OF DISCLOSURE**

**MEMORANDUM** April 4, 1994

**TO:** Nicholas Djeu
Professor
Department of Physics
College of Arts & Sciences

**FROM:** Office of the Vice President for Research

**SUBJECT:** **Disclosure Regarding *Apparatus for the Rapid Growth of Optical Sapphire Fibers***

**Disclosure Number 94/03/017**

This will confirm that the above referenced Disclosure of Invention was received on April 1, 1994, by the Office of the Vice President for Research.

As a result of our initial evaluation of the Disclosure, the Office claims an interest in the invention as described in Rule 6C4-10.012 and/or the pertinent collective bargaining agreement. Further in accordance with the foregoing Rule, two copies of the USF Revenue Allocation Agreement are enclosed for your signature. This Agreement is intended to individually recognize the terms of above Rule as required in Section 4 thereof, as well as to more fully explain the obligations of the parties. Please sign the Agreements, retain a copy for your files, and return one signed copy to my office.

For and on behalf of the University, we will be continuing our evaluation of the Disclosure with respect to the appropriate protection to be sought, marketability and the rights that various interested parties may have.

GRN:KGP:alf