Changes in etch rate due to hydrogen loading and UV-irradiation in phosphorus-doped fibers

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Abstract: Changes in etch rate due to hydrogen loading and subsequent UV-irradiation have been observed for phosphorus-doped fiber cores using an atomic force microscope. The etch rate of the core is found to decrease after hydrogen loading. UV-irradiation of the hydrogenated fiber enhances the core etch rate considerably, resulting in a higher etch rate as compared to the pristine fiber. The change in etch rate does not depend on pulse fluence, but only on total dose. We attribute the changes in etch rate to a hydrogen- and radiation-induced modification of color center population.

1. Introduction

Atomic force microscopy (AFM) of etched fiber end-faces yields topographic information on a nanometer scale that can be related to the fibers light guiding properties [1,2]. However, the exact dependence of etch rate on dopant and defect concentration on the one hand and mechanical properties of the fiber on the other hand has not been clarified so far. Particularly, the origins of changes in etch rate due to UV-illumination [2-4] still require an appropriate explanation. Inniss *et al.* reported an asymmetric etch profile for hydrogen loaded standard telecommunication fiber caused by side exposure to UV at 244 nm [2]. For hydrogenated phosphorus-doped fibers, preferential etching at the core-cladding boundary has been observed and attributed to stress-assisted UV-initiated bond breaking at the interface [4].

Illumination of hydrogen loaded phosphorus-doped fibers with ArF-laser irradiation at 193 nm results in an increase of the fibers refractive core index [5]. Highly phosphorus-doped low-loss optical fibers are an attractive gain medium for Raman fiber amplifiers and lasers [6], where photosensitivity can be exploited by writing Bragg gratings as reflectors directly in the active fiber. As for Ge-doped fibers, the origins of photosensitivity in phosphorus-doped fibers have not yet been completely clarified. Generally, the interaction of UV-light with glass leads to color center as well as to density changes of the matter, both modifying the refractive index of the glass. Additionally, the change in density results in a modification of the stress distribution introduced in the fiber during the fabrication process [7]. The stress change contributes negatively to the net index change via the photoelastic effect [7]. For phosphorus-doped fibers, densification has been proposed to be the dominating photosensitive mechanism [8]. A corresponding increase in axial core stress has been confirmed experimentally [9].

In this paper, we present changes in etch rate of phosphorus-doped fibers due to hydrogen loading and subsequent UV-irradiation. Our results suggest that the etch rate is predominately governed by the color center population in the fiber core.

2. Experiment and results

The fiber investigated within this work is a phosphorus-doped single-mode fiber drawn from a preform manufactured by the modified chemical vapor deposition (MCVD) method. The core layers of the preform are doped with 12 mol% P_2O_5 and are surrounded by inner cladding layers doped with 1 mol% P_2O_5 and 1 at% F. The corresponding peak core index change is $\Delta n=1\times10^{-2}$. Before irradiation, the fiber is hydrogen loaded for about two weeks at a pressure of 110 bar at room temperature. Two pieces of hydrogenated fiber are exposed to 193 nm light from an Excimer-laser under different radiation conditions, both resulting in a total fluence of 1 kJ/cm^2. For the first fiber, pulse fluence is 50 mJ/cm^2 at a repetition rate of 1 Hz, resulting in an exposure time of about 330 minutes. The second fiber is illuminated with a pulse fluence of 350 mJ/cm^2 at 20 Hz repetition rate and a corresponding exposure time of 142 seconds.

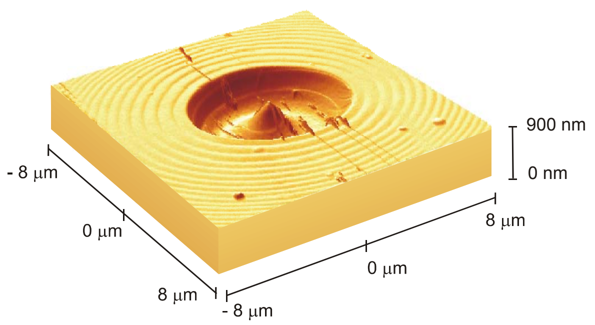

Fig. 1. AFM image of phosphorus-doped fiber after 180 seconds etching in hydrofluoric acid. The inner cladding layers are etched slower than the core layers and can clearly be observed.

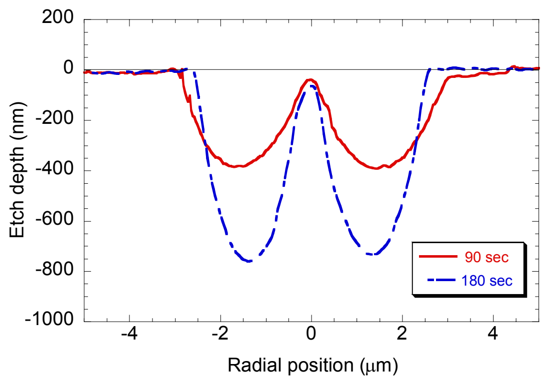

Fig. 2. Etch dynamics of the pristine phosphorus-doped fiber. Etch depth changes linearly with etch time. The difference in diameter for the two etch times reflects the ellipticity of the fiber core.

For AFM-observation, the samples are cleaved and etched in a 5% HF solution for 90 and 180 seconds, respectively. The AFM (TopoMetrix Explorer) is operated in contact mode in air with a standard V-shaped silicon nitride cantilever. The topography of the pristine fiber after 180 seconds of etching is shown in Fig. 1. The highly phosphorus-doped core layers are etched faster than the surrounding inner cladding layers. The dip in the center of the core is caused by dopant out-diffusion during collapsing of the preform tube. The core is slightly elliptical and has a diameter (FWHM) of 4.5 µm.

In Fig. 2, the etch profile of the pristine fiber is illustrated for two different etch times. A linear dependence of etch depth on etch time is found, as has already been reported for Ge-doped fibers [1]. The difference in diameter between the two profiles shown in Fig. 2 reflects the ellipticity of the fiber core. A linear dependence of etch depth on time was also observed for the hydrogen loaded and UV-irradiated fibers. However, the change in etch depth over time is found to change significantly compared to the pristine fiber.

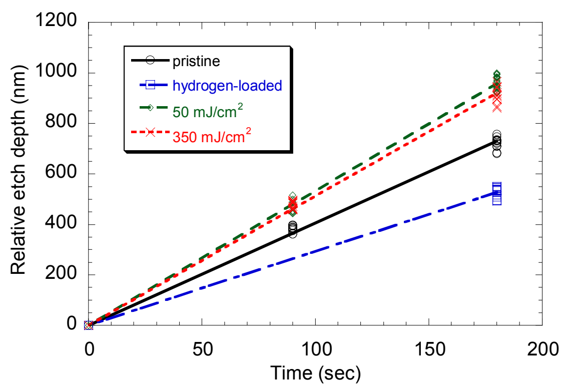

Fig. 3. Relative etch depth as function of time for the etch maxima. The etch maxima depend linearly on time.

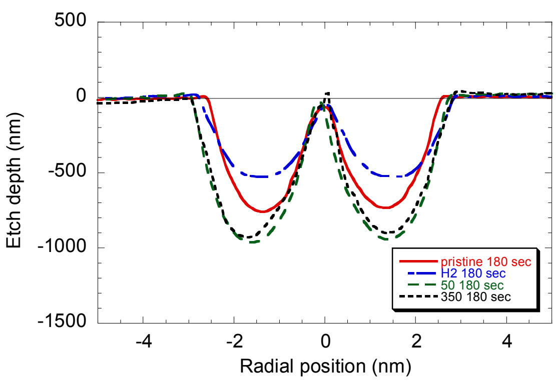

Fig. 4. AFM cross-sectional profiles of the four different samples under investigation..

In Fig. 3, the maximum etch depth occurring at a radial position of about ± 1.5 µm is plotted as a function of etch time for all samples under investigation. The slope of the linear fit defines the etch rate of the corresponding sample. The etch rate is about 4 nm/s for the pristine fiber and is reduced by almost 27% due to hydrogen loading. The etch rates of the two UV-irradiated fiber samples are also shown in Fig. 3. In comparison not only to the etch profile of the hydrogenated, but also to the pristine fiber, the etch depth of the core region has increased for both pulse fluences. No significant dependence of etch rate on pulse fluence can be observed. The etch rate of the irradiated samples has increased by about 30% compared to the pristine sample and by almost 80% compared to the hydrogenated sample. In Fig. 4, the etch profiles for the four samples are shown for comparison. Apart from the differences in diameter caused by the ellipticity of the core, the four profiles scale almost linearly. Particularly, no preferential etching at the core-cladding interface [4] can be found.

3. Discussion

For the fiber under investigation, index changes on the order of several 10^{-4} have been found after irradiation with a total fluence of 1 kJ/cm^2 [9]. As the total index change between core and inner cladding of the fiber is $\Delta n=1\times10^{-2}$, this corresponds to an increase of several percent in core refractive index. If etch rate was linearly related to refractive index, as reported for Ge-doped fibers in [1], the increase in etch rate after UV-irradiation should almost be one order of magnitude smaller than the 27% reported in the previous section. Furthermore, hydrogen loading increases the core refractive index of optical fiber [10], which should result in an increasing etch rate and not in an etch rate reduction of 30%. We thus conclude that the etch rate is not governed predominately by the refractive index of the fiber core.

In [3], an increase in etch rate with fiber drawing tension has been reported for nitrogen-doped fibers drawn from the same perform. This result could indicate an increase in etch rate with compressive core stress, as the fibers core stress was found to decrease linearly with drawing tension [3,7]. However, for phosphorus-doped fibers, no change in stress with hydrogen loading and an increasing core stress with UV-exposure has been found [9]. If stress was the parameter dominating the etch rate, we would thus expect no change in etch rate with hydrogen loading and a decreasing etch rate after UV-illumination. This is in contradiction with the results presented in the previous section, so we conclude that stress does not influence the etch rate in our case.

UV-irradiation alters the defect population introduced into optical fibers during the fabrication process [11]. The resulting change in absorption is known to contribute significantly to refractive index change via the Kramers-Kronig relationship. Several defects are characterized by an unpaired electron, which could facilitate the adsorption of hydrofluoric acid and thus speed up the etching process. In contrast, the interstitial hydrogen present in the fiber after hydrogen loading might saturate the defects and slow down the etching. We thus suggest that the etching characteristics reported in this paper might be governed by color center concentrations and their modification due to hydrogen loading and UV-illumination.

In contrast to [4], we did not observe any preferential etching at the core/cladding interface after UV-irradiation. The fiber used within this study has about the same phosphorus concentration as the fiber investigated in [4], but no details about irradiation conditions were given in the article. The effect reported in [4] might thus only occur for a limited range of irradiation parameters.

4. Conclusion

Changes in etch rate due to hydrogen loading and subsequent UV-irradiation have been observed in phosphorus-doped fibers. Etch rate decreases after hydrogen loading by about 27% and increases by about 30% after UV-irradiation with respect to the pristine fiber. No etch rate dependence on pulse fluence is observed. Changes in defect population might be the reason governing the observed etching behavior.

5. References

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