



Copyright Notice

This paper was published in *Optics Express* and is made available as an electronic reprint with the permission of OSA. The paper can be found at the following URL on the OSA website: <http://dx.doi.org/10.1364/OE.20.014508>. Systematic or multiple reproduction or distribution to multiple locations via electronic or other means is prohibited and is subject to penalties under law.

(Article begins on next page)

Fabrication of corrugated Ge-doped silica fibers

P. Wróbel,^{1,*} T. Stefaniuk,¹ T. J. Antosiewicz,² A. Libura,³ G. Nowak,³ T. Wejrzanowski,⁴ M. Andrzejczuk,⁴ K. J. Kurzydłowski,⁴ K. Jedrzejewski,⁵ and T. Szoplik¹

¹ University of Warsaw, Faculty of Physics, Pasteura 7, 02-093 Warsaw, Poland

² Chalmers University of Technology, Department of Applied Physics, SE-412 96 Gothenburg, Sweden

³ Institute of High Pressure Physics, Polish Academy of Sciences, Sokolowska 29/37, 01-142 Warsaw, Poland

⁴ Warsaw University of Technology, Faculty of Materials Science and Engineering, Woloska 141, Warsaw 02-507, Poland

⁵ Warsaw University of Technology, Faculty of Electronics and Information Technology, Institute of Electronic Systems, Nowowiejska 15/19, 00-665 Warsaw, Poland

*pwrobel@igf.fuw.edu.pl

Abstract: We present a method of fabricating Ge-doped SiO₂ fibers with corrugations around their full circumference for a desired length in the longitudinal direction. The procedure comprises three steps: hydrogenation of Ge-doped SiO₂ fibers to increase photosensitivity, recording of Bragg gratings with ultraviolet light to achieve modulation of refractive index, and chemical etching. Finite-length, radially corrugated fibers may be used as couplers. Corrugated tapered fibers are used as high energy throughput probes in scanning near-field optical microscopy.

©2012 Optical Society of America

OCIS codes: (230.0230) Optical devices; (060.2380) Fiber optics sources and detectors; (060.3738) Fiber Bragg gratings, photosensitivity; (180.4243) Near-field microscopy; (180.5810) Scanning microscopy.

References and links

1. G. Brambilla, "Optical fibre nanowires and microwires: a review," *J. Opt.* **12**(4), 043001 (2010), <http://dx.doi.org/10.1088/2040-8978/12/4/043001>.
2. X. Zeng and D. Fan, "Electromagnetic fields and transmission properties in tapered hollow metallic waveguides," *Opt. Express* **17**(1), 34–45 (2009).
3. Y. Wang, W. Srituravanich, C. Sun, and X. Zhang, "Plasmonic Nearfield Scanning Probe with High Transmission," *Nano Lett.* **8**(9), 3041–3045 (2008).
4. C. C. Neacsu, S. Berweger, R. L. Olmon, L. V. Saraf, C. Ropers, and M. B. Raschke, "Near-Field Localization in Plasmonic Superfocusing: A Nanoemitter on a Tip," *Nano Lett.* **10**(2), 592–596 (2010).
5. S. Mühlig, C. Rockstuhl, J. Pniewski, C. R. Simovski, S. A. Tretyakov, and F. Lederer, "Three-dimensional metamaterial nanotips," *Phys. Rev. B* **81**(7), 075317 (2010).
6. A. Leung, P. M. Shankar, and R. Mutharasan, "A review of fiber-optic biosensors," *Sens. Actuators B Chem.* **125**(2), 688–703 (2007).
7. R. Gumenyuk, C. Thür, S. Kivistö, and O. G. Okhotnikov, "Tapered fiber Bragg gratings for dispersion compensation in mode-locked Yb-doped fiber laser," *IEEE J. Quantum Electron.* **46**(5), 769–773 (2010).
8. T. Allsop, F. Floreani, K. P. Jedrzejewski, P. V. S. Marques, R. Romero, D. J. Webb, and I. Bennion, "Spectral Characteristics of Tapered LPG Device as a Sensing Element for Refractive Index and Temperature," *J. Lightwave Technol.* **24**(2), 870–878 (2006).
9. G. Nemova and R. Kashyap, "Fiber-Bragg-grating-assisted surface plasmon-polariton sensor," *Opt. Lett.* **31**(14), 2118–2120 (2006).
10. K. C. Patra, R. Singh, E. K. Sharma, and K. Yasumoto, "Analysis of transmission characteristics of long period gratings in tapered optical fibers," *Appl. Opt.* **48**(31), G95–G100 (2009).
11. R. W. Ziolkowski and J. B. Judkins, "Nonlinear finite-difference time-domain modeling of linear and nonlinear corrugated waveguides," *J. Opt. Soc. Am. B* **11**(9), 1565–1575 (1994).
12. J. M. Lázaro, A. Quintela, W. Urbanczyk, J. Wojcik, and J. M. Lopez-Higuera, "Bragg Gratings Written in Tapered Solid-Core Photonic Crystal Fibers," *IEEE Photon. Technol. Lett.* **22**(14), 1048–1050 (2010).
13. K. O. Hill, Y. Fujii, D. C. Johnson, and B. S. Kawasaki, "Photosensitivity in optical fiber waveguides: Application to reflection filter fabrication," *Appl. Phys. Lett.* **32**(10), 647 (1978).
14. R. Stöckle, Ch. Fokas, V. Deckert, R. Zenobi, B. Sick, B. Hecht, and U. P. Wild, "High-quality near-field optical probes by tube etching," *Appl. Phys. Lett.* **75**(2), 160–162 (1999).
15. S. K. Mondal, A. Mitra, N. Singh, S. N. Sarkar, and P. Kapur, "Optical fiber nanoprobe preparation for near-field optical microscopy by chemical etching under surface tension and capillary action," *Opt. Express* **17**(22), 19470–19475 (2009).

16. P. J. Lemaire, R. M. Atkins, V. Mizrahi, and W. A. Reed, "High pressure H₂ loadings as a technique for achieving ultrahigh UV photosensitivity and thermal sensitivity in GeO₂ doped optical fibres," *Electron. Lett.* **29**(13), 1191 (1993).
17. J. Albert, K. O. Hill, B. Malo, D. C. Johnson, F. Bilodeau, I. M. Templeton, and J. L. Brebner, "Maskless writing of submicrometer gratings in fused silica by focused ion beam implantation and differential wet etching," *Appl. Phys. Lett.* **63**(17), 2309–2311 (1993).
18. V. Grubsky, D. S. Starodubov, and J. Feinberg, "Photochemical reaction of hydrogen with germanosilicate glass initiated by 3.4–5.4eV ultraviolet light," *Opt. Lett.* **24**(11), 729–731 (1999).
19. M. Fokine, "Manipulating glass for photonics," *Phys. Status Solidi A* **206**(5), 880–884 (2009).
20. F. Dürr, G. Kulik, H. Limberger, R. Salathé, S. Semjonov, and E. Dianov, "Hydrogen loading and UV-irradiation induced etch rate changes in phosphorus-doped fibers," *Opt. Express* **12**(23), 5770–5776 (2004).
21. P. Pace, S. Huntington, K. Lyytikäinen, A. Roberts, and J. Love, "Refractive index profiles of Ge-doped optical fibers with nanometer spatial resolution using atomic force microscopy," *Opt. Express* **12**(7), 1452–1457 (2004).
22. L. Novotny and B. Hecht, *Principles of Nano-Optics* (Cambridge University Press, 2006).
23. A. Drezet, A. Cuche, and S. Huant, "Near-field microscopy with a single-photon point-like emitter: resolution versus the aperture tip," *Opt. Commun.* **284**(5), 1444–1450 (2011).
24. T. J. Antosiewicz and T. Szoplik, "Corrugated metal-coated tapered tip for scanning near-field optical microscope," *Opt. Express* **15**(17), 10920–10928 (2007).
25. T. J. Antosiewicz and T. Szoplik, "Corrugated SNOM probe with enhanced energy throughput," *Opto-Electron. Rev.* **16**(4), 451–457 (2008).
26. V. Lotito, U. Sennhauser, and Ch. Hafner, "Effects of asymmetric surface corrugations on fully metal-coated scanning near field optical microscopy tips," *Opt. Express* **18**(8), 8722–8734 (2010).
27. F. I. Baida and A. Belkhir, "Superfocusing and Light Confinement by Surface Plasmon Excitation Through Radially Polarized Beam," *Plasmonics* **4**(1), 51–59 (2009).
28. V. Lotito, U. Sennhauser, Ch. Hafner, and G.-L. Bona, "Fully Metal-Coated Scanning Near-Field Optical Microscopy Probes with Spiral Corrugations for Superfocusing under Arbitrarily Oriented Linearly Polarised Excitation," *Plasmonics* **6**(2), 327–336 (2011).
29. D. R. Turner, "Etch procedure for optical fibers," US patent 4,469,554, AT&T Bell Laboratories, Murray Hill, NJ, USA, 1983.
30. H. Muramatsu, K. Homma, N. Chiba, N. Yamamoto, and A. Egawa, "Dynamic etching method for fabricating a variety of tip shapes in the optical fibre probe of a scanning near-field optical microscope," *J. Microsc.* **194**(2-3), 383–387 (1999).
31. T. Osuch and Z. Jaroszewicz, "Numerical analysis of apodized fiber Bragg gratings formation using phase mask with variable diffraction efficiency," *Opt. Commun.* **284**(2), 567–572 (2011).
32. T. J. Antosiewicz, P. Wróbel, and T. Szoplik, "Magnetic field concentrator for probing optical magnetic metamaterials," *Opt. Express* **18**(25), 25906–25911 (2010).

1. Introduction

In the past decade, nanotechnological means to fabricate structures smaller than the wavelength of light have been growing and addressing various applications. The common point of almost all of them is connected with concentration of light signal due to transformation of waves propagating in uniform media to surface plasmon-polariton (SPP) ones at metal-dielectric interfaces. This transformation leads to miniaturization of information channels. Light concentrators have different forms from tapered fibers [1], either all-dielectric with a large core-cladding index contrast or metal coated ones [2], through tapered plasmonic waveguides with photon-plasmon couplers [3,4] ending at metamaterial nanotips [5]. Applications of light concentrators are numerous. In chemistry and biology sensors of molecules, cells, proteins and DNA have a form of tapered fibers [6]. In fiber laser applications tapered fibers with distributed Bragg reflectors (DBRs) are used for dispersion compensation [7]. Fiber tapers with long-period DBRs are used for sensing different physical properties such as temperature, stress or refractive index [8–10]. Tapered and corrugated waveguides have many potential uses in fiber output couplers and beam steerers [11] and tailoring the properties of photonic crystal fibers [12].

In this paper we describe a method of corrugating a desired length the core surface of Ge-doped silica fibers of constant diameter as well as tapering and corrugating such fibers. The process consists of the following three steps. To increase sensitivity of a fiber core of amorphous, photosensitive germanosilicate glass to UV illumination the fiber is hydrogenated. Then, a DBR is recorded in a direct grating writing process. Finally, the fiber is etched in an aqueous solution of HF acid to obtain equidistant circular grooves at the core surface.

2. Photosensitivity of germanium doped silica

Photosensitivity of germanosilicate glass is known since 1978 when Hill *et al.* [13] recorded Bragg gratings in Ge-doped silica-core fibers. Photosensitivity of such glass is observed in a wide range of Ge-dopant concentrations from 1 ÷ 30 mol. %. In our experiment the Ge-dopant concentration is chosen not because of maximum photosensitivity but because of desired isotropic etching rate – and more precisely – differential etch rate of exposed and unexposed glass. For small dopant concentrations depth of grooves is controlled by exposure time. For high dopant concentrations the etch rate of the core is much higher than that of the cladding and Turner type etching evolves to tube etching [14,15]. The best etch rate of a core of 8 μm diameter is achieved for 5.4 mol. % Ge-dopant concentration (6.5% by weight).

The photosensitivity can be drastically increased by the hydrogenation process [16]. In our experiment, the glass fiber is placed in a 120 bar pressure hydrogen atmosphere at room temperature for a period of two weeks, during which H_2 molecules interstitially, without appreciable chemical interaction, diffuse into the sample. Application of an external trigger like focused ion beam implantation [17] or ultraviolet illumination [16,18] induces chemical reactions which form hydroxyl groups within the glass structure. UV photons excite the O–Ge bond of a Si–O–Ge site which reacts with a hydrogen molecule forming Si–OH and a germanium defect center leaving one H atom to react with another Si–O–Ge site [18,19]. The defect centers are responsible for refractive index changes which form a DBR, and consequently for a different etch rate of the exposed areas relative to unexposed ones. This differentiated reaction speed is the basis for fabricating grooves in the fiber core surface.

3. Fabrication method

3.1 Bragg grating recording

A Bragg grating in a photosensitive fiber core deprived of acrylic cladding is recorded by exposure to ultraviolet 244 nm wavelength argon ion 100 mW CW laser light, which is modulated by phase masks with grating periods $\Lambda = 709$ or 1061 nm. The relative mask – fiber position is fixed during exposure so the length of the Bragg grating is determined by the length of the mask which in our case is 15 mm. Figure 1 presents a scheme of the exposure setup. A hydrogenated photosensitive fiber is brought into close proximity of the phase mask and both are placed on a translation stage which moves the pair across the light path so the entire interference pattern is permanently recorded in the fiber core. The phase mask is designed in such a way that the efficiency of radiation into the ± 1 st diffraction orders is maximized and equal. In an ideal case of interference only between these two diffraction orders the Bragg period would be half of the grating period. However, it is never possible to eliminate the 0th order completely and thus the interference pattern is not perfectly sinusoidal. This issue is commented in Section 4.

3.2 Wet chemical etching of germanosilicate fibers

Dürr *et al.* [20] proved experimentally that both hydrogen loading and UV-irradiation influence defect population in doped glass and, respectively, decrease and increase etch rate. Pace *et al.* [21] showed that in Ge-doped glass the etch rate nonlinearly depends on molar concentration of the dopant. Taking into account the above relations we describe a fabrication of thin dielectric corrugated waveguides by means of HF-etching of hydrogenated UV-exposed Ge-doped silica fibers. In Fig. 2 we present scanning electron microscope (SEM) images of a silica waveguide corrugated with periodicity of 709 nm etched at temperature 23.7°C for 54.5 min with final diameter 2.54 μm . The grooves are about 40 nm deep. Increasing the reaction time produces deeper corrugations at the same time giving a smaller core radius, so in effect increasing the surface modulation, i.e. the groove-depth to core-radius ratio. For the waveguide in Fig. 2 the ratio is 3.3%, however, this value may be increased by changing such process parameters as exposure time and Ge concentration.

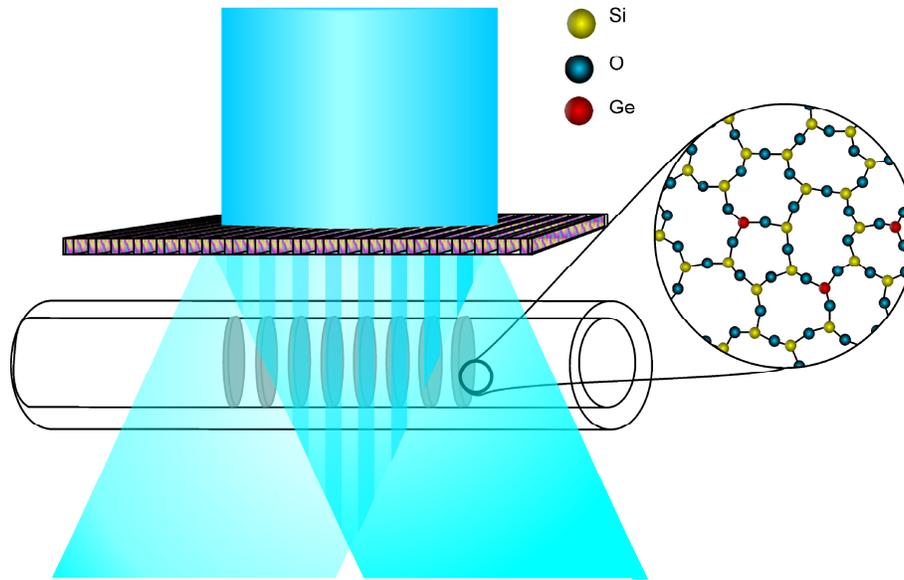


Fig. 1. Recording scheme of a distributed Bragg reflector in a Ge-doped silica-core fibers.

A wide practical use of light concentrators is in scanning near-field optical microscopes (SNOMs) as tapered fiber probes, which inspect a sample from a distance of about ten nanometers [22]. Stability of this distance, which assures a constant resolution along a scan is controlled with shear-force technique. In aperture metal-coated tapered SNOM probes resolution inversely depends on aperture size [23], and can be increased due to corrugation of the interface between the fiber core and metal coating what enhances energy throughput and allows for aperture diameter reduction [24,25]. In tapered fully metal-coated apertureless or all metallic SNOM tips surface corrugations enhance excitation and traveling of surface plasmons toward the tip apex [26–28].

Tapering fibers is possible with different techniques. The classical one is local heating of a fiber to temperatures higher than the Littleton softening point and stretching it. The other one consists of chemical etching in aqueous solution of HF acid usually realized with the Turner method useful for glasses which contain not less than 80% of SiO_2 by weight [29] or tube etching [14]. This lower limit of silica content in glass agrees with the before mentioned upper limit of Ge dopant concentration for which induced photosensitivity saturates. In our experiment tube etching, which gives tapered tips with relatively small surface roughness and of highly reproducible shape, cannot be used because in the recording of Bragg gratings the acrylic coating which absorbs UV light is removed. As a result, the Turner method, which is quite sensitive to vibrations and produces tapers with large surface roughness due to a step-like height decrease of the HF meniscus, is used. The etching is made on a super stable holographic table used for recording of holograms with up to 5000 line pairs/mm and exposure times up to a few minutes. A mixture of H_2O and HF (3:2) is covered with a thin layer of less dense and nonmiscible in HF organic isooctane, that is 2,2,4-trimethylpentane. Thus, the initial meniscus height dependent on density of the etchant and the top layer defines the maximum taper length. The taper angle can be controlled by pulling the fiber up or down into HF [30].

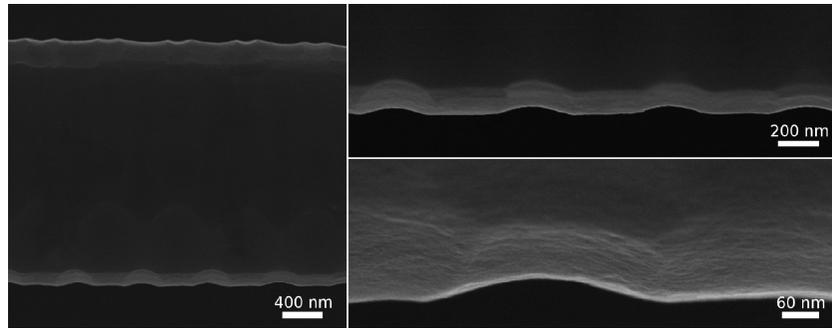


Fig. 2. SEM pictures of corrugated silica waveguides fabricated from a Ge-doped UV-exposed fiber using a phase mask with periodicity of 709 nm etched at 23.7 °C for is 54.5 min, final diameter is 2.54 μm .

The Ge-defect centers are responsible for changing the etch rate of the exposed volumes of the core relative to unexposed ones. This differentiated reaction speed is the basis for forming grooves while the fiber core is being etched. If the process continues until the taper is formed, then the result is a corrugated conical surface as illustrated by SEM pictures in Fig. 3. A fiber with an interference pattern from the phase mask with a 1061 nm period was etched in 40% HF at 22.4°C for 75 minutes (Fig. 3a). As seen from SEM measurements the groove period is approximately equal to the phase mask period and the groove depth is about 120 nm. A fiber with DBR recorded through phase mask with a 709 nm period was etched in 40% HF at 24.1°C for 67 minutes (Fig. 3b). In both cases the grooves are not perfectly perpendicular to the axis what results from a small misalignment during UV exposure. They are also not fully radially symmetric what is explained by a structure of interference pattern behind imperfect phase mask with not fully eliminated the 0th order.

4. Discussion and conclusions

Imperfect phase masks generate interference patterns which have their periodicity equal to the periodicity of the grating with every second fringe achieving maximum intensity [31]. In between these intensity maxima one can observe peaks of lower intensity and their decrease is dependent on the discretization and fabrication quality of phase levels of the mask. Moreover, in accordance to the Talbot self-image effect the interference field is periodic with a lattice constant of 6 and 13.4 μm for periods $\Lambda = 709$ and 1061 nm, respectively. If the fiber core is a lot smaller than the self-image length z then the irradiation efficiency should be quite uniform throughout the core, but will depend on the core – image alignment. In our case, the core diameter is 8 μm and is larger or comparable to z , so the interference intensity varies across the core. This in turn affects the concentration of germanium defect centers, the etch rate and finally the surface profile. To assure symmetric grooves it is necessary either place the fiber so that its axis coincides with a multiple of the self-image distance z or to assure that z is considerably greater than the diameter of the taper [31]. The former condition might be hard to achieve, however, the latter one will require the use of phase masks with larger periods. A larger Λ increases the corrugation period, however, the lattice does not need to contribute a large amount of momentum to couple light to plasmons at the core-coating interface, because the photon wavevector has a considerable parallel component.

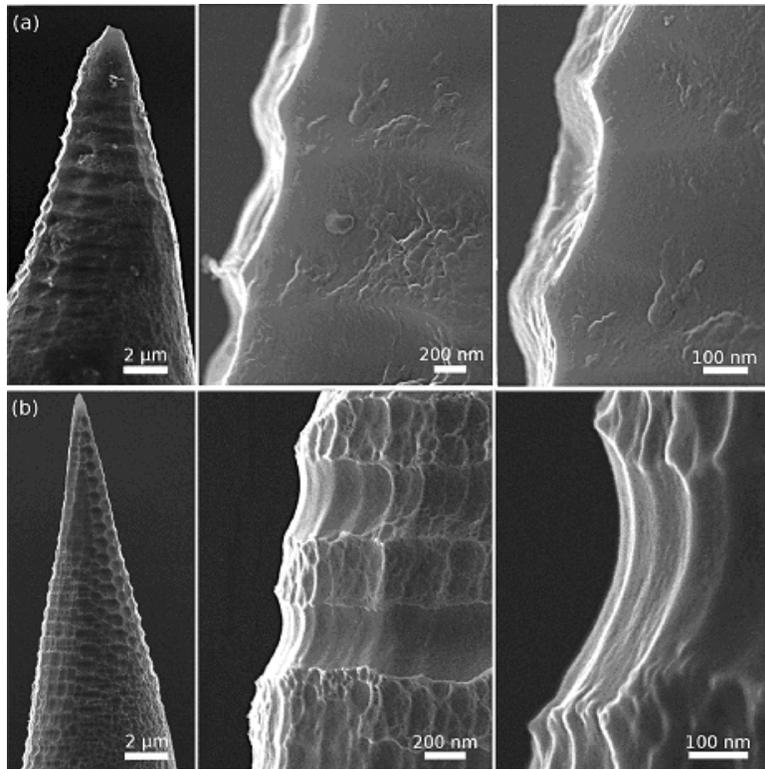


Fig. 3. SEM pictures of corrugated tapered fiber probes with (a) an average groove periodicity $1.03 \mu\text{m}$ equal approximately to the period of the phase mask $1.061 \mu\text{m}$ and groove depth about 120 nm and (b) an average groove periodicity 700 nm equal approximately to the period of the phase mask 709 nm and groove depth about 100 nm .

The most important use of tapered Ge-doped silica fibers with axially symmetric corrugations on desired length in the longitudinal direction is in scanning near-field optical microscopy for enhancing the energy efficiency by one order of magnitude [24,25]. For aperture metal-coated probes with axially symmetric corrugations internal either linearly or radially polarized illumination is used. Linearly polarized light excitation is adequate for fully metal-coated probes with single-side asymmetric corrugations fabricated with FIB [26]. If in a metal-coated tapered fiber corrugations along the probe length, that is axially symmetric, are replaced with corrugations along the angular dimension, that is longitudinal, and internal azimuthally polarized illumination is used, then such a probe focuses the longitudinal magnetic component of evanescent field of light [32]. A probe of this kind may be used in a future scanning near-field magnetic microscope for studies of magnetic responses of subwavelength elementary cells of metamaterials.

Acknowledgments

This work was supported by the Polish Ministry of Science and Higher Education under the project Iuventus Plus 0480/H03/2010/70, the National Centre for R&D under the project N R15 0018 06 and the National Science Centre under the projects DEC-2011/01/M/ST3/05734 and DEC-2011/01/B/ST3/02281.