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Etching of fused silica fiber by metallic laser-induced backside wet etching technique

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ABSTRACT

The tip of multimode fused silica fiber (core diameter: 550 μm) was etched by metallic laser-induced backside wet etching (M-LIBWE) method. Frequency doubled, Q-switched Nd:YAG laser (λ = 532 nm; τ_{FWHM} = 8 ns) was used as laser source. The laser beam was coupled into the fiber by a fused silica lens with a focal length of 1500 mm. The other tip of the fiber was dipped into liquid gallium metallic absorber. The etching threshold fluence was measured to be 475 mJ/cm^2, while the highest fluence, which resulted etching without breaking the fiber, was 1060 mJ/cm^2. The progress of etching was followed by optical microscopy, and the etch rate was measured to be between 20 and 37 nm/pulse depending on the applied laser energy. The surface morphologies of the etched tips were studied by scanning electron microscopy. A possible application of the structured fibers was also tested.

1. Introduction

The laser-induced backside wet etching (LIBWE) method [1,2] is a powerful, effective and flexible tool for micro- and submicrometer machining of transparent materials. LIBWE is used for the fabrication of microoptical elements e.g. micro-lens arrays, Fresnel lenses, micro-prisms [3–8]. This technique is also applicable for the preparation of various structures for biological, or biotechnological use [9–13]. High quality, high resolution (transmission) gratings can be fabricated by LIBWE technique [14–19]: the reachable minimal line width of the structure is near 50 nm [16,19]. Fabrication of deep micro trenches in transparent dielectrics by indirect laser etching is also promising application area of LIBWE [20–27]. In the LIBWE procedure the backside of the transparent target plate is in contact with a liquid absorber (hydrocarbon solution or liquid metal) having high absorption coefficient at the wavelength of the applied laser. The target-liquid boundary is irradiated through the transparent dielectric. The material removal can be attributed to a complex combination of thermal (high temperature target surface), mechanical (high pressure jet and bubble) and chemical (chemical modification of target surface) effects.

In metallic LIBWE (M-LIBWE) the liquid absorber is a liquid metal (gallium, mercury, tin) [28–38]. The most important advantage of M-LIBWE is that various lasers (UV, vis, IR) can be used for the etching due to the high absorption coefficients of the applied metals in a wide range of wavelengths.

In previous LIBWE experiments [1–38] the target was transparent plate and the structures were prepared on the backside of these plates. In this study our main motivation was to introduce and study a novel, non-conventional LIBWE setup. We applied a fiber with fused silica core as target and studied the progress of the etching process (etch rate vs. fluence), the changes in surface morphology of the fiber tip, and the application possibilities of the M-LIBWE processed fibers.

2. Experimental

In our novel setup the laser beam was coupled into the optical fiber at one end of it (input tip), while the other end of the fiber (output tip) was dipped into a liquid absorber. The dipped fiber tip could be etched by LIBWE mechanism. We used multimode fiber (*NA* = 0.22; type: Thorlabs BFH22-550) having 550 μm diameter fused silica core. The core was covered by fluorine silica cladding (thickness: 25 μm), hard polymer puffer (thickness: 15 μm) and Tefzel coating (thickness: 205 μm). The latter was removed before etching.

In our first experiments we applied 4th harmonic of Q-switched Nd:YAG laser pulses (λ = 266 nm; τ_{FWHM} = 8 ns, rep. rate: 10 Hz) and saturated naphthalene/methyl-methacrylate solution (c = 1.71 mol/dm^3) as liquid absorber. The laser beam was coupled into the fiber by a lens with a focal length of 700 mm (the beam diameter on the input tip was fitted to the fiber core diameter). After several experiments, we experienced that by increasing the laser intensity, the fiber became damaged at the input tip end before the etching could have started at the output tip end, which was dipped into the hydrocarbon absorber. This means that the laser-induced

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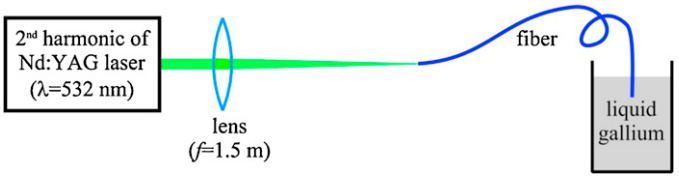

Fig. 1. The scheme of the experimental arrangement.

damage threshold (LIDT) of the fiber core is lower than the etching threshold of M-LIBWE.

In our second experiment we used higher wavelength laser pulses which resulted in a higher LIDT of the fiber core. We applied the 2nd harmonic of the previously used Q-switched Nd:YAG laser pulses ($\lambda = 532$ nm; $\tau_{FWHM} = 8$ ns, rep.rate: 10 Hz). In order to achieve effective absorption at 532 nm, the liquid absorber was also changed to liquid gallium absorber (the temperature of the gallium was approx. 35 °C; melting point of absorber is 29.8 °C). The scheme of the experimental arrangement can be seen in Fig. 1. The output fiber tip was dipped into the liquid gallium (5 mm deep).

The change in length of the etched fiber was measured by optical microscopy (using a reference marker) after etching by 10,000 laser pulses. The etched fiber tip was cut and cleaned by sulphuric acid in ultrasonic bath to remove the remained gallium.

The surface morphology of the etched tips was studied by field-emission scanning electron microscopy (FESEM, Hitachi-S4700). The samples were mounted on Al stubs by carbon tape and coated with gold–palladium film (thickness: 3–5 nm) to avoid the charging of the dielectric fibers.

We applied Raman microcopy (Thermo Scientific DXR Raman Microscope, operated at $\lambda = 780$ nm) in order to ascertain whether the etched surface of the fiber can serve as substrate for the so called surface enhanced Raman spectroscopy (SERS). For our test experiment we choose the solution of Rhodamine 6G (concentration: 1 mmol/dm^3), a well known Raman active compound. For the Raman measurements, we dipped the fibers in the Rhodamin 6G solution.

3. Results

The applied laser energy was varied between 1.4 and 3.2 mJ, which resulted in 450–1060 mJ/cm^2 fluence values in average at the output of the fiber (i.e., output energy divided by the cross-sectional area of the fiber core). The M-LIBWE etching threshold of the fiber was measured to be 450 mJ/cm^2 (this value is similar to the thresholds measured for plate targets [3,39–45]). If the output fluence was higher than 1060 mJ/cm^2, the fiber broke up into sub-millimeter fragments, and the etching was not controllable. This observation could probably be accounted for by the mechanical stress generated in the fiber tip by the high temperature gradient induced in the gallium nearby the tip.

In the fluence region of 475–1060 mJ/cm^2 the etch rate (i.e., decrease in length divided by the number of laser pulses) was measured to be between 20 and 37 nm/pulse (Fig. 2). This behavior is similar to the conventional LIBWE arrangements in the case of plate targets [3,39–45].

The surface morphologies of the fiber tips were studied by SEM. The original surface can be seen in the Fig. 3 as a reference.

The application of laser fluence (450 mJ/cm^2) resulted in only local damage of the tip, and the length of the fiber did not decrease. At the tip of the fiber modified, porous surface structure could be observed (Fig. 4a). The high resolution SEM image (Fig. 4b) shows the "cauliflower like" fine structure of the etched surface.

In our next experiment the laser fluence was set above the etching threshold. Fig. 5a shows, that the damage caused by the laser

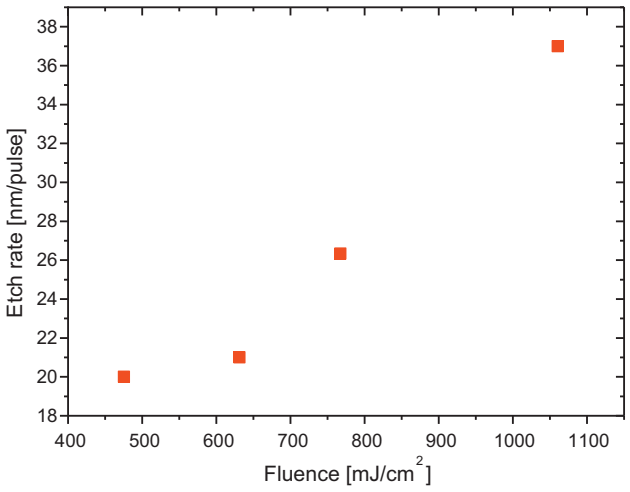

Fig. 2. Etch rate vs. fluence in the case of fused silica fiber etching by M-LIBWE.

fluence of 630 mJ/cm^2, is more severe at the tip of the fiber, than it was in the previous case. The porous fine structure of the etched surface exhibits somewhat smaller (~sub-100 nm) "grains" as well (Fig. 5b).

The main difference between the conventional LIBWE etched surface and the fiber M-LIBWE etched surface is their morphologies: while the conventional LIBWE provides smooth and high quality surface, the fiber M-LIBWE resulted in rough and porous structures.

Although the presented M-LIBWE etched fused silica fibers have not had any direct optical applications yet – contrary to the results of Dou, Zinn, Ihlemann and their co-workers who presented impressive study about the machining of fused silica fiber tip by direct ablation with VUV F_2 laser [46,47] – they might be used in those applications, where highly porous, submicrometer sized structures are needed.

According to the relevant scientific literature, the observed sub-100 nm sized structures could be suitable substrates for surface enhanced Raman spectroscopy (SERS) [48]. The Raman activity is strongly influenced by the size and the shape of the surface structures. This encouraged us to try to apply our M-LIBWE etched surfaces for SERS. The Raman-spectra of Rhodamine 6G solution taken from un-etched and etched fiber tip surfaces are shown in Fig. 6. While no specific spectra could be observed from the un-etched fiber tip, the characteristic peaks of Rhodamine 6G appeared in the spectra taken from the etched surfaces. The Raman spectra taken from the fiber tip that was etched by higher laser fluence is

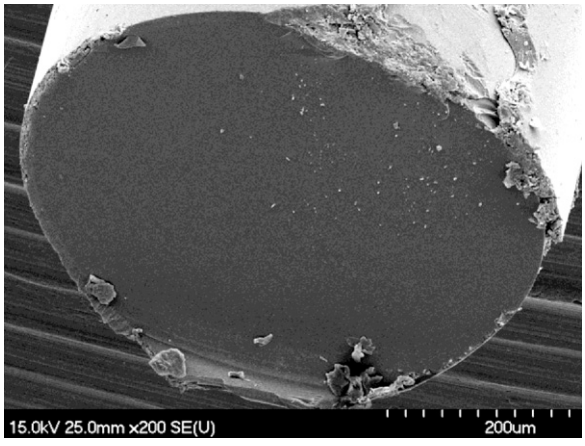

Fig. 3. SEM image of the original fiber tip before etching.