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A novel 2 µm ultrashort pulsed laser source for selective laser-induced etching of glass

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Abstract

We present a novel source of ultrashort light pulses in the 2 µm wavelength range and demonstrate its application for selective laser-induced etching of different glasses, such as borosilicate glass and fused silica. A chirped pulse master oscillator power amplifier (MOPA) system is based on a mode-locked seed oscillator followed by a chain of Thulium doped fiber amplifiers. The laser system delivers pulse energies of up to 2 µJ at repetition rates between 30 kHz and 2 MHz and pulse durations as short as 540 fs. The output beam of laser light features an M^2 better than 1.3.
Focusing ultra-short laser pulses into a glass work piece generates material modifications at the position where the field intensity exceeds the threshold for modification. This laser-induced structure alteration of the glass material is attributed to a highly nonlinear process involving ultrafast multi-photon excitation of electrons and subsequent energy transfer to the lattice. Here, we demonstrate that modifications inscribed by the 2 µm laser into the different glasses can be selectively removed in a wet chemical etching process. This manufacturing procedure is termed “selective laser-induced etching” or SLE. The key is that the etching rate of modified material is higher than the etching rate of unmodified material. For borosilicate glass we demonstrate etching selectivities of 450 and for fused silica of 500. These high selectivities will allow manufacturing of tunnels, three-dimensional cavities, and even complex networks for microfluidic devices in both materials. The ultrashort 2 µm laser pulse thus makes borosilicate glass – the most important glass for chemical laboratory equipment due to its superior durability, chemical and heat resistance – accessible to manufacturing via selective laser-induced etching.

Keywords: ultrashort pulsed fiber laser; 2 micron laser; selective laser induced etching; borosilicate glass; glass processing

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1. Introduction

Ultrashort pulsed laser sources are becoming more and more important in industrial manufacturing. Their main advantage is the high quality of manufacturing results, which is by far superior to the quality achieved with conventional laser sources or mechanical approaches. In this regard, the development of new ultrashort pulsed laser sources is of importance, if they expand the range of available parameters for material processing.

High energy ultrafast lasers in the 2 µm wavelength range bear great potential for application in material processing, gas sensing, spectroscopy and medical surgery. In particular, the 2 µm wavelength opens up the possibility to expand established processing techniques to materials which are opaque in the visible spectral range but optically transparent at wavelengths of 2 µm (e.g. silicon). However, until today ultrashort laser pulses (< 1 ns) in the 2 µm wavelength range were mainly realized via the combination of an ultrashort pulsed laser sources at 800 nm or 1064 nm with an optical parametric amplifier. Besides significant costs and physical dimensions of these setups, a limiting factor of this approach is the low quality of the beam profile.

Here, we present a novel, compact source of ultrashort light pulses in the 2 µm wavelength range which is based on Thulium (Tm) doped silica fibers and a chirped pulse master oscillator power amplifier (MOPA) system (Sotier et al., 2016). Furthermore, we demonstrate that this novel laser system is a promising tool for manufacturing hollow 3D microstructures in glass.

Precise manufacturing of glass work pieces is of particular interest, because glass is one of the most important high-performance materials in industry, scientific research and society (Kotz et al., 2017) In particular, glass has an unmatched optical transparency, features outstanding mechanical, chemical and thermal resistance as well as thermal and electrical insulating properties. These properties make glass an ideal material for microfluidic devices for micro-sized chemical reactions or medical diagnostics. However, such glass chips comprising hollow 3D microstructures are challenging to manufacture. The most advanced approach to achieve such structures in glass is selective laser-induced etching (SLE) (Bellouard et al., 2004, Hnatovsky et al., 2005, Gottmann et al., 2017, Meineke et al., 2016). This approach uses ultrashort laser pulses to locally modify the glass material in the laser focus. Scanning the laser focus inside the glass material allows to inscribe complex 3D structures. In a subsequent step, the modified material is selectively removed in a wet chemical etching process (Kiyama et al.,2009, Hermans et al., 2014). Key is that the etching rate of modified material is higher than the etching rate of unmodified material. To date, this approach is successfully applied to process fused silica glass. Other glasses, and in particular borosilicate glass, which is the most important glass for chemical laboratory equipment, remained largely inaccessible to this approach because of insufficient etching selectivities of modified versus intact material.

Here, we demonstrate that structures inscribed by the novel 2 µm laser system into borosilicate glass can be chemically etched with high selectivities. This is an important step towards large scale processing of this important glass material via SLE.

2. Experimental and results

2.1. 2 µm ultrashort pulsed laser source

The high energy ultrafast laser system in the 2 µm wavelength range is based on Thulium (Tm) doped silica fibers. These fibers exhibit a broad amplification bandwidth between 1850 nm and 2100 nm enabling straightforward access to ultrashort laser pulses in this spectral region (Engelbrecht et al., 2008, Stutzki et al., 2015).

The conceptual layout of the laser system has previously been described in reference Sotier et al., 2016. Briefly, the system consists of a novel chirped pulse master oscillator power amplifier (MOPA) system (Yang et al., 2012) based on a mode-locked seed oscillator followed by a chain of three Thulium (Tm) doped fiber amplifiers. The seed pulses are supplied via super continuum generation in a highly nonlinear fiber seeded by an Erbium doped fiber oscillator (Kumkar et al., 2012). After a preamplifier the pulses are free space coupled and directed to a large area chirped volume Bragg grating (CVBG). With this grating a narrow spectrum (25 nm) is selected and the pulses temporally stretched by applying a linear chirp of 18 ps/nm. Consecutively, the pulses are coupled back into the fiber and an optical circulator directs them to a second Tm doped fiber amplifier. In order to gain the high pulse energies necessary for material processing a fiber coupled acousto-optic modulator reduces the 30 MHz repetition rate of the oscillator to a user selectable frequency between 30 kHz and 2 MHz prior to the last amplification step. The main amplifier is pumped by diode lasers operating at 793 nm emission wavelength providing a maximum power of 16 watt. After the main amplifier, the pulses are collimated and reciprocally sent to the CVBG (Liao et al., 2007). The reflection in the CVBG exactly compensates for the dispersion imposed on the pulses before the amplification. In order to prevent cross talk between amplifiers the beams are offset laterally in the CVBG. Incident and reflected beam are separated using a polarizing beam splitter cube and a quarter wave plate. In Figure 1a) the energy of the compressed pulses is plotted versus the pump power for different repetition frequencies. A maximum average power of 600 mW is measured for the laser pulses at a repetition rate of 500 kHz. Autocorrelation measurements demonstrate that the pulses can be compressed down to a temporal duration of 540 fs by removing the remaining fiber dispersion (see Figure 1b). In summary, the laser system delivers ultrashort femtosecond laser pulses with pulse energies of up to 2 µJ. The repetition rate can be set to values between 30 kHz and 2 MHz, and the output beam features an $M^2 < 1.3$.

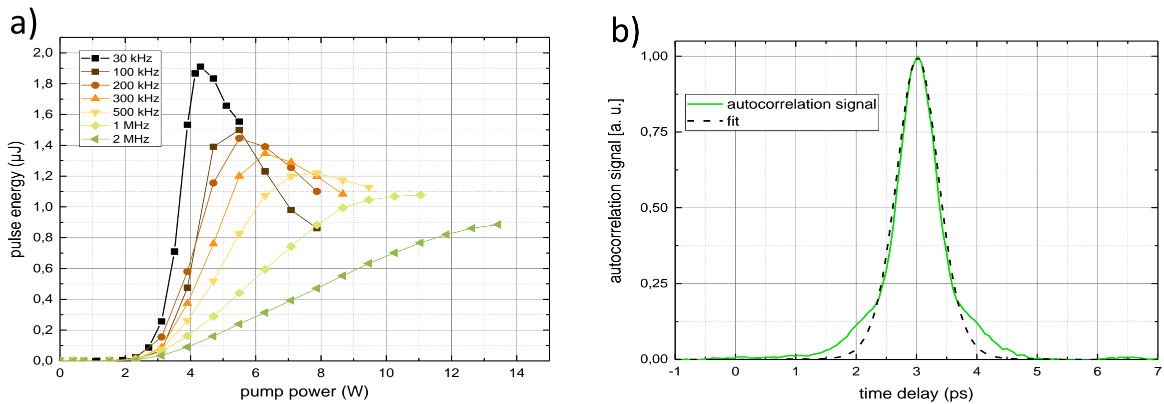

Fig. 1. (a) Pulse energies after recompression for different repetition frequencies plotted versus the pump power in the main amplifier. (b) Autocorrelation signal measured after removing residual dispersion. The signal is fit by a $sech^2$-function (dotted line).

2.2. Laser induced modification of glass and selective chemical etching

We have recently reported that the 2 µm ultrashort pulsed laser source can be used to inscribe lines into glass work pieces (Sotier et al., 2016). Such a localized laser-induced material modification by itself is already of interest with regard to the fabrication of waveguides as demonstrated by Nolte et al., 2003. Here, we further demonstrate that these inscribed structures can be selectively removed via chemical etching.

In our experimental setup, a lens with f = 1.8 mm focuses the laser light into the glass work piece with a numerical aperture of NA = 0.42. In the present case, we used pulse energies of 1 µJ/pulse (measured in front of the lens), a repetition rate of 300 kHz and determined a pulse length of 4.5 ps. With these parameters, the field intensity in the focus reaches values of about 2×10^{12} W/cm^2, which exceeds the threshold for modification in glass (Sotier et al., 2016). The glass work piece is mounted to a computer controlled 3-axis manipulator, which allows to position and move the glass precisely with respect to the laser focus. The stage moved at a velocity of 0.1 mm/s during the inscription of lines into the glass materials.

The microscope image shown in Figure 2a) demonstrates that focusing the light into borosilicate glass (GVD GmbH, new boroplate, floated borosilicate glass 3.3) leads to a modification of the glass at the position of the laser focus. We attribute this laser-induced modification to the results of highly nonlinear processes involving ultrafast multi-photon excitation of electrons and subsequent energy transfer to the lattice (Gattas and Mazur, 2008).

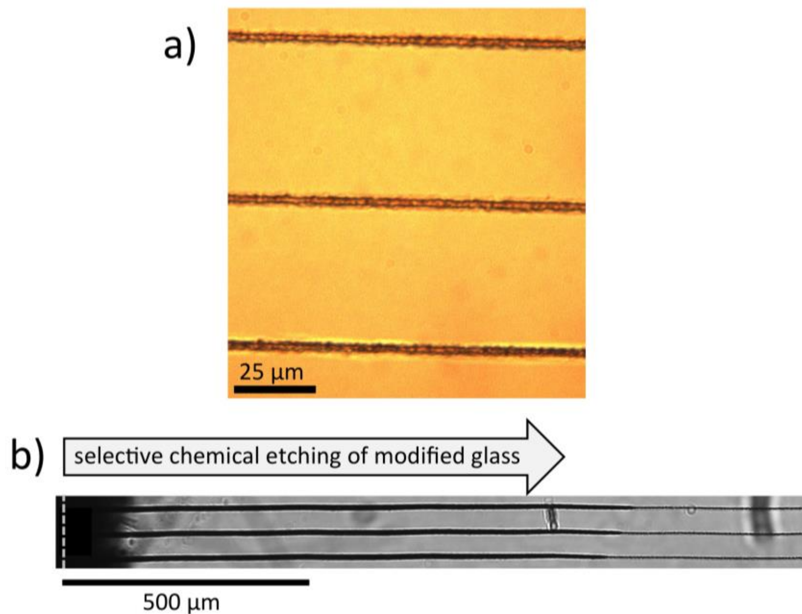

Fig. 2. (a) Microscope image of laser induced modifications in borosilicate glass; (b) Microscope image after chemical etching of laser-inscribed modifications. The white dashed line marks the surface of the work piece at which the inscribed lines are in contact with the chemical etching reagent and from where selective chemical etching of modified glass begun.

In the next step, the work piece is cut perpendicular to the inscribed lines so that the modified material has a contact to the surface of the work piece. The work piece is then exposed to an 8 mol/L aqueous solution of potassium hydroxide at a temperature of 85°C for 6 hours (Hermans et al., 2014). In this solution the modified material is etched much faster than the unmodified material and is selectively removed resulting in holes which deeply extend into the glass piece (see Figure 2b). For the example shown in Figure 2 we determine an etching selectivity of 450. This selectivity is calculated according to the method presented in (Hermans et. al., 2014). The etching rate of unmodified material is 0.43 µm/s and was determined by

measuring the thickness of the work piece before and after exposure to the etching solution. These etched holes feature a length of about 1.1 - 1.2 mm and a diameter of about 10 µm. This corresponds to an aspect ratio better than 110:1.

We note that experiments on fused silica showed similar results with etching selectivities as high as 500. This opens up the possibility to use the 2 µm ultrashort pulsed laser source to process both of these technically relevant materials.

3. Conclusion

We present a novel, compact source of ultrashort light pulses in the 2 µm wavelength range, which delivers pulse energies of up to 2 µJ at repetition rates between 30 kHz and 2 MHz. Pulse durations are determined to 540 fs and the output beam of laser light features an M^2 better than 1.3. Furthermore, we demonstrate that this novel laser system is a promising tool to manufacture hollow 3D microstructures into borosilicate glass and fused silica via selective laser induced etching (SLE). The high etching selectivities achieved in borosilicate glass are an important step towards making this material accessible to large scale processing via SLE. Besides the fabrication of micro scale structures into borosilicate and fused silica glass, the 2 µm wavelength opens up the exciting possibility to process a range of materials (e.g. silicon) which are opaque in the visible spectral range but optically transparent at wavelengths of 2 µm.

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