

Fluorescence study of doped optical fibers

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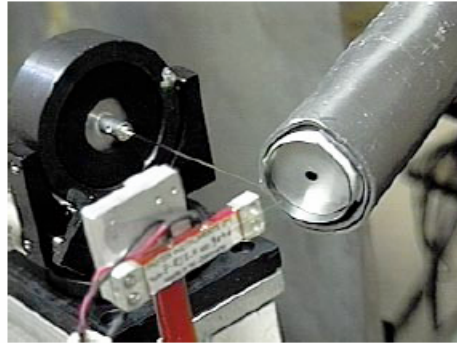
As materials engineering and fabrication descends into the sub-micron regime, new tools must be developed to both view and analyze the novel structures. Many of the excellent techniques that provide atomic resolution are not best suited to study the features the forefront of integrated circuit technology, where interest now lies in the 0.1 micron regime. Most scanning techniques are also limited to surface structures, whereas in most solid-state devices the interesting properties depend upon details at buried interfaces. And because the electron interacts so strongly with matter, electron microscopy and other excitation techniques can be also limited to surfaces or sectioned, thinned and stained specimens.

Interacting much more weakly, high-energy x-rays penetrate deeply into materials, making it possible to study both surface and bulk properties using any combination of absorption, diffraction, and fluorescence excitation. Absorption can be used to determine local atomic environments and valance. Diffraction provides a means to understand or disorder, and micro-crystalline state. And fluorescence excitation spectra, unique to each atom, can be used to determine atomic spatial distributions.

In this article we will describe an experiment using fluorescence excitation to determine the spatial distribution of a germanium atoms selectively doped into a communications grade optical fiber. Communications fibers are doped in an attempt to create a perfect optical channel, having no attenuation or loss over hundreds of kilometers distance. Because the dopant atoms are introduced into the glass early in the fabrication process, subsequent high temperature treatments could result in sublimation and/or migration of the Ge atoms. Since the final dopant concentration profiles determine the performance of the fiber, a diagnostic tool is needed to characterize those profiles during the manufacturing process.

The fiber samples used in this study are commercially made by AT&T. They were fabricated starting with a hollow, cylindrical borosilicate glass form. Ultra-high purity silica is deposited from vapor on inner surface, followed by a multiple deposition cycle of pure germania. The form is then heated and collapsed into a preform, which resembles a solid glass rod approximately 2 cm in diameter. The preform is then loaded onto the drawing tower, heated in a controlled environment, and many hundreds of kilometers of fiber 125 microns in diameter are sequentially pulled, clad, and bundled.

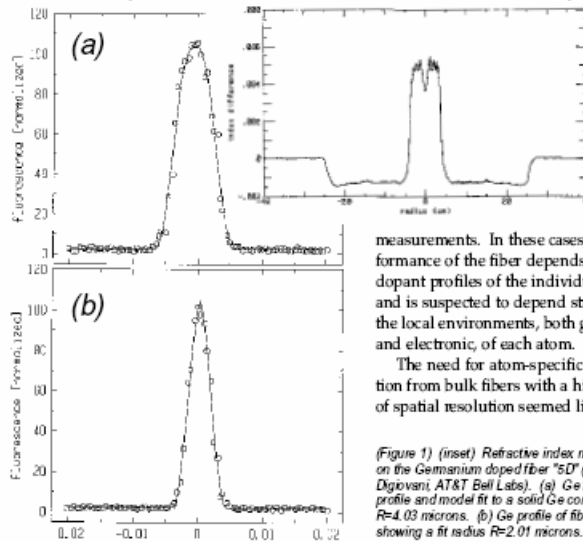
The collapse of the form requires local heating to high temperatures. At that point, Ge sublimation from the inner-surface leads to a core deficit in the final fiber. Most commonly, the fibers are characterized by measurements of the index



(Figure 2) Close-up photo of the x-ray capillary concentrator (left) and the Ge doped fiber close to its tip (mounted on a piezo-electric transducers stage). The aperture of a solid-state detector is shown at right.

of refraction as a function of position across the diameter (see inset, figure 1) The loss of Ge at the center is apparent, although the index measurements can be difficult to interpret because the wavelength of the light is such that it can propagate into the fiber, resulting in an erroneous index measure.

Some of the newer fiber designs utilize multiple dopant atoms. The performance of these fibers, some of which act as signal concentrators and amplifiers, cannot be characterized only by index



measurements. In these cases, the performance of the fiber depends on the dopant profiles of the individual species, and is suspected to depend strongly on the local environments, both geometric and electronic, of each atom.

The need for atom-specific information from bulk fibers with a high degree of spatial resolution seemed like an ap-

(Figure 1) (inset) Refractive index measurement on the Germanium doped fiber "5D" (from D. D'giovanni, AT&T Bell Labs). (a) Ge fluorescence profile and model fit to a solid Ge core with radius $R=4.03$ microns. (b) Ge profile of fiber "1024", showing a fit radius $R=2.01$ microns.

appropriate application for a tapered capillary concentrator and an x-ray fluorescence measurement. The capillaries we used were fabricated at CHESS as discussed in the article on page 41. The x-ray measurements were done on a piece of fiber whose cladding had been removed by dipping in sulfuric acid. Shown briefly in figure 2, the fibers were affixed to a vertical translation stage with both a piezo-electric element and a gear-reduced mechanical z-jack. One of the most useful features of the capillary optic for fluorescence work is that all energies can be simultaneously concentrated by the capillary. To get the highest x-ray flux onto the specimens as possible, therefore, we utilized the white x-ray beam into the C2 station at CHESS and a W/Si multilayer monochromator that passed a 1.6% energy bandpass at 15 keV. This report summarizes two experiments; one using a capillary with a 4 micron diameter exit opening, and the second experiment with a capillary of approximately 1 micron diameter.

The output of the capillary was first characterized by scanning a knife-edge at the tip of the capillary while measuring the transmitted beam. A model fit to a circular aperture establishes the radius of the incident beam to be 2.1 microns.

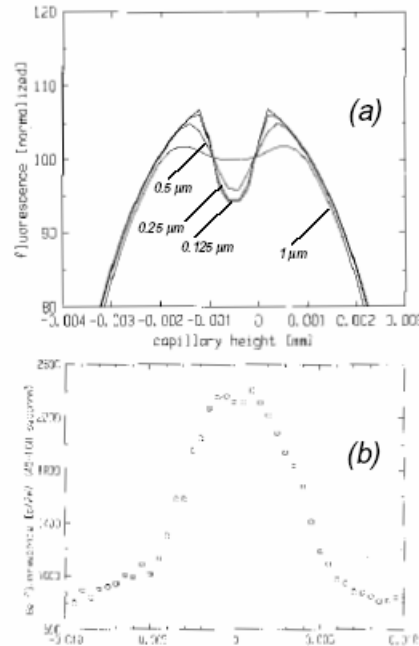
Taken care to place the fiber specimens at the same location as the knife-edge, this radius was used, without adjustment, to analyze the Ge fluorescence profiles. Shown in figure 1, the data are fitted using a model of a uniform circular beam of x-rays impinging onto a cylinder of Ge atoms that is also uniform in density. The fitted lineshape is calculated by integrating the x-ray flux over the circular beam emitted from the capillary traveling through the cylindrical Ge core; the integral is similar to convolution of two cylinders at right angles. The resulting lineshape is distinct in having a true discontinuity from zero at the edges and a broadened central peak. This shape is different from models using gaussian or lorentzian functions, which both have smooth continuous tails and fairly sharp peaks. The measured Ge core radius of 4.03 ± 0.06 microns for the "SD" fiber is in good agreement with the index of refraction plot (see inset, figure 1).

A second fiber, referred to as "1024," had both Ge and Er dopants. With a concentration below the 100 part-per-million levels, and we were unfortunately not

able to measure Er fluorescence in this first experiment. Using the same procedure discussed above, the Ge core radius was measured to be 2.01 ± 0.06 microns (figure 1b). This result is again in good agreement with the index measurements. The fitted lineshape is nearly triangular, which results from the fact that the x-ray beam and the Ge core have the same dimension. As the Ge core is translated by the capillary tip, the Ge fluorescence increases almost linearly until the core is exactly centered in the beam, at which point the overlap decreases almost linearly to zero. In other words, a triangle is the result of the convolution of the two square functions, one for the uniform circular x-ray beam aperture, and the second for the uniform cylindrical Ge core.

Although the following results are preliminary, they show the steps we must take to achieve higher spatial resolution. Figure 3 shows a scan using a one micron exit diameter capillary. At that small size, we can begin to probe the Ge deficit in the center of the fiber. The statistics and the beam size were not sufficient for a good definition of the deficit, but the analysis model can be used to explore what size capillary is needed for best spatial resolution.

Figure 3a shows the simulated experimental fluorescence profiles that would result from a scan of a .55 micron radius of constant density. The experimental data in Figure 3b correspond to the scan with a 1 micron capillary. For comparison, a profiles for 0.5, 0.25, and 0.125 micron capillaries have been calculated. Since smaller x-ray beam sizes yield less incident intensity, the most efficient experimental arrangement would use the largest possible capillary. This consideration would be especially important for those measurement involving two-dimensional imaging. The plotted curves show that going from a .25 to .125 micron x-ray beam yields very little gain in spatial



(Figure 3) (a) Calculated fluorescence profiles for a Ge core ($R=4.0$ microns) with a central void of radius 0.55 microns, comparing what data could be collected using an x-ray beam with the diameter indicated. The 1 micron plot corresponds best to the preliminary experimental data (b) collected from fiber "1024".

definition, whereby most probably such a choice would reduce the x-ray flux by a factor of 4.

Of course, the contrast shown in Figure 3b is limited by collecting data using an x-ray beam traveling through the entire diameter of the fiber. For the greatest detail of the core, it would be best to record a two-dimensional scan through a thin transverse section of the fiber. While that measurement would be fine for a post analysis of a fiber, one of the goals of this experiment was to consider a measurement probe that might be useful in a non-destructive mode, as in, for example, a non-destructive diagnostic probe *in situ*, on the manufacturing line. In that circumstance, a non-contact x-ray probe is the only available means of dopant density profiling.