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The Preparation of Optical Waveguide Preforms by Plasma Deposition

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Optical fiber preforms are prepared by a technique similar to the modified CVD process, except that an RF plasma is used. Optical losses as low as 6.5 dB/km at 1.06 μm were achieved. Higher reaction efficiencies resulted in deposition rates more than three times greater than the modified CVD process.

I. INTRODUCTION

The modified chemical vapor deposition (MCVD) process[1] has achieved acceptance as a means of preparing low-loss optical waveguides. A recent study of the preparation of silica-clad germania borosilicate optical fibers by this process[2] indicates that it can provide low-loss optical fibers having properties which surpass minimum requirements for wide-band communications applications. Typical fibers produced by MCVD are characterized by low loss (<4 dB/km at 1.06 μm), a reproducible graded index profile ($\alpha \sim 2.0$) and controlled core/cladding ratio, circularity and concentricity.

Although current deposition rates are competitive with other processes, higher rates are desirable. Furthermore, the efficiency, defined as the ratio of glass deposited/reactants in, is generally below 50 percent for most GeO_2 containing compositions. These considerations stimulated our investigation into using the ionized environment of a plasma to increase the rate and efficiency of the MCVD process. Initially both microwave and RF plasmas were considered. In the former, ionic species are produced which deposit as a vitreous deposit directly on the tube walls as the result of a heterogeneous reaction similar to conventional CVD.[3] This advantage, however, is purchased by the use of reduced pressures which dictates a low flux of reactants. As a result, even at 100 per cent efficiency, the deposition rates in the microwave method need be no higher than MCVD and frequently are lower.

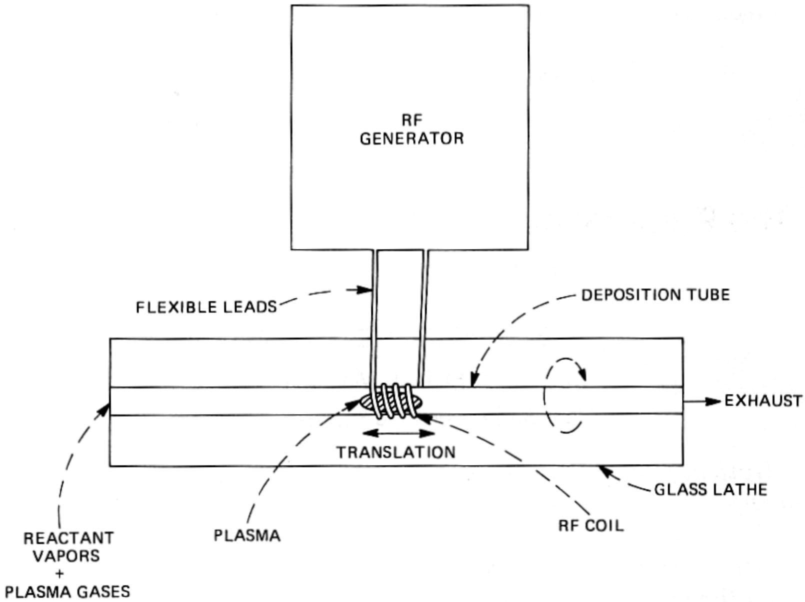

Fig. 1—Apparatus for preparing preforms.

In contrast, the use of an RF argon-oxygen plasma operating at a pressure of 1 atmosphere provides the potential for higher deposition rates as well as increased efficiency. In this type of plasma, temperatures in the vicinity of 10,000–20,000°K exist, producing an intense reaction region in which highly ionized species occur. Under these conditions, reactions between ionized species occur resulting in a deposit on the tube wall which is similar to that produced by MCVD. Significant improvements in the efficiency and deposition rate achieved by the RF plasma MCVD process are derived from the ability to produce gas phase reactions in a highly ionic environment.

II. EXPERIMENTAL

The apparatus consists of a glass working lathe positioned adjacent to an RF generator as shown in Fig. 1. An RF coil, mounted to a motorized carriage on the lathe was connected to the generator via water-cooled flexible leads. In a typical deposition, a 25 × 28 mm TO8 silica tube was positioned in the lathe and a flow of argon was started. All gas flows were left to right and were controlled with rotometers. The plasma was initiated at a frequency of 4.5 MHz by striking an arc with a graphite rod inserted into the tube in the vicinity of the coil. The coil was traversed at a constant rate over ~25 cm length and the tube was rotated as shown. Gaseous $SiCl_4$ and $GeCl_4$ were introduced to the reaction zone using an

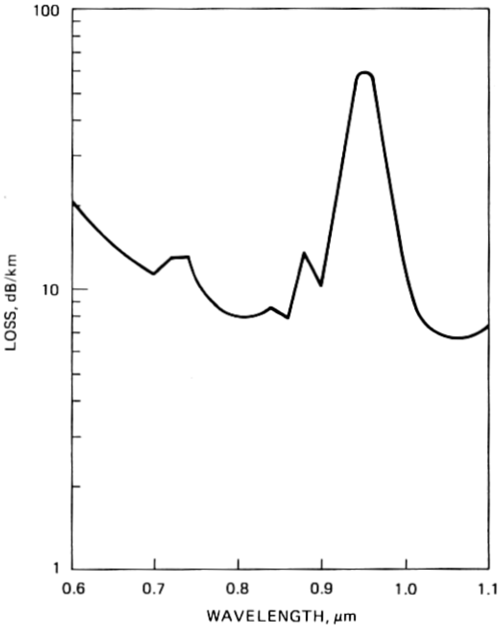

Fig. 2—Loss spectrum.

oxygen carrier which was bubbled through room temperature chloride liquids. BCl_3 gas was directly introduced from a cylinder of liquid BCl_3. $SiCl_4$ at 92 cc/min and BCl_3 at 10 cc/min were initially introduced into the gas stream to produce a borosilicate "barrier layer." Deposition was carried out for 5 minutes before $GeCl_4$ at 65 cc/min was added to produce a germanium borosilicate core. Core deposition continued for 15 minutes, at which time the RF power and all gas flows were turned off and the tube collapsed using an oxyhydrogen burner.

III. RESULTS AND DISCUSSION

It is well known that the stability of an RF plasma may be related to the diameter of the tube in which it is being sustained. We found that under our operating parameters, stable operation could be obtained at a frequency of 4.5 MHz by using 25 mm ID tubes. Fundamental considerations indicate that, in general, it is desirable that the tube radius be greater than the skin depth[4] of the plasma, which is proportional to the ½ power of the ratio of the plasma resistivity to the operating frequency. Hence, higher frequencies would be one variation that would simplify the use of smaller tubes.

Figure 2 gives the loss spectrum for a 1100 meter length of a plasma-deposited fiber with a 35 μm core and a 110 μm OD having a minimum loss of 6.5 dB/km at 1.06 μm. The peaks are due to overtones and com-

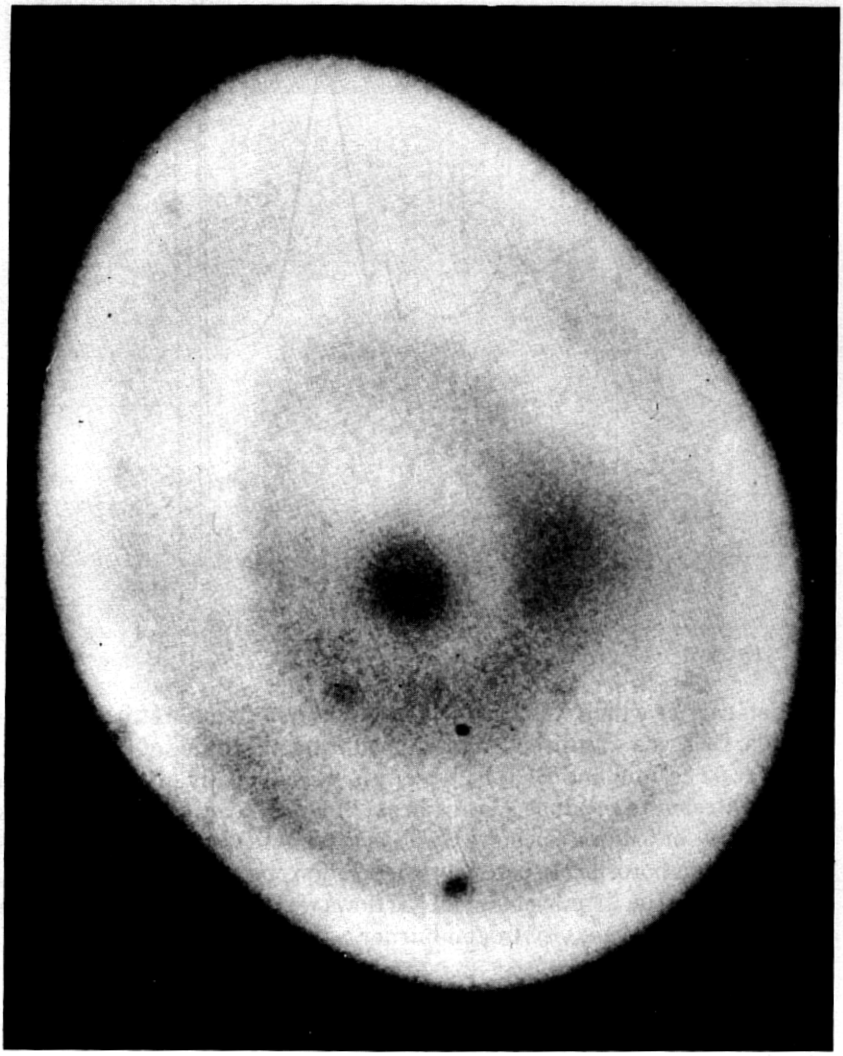

Fig. 3—Unetched microstructure.

binations of the fundamental OH absorption band and the loss at 0.95 μm indicates a water content of ~50 ppm.[5] Since typical MCVD fibers exhibit a minimum loss below 4 dB/km and contain only a few ppm water, an attempt was made to determine the extent to which the current plasma process parameters contributed to the excess water content and loss, by preparing a preform in the conventional manner using an oxyhydrogen torch as the heat source. This resulted in a fiber having the same OH content but with a minimum loss < 3 dB/km at 1.06 μm, suggesting that the excess loss at this wavelength is related to specific process parameters while the OH concentration may be due to the general lack of humidity control in the surrounding environment.

The unetched microstructure of a core deposit for a 200 μm fiber viewed in an optical microscope, shown in Fig. 3, indicates some of the limitations encountered under the present operating conditions. The noncircularity of the core reflects the difficulty in collapsing a 25 mm tube while the rings result from compositional variations in the deposit. Examination of an etched core deposit in a scanning electron microscope revealed that the dark rings are GeO_2 deficient regions. Thus, the excess loss in this fiber may result from scattering caused by inhomogeneity of the deposit and/or compositional variations in the core. In either case, large excursions of the tube wall temperature during the deposition are the probable cause. However, other loss phenomena have not been ruled out.

In order to make very low loss preforms, it may be desirable to heat the deposit on the wall to a temperature where fining and homogenization of the glass take place with minimal vaporization of the more volatile constituents or heating the tube to temperatures where it will deform. Temperature control in the plasma deposition process was attained primarily by traversing the plasma ball along the tube at a rate sufficient to accomplish these objectives. Additional cooling of the outside of the tube to prevent deformation was provided by an air stream directed at the region surrounding the plasma. However, other important parameters that affect the wall temperature include the composition and velocity of the plasma gases and the RF power levels. For example, increasing the oxygen/argon ratio in the plasma requires increased RF power, both of which produce a high plasma temperature that is transferred to the walls. The evaluation and control of these parameters is currently underway.

The principal advantage of plasma deposition is the potential for increased deposition rates and improved overall reaction efficiency. For approximately equivalent reactant flows a rate of deposition of 0.19 g/min was obtained compared to a rate for MCVD of 0.05 g/min. This increase in the deposition rate supports the belief that the overall reaction efficiency is markedly increased for the RF plasma process. In addition, the NA of the plasma-deposited fibers were 0.28 compared to 0.24 for the conventional process, indicating increased efficiency for GeO_2 incorporation. Furthermore, reasonable extension of the results achieved to date suggest that deposition rates perhaps an order of magnitude greater than normally achieved by the MCVD process may be obtainable.

IV. CONCLUSIONS

The RF plasma CVD process is capable of producing low loss (< 10 dB/km) optical fibers at high deposition rates. A better understanding of the effect of various operating parameters on the nature of the deposition process is necessary to exploit the full potential of this process.

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REFERENCES

1. J. B. MacChesney, P. D. O'Connor, F. V. DiMarcello, J. R. Simpson, and P. D. Lazay, Proc. 4th Int. Cong. on Glass, Kyoto, Japan (1974), pp. 6–40.
2. J. B. MacChesney, P. B. O'Connor, and H. M. Presby, Proc. IEEE, *62* (1974), p. 1280.
3. F. V. DiMarcello and J. C. Williams, Conf. Pub. No. 132, IEEE, First European Conf. on Optical Fiber Communications, (1975), p. 36.
4. J. Koenings, D. Kuppers, H. Lydtin, and H. Wilson, Proc. of 5th International Conf. of CVD (Sept. 21–26, 1975), Fulmer, England.
5. P. Geittner, D. Kuppers, and H. Lydtin, Applied Physics Letters, *28,* No. 11, p. 545 (1976).
6. D. Kuppers, J. Koenings, and H. Wilson, J. Electrochem. Soc., Solid-State Science and Technology, *123,* No. 7 (1976), p. 1079.
7. *High Temperature Technology,* Proc. of an Int. Symp. on High Temperature Technology, Asilomar, California 1963, Session I, Buttersworths, Wash. (1964), p. 525.
8. G. R. Newns and H. N. Daglish, Symp. on Electrotech. Glasses, London (1970).