Imaging cleaved surfaces of SiO2:GeO2 optical fibers by atomic force microscopy

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Short range structural order is now well known in silica glass, but a lot of investigations are still in progress to characterise and understand medium range order. Here, we investigate by atomic force microscopy (AFM) the morphology of freshly cleaved surfaces of Ge-doped silica optical fibers. The comparative study of the core and the cladding shows that measurements are sensitive to Ge doping. This sensitivity depends on the imaging environment and mode. We carry out investigations, down to the nanometric scale, in air and liquid with both contact (topography, friction) and tapping modes.

Introduction

In silica optical fibers Ge-doping is used to increase the refractive index of the core which constitutes the guiding part of the fiber. One expects that Ge-doping modifies the intrinsic inhomogeneities in the glass structure at medium length scale. These inhomogeneities may have several origins such as, for instance, frozen-in density fluctuations, compositional fluctuations or microscopic intrinsic stresses which are postulated to exist in topologically disordered structures. They are expected to be of the order of few nanometers. At this length scale, AFM has emerged as an appropriate tool for probing the local characteristics of oxide glasses [1,2]. Here, we investigate by AFM the surface properties of fiber cross sections carefully cleaved with a diamond. The atomic force microscope is used under both tapping mode and contact mode. In addition to the usual topographic images, we record images representing the surface friction-behavior (using the lateral force signal under contact mode) and the adhesion forces (using the phase signal under tapping mode). At first, we compare the sensitivity of these different modes to the Ge-doping by recording large-scale images including both the core and the cladding of the fibers. Then, 500 nm-wide images are achieved to study small length scale properties of the fracture surface.

Experimental details

Two fibers of different Ge-concentration have been studied. Fig. 1 shows their simplistic refractive index profile. Hereafter, the fibers are labelled by the step of index between the core and the cladding. The F24 fiber has the greatest germanium concentration and an inner cladding doped with fluor. F-doping is used to decrease the refractive index. Fibers are cleaved with a cutting tool producing a "mirror" region all over the surface as shown in Fig. 2. Note that the crack front propagates from the defect clearly visible on the edge of the fiber. Freshly cleaved surfaces were imaged under ambient conditions using a Nanoscope IIIa, Dimension 3100 from Digital Instruments, using standard silicon tips under tapping mode and standard Si_3N_4 tips under contact mode.

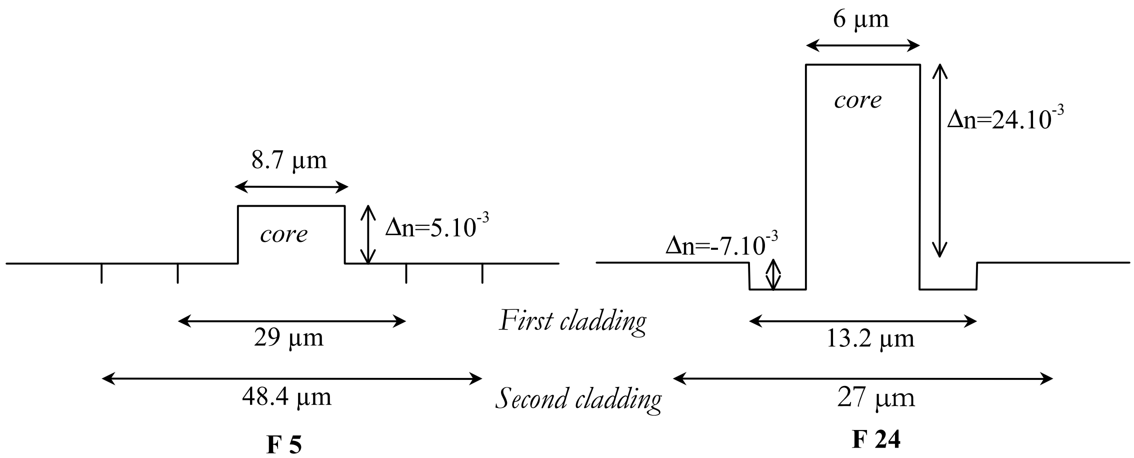


Figure 1 : schematic plot of the radial profile of refractive index of the two studied fibers, F5 (left) and F24 (right).

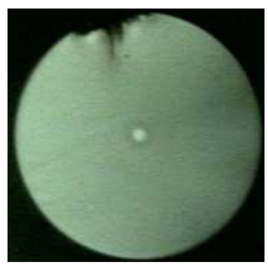
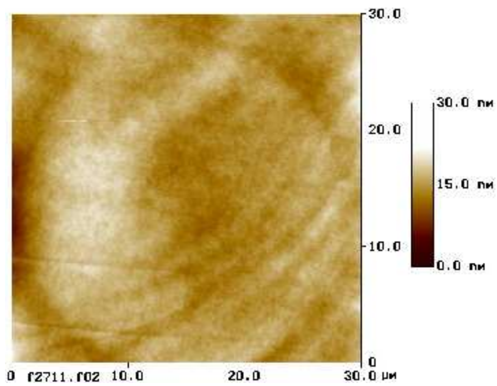


Figure 2 : typical view of one F24 fiber cross-section with an optical microscope. The outer diameter of the fiber is 125 µm.

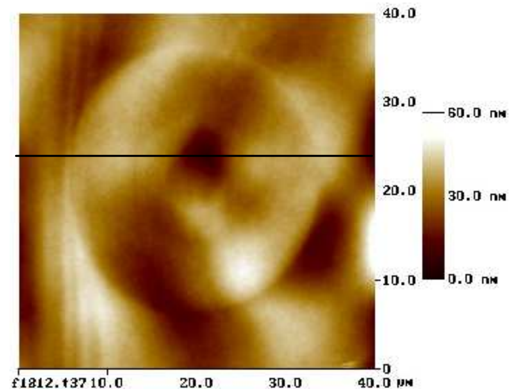
AFM investigations at micrometric length scale

Topography observations

Typical images of the fracture surface topography around the core and the cladding are shown in Fig. 3. The images are very similar using either the tapping mode or the contact mode. One observes clearly a quite strong contrast between the core, the cladding and the supporting tube. For both fibers, the cladding appears above the mean level of the supporting tube and the core forms a depression at the centre. It is well known that this topography originates in the release, in the plane of the fracture, of residual internal stresses stored in the fiber [3]. The stress field is in part produced by the radial variation of expansion coefficient occurring in the fiber due to the radial variation of glass composition. The cladding is generally under compressive stress whereas the core is under tensile stress. The characteristic diameters of the different regions revealed in these measurements are in agreement with those deduced from the index profile. One notes in Fig. 3 the presence of unwanted waves all over the fracture surface which slightly distort the characteristic profile.



F5



F24

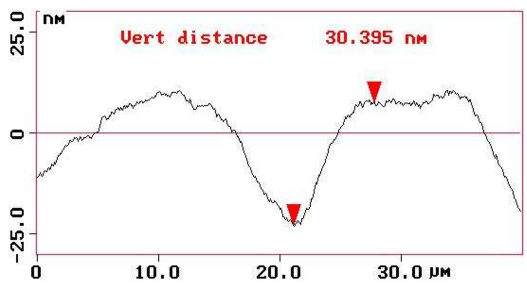
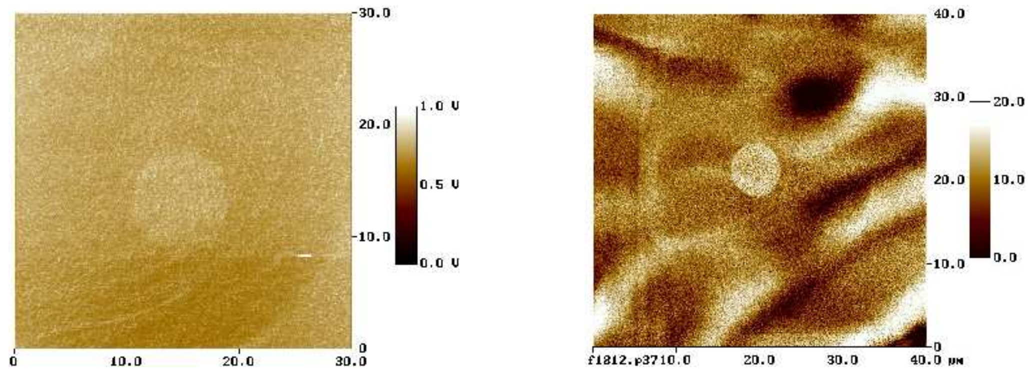


Figure 3 : typical large length scale topography measurements of F5 (left) and F24 (right) fibers under contact mode. The first cladding of F5 fiber is clearly visible above the mean level of the image. Its diameter matches the one deduced from index profile (~ 29 µm). The first and the second cladding of F24 fiber are indistinguishable, both appearing above the mean level of the image. For both fibers, the core appears as a depression at the centre. This depression is nearly 30 nm deep in F24 fiber as seen on the profile extracted from the topographical image of F24 (bottom).

Friction mapping

Besides the ordinary topographical signal (resulting from normal force fluctuations), lateral force fluctuations can be simultaneously recorded on the same area. It allows to image frictional properties of the fracture surfaces. Fig. 4 shows such images simultaneously registered with those of Fig. 3. A quite strong contrast is clearly evidenced between the core and the cladding which can be unambiguously attributed to variation in Ge-concentration. An accurate measurement of the core diameter is then possible. Moreover, a weak contrast is observed at the interface between the cladding and the supporting tube which also allows its diameter determination. Although this frictional signal is not quantitatively understood, one knows that it directly relates to the chemical composition of the surface. Its amplitude can be enhanced or decreased by varying the experimental environment. For example, the frictional contrast observed between the core and the cladding of F5 fiber under air environment completely disappears under aqueous electrolyte of pH ~ 5.

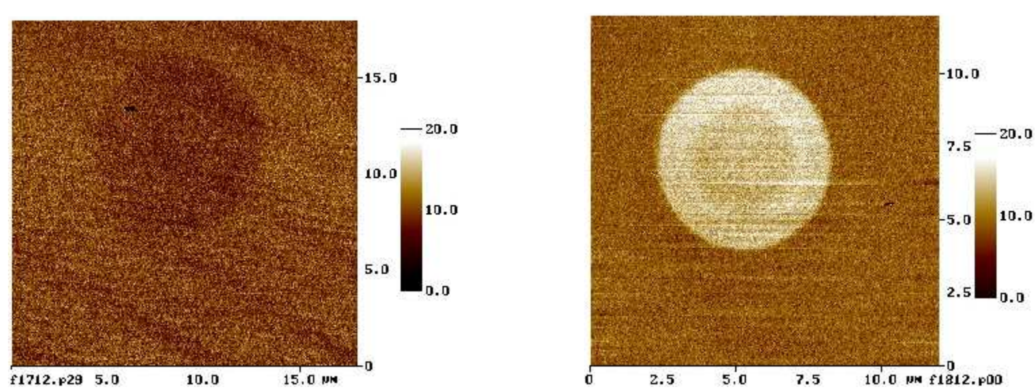


F5 **F24**

Figure 4 : typical frictional images of F5 (left) and F24 (right) fibers. One can easily recognise the core of each fiber. Their diameter nicely coincides with that shown on the index profile plot of Fig. 1.

Phase imaging

Finally, the phase of the cantilever oscillation under tapping mode can be recorded simultaneously with the topographical image. The phase signal relates to the dissipation energy associated to the tip-sample contact. It may vary with many properties of the surface such as for instance the adhesive or the frictional properties. As shown in Fig. 5, the amplitude of the phase signal is highly sensitive to the surface chemical composition. The phase image of F24 fiber in Fig. 5 highlights the existence of two regions inside the core itself probably related to different Ge-concentration, which were not mentioned by the manufacturer.



F5 **F24**

Figure 5 : typical phase images recorded under tapping mode on F5 (left) and on F24 (right) fibers.

nanometre scale investigations

We acquired 500 nm-wide and 250 nm-wide images both inside the core and the cladding regions of the F5 and F24 fibers. All surfaces exhibit a similar nanoscale granular structure in topography. However, one should remind that the spatial resolution is limited by the shape and the size of the tip. The average lateral dimension of the hillocks measured with Si_3N_4 tips under contact mode is indeed more than twice as large as that observed with silicon tips (~ 10 - 20 nm) using the tapping mode. The calculated power spectral density curves of these images are very similar when moving from the core to the cladding of the fibers. We can deduce from these observations that the ultimate nanoscale structure of the cleaved surfaces is unfortunately not resolved so we do not learn much of information at the nanometre scale. Finer tips should be tested.

Conclusion

We have shown the relevance of AFM measurements to control the morphology of both the core and the cladding of Ge-doped silica optical fibers. However, obtained results are mainly qualitative. The phase signal under tapping mode and the frictional signal under contact mode are highly sensitive to variation in chemical composition. The amplitude of these signals could be monitored by operating under controlled environment. At the nanometre scale, the different regions of the fibers are indistinguishable since the standard Si-tips of radius ~ 10 nm do not allow to resolve the intrinsic nanostructure of the surfaces.

Acknowledgment

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