

Refractive index profiles of Ge-doped optical fibers with nanometer spatial resolution using atomic force microscopy

P. Pace¹, S. T. Huntington¹, K. Lyytikäinen², A. Roberts³, J. D. Love⁴

¹ NANO-MNRF, School of Physics, The University of Melbourne, Parkville, 3010, Australia.

² Optical Fibre Technology Centre & Australian Photonics Cooperative Research Centre, University of Sydney, 206 National Innovation Centre, ATP, Eveleigh, 1430, Australia.

³ School of Physics, The University of Melbourne, Parkville, 3010, Australia.

⁴ Australian Photonics Cooperative Research Centre, Research School of Physical Sciences & Engineering, Australian National University, Canberra ACT, 0200, Australia.

shaneth@unimelb.edu.au

Abstract: We show a quantitative connection between Refractive Index Profiles (RIP) and measurements made by an Atomic Force Microscope (AFM). Germanium doped fibers were chemically etched in hydrofluoric acid solution (HF) and the wet etching characteristics of germanium were studied using an AFM. The AFM profiles were compared to both a concentration profile of the preform determined using a Scanning Electron Microscope (SEM) and a RIP of the fiber measured using a commercial profiling instrument, and were found to be in excellent agreement. It is now possible to calculate the RIP of a germanium doped fiber directly from an AFM profile.

©2004 Optical Society of America

OCIS codes: (060.2270) Fiber characterization; (290.3030) Index measurements; (999.9999) Atomic force microscopy

References and links

1. S.T. Huntington, P. Mulvaney, A. Roberts, K.A. Nugent and M. Bazylenko, "Atomic force microscopy for the determination of refractive index profiles of optical fibers and waveguides: A Quantitative study," *J. Appl. Phys.* **82**, 2730-2734 (1997).
2. Q. Zhong and D. Inniss, "Characterisation of lightguiding structure of optical fibers by atomic force microscopy," *J. Lightwave Technol.* **12**, 1517-1523 (1994).
3. D.J. Monk, D.S. Soane, R.T. Howe, "A review of the chemical reaction mechanism and kinetics for hydrofluoric acid etching of silicon dioxide for surface micromachining applications," *Thin Solid Films*, **232**, 1-11 (1993).
4. G.A.C.M. Spierings, "Wet chemical etching of silicate glasses in hydrofluoric acid based solutions," *J. Mater. Sci.* **28**, 6261-6273 (1993).
5. D.P. Tsai, Y.L. Chung, "Study of optical fibre structures by atomic force microscopy," *Opt. Quantum Electron.* **28**, 1563-1570 (1996).

1. Introduction

During the past decade, extensive research has focused on the accurate characterization of dopant location and its associated concentration in optical fibers [1-2]. The optimization of optical fiber design relies heavily on the fabrication techniques used, which in turn depends on the accurate characterization of the dopant profiles. GeO₂ is a dopant commonly used for doping the core region, raising the refractive index. On the other hand B₂O₃ or F are dopants chosen for doping the cladding region that in turn lower the refractive index. Consequently, doping the core and cladding results in a difference in structure between the two regions. If silica fiber is exposed to an etching solution such as hydrofluoric acid (HF) or buffered hydrofluoric acid (ammonium bifluoride), the two regions will be etched differently. By taking into consideration the different etch rates of these materials, the differences in the type and level of the dopant can be determined [1].

The etching/AFM procedure was first reported by Zhong and Inniss [2]. This technique involves the selective chemical etching of a cleaved end of a fiber by exposure to either HF acid, ammonium bifluoride solution or any solution capable of etching glass. The procedure also utilizes an Atomic Force Microscope that has been used extensively to measure the resulting topography of a wide range of etched optical fiber structures. Previous work performed by Huntington et al. [1] and others has shown various qualitative comparisons between AFM profiles and preform index profiles to be in good agreement. However, this direct method of comparing the two profiles is incomplete and insufficient because it does not take into consideration the effect of dopant concentration or etchant concentration when scaling AFM data to index.

In this paper we report for the first time a precise technique for quantitatively calculating a refractive index profile from an AFM profile. We examine the use of the AFM to accurately map the structure of a germanium-doped core fiber and model the wet etching characteristics of germanium. We demonstrate that an AFM profile can provide information such as the dopant concentration and the refractive index change by extracting the concentration and refractive index profiles from a depth profile that is governed by the chemical kinetics of doped silica. The application of the AFM to this problem provides a means by which dopant concentration can be mapped with a lateral resolution of ~ 1 nm. This represents a fundamental enhancement of the optical resolution typically associated with the characterization of fiber samples, such as the refracted near field technique.

2. Wet chemical etching

When a cleaved endface of a fiber is etched chemically in HF solution, the etch rate of SiO_2 is a first-order reaction in the regime where the molar concentration (C) of the solution is less than 4M HF [3]; for $C > 4$ M HF, non-linear reactions will start to occur [3]. The expression relating the etch rate of doped silica fibers to the molar concentration of the dopant is presented by Eq. (1) [3]:

$$\frac{dx}{dt} = k[C]^n \quad (1)$$

where x is the etch depth, dx/dt is the etch rate of the sample, k is the reaction rate constant, C is the molar concentration of the dopant and, n is the reaction order. Eq. (1) can be applied to both doped and undoped silica. It is also known that the etch rate of doped silica is dependent on factors such as the type of dopant used and the dopant concentration [1,4]. The above equation shows that the etch rate is a non-linear function of the concentration unless $n=1$.

3. Characterization and discussion

By investigating the etching mechanisms in Ge-doped fibers, a quantitative comparison can be made between an AFM profile and the refractive index profile of the fiber. For this comparison, a special Ge-doped, three-level ring fiber (OFTC OD0032_01_A) was deliberately chosen because of the known variation in the molar concentration of Ge-doped silica across the fiber. The Ge-doped rings will have a higher etch rate than for the silica regions, so that a depressed region in the AFM profile represents a peak in the refractive index profile of the fiber.

The fiber was stripped of its coating and cleaved with a Siemens S46999-M9-A8 cleaver. The endface of each fiber was immersed in HF solution at a concentration of between 48vol-%-2vol-% (24M-1M) for various etch times at ambient temperature. The fiber samples were measured with an AFM (Digital Instruments Dimension 3100). Contact mode imaging was used utilizing NT-MDT (CSC21/Si3N4/Al) cantilever tips. Figure 1 shows an AFM image of a Ge-doped, three-level ring fiber etched in 2M HF solution for 3 min with the inner, middle and outer rings having GeO_2 molar concentration in percent of 17, 8 and 4, respectively. A

radial cross-section of the Ge-doped, three-level ring fiber is shown in Fig. 2, showing the three depressed regions due to the variation of germanium concentration.

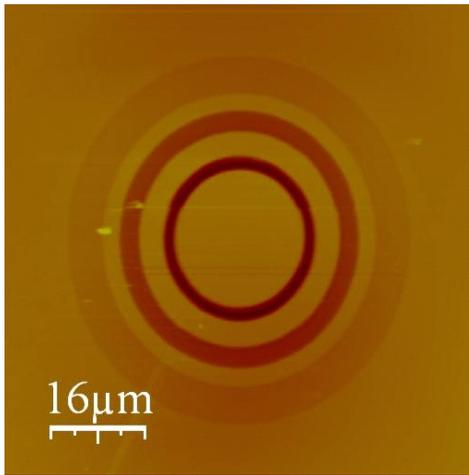


Fig. 1. AFM image of Ge-doped, three-level ring fiber etched in 2M HF solution for 3 min.

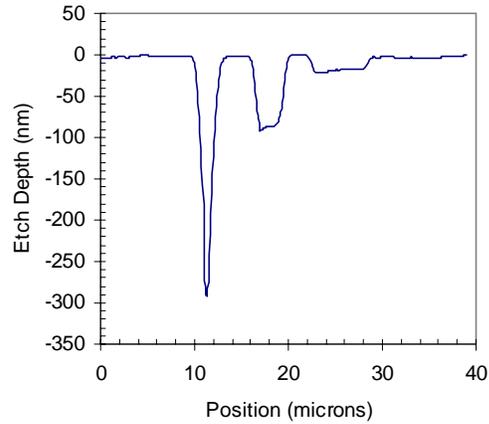


Fig. 2. Radial cross-section of the etched rings shown in Fig. 1

As explained previously the fibers were etched in several HF solutions for various etch times and this enabled the measurements of the etch rate for the inner, middle and outer rings to be performed. The etch depth exhibited a linear dependence on the etch time and as no etching occurs until the fiber is placed in the HF solution, the line of best fit necessarily passes through the origin as shown in Fig. 3. The errors presented in Fig. 3 represent the uncertainty in the height measurements from the AFM obtained by employing a calibration grid. The timing error was assumed to be 2 sec, representing the time it takes the etched fiber to be rinsed free of the acid.

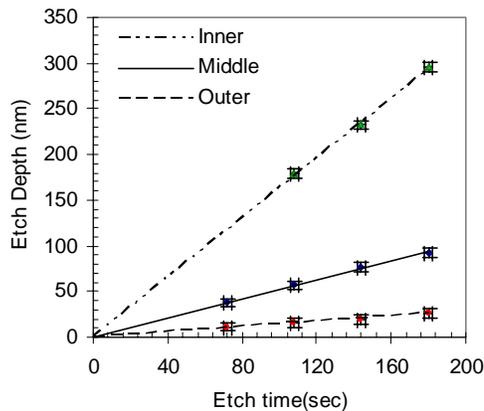


Fig. 3. Etching of Ge-doped, three-level ring fiber in 2M HF.

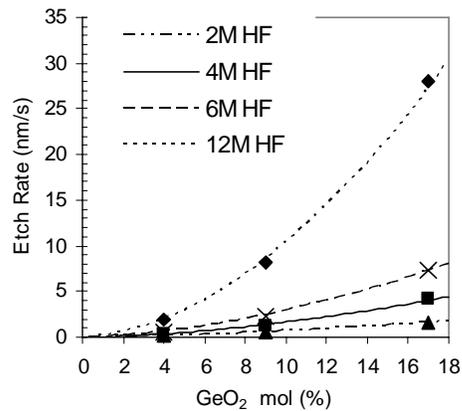


Fig. 4. Measured relationship of the etch rate of Ge-doped core fibers as a function of GeO_2 content etched in HF solution.

The Ge-doped core fiber was found to exhibit a monotonic etch rate dependence with GeO_2 concentration as presented in Fig. 4. Using Eq. (1), a power curve was fitted to this data using

a least squares fitting routine enabling the calculation of the reaction constants to be performed as shown in Table. 1.

Table 1. Summary of measured reaction constants.

HF (M)	k	\pm	n	\pm
2	0.014	0.004	1.67	0.13
4	0.035	0.025	1.67	0.25
6	0.055	0.013	1.73	0.09
12	0.154	0.065	1.83	0.15

We have showed that the etch rate for germanium is a non-linear function of the dopant concentration and the reaction constants are dependent on the molar concentration of HF. These important outcomes were used in the extraction of a dopant concentration profile from a depth profile which allows us to relate etch depth to refractive index presented in the next section.

The second part of this experiment was to compare quantitatively an AFM profile to a refractive index profile of the fiber. Firstly, a Scanning Electron Microscope (SEM) equipped with an energy dispersive spectrometer (EDS) was used to obtain a dopant concentration profile of the preform. A section of the preform was cut and polished with 6 μm and 1 μm diamond paste and carbon coated for microanalysis. The germanium concentration profile was obtained by EDS on a Philips Scanning Electron Microscope XL30. Automatic line scan operation was used to obtain points every 10 μm across each Ge-doped ring of the preform at 300x magnification. A 20kV accelerating voltage was used with 125nm beam spot size with an estimated spatial resolution at this voltage for silica was about 2-3 μm . The spatial data for the concentration profile was calibrated against a calibration mesh. In comparing both profiles the measured data relating both etch rate and GeO₂ concentration were used as shown in Fig. 4. This enabled an AFM profile of a Ge-doped, three-level ring fiber etched in 2M HF solution to be converted from depth values to concentration values using Eq. (1) and the measured reaction constants shown in (Table. 1). Figure 5 shows a conversion of a depth profile to a concentration profile with improved spatial resolution.

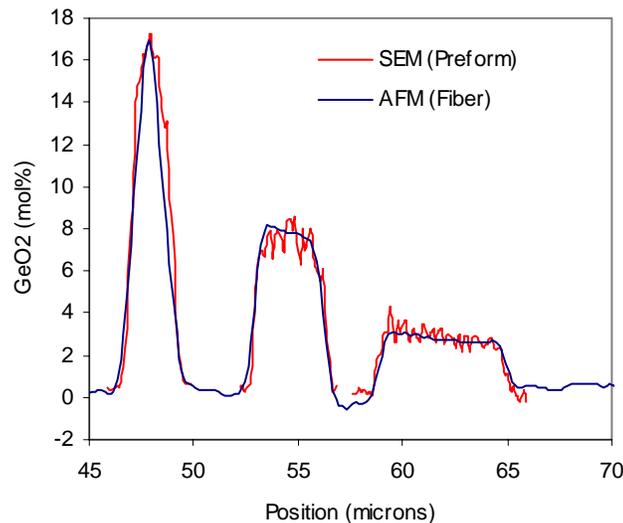


Fig. 5. Comparison between GeO₂ concentration profiles calculated from AFM data of the fiber and measured SEM data measured from the preform.

The above diagram shows excellent agreement between the measured dopant concentration profile of the preform and the calculated concentration profile of the fiber. The difference between the two profiles is mainly due to the lateral resolution achieved by the two techniques. Also, the preform is three orders of magnitude greater in size than the fiber so a scaling factor was introduced to convert it to fiber dimensions for comparison.

Now that the concentration profile has been accurately determined, the conversion to refractive index values can be made using the fact that the GeO_2 doping ratio of 1 mol% corresponds to a relative difference in refractive index, $\Delta n = 0.1\%$ [5]. The quantitative comparison was successfully made between an AFM profile and refractive index profile of the fiber using the data obtained from Fig. 5 and assuming no stress effects. This is shown in Fig. 6 and the refractive index profile of the fiber was measured by cleaving the fiber sample and utilizing an EXFO HR-NR9200 Fiber Refractive Index Profiler.

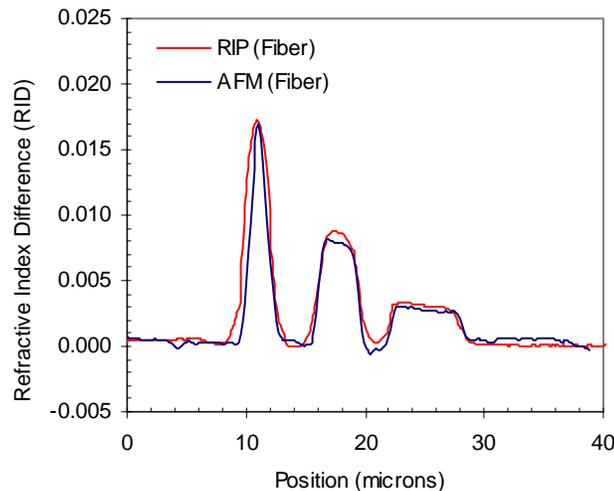


Fig. 6. Quantitative comparison between refractive index profiles calculated from AFM data and measured refractive index data.

Figure 6 shows that the calculated refractive index profile from a depth profile was successfully performed with enhanced spatial resolution. The refractive index profile calculated from AFM data shown in the above diagram shows excellent agreement when compared to the measured refractive index profile. By exploiting the superior lateral resolution exhibited by the AFM profile we have improved the spatial resolution of the refractive index profile to the nanometer scale.

The acquisition of accurate information from the etching technique relies heavily on a clear understanding of the types of errors involved. During the process of etching, as the depressed regions are created there is a small amount of side etching that occurs which effectively changes the final profile. The location of index peaks in the profile do not change, but the actual measured core width may be increased due to this effect. Figure 7 shows the measured core width and percentage error of a Corning SMF-28 fiber using 24M HF for varying times. For short etch times the core width has a percentage error of only $\sim 1\%$. For larger times this error becomes significant and the technique can no longer be relied upon for accurate profiles. The nominal manufacturers core width is $8.3\mu\text{m}$ which is in good agreement with that determined from Fig. 7. The etching performed to obtain the data shown in Fig. 6 has an error $< 1\%$ due to the short times used and the low concentration of HF. With

care, the side etching problem can be readily controlled, and superior resolution data such as that shown in Fig. 6 can be produced.

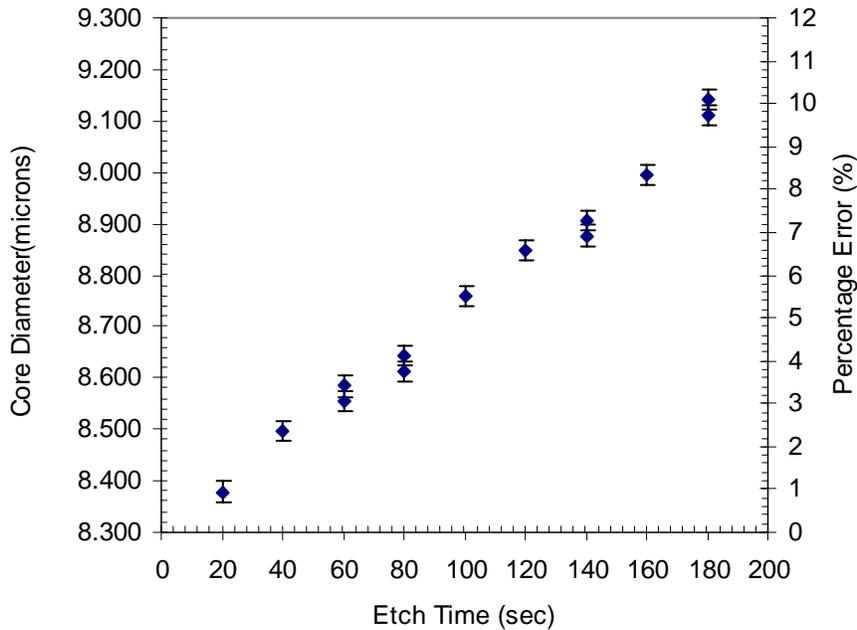


Fig. 7. Variation of core diameter with etch time for a standard SMF-28 fiber.

4. Conclusion

We conclude that the characterization of the wet chemical etching of germanium doped fibers in HF solution was successfully performed. The accurate measurements obtained by AFM showed that the etch rate for germanium is a non linear function of the dopant concentration. A method was employed in calculating the refractive index profile from a depth profile and showed that a spatial resolution of $\sim 1\text{nm}$ was achievable. This represents a fundamental enhancement of the resolution typically associated with the characterization of fiber samples and will assist in the development of fibers for new photonic devices.

Acknowledgments

The authors acknowledge the support of the University of Melbourne and University of Sydney Nodes of the NANO-MNRF. P.Pace acknowledges the financial support of the Australian Research Council and the Particulate Fluids Processing Centre, School of Chemistry, University of Melbourne for the use of the atomic force microscope. K. Lyytikainen acknowledges the fiber fabrication team of Optical Fibre Technology Centre (OFTC) for manufacturing the preform, Ian Kaplin from the Electron Microscopy Unit, University of Sydney for help in SEM measurements and assistance from S. Ringer, P. McNamara, A. Carter and S. Fleming. Jarek Abramczyk from Nufern is acknowledged for supplying the fiber RIPs. A. Roberts acknowledges the financial support of the Australian Research Council.