Quantum Electronics **43** (4) 300 – 303 (2013)

PACS numbers: 81.40.Wx; 79.20.Ds; 42.70.Ce; 42.65.Re
DOI: 10.1070/QE2013v043n04ABEH015053

Fabrication of microcapillaries in fused silica using axicon focusing of femtosecond laser radiation and chemical etching

D.A. Yashunin, Yu.A. Malkov, A.N. Stepanov

Abstract. **Fabrication of microcapillaries with a diameter of 50 – 80 μm and a length up to 2.5 mm in fused silica by axicon focusing of femtosecond laser radiation and subsequent chemical etching in a 8 % hydrofluoric acid solution is demonstrated. The etching rate is ~6 μm min^{-1}. It is shown that the microcapillaries have optical waveguiding properties, which testifies to the optical quality of the walls of obtained structures.**

Keywords: *femtosecond laser radiation, axicon focusing, microchannels, micromodification of materials, chemical etching.*

1. Introduction

One of the promising ways to advance the modern biochemical and medical diagnostics is the development and fabrication of devices based on a system of microchannels formed in a single substrate (lab-on-a-chip systems) [1, 2]. Such microchips allow one to produce, transport, separate, and study small (from nano- to picolitres) volumes of solutions, as well as to conduct reactions between various substances. The advantages of these devices are high sensitivity, high analysis rate, low reagent and sample consumption, and the possibility of automation and standardisation of measurements. In numerous applications, the most preferable materials for microchip substrates are transparent dielectrics (optical glasses) [3] because they are hydrophilic, optically pure, chemically inactive, and biologically neutral; in addition, some of them, for example, fused silica, are optically transparent up to the UV wavelength region.

Relatively recently, as a tool for formation of microchannels in transparent dielectrics, it was proposed to use focused laser radiation with a femtosecond pulse duration [4]. An important advantage of femtosecond laser pulses is high radiation intensity, which makes it possible to achieve nonlinear interaction with the substrate material and thus to decrease the size of the structures produced and decrease the region exposed to heating. In particular, femtosecond radiation can be used to fabricate three-dimensional structures in transparent samples [5].

To date, there is a large number of works devoted to the formation of microchannels in fused silica by femtosecond laser pulses with subsequent etching [6] or by direct laser ablation of materials [7]. The overwhelming majority of works use focusing of laser radiation by microscopic objectives inside the material with simultaneous movement of the sample. However, even for laser systems with kilohertz pulse repetition rates, the typical material modification rate is ~10 μm s^{-1} [8]. In [9], three-dimensional microchannel structures in fused silica were formed by irradiation with femtosecond laser pulses and subsequent etching of samples in a hydrofluoric acid solution.

An alternative to laser beam focusing by spherical optics is focusing by axicon (conical) lenses, which forms in the sample volume a long (up to several centimetres) region of modified material with a cross section of several microns. For example, the authors of [10] demonstrated the possibility of forming microchannels in fused silica by multiple irradiation of one and the same point of the sample by relatively low-energy femtosecond laser pulses focused by a diffractive axicon with subsequent etching of the sample in hydrofluoric acid.

In the present work, we study the formation of channels of modified material in fused silica samples using axicon focusing of femtosecond laser pulses with a rather high energy (exceeding 1 mJ). It is shown that, in this case, it is possible to produce uniform microchannels by one laser pulse, which considerably increases the processing rate. Chemical etching of the fused silica samples after this laser treatment forms in them hollow microchannels, which possess optical waveguiding properties.

2. Experimental method and results

The experimental scheme for microstructuring of transparent optical materials using femtosecond laser radiation is shown in Fig. 1. The laser beam of a Ti : sapphire laser system (pulse duration ~60 fs, wavelength 800 nm, pulse energy up to 10 mJ, pulse repetition rate 10 Hz) [11] was focused by a quartz

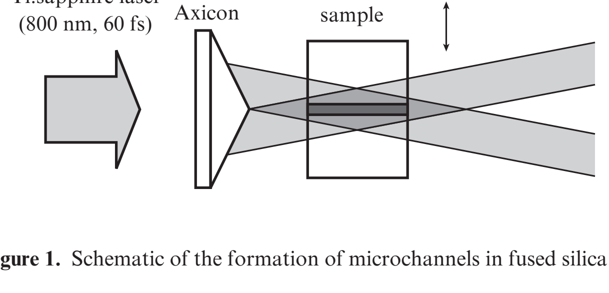

Figure 1. Schematic of the formation of microchannels in fused silica.

D.A. Yashunin, Yu.A. Malkov, A.N. Stepanov Institute of Applied Physics, Russian Academy of Sciences, ul. Ul'yanova 46, 603950 Nizhnii Novgorod, Russia;
e-mail: yashuninda@yandex.ru, yurymalkov@mail.ru

Received 12 November 2012; revision received 12 February 2013
Kvantovaya Elektronika **43** (4) 300 – 303 (2013)
Translated by M.N. Basieva

axicon lens with a cone base angle of 30° inside a fused silica sample in the form of a parallelepiped 5 mm thick. The pump beam diameter at the 1/e^2 level was ~15 mm. The sample was mounted on a translator, which allowed precision two-coordinate motion in the plane perpendicular to the incident beam. The sample face was placed at a distance of 4.5 mm from the axicon top so that the laser intensity along the axicon axis was maximal inside the target material.

In the experiment, we used two regimes, namely, we irradiated a region of the sample either by a single laser pulse with the energy $W = 0.2-7.0$ mJ or by a train of laser pulses (~30 pulses with the energy $W = 5-7$ mJ and a repetition rate of 10 Hz). In both cases, the sample after the laser beam action was shifted by about 200 μm for formation of a new channel. The appearance of channels in the sample was observed with an optical microscope. In the regime of a single laser pulse with an energy exceeding a threshold value ($W_{\text{th}} \sim 1.2$ mJ), a uniform (over the length) channel was formed in the sample in the axicon focal plane (Fig. 2a). The channel formation was visually observed as the appearance of a glowing filament in this region. The microchannel diameter was several microns, which is comparable with the characteristic transverse size of the Bessel beam formed by axicon focusing [12]. The length of channels was determined by the length of the fused silica samples and was equal to 5 mm.

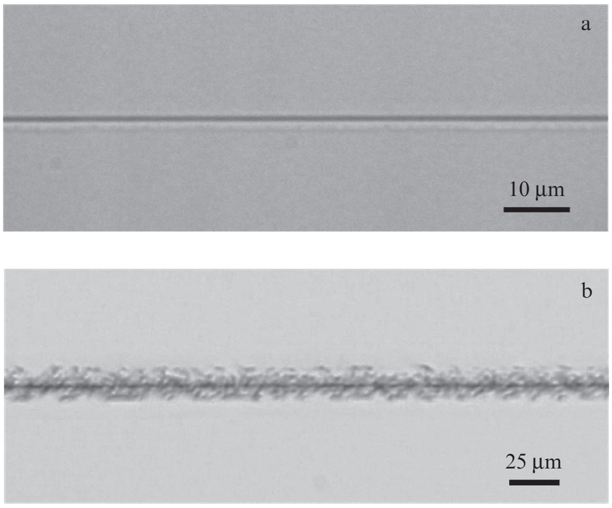

Figure 2. Typical image (side view) of channels formed in fused silica by (a) single and (b) multiple (~30) laser pulses.

In the multipulse regime, each channel was formed by a train of pulses with identical energies (5–7 mJ). This led to the formation of nonuniform channels consisting of numerous microscopic inhomogeneities (Fig. 2b). These nonuniform channels were about 20 μm in diameter, i.e., noticeably larger than the transverse size of the field in the axicon focus, which is obviously caused by scattering of the incident laser radiation by a structure formed inside the sample by preceding laser pulses.

After laser irradiation, the samples were polished to a thickness of 2.5 mm. Figure 3a presents the optical image of a polished sample face with a typical uniform channel (obtained with a laser pulse energy of 4.4 mJ). One can see an elongated structure, i.e., the channel cross section has a form of a crack. In the case of nonuniform channels formed by a train of laser pulses, the polished surface has noticeable cavities (Fig. 3b), which were observed by scanning with an atomic force microscope.

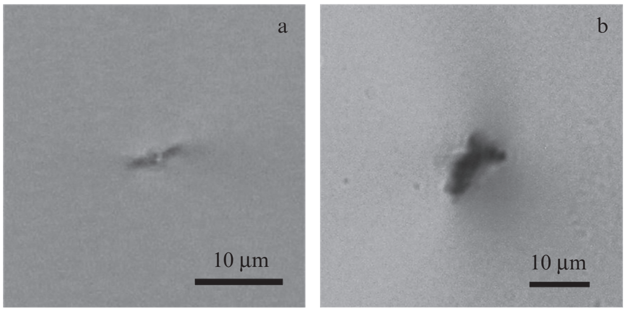

Figure 3. Typical image (front view) of channels after polishing of the sample. The channels are formed by (a) single and (b) multiple laser pulses.

After polishing, the samples were chemically etched in a 8% aqueous solution of hydrofluoric acid HF in an ultrasonic bath at a temperature of 40 °C. It was found that the microchannels are etched faster than the surrounding material, which was not exposed to laser irradiation. The effect of an ultrasonic bath on the etching processes was studied in [8] on the example of etching of channels formed in a fused silica sample by a train of pulses with a kilohertz repetition rate focused by a microscopic objective. As was shown in [8], the use of an ultrasonic bath leads to a more efficient removal of reaction products and to a faster renewal of the acid solution in the etched regions, thus improving the aspect ratio of obtained microcapillaries.

We measured the dependence of the microcapillary length on the etching time (Fig. 4). As is seen, the etching rate almost does not depend on the microcapillary length (up to a length of ~1.25 mm, which is equal to half of the sample thickness after polishing) and is ~6 μm min^{-1}. It should be noted that, in the case of microstructuring by single femtosecond laser pulses, the etching rate slightly increases with increasing laser pulse energy. The etching rate also depends on the laser irradiation regime and is the highest in the case of multiple pulses. The weak dependence of the etching rate on the microcapillary length indicates that the diffusion of the reagent (hydrofluoric acid) upon etching in an ultrasonic bath at a high temperature is not the factor that limits etching.

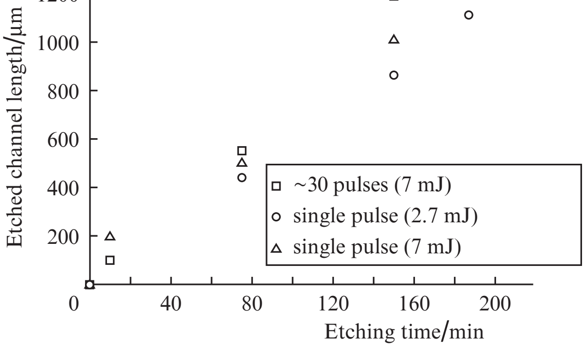

Figure 4. Dependences of microcapillary length on the time of etching in a 8% HF solution.

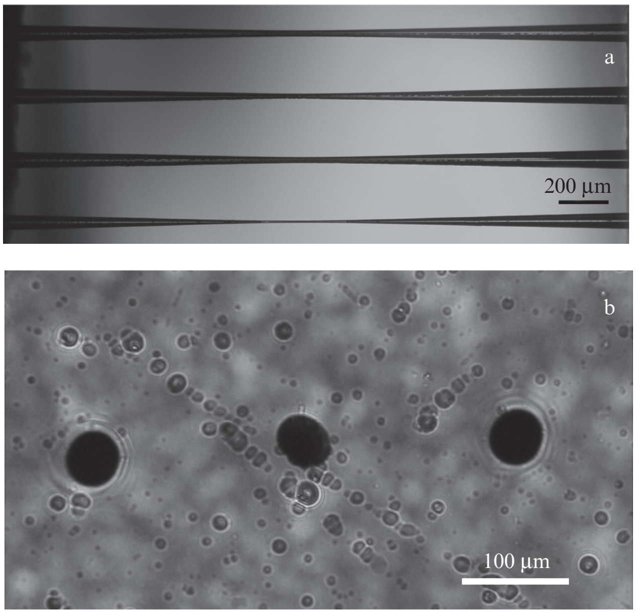

Figure 5. Optical images of channels after etching, (a) side and (b) front views.

After etching of the sample during three hours, we obtained straight microcapillaries, which converged to the sample centre and extended over the entire polished sample thickness (~2.5 mm). The larger transverse sizes were observed for capillaries etched after multipulse laser irradiation. Typical diameters of microcapillaries were 50 and 80 μm near the sample surface and 8 and 50 μm in the centre in the case of single and multipulse laser irradiation, respectively (Fig. 5).

The formed microcapillaries had a high aspect ratio (the ratio of the microcapillary length to its diameter near the sample surface), up to 50 : 1. A similar aspect ratio was observed in [3, 8] for microcapillaries formed using focusing of laser pulses with a kilohertz repetition rate by a microscopic objective in a fused silica sample, which was shifted during irradiation and afterwards etched. The high channel etching rate is obviously determined by the laser-induced modification of the silica properties in the region of channels; an additional factor can be an easier transfer of the acid solution along the crack-like channels (see Fig. 3a).

To determine the optical quality of the walls of microcapillaries, we studied their waveguiding properties (Fig. 6). A He – Ne laser beam was focused by a lens ($F = 5.5$ cm) on the entrance of a microcapillary. An aperture of a variable diameter placed in the beam was used to optimise the laser beam propagation by changing its diameter in the focus. The beam passed though the capillary was transferred by an objective ($F =$ 12 mm) with $20^{\times}$ magnification from the capillary exit to a digital CCD camera to record the spatial intensity distribution and measure the propagation factor. The formed microcapillaries turned out to have good optical waveguiding properties; for example, a microcapillary with characteristic diameters of 60 μm at the surface and 15 μm in the centre had a transmission coefficient of 60 %. A typical spatial intensity distribution of a beam passed though a capillary is shown in Fig. 7. One can see a smooth spatial distribution at the exit from the capillary. The high transmission coefficient of microcapillaries for optical radiation testifies that their walls are optically smooth.

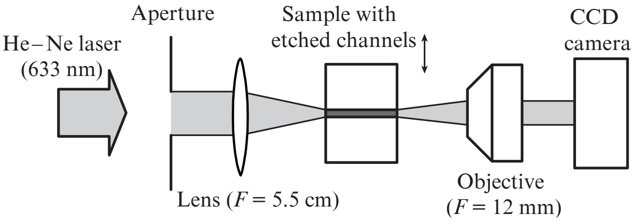

Figure 6. Scheme for the investigation of the efficiency of beam propagation through a microcapillary.

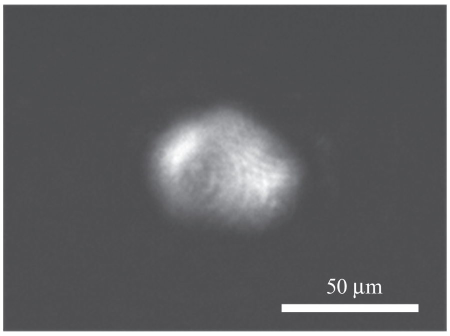

Figure 7. Typical image of the spatial distribution of radiation passed through a microcapillary.

3. Conclusions

The possibility of forming microchannels in fused silica using axicon focusing of femtosecond laser radiation in the regimes of single and multiple laser pulses is demonstrated. Subsequent chemical etching of the samples with channels in a hydrofluoric acid solution in an ultrasonic bath made it possible to obtain microcapillaries extending through the entire sample thickness up to 2.5 mm and having a high (30 – 50) aspect ratio. The etching rate was ~6 μm min^{-1}. Investigation of propagation of a He – Ne laser beam through the capillaries showed that they have good waveguiding properties, which testifies to an optical quality of the walls of the obtained structures. The formed microcapillaries can be of interest for developing devices for microanalysis of solutions in biological and medical researches.

Acknowledgements. This work was partially supported by the programme 'Extreme Light Fields and Their Applications' of the Presidium of the Russian Academy of Sciences.

References

1. Reyes D.R., Iossifidis D., Auroux P.-A., Manz A. *Anal. Chem.*, **74**, 2637 (2002).
2. Whitesides G.M. *Nature*, **442**, 368 (2006).
3. Osellame R., Maselli V., Vazquez R.M., Ramponi R., Cerullo G. *Appl. Phys. Lett.*, **90**, 231118 (2007).
4. Davis K.M., Miura K., Sugimoto N., Hirao K. *Opt. Lett.*, **21**, 1729 (1996).
5. Marcinkevicius A., Juodkazis S., Watanabe M., Miwa M., Matsuo S., Misawa H., Nishii J. *Opt. Lett.*, **26**, 277 (2001).

6. Hnatovsky C., Taylor R.S., Simova E., Rajeev P.P., Rayner D.M., Bhardwaj V.R., Corkum P.B. *Appl. Phys. A*, **84**, 47 (2006).
7. Kim T.N., Campbell K., Groisman A., Kleinfeld D., Schaffer C.B. *Appl. Phys. Lett.*, **86**, 201106 (2005).
8. Maselli V., Osellame R., Cerullo G., Ramponi R., Laporta P. *Appl. Phys. Lett.*, **88**, 191107 (2006).
9. Vishnubhatla K.C., Bellini N., Ramponi R., Cerullo G., Osellame R. *Opt. Express*, **17**, 8685 (2009).
10. Amako J., Sawaki D., Fujii E. *J. Opt. Soc. Am. B*, **20**, 2562 (2003).
11. Babin A.A., Kiselev A.M., Sergeev A.M., Stepanov A.N. *Kvantovaya Elektron.*, **31** (7), 623 (2001) [*Quantum Electron.*, **31** (7), 623 (2001)].
12. Korobkin V.V., Polonskii L.Ya., Poponin V.P., Pyatnitskii L.N. *Kvantovaya Elektron.*, **13** (2), 265 (1986) [*Sov. J. Quantum Electron.*, **16** (2), 178 (1986)].