Refractive index dispersion and related properties in fluorine doped silica

J. W. Fleming and D. L. Wood

Refractive index dispersion has been determined for fluorine doped fused silica containing 1 and 2% fluorine. Addition of fluorine is found to reduce the refractive index and material dispersion. Fibers modeled for minimization of modal dispersion having fused silica cores and fluorine doped silica cladding show a strong composition dependence of the optimum refractive index profile. Comparison of results with those in the literature based on N.A. vs λ measurements reveal minor discrepancies probably due to the drawing process.

I. Introduction

The refractive index dispersion has been measured on bulk samples of commercial fluorine doped silica glasses, which were prepared by a plasma torch in the same way the glasses are for use in certain optical waveguides.^{1,2} Fluorine doping lowers the refractive index of fused silica and, therefore, is a suitable dopant for preparation of multimode and single-mode lightguides having fused silica cores. Numerical apertures of 0.2 have been achieved with only 3 wt. % addition of fluorine to the glass.²

Bulk measurement of refractive index dispersion continues to be of importance for accurate characterization of dispersion in glasses used in lightguides. Although techniques have been developed which measure the dispersion on fiber specimens, their accuracy is in doubt particularly with respect to the composition or state of annealing to which the measurements apply. It is well known that step index lightguides made by MCVD or other related fabrication techniques produce lightguiding material in layers which exhibit significant interlayer and intralayer compositional variation. Many of these lightguide analysis techniques, particularly those using interferometry, are insufficiently precise to discern adequately the index change between layers (for example, see Ref. 3). Thus index measurements on bulk glasses are important to supplement the corresponding measurements on fibers even though the

The authors are with Bell Laboratories, Murray Hill, New Jersey 07974.

Received 18 May 1983. 0003-6935/83/193102-03\$01.00/0. © 1983 Optical Society of America. glasses may be made by different techniques in the two cases. They are particularly necessary for characterization of the materials apart from the core-cladding stress,⁴ compositional variation, and unknown thermal history involved in fiber measurements.

II. Experimental

The bulk fused silica samples doped with 1 and 2 wt. % fluorine were obtained from Heraeus Quartzschmelze GmbH. These were reportedly made with a plasma chemical vapor deposition apparatus,¹ which is the same equipment used by them for making the F-doped optical waveguide preforms. Without additional heat treatment these pieces were prepared as prisms with apex angles of ~45° and faces optically polished to 1/10 wavelength flatness.

Refractive index was measured using a recently developed computerized refractive index measurement apparatus,⁵ which measures the deviation of monochromatic radiation through a prism specimen. The standard deviation of these measurements is 3.5×10^{-5} .

Indices were determined for 24 wavelengths from 0.36 to 2.3 μ m on the 1% sample and for 17 wavelengths from 0.36 to 1.7 μ m for the 2% sample. Two to four measurements were made at each wavelength. Wavelengths were not used between 1.7 and 2.3 μ m for the 2% sample because its small size gave a very low transmitted signal, especially at the longer wavelengths.

The temperature of the prisms varied several degrees Celsius, about 25°C during the periods in which the measurements were made on each prism. To compensate for this variation and facilitate comparisons of the data with other measurements on fused silica, the index values were corrected to 20°C using the temperature coefficients for fused silica determined by the National Bureau of Standards.⁶ The index in each case was fitted to a three-term Sellmeier dispersion formula of the form

$$n^{2} - 1 = \sum_{i=1}^{3} \frac{A_{i} \lambda^{2}}{\lambda^{2} - l_{i}^{2}},$$

where A_i is an oscillator strength,

 l_i is an oscillator wavelength, and

 λ is the wavelength at which the index is *n*.

The average absolute values and standard deviations of the residuals are, respectively, 2.4×10^{-5} and 3.6×10^{-5} for the 1% sample and 2.4×10^{-5} and 2.6×10^{-5} for the 2% sample. A standard F test of the residuals indicates that there is no reason to doubt that the formula accurately represents the data.

III. Results and Discussion

Measured refractive indices for the two fluorine doped glasses and for pure SiO_2 are listed in Table I. As indicated, the addition of fluorine decreases n. The computed Sellmeier coefficients are given in Table II. The third term of the relationship, which is correlated with an intrinsic absorption of the material in the infrared, has an oscillator strength close to unity and an oscillator wavelength very near a predominant absorption wavelength for fused silica. This is similar to the behavior found previously⁷ and is consistent with

Table I. Refractive index vs Wavelength for the Measured F Doped Glasses

| | <i>n</i> | | | |
|----------|----------|---------|---------|--|
| λλ | 1% F | 2% F | SiO_2 | |
| 0.365015 | 1.46941 | 1.46485 | 1.47486 | |
| 0.404656 | 1.46458 | 1.46009 | 1.46994 | |
| 0.435835 | 1.46174 | 1.45726 | 1.46701 | |
| 0.479991 | 1.45855 | 1.45414 | 1.46382 | |
| 0.508582 | 1.45694 | 1.45257 | 1.46218 | |
| 0.546074 | 1.45521 | 1.45083 | 1.46040 | |
| 0.578012 | 1.45395 | 1.44962 | 1.45913 | |
| 0.587561 | 1.45363 | 1.44924 | 1.45878 | |
| 0.589262 | 1.45357 | 1.44916 | 1.45872 | |
| 0.643847 | 1.45187 | | 1.45702 | |
| 0.852110 | 1.44767 | 1.44344 | 1.45279 | |
| 0.894350 | 1.44704 | 1.44282 | 1.45216 | |
| 1.0074 | 1.44560 | 1.44137 | | |
| 1.01398 | 1.44555 | 1.44132 | 1.45056 | |
| 1.08297 | 1.44470 | 1.44050 | 1.44973 | |
| 1.12866 | 1.44420 | 1.43999 | 1.44919 | |
| 1.19382 | 1.44346 | | | |
| 1.3589 | 1.44158 | | | |
| 1.3622 | 1.44158 | 1.43743 | 1.44653 | |
| 1.4695 | 1.44029 | | 1.44530 | |
| 1.52952 | 1.43973 | | 1.44459 | |
| 1.6932 | 1.43761 | 1.43344 | 1.44258 | |
| 1.9700 | 1.43400 | | 1.43884 | |
| 2.3253 | 1.42852 | | 1.43325 | |

Table II. Sellmeier Parameters Determined from a Nonlinear Least-Squares Fit of Experimental Index vs Wavelength Data

| <i>A</i> ₁ | l_1 | A_2 | l_2 | A ₃ | l_3 |
|---------------------------|--------|---------|--------------------|----------------|--------|
| 67744 0 | .06135 | 0.40101 | 0.11714 0.12030 | 0.87193 | 9.8563 |

^a From Ref. 9.

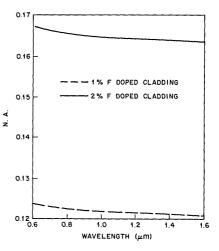


Fig. 1. Numerical aperture as a function of wavelength for fibers with silica cores and F doped silica cladding.

the observation that the dispersion of the F doped glasses is not very different from that of fused silica.

From a calculation of the second derivative of the fitted Sellmeier relationship using the analytical expression for the derivative, the wavelength λ_0 of zero material dispersion was determined for these glasses. For the 1% F doped glass, $\lambda_0 = 1.272$, and for the 2% F doped glass, $\lambda_0 = 1.265$, while for pure SiO₂ $\lambda_0 = 1.276$. The addition of fluorine to silica thus lowers the material dispersion shifting λ_0 to shorter wavelengths.

The dispersion data in the form of the fitted Sellmeier coefficients can be used to model the propagation properties of lightguides comprised of these materials as, for example, in Ref. 8. For fibers with the 1 and 2% F doped silica as a cladding material and with silica cores, Fig. 1 shows the numerical aperture as a function of wavelength λ . For the core data the fused silica results obtained for a quenched sample⁹ were used. This is believed to be more appropriate for waveguide modeling than the NBS data⁶ on well-annealed SiO₂, since measurements of the refractive index of drawn silica fibers indicate that the silica is definitely quenched.¹⁰

Employing an approximation for modal propagation in multimode lightguides,^{11,12} one can determine from core and cladding dispersion data an index profile shape which for a smooth profile (no index steps) should minimize modal dispersion in lightguides comprised of the core and cladding material. The profile shape is characterized by the well-known parameter α

$$n(r,\lambda) = n_1(\lambda) \left[1 - 2\Delta \left(\frac{r}{a}\right)^{\alpha}\right]^{1/2},$$

where n_1 is the axial refractive index;

- *a* is the core radius; and
- Δ is the fractional index difference between axis and cladding.

Figure 2 shows optimum α as a function of λ for the two waveguide structures indicated above. As can be seen in the figure the profile shape which minimizes modal dispersion (optimum α) at any wavelength is a function of the amount of fluorine doping in the

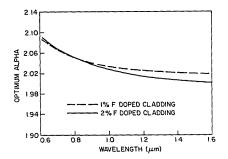


Fig. 2. Optimum α as a function of wavelength for fibers with silica cores and F doped silica cladding.

cladding with lower optimum α as the fluorine concentration of the cladding increases.

In the case of fabricated fibers¹³ with silica cores and fluorine doped silica cladding the results for N.A. or optimum α as a function of λ do not agree completely. The bulk glass measurements indicate that the N.A. decreases monotonically with increasing wavelength, although the index change from 0.6 to 1.6 μ m is only \sim 0.003. Reference 13 reports a minimum in N.A. at $\sim 0.7 \,\mu m$ for a sample which has a cladding of effective composition of ~ 1.5 mole % fluorine. While a true N.A. minimum is not impossible it is unlikely to occur in this wavelength range particularly in fibers comprised of materials as similar as silica and F doped silica. In general, a difference in the inflection point in the n vs λ curves for the core and cladding, capable of causing a minimum in N.A. = $(n_1^2 - n_2^2)^{1/2}$, is more likely to occur near λ_0 of the core and cladding glasses (1.27 μ m) than at the lower wavelength of Ref. 13. The variation in N.A. with wavelength measured on fibers was used in Ref. 13 to obtain an optimum α for fluorine doped glasses of $< \alpha = 2$ for wavelengths between 0.6 and 1.2 μ m with optimum $\alpha = 1.86$ at 1.2μ m. Reference to Fig. 2 shows that this is not the case where bulk index values are used.

This lack of detailed agreement between the bulk and the fiber results is important, as fiber design based on bulk measurements is very convenient. One possible source of discrepancy arises from the difference in refractive properties which exists between annealed and unannealed glasses, the fiber being in a state of quenching as yet unaccounted for in model calculations. Other possible differences between fiber and bulk measurements might arise from the layer structure which is a by-product of the fiber fabrication technique. It is not possible at this time to resolve quantitatively the disagreement between the two methods of measuring, but the resolution will surely reveal a more detailed understanding of actual fiber structures.

IV. Conclusion

Accurate refractive index dispersion data on two F doped silica bulk specimens have been presented and compared with similar results for pure silica. The results of modeling fibers with F doped silica cladding and silica cores using the bulk data indicate a compositional dependence of the optimum profile shape. These results differ from those experimentally measured on a fiber of the same structure as that modeled and having an F doping level between those of the two bulk samples. The differences cannot be resolved quantitatively at the present time, but alterations in refractive properties due to the drawing process, quenching, or diffusion of dopants may contribute.

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