

# Synchrotron X-Ray Microprobe Analysis of Optical Fibers

K. K. Soni,<sup>1</sup> C. W. Ponader,<sup>1</sup> B. Lai,<sup>2</sup> Z. Cai,<sup>2</sup> G. Shenoy<sup>2</sup>

<sup>1</sup>Corning Incorporated, Science & Technology Division, Corning, NY 14831.; <sup>2</sup>Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, U.S.A.

## Introduction

An optical fiber typically consists of a silica-based fiber 125 $\mu\text{m}$  in diameter with a central core region  $\sim 5\ \mu\text{m}$  in diameter with higher refractive index than the surrounding cladding. The increase in refractive index is achieved by using germania as a dopant in the core region. Fibers can also be doped with rare earths (e.g., Er) for amplification purpose. It is important to understand the distribution of these dopants at sub- $\mu\text{m}$  level including those of trace impurities that can affect the optical attributes of the fiber such as attenuation [1]. We have explored the capabilities of SR in analyzing optical fibers. Our goal is to map the distributions of dopants and impurities with submicron resolution in the fiber core using the x-ray microprobe at the Advanced Photon Source and relate this chemical information to optical properties of the fiber. Previously, x-ray fluorescence measurements have been performed at low resolution (1-4  $\mu\text{m}$  capillary optics) to map the Ge core of the fiber [2]. X-ray absorption measurements with a 0.5 X 0.5 mm<sup>2</sup> beam has been performed to determine the impurity concentration along the diameter of the fiber preform several mm in diameter [3].

## Methods and Materials

Experiments were performed at the Advanced Photon Source using beamline 2-ID-D. Fiber samples were mounted and aligned so as to make the optic axis parallel to the incoming X-ray beam. The energy of the incoming beam was set at 11keV below the Ge K-edge for the results shown here. Ge mapping was still possible using the Ge loss peak which was caused by the incident x-ray losing an energy corresponding to the Ge L-edges. The fluorescence radiation was detected using a Ge detector. A single mode fiber was thinned using mechanical polishing.

## Results

Fig. 1 shows Si and Ge maps of a single mode fiber containing germania as a dopant. For bulk glass fibers containing high Z elements such as Ge, the inelastic scatter peak dominated the spectrum and caused spurious signals from hutch components, making it impossible to accurately detect and map trace metallic impurities. Therefore, a thin slice  $\sim 80\ \mu\text{m}$  thick was analyzed that reduced scattering and consequently the spectral background. The feature at the center of the Ge-rich core is caused by collapse of the airline during fiber manufacture. Fig. 2 shows Ge and Er maps of an erbium doped fiber amplifier (EDFA). These maps were acquired from a cleaved fiber without thinning (fiber length  $\sim 5\text{mm}$ ) by scanning the fiber core region (5 $\mu\text{m}$ )<sup>2</sup> in 0.16 $\mu\text{m}$  steps.

## Discussion

The core of the fiber is circular but appears elliptical in these maps because the emitted X-ray are detected at nearly a normal angle to the fiber optic axis. Given the high concentration of

these dopants, their distribution can be easily mapped using X-ray microprobe and other microanalytical techniques. An important advantage of the current approach is that sub-micron resolution approaching probe size (150-200nm) can be obtained which is difficult with other techniques. The Ge distribution can be used to indirectly measure the refractive index profile with 150-200nm detail. Detection of trace elements can be improved by using still thinner samples and shielding of fluorescent components in the hutch. Fig. 2 shows that while Ge and Er maps are concentric, the Er-rich region is smaller than Ge-rich region in the core. Moreover radial gradients in the doping of these elements are also visible in these maps. We have also applied this technique to analyze other types of fibers including multi-mode, photonic band-gap fibers.

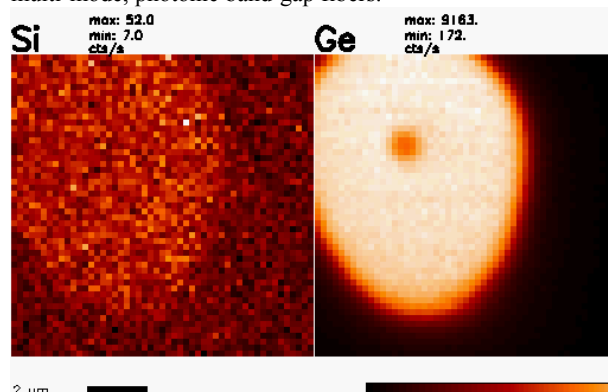


Fig. 1. Si (left) and Ge (right) fluorescence maps of the core region of a single mode fiber. Image width=10 $\mu\text{m}$  (0.2 $\mu\text{m}$  step).

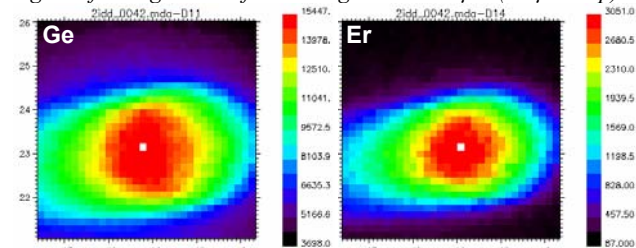


Fig. 2. Ge (left) and Er (right) fluorescence maps of the core region of a EDFA fiber. Image width=5 $\mu\text{m}$  (0.16 $\mu\text{m}$  step).

- [1] P.C. Shultz, J. Am. Ceramic Soc. 57, 309 (1974).
- [2] E. Fontes, J.R. Patel, and P. Pahl, "Fluorescence study of doped optical fibers," CHESS Newsletter, 1995.
- [3] P.H. Citrin, P.A. Northrup, R.M. Atkins, P.F. Glodis, L. Niu, M.A. Marcus, D.C. Jacobson, Mat. Res. Soc. Symposium, Proc. 524, 251 (1998).

## Acknowledgments

Use of the Advanced Photon Source was supported by the U.S. Dept. of Energy, Office of Science, Basic Energy Sciences, under Contract No. W-31-109-Eng-38.