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Selective etching of sapphire and fused silica by double pulse femtosecond laser radiation

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Abstract

During the few past years, the laser processing of transparent materials has attracted more and more demand. The new approaches that can improve the processing efficiency and elucidate unexplained mechanisms are required. The femtosecond laser-induced selective etching (FLSE) is a promising technology for a wide range of optical mechanical and microfluidic applications. It is already investigated various etching dependences, and significant optimisations are done. The most used material for chemical etching is fused silica. However, there are some limitations like position dependent etching, low processing speed etc. Recently introduced double-pulse processing attracted a lot of attention in the field of direct materials ablation as a technology that can discover the ultrafast pulse-ablation dynamics. In this work, we investigate in details the double-pulse processing approach for microchannels formation in fused silica and sapphire with crossed polarisations. We demonstrate the pulse delay influence on the etching rate inside the fused silica. The etching rate improvement comparing to the single pulse FLSE technology is demonstrated, and the recording speed of the microchannels is tending to be increased.

Keywords: Double pulse; femtosecond; chemical etching; fused silica; sapphire

1. Introduction

During the past two decades when the femtosecond pulse processing of the transparent materials has shown the promising results (Shimotsuma et al., 2006, Gattass et al., 2008), many different processing technologies of transparent materials were developed for marking, dicing, cutting and intravolume modifications. In 2001, Marcinkevičius et al. demonstrated the enhanced chemical etching along the

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femtosecond laser modified track inside the fused silica (Marcinkevičius et al., 2001). This achievement involved the formation of microchannels and 3D structures in the bulk of fused silica. After two years, it was found that the modifications are composed of the self-organised nanogratings oriented perpendicular to the laser polarisation (Shimotsuma et al., 2003). Hereafter, the detailed investigations have shown that the selective etching is related to the nanogratings orientation and various etching dependences have been investigated (Hnatovsky et al., 2005). During the past decade, the etching optimizations were done (Bellouard et al., 2004, Ho et al., 2012, Lo Turco et al. 2013, Hermans et al., 2014), and the femtosecond laser-induced selective etching (FLSE) has become a very promising technology for wide range of complex applications such as internal 3D structures for mechanical, optical and microfluidic devices (Belloard et al., 2005, Sugioka et al., 2014, Osellame et al., 2011). Mainly, the most used and investigated material for chemical etching is fused silica. As an alternative there are some materials such as sapphire that demonstrated very high etching selectivity of 1:10000 (Hörstmann-Jungemann et al., 2010), however still it is very complicated to etch complex 3D structures due the extreme etching conditions in 40% HF acid heated to 80˚C and crystalline structure of the sapphire. It was demonstrated that selective etching in sapphire appears due to the formation of the amorphous phase of the sapphire (Juodkazis et al., 2006). However, the conditions to get this property enhancement needs new investigations and experimental approaches.

Recently introduced double-pulse processing technology for materials ablation (Schilie et al., 2016), gained increased attention for the transparent materials processing (Chu et al., 2017). The few double pulse approaches for processing of different materials were already used: double pulse with different wavelength or double pulse with the separated laser beam for pump-probe experiments. It was demonstrated the improved etching rate for the formation of high-aspect-ratio channels (Wang et al., 2018). However, there are still some limitations like position-dependent etching and low processing speed that need to be overcome. In this work, we investigate the double-pulse processing approach in details. We show the influence of the pulse duration and pulse delay to the selective etching of the microchannels and through structures in fused silica and sapphire. We introduce a new variable parameter that can help further understanding of the etching relations and nanogratings formations.

2. Experimental and results

The commercially available fused silica (FS) and sapphire samples were used for the experiments. To have the required dimensions, FS samples were cut from the bulk slab with a nanosecond laser. The size of the FS samples was 20x3x2 mm^3, and the sapphire samples were scribed to 12x8x0.4 mm^3.

The micromachining station with the integrated femtosecond laser system based on the Yb:KGW active medium operating at maximum 600 kHz repetition rate and the shortest pulse duration of ~ 290 fs was used. The operating wavelength was set to 1030 nm. The laser beam was divided into two laser pulses by the polarising beam splitter, and the energy of each pulse was controlled with the a rotating half-wave plate. The double pulses were combined applying the Mach-Zender interferometer setup composed from delay line (DL) and reference arm (RA). Each arm has polarisation control. The polarisation in the reference arm was set to **Ey** and one in the delay line to **Ex**. Therefore, the orthogonal polarisations were achieved. For the current experiment, the pulse energy in both arms was set equal. The combined two pulses were focused to bulk fused silica or sapphire with a 100x focusing objective (Mitutoyo Mplan NIR 0.5 NA). A sample was translated with the high accuracy positioning system (Aerotech ANT).

The delay line was calibrated in order to get the 0 fs delay position. The smallest delay resolution was 7 fs. The delay was estimated relative to the reference arm; therefore, the negative delay was achieved when the delay arm was shorter in respect to the reference arm. During the experiments, the delay was changed from -10 ps to +10 ps. First, the series of experiments were performed where the single microchannels were recorded at various focusing depths (50 to 500 µm below the sample surface). The pulse energy and scan speed at the constant repetition rate were varied. Next, the vertical modifications from bottom to top were fabricated by stacking the single modifications in the vertical direction with the 7 µm vertical z step. The modification in the XY plane formed semi-circle that elucidated the directional etching dependence. The fabricated samples were immersed in HF or KOH solutions. In the fused silica case, the 5% HF and 10 M KOH solutions were used. The sapphire samples need more hazardous environment, and they were immersed in the 40% HF solution and heated up to 80˚C. After chemical etching, the microscope pictures of the microchannels were analysed, see *Fig. 1*.

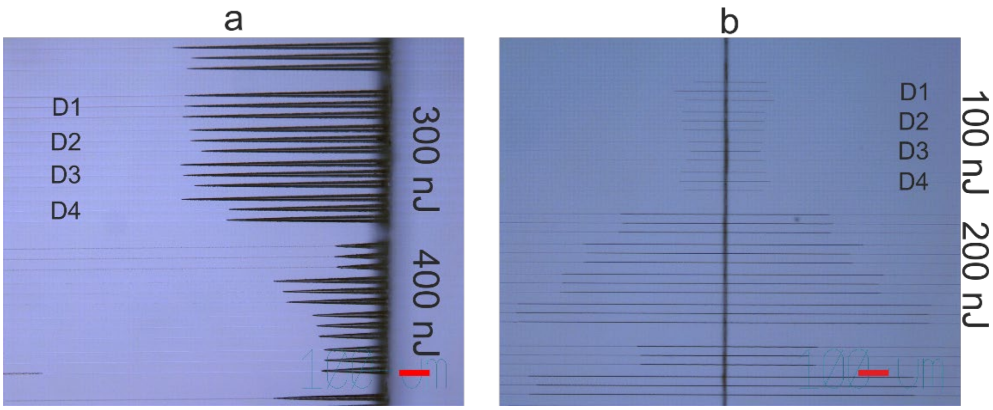

Fig. 1. The microscope pictures of the etched microchannels: a) fused silica, recording at 0 fs delay, etching for 30 min in 10% HF; b) sapphire, recording at 3.7 ps delay, etching for 4 hours in 40% HF. D1-D4 is the used pulse density (pulses/µm). Scale bar represents 100 µm.

For short pulse duration of ~ 290 fs, the delay has a critical influence for the etching rate. As can be seen in *Fig. 2*. For 0 fs delay the etching rate decreases >10 times in the fused silica case and >8 times in the sapphire case. The etching rate rised when the pulse delay was changed to +/-10 ps and reached its maximum at the 10 ps delay. It was noted that the etching rate for the single-pulse and double-pulse processing had similar values (for double pulses ~ 20% higher). However, the main advantage of the double-pulse processing was that the same etching rate was achieved for faster fabrication speed.

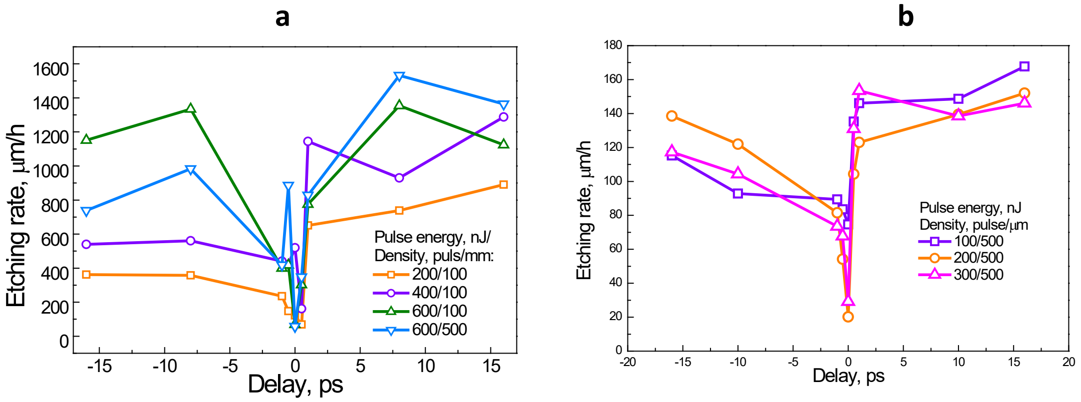

Fig. 2. Etching rate dependence on the delay between two pulses: (a) in fused silica; (b) in sapphire.

The etching rate of the sapphire samples is much slower than for FS samples; however, compared to the single-pulse fabrication, the etching rate in sapphire was improved twice. As the pristine sapphire is almost resistant to the HF acid, the etching selectivity of the modified area is extremely high and can reach more than 1:10000 selectivity. The FS samples are less resistant to the HF solution; therefore, the etching selectivity is in the range of 1:100.

3. Conclusion

In this study the femtosecond double-pulse approach was puzzled out to investigate the etching rate dependence on the pulse delay and find the optimal parameters (pulse energy, translation speed and pulse duration). It was demonstrated that double pulse fabrication could improve the processing speed for fused silica samples and at least twice rise the etching rate for sapphire samples.

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