Kumkar, M., *et al*. "Ultrafast laser processing of transparent materials supported by in-situ diagnostics." *Laser Applications in Microelectronic and Optoelectronic Manufacturing (LAMOM) XXI*. Vol. 9735. International Society for Optics and Photonics, 2016.
https://doi.org/10.1117/12.2209507

# Ultrafast laser processing of transparent materials supported by in-situ diagnostics

M. Kumkar*[a], M. Kaiser[a], J. Kleiner[a], D. Grossmann[a,c], D. Flamm[a], K. Bergner[b], S. Nolte[b]
[a]TRUMPF Laser- und Systemtechnik, Johann-Maus-Str. 2, 71254 Ditzingen, Germany; [b]Institute of Applied Physics, Friedrich-Schiller-University Jena, Albert-Einstein-Str. 15, 07745 Jena, Germany; [c]Chair for Laser Technology, Technical University Aachen, Steinbachstr. 15, 52074 Aachen, Germany

## ABSTRACT

For the development of industrial NIR ultrafast laser processing of transparent materials, the absorption inside the bulk material has to be controlled. Applications we aim for are front and rear side ablation, drilling and inscription of modifications for cleaving and selective laser etching of glass and sapphire in sheet geometry.

We applied pump probe technology and in situ stress birefringence microscopy for fundamental studies on the influence of energy and duration (100 fs – 20 ps), temporal and spatial spacing, focusing and beam shaping of the laser pulses.

Applying pump probe technique we are able to visualize differences of spatio-temporal build up of absorption, self focusing, shock wave generation for standard, multispot and beam shaped focusing. Incubation effects and disturbance of beam propagation due to modifications or ablation can be observed.

In-situ imaging of stress birefringence gained insight in transient build up of stress with and without translation. The results achieved so far, demonstrate that transient stress has to be taken into account in scaling the laser machining throughput of brittle materials. Furthermore it points out that transient stress birefringence is a good indicator for accumulation effects, supporting tailored processing strategies.

Cutting results achieved for selective laser etching by single pass laser modification exemplifies the benefits of process development supported by in situ diagnostics.

**Keywords:** Ultra-short pulse, laser, materials processing, glass, pump-probe, nonlinear absorption

## 1. INTRODUCTION

Nonlinear absorption of ultrafast laser pulses enables a plurality of different methods for machining of transparent materials. There are well established applications based on induced absorption in the region of the beam entrance surface, the exit surface or inside the volume of the transparent workpiece[1] (Fig. 1a-c). In case of entrance side machining detrimental effects of parasitic light penetrating into the volume are often an issue[1,2,3]. For processing at the exit surface disturbance of propagation due to effects of the entrance surface or the volume traversed has to be avoided. Processing inside the bulk material (Fig. 1e) requires both, undisturbed propagation towards the intended region of interaction and avoiding damage elsewhere. High NA focusing is typically used for such applications, like for instance selective laser etching[4].

Beam shaping offers the potential for tailoring the region of interaction inside the transparent workpiece[5]. Figure 1d for example illustrates an in elongated interaction region extending from the entrance surface through the complete thickness to the exit surface[6,7].

Beside the focusing conditions, the pulse energy, the pulse duration, additionally the temporal and spatial intervals of the pulses have a major influence on the processing. Supplementary to incubation or accumulation effects in overlapping light-matter interaction regions, it is recognized, that the material may be influenced outside of this primary interaction region. It is for instance well known, that by accumulation and diffusion a melt volume can be generated by far larger than the primary interaction region (Fig. 1e), useful e.g. for welding applications[8].

*malte.kumkar@de.trumpf.com; phone +49 7156 321 98; fax +49 7156 9321 98; www.trumpf-laser.com


https://doi.org/10.1117/12.2209507

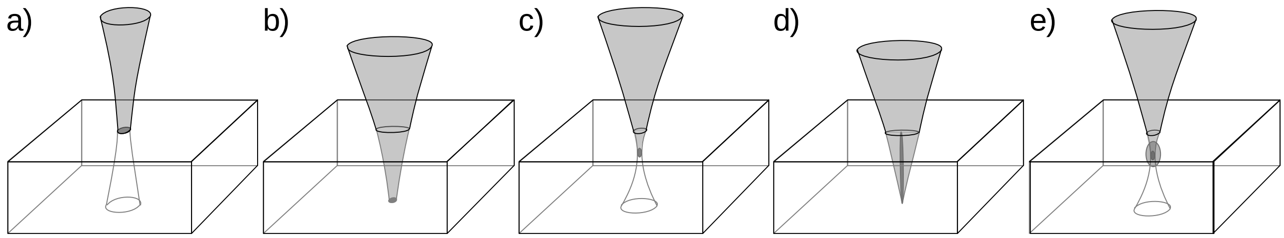


Figure 1. Laser processing of transparent workpieces by nonlinear absorption, a) on the beam entrance surface, b) on the beam exit surface and c) inside of the transparent volume. d) Beam shaping for tailored interaction region. e) Extended modification volume by accumulation effects.

For processing of brittle materials by nonlinear absorption, the control of the region and energy density of absorption is crucial. Transparent materials offer the potential for direct optical in situ diagnostics of the interaction volume. We presented results of pump probe technology applied for process development towards separation by elongated modifications earlier[1,7]. In this work we present additional pump probe results for multi pulse applications, showing the influence of incubation and craters at a low repetition rate. Furthermore we apply in situ stress birefringence microscopy[9] in an accumulation regime at elevated repetition rates.

## 2. EXPERIMENTAL SETUP

### 2.1 Pump Probe Setup

Pump probe technology was used to investigate the spatial and temporal evolution of the electron density inside the material (Fig. 2a). The pulse of a Coherent Libra laser source with 2 mJ energy and 100 fs duration at 800 nm wavelength was split into pump and probe beam lines. We adjusted the pump pulse energy by a combination of half wave plate and polarizer, the duration by a double pass grating stretcher. The beam was collimated to a waist diameter of 6 mm. We mounted the sample on a translation stage and focused the pump beam towards the sample. The probe beam was directed through the sample perpendicular to the pump beam after passing a second harmonic generation unit and a delay line. To observe changes in the transmitted light, the focal region was imaged onto a camera.

### 2.2 Stress Birefringence microscopy

During laser processing by beam shaped laser pulses with a wavelength of 1030 nm, stress birefringence was imaged perpendicular to the beam propagation of the pulse (Fig 2b). We applied a liquid crystal spatial light modulator (HAMAMATSU X10468-03 800x600 Pixel) for beam shaping. The processing beam with diameter of 5 mm was focused through a polished edge into the sample by a microscope objective with a focal length of 10 mm and a NA of 0.4. A fiber coupled randomly polarized laser diode system (CAVITAR Cavilux HF) with a wavelength of 810 nm, a peak power of 500 W and a pulse duration of 300 ns was used to illuminate the sample after passing a polarizer. The polarization was adjusted in 0°, 30 ° and 60° with respect to the propagation of the processing beam. We imaged the focal region through an analyzer, orientated in 90° with respect to the polarizer, onto a high speed camera (Photron FASTCAM SA-X). The sample was mounted on a translation stage. Images were taken under translation perpendicular to the propagation direction of the processing and the imaging beam. Stress birefringence images were calculated by taking three images for a polarization of 0°, 30° and 60°during separate passes and adding the signal pixel by pixel.

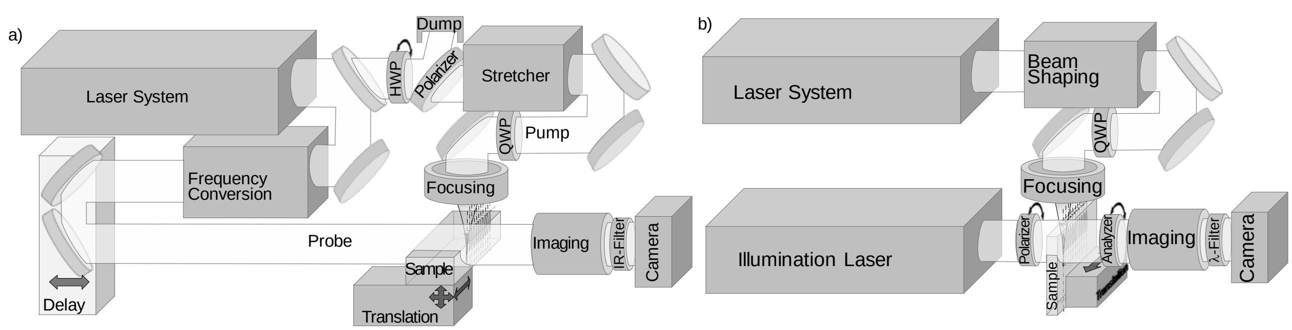


Figure 2. Experimental setup. a) Pump probe. b) In situ stress birefringence microscopy

## 3. EXPERIMENTAL RESULTS AND DISCUSSION

### 3.1 **Pump Probe imaging for ablative processing**

Pump probe imaging (Fig. 2a) was applied for ablative processing at the beam entrance and the beam exit side with a repetition rate of 1 Hz. To determine the attenuation induced by individual pulses, for each shadowgraph image $\boldsymbol{I}$ an additional background image $\boldsymbol{I_0}$ was recorded. For the pump probe images an equivalent optical depth $\boldsymbol{\tau}$ was calculated for each pixel of the camera.

$$\tau = \ln ( I_0 / I )$$

Figure 3 shows pump probe images for entrance side processing with a pulse energy of 50 µJ focused onto the entrance surface by a focal length of 100 mm. The delay was chosen for maximum extend of the absorption region, a few picoseconds after the first signal was visible.

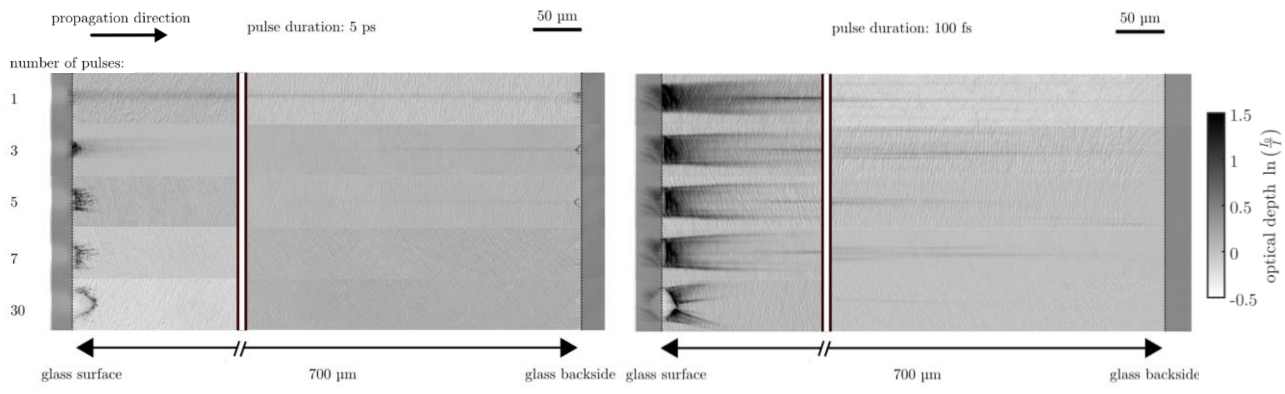


Figure 3: Pump probe images for percussion drilling of non-strengthened, 700 µm thick Corning Gorilla® glass with a pulse energy of 50 µJ and a pulse duration of 5 ps (left) and 100 fs (right).The images were taken from the first to the 30th pulse.

For a pulse duration of 5 ps just a slight absorption became visible in the volume at the entrance surface after the first pulse. This absorption was similar to the signal observed within the material along the beam pass towards the rear side. A remarkable absorption was observed near this exit side. The third pulse caused a stronger absorption near the entrance side, and a reduced absorption in the adjacent region. Nearby the rear side the absorption on the beam path showed up again and an increased absorption could be observed near the surface of a crater which was formed during the first two pulses. For the following pulses a crater on the entrance side was growing in diameter and depth, showing a subsurface absorption. Since this led to an increasing disturbance of the beam propagation the absorption in the volume and on the rear side was no longer visible.

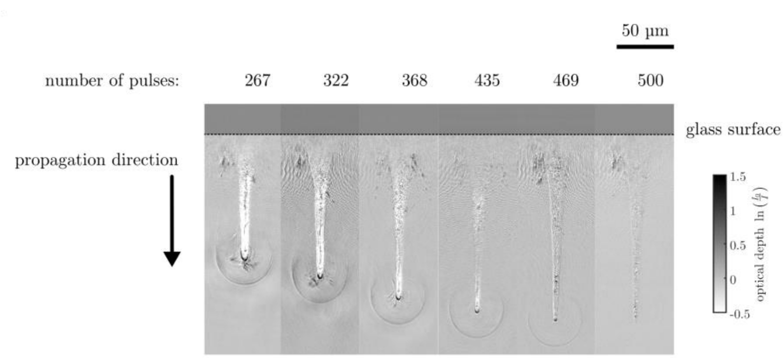


Figure 4: Pump probe images for percussion drilling of non-strengthened Corning Gorilla® glass with increasing number of pulses with an energy of 50 µJ, pulse duration of 5 ps using a focal length of 100 mm. The probe delay was set to approximately 4 ns.

For a pulse duration of 100 fs the first pulse resulted in a strong and extended region of absorption beneath the entrance surface, followed by a filament reaching the exit side. Interestingly no increased absorption near the rear side was observed. The crater formation during the following pulses is smaller compared to the one generated with longer pulses. The influence of the crater on the absorption region became more pronounced with increasing number of pulses.

Whereby with increasing delay the absorption ceased, other effects became visible. In Figure 4 pump probe images for drilling with increasing number of pulses with an energy of 50 µJ at a duration of 5 ps and a focal length of 100 mm are shown. The delay of the probe pulse is set to 4 ns. A shockwave emitted from the tip of the crater could be observed, as long as the drilling process did not saturate. The structures within the shockwave region indicated cracks induced by the shockwave. Since these structures were not observed in the background images, we assume a transiently opened gap during the shockwave. Within the crater plasma emission at 400 ± 40 nm was detected due to the long integration time of the camera.

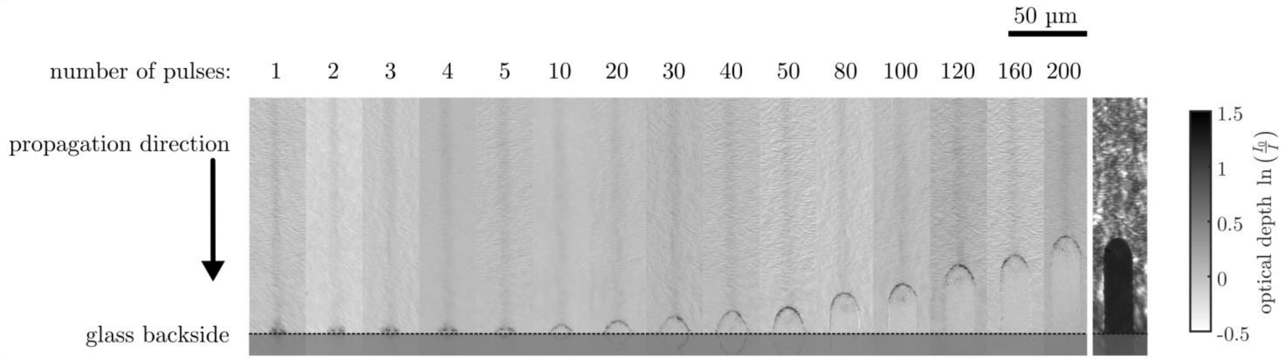


Figure 5. Percussion drilling from the glass backside of 700 µm thick Coring Gorilla® glass using pluses with an energy of 20 µJ at a pulse duration of 5 ps and a repetition rate of 1 Hz. The beam was focused applying a focal length of 100 mm and the beam waist was located on the exit side. Pump probe images for an increasing number of pulses are shown on the left, the shadowgraph image of the crater processed by 200 pulses can be seen on the right.

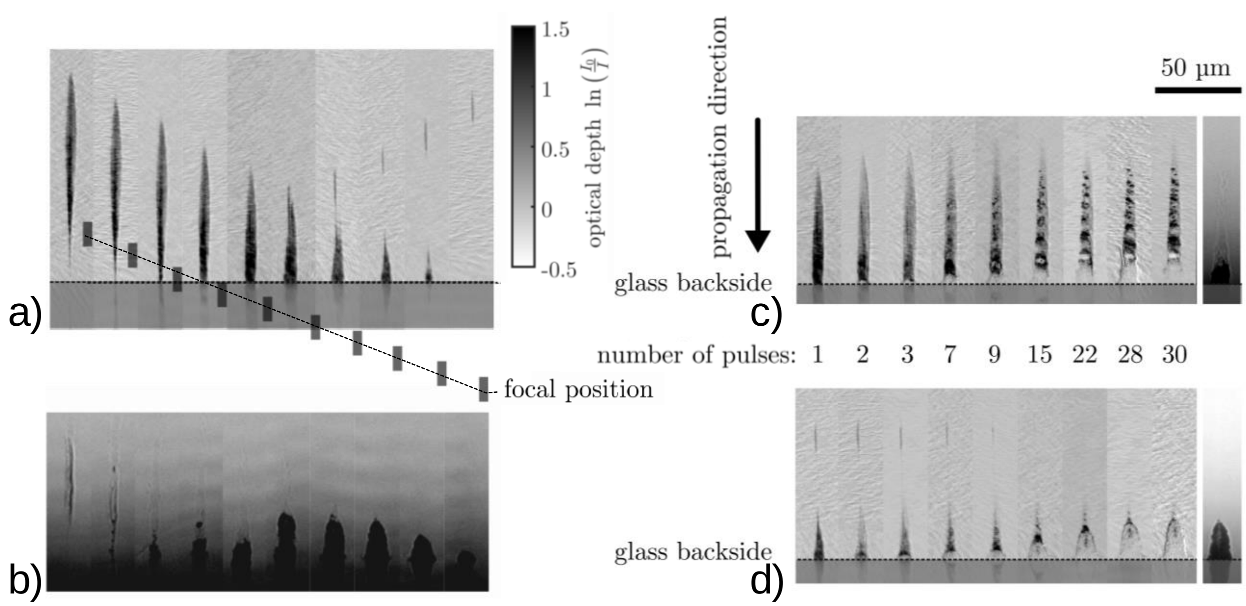


Figure 6: Percussion drilling from the glass backside of 700 µm thick Coring Gorilla® glass by focusing pulses with an energy of 10 µJ, a pulse duration of 5 ps and a repetition rate of 1 Hz applying a focal length of 10 mm. a) Influence of the focus position. The focus position with respect to the beam exit side is indicated in the upper picture showing pump probe images of the first pulse. b) Shadowgraph images for processing by 30 pulses are given for focus positions corresponding to a). c) Pump probe images for increasing number of pulses illustrate the absorption for a focus position about 20 µm below surface and d) 60 µm below surface. The added shadowgraph images revealed some modification next to the crater. The delay was chosen for maximum extend of absorption region.

By locating the focus on the exit side of the workpiece, drilling from the backside could be achieved. For a pulse energy of 20 µJ and a pulse duration of 5 ps some absorption was visible on the beam path towards the exit side, but no modification due to this absorption appeared. The absorption located near the backside resulted in ablation. For increasing number of pulses a crater and absorption nearby its surface could be observed. The focus position was not changed during processing.

To ensure pure rear side ablation even for reduced sample thickness, we carried out tests with tighter focusing (Fig. 6). For a focal length of 10 mm pump probe images of the first pulse are shown for different focal positions in Figure 6a. Beside the absorption induced by the forward propagating beam, some absorption in the focal region of the reflected beam became visible. The corresponding shadowgraph images of processing by 30 pulses revealed modification regions next to the ablation craters for focal positions not to far below the surface (Fig. 6b). Pump probe images for increasing number of pulses for a focus position of about 20 µm below the exit surface indicated a spatial modulation of the absorption by incubation effects (Fig. 6c). Under these incubation conditions a modification region located in the beam pass towards the crater could be observed. Locating the focus about 60 µm below the surface resulted in a larger crater and a smaller visible modification region (Fig. 6d).

The presented results on pump probe technology exemplify the benefits for process development by observing transient effects on slightly different timescales with temporal resolution below the duration of the processing pulse.

### 3.2 In situ stress birefringence microscopy applied for in-volume absorption

For scaling throughput at elevated repetition rate, the impact of transient effects on regions of the sample extending the volume of direct laser matter interaction becomes more crucial. The same holds for processes based on accumulation effects. Transient stress birefringence microscopy offers a high potential for process development of brittle, transparent materials.

The setup shown in Figure 2b was applied to generate stress birefringence images for processing of non-strengthened Corning Gorilla® glass.

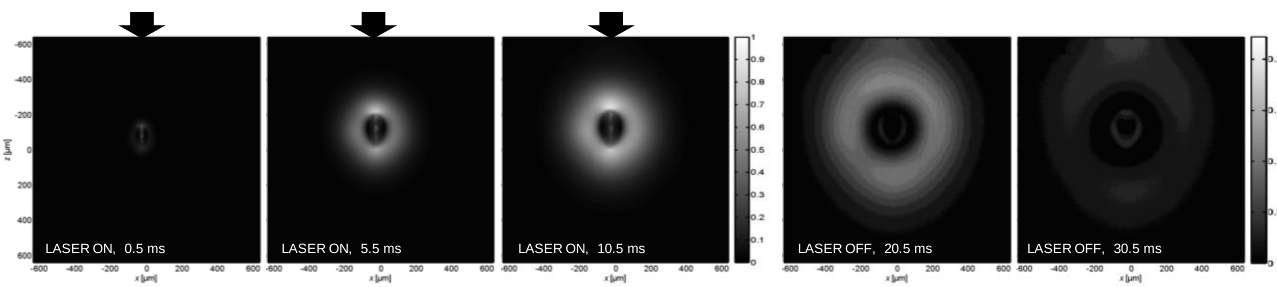


Figure 7: Evolution of stress birefringence for single spot focusing without translation. Laser pulses of a wavelength of 1030 nm, an energy of 10.9 µJ, a pulse duration of 300 fs, a repetition rate of 200 kHz, switched on at 0 ms and off at 13 ms. The grayscale was expanded for the images at 20.5 and 30.5 ms by a factor of 4.5 with respect to the images taken during laser emission.

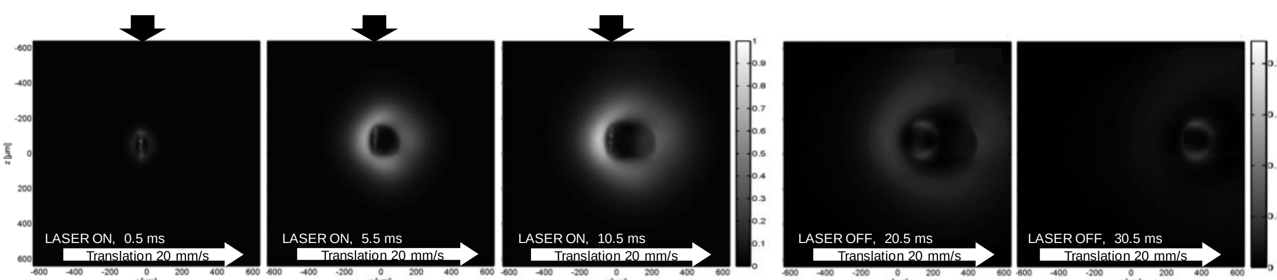


Figure 8: Evolution of stress birefringence for single spot focusing with translation of 20 mm/s perpendicular to the direction of observation. Laser pulses with an energy of 10.9 µJ, a pulse duration of 300 fs and a repetition rate of 200 kHz, switched on at 0 ms and off at 13 ms. The grayscale was expanded for the images 20.5 and 30.5 ms by a factor of 4.5 with respect to the images taken during laser emission.

In Figure 7 the development of stress birefringence is shown for focusing pulses of 10.6 µJ at a repetition rate of 200 kHz about 600 µm into the sample. The laser was switched on at 0 ms for 13 milliseconds, corresponding to the time given in the images. As long as the laser was on, some process light could be seen in the focal region. During laser emission phase, stress was building up surrounding this absorption region and showing a maximum on the beam axis. We assume that the stress during this phase was generated in a region adjacent to a growing melt zone. After switching off the laser, the stress resulting from temperature gradients decreased while some permanent stress was built up mainly in the solidifying material. The grey scale of the signal for the images without laser emission was stretched, indicating a by far higher transient stress compared to the permanent stress induced.

Under the same conditions but with a translation speed of the sample of 20 mm/s, the melt and the maximum transient stress could be observed in front of the beam axis, adjacent to a small melt zone (Fig. 8). Behind the beam axis, the melt zone expanded. Slight stress was observed rearward at 15.5 ms, indicating solidification during laser exposition. Again, the grey scale was stretched for the images taken after the end of emission, indicating dominant transient stress. The scale is the same as in Figure 7.

We furthermore investigated the influence of multisport arrangements on the stress evolution. The same signal scale holds for the stress birefringence images in Figures 9 – 11. Figure 9 displays results achieved for locating two spots with a distance of 165 µm on the beam axis inside the sample by translating the sample with a speed of 200 mm/s. A connected stress region with a substructure developed during the laser emission of 5 ms from two separate stress regions of the individual spots.

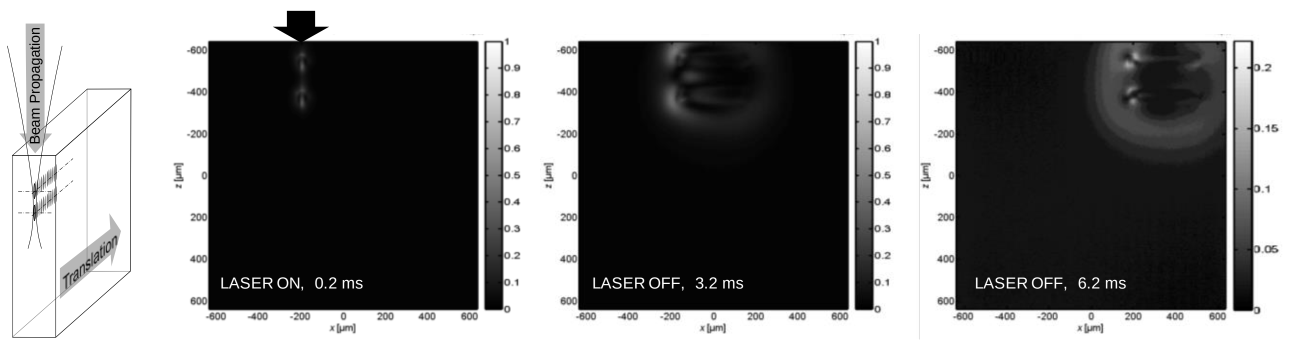


Figure 9: Evolution of stress birefringence for dual spot focusing under translation with 200 mm/s. The two spots with a pulse energy of 10.5 µJ per spot were separated 165 µm in direction of beam propagation. Laser emission with a repetition rate of 200 kHz started at 0 ms and stopped at 3 ms. Grey scale expanded by a factor of 4.5 for the image after 6.2 ms.

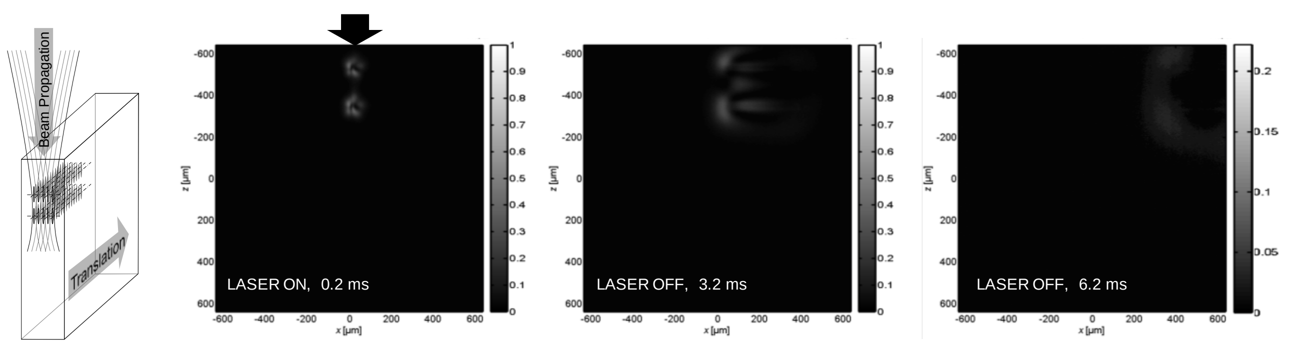


Figure 10: Evolution of stress birefringence for ten spot focusing. The spots were arranged in two lines separated by 165 µm in direction of beam propagation, the lines with a spot separation of 20 µm were orientated perpendicular to the direction of translation. The laser emission of a pulse energy of 2.1 µJ per spot with a repetition rate of 200 kHz started at 0 ms and stopped at 3 ms. Grey scale expanded by a factor of 4.5 in the image for 6.2 ms.

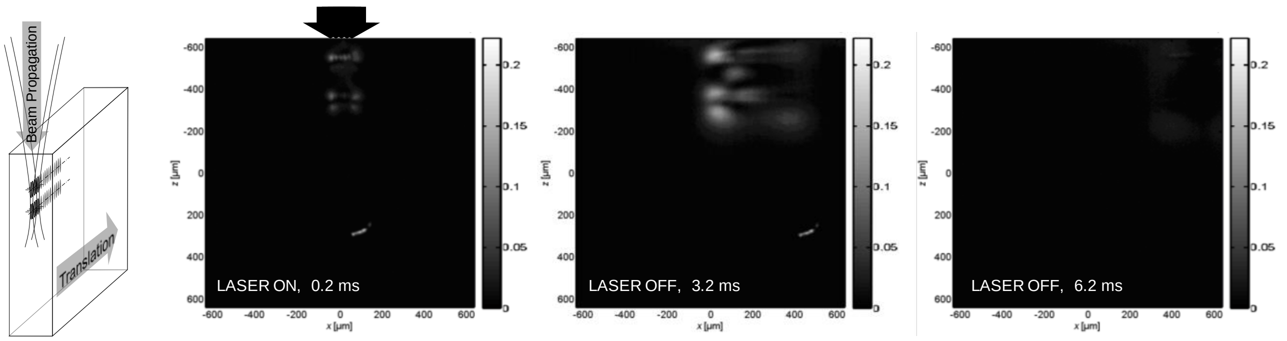


Figure 11: Evolution of stress birefringence for ten spot focusing. The spots were arranged in two lines separated by 165 µm in direction of beam propagation, the lines with spot separation of 20 µm were orientated in direction of translation. The laser emission of a pulse energy of 2.1 µJ per spot with a repetition rate of 200 kHz started at 0 ms and stopped at 3 ms. Grey scale expanded by a factor of 4.5 for all images.

By splitting each of the two spots into five spots orientated perpendicular to both, the direction of beam propagation and of translation (Fig. 10), a similar transient stress birefringence image for ten spots was achieved as for two spots. Interestingly nevertheless the permanent stress seemed to be reduced by this split of energy into five separate spots. Orientation of the five spots per line in direction of translation (Fig. 11) resulted in remarkably reduced signal due to transient stress.

The results presented on transient stress birefringence microscopy indicate the potential for supporting process development especially in the accumulation regime, when, beside sample parameters and the geometry of the absorption region, the rate of repetition and feed show a major influence.

### 3.3 **Cutting and drilling by selective laser etching**

Single pass modification for cutting by selective etching exemplifies the development of ultrafast laser processing of transparent materials supported by in-situ diagnostics.

The laser process is based on the inscription of a series of modification profiles during translation, extending from the beam entrance side to the exit side of the component. After processing, a connected modification zone along the contour resulted, having a low lateral extend perpendicular to this area (Fig. 12a). The modified volume showed typically at least a 100 times higher etching rate compared to the etching rate of the base material (Fig. 12b).

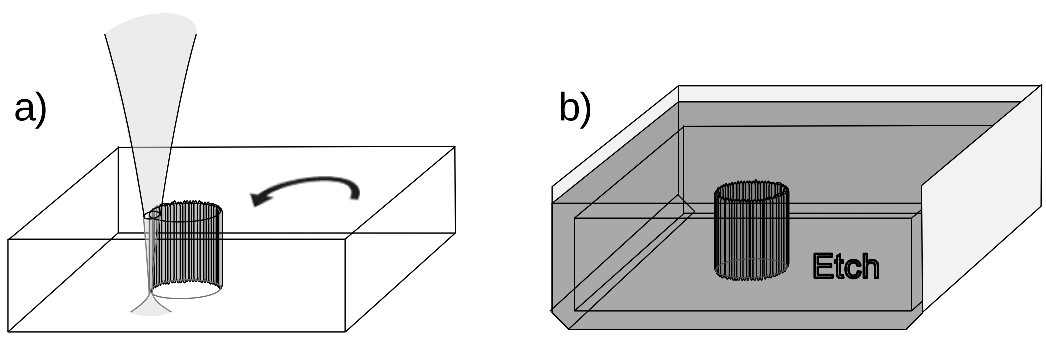


Figure 13. Schematics of cutting by single pass laser modification (a) followed by wet etching (b)

Pump probe technology was applied in the development of suitable modification profiles by beam shaping. The pump probe images in Fig. 13 illustrate the difference in spatio-temporal evolution of absorption for a Gauss-Bessel beam and a Gaussian beam with a pulse duration of 5 . The Gauss-Bessel beam was generated by imaging the beam profile originating from a refractive axicon into the sample. The same microscope objective of 10 mm focal length was used for both beam profiles. At time of initial absorption appearance, for the 10 µJ Gauss-Bessel beam absorption became visible about 70 µm behind the entrance surface along a 80 µm long region with low lateral extend (Fig. 13a, “absorption start”). For the Gaussian beam absorption started in the focal region, located about 130 µm inside the sample (Fig 13b, “absorption start”). 500 fs later the length of the absorption region for the Gauss-Bessel beam increased, mainly in direction of pulse propagation, to about 200 µm, without remarkable increase of the lateral extension (Fig 13a, “0.5 ps delay”). For the Gaussian beam just a slight increase in the length of the absorption region towards the entrance side could be observed (Fig 13b, “0.5 ps delay”). 2.4 ps after the first appearance, the absorption region for the Gauss-Bessel beam expanded from the entrance surface more than 350 µm into the material, showing increased absorption in the central region. At the same time the absorption region of the Gaussian beam reached about 50 µm in length already showing increased lateral extension. Another 2.5 ps later the absorption region of the Gauss-Bessel beam further expanded in direction of beam propagation, showing further increase of absorption with just marginal lateral expansion. For Gaussian beam focusing the final length of the absorption region stayed below 150 µm in length, reaching a diameter of about 10 µm.

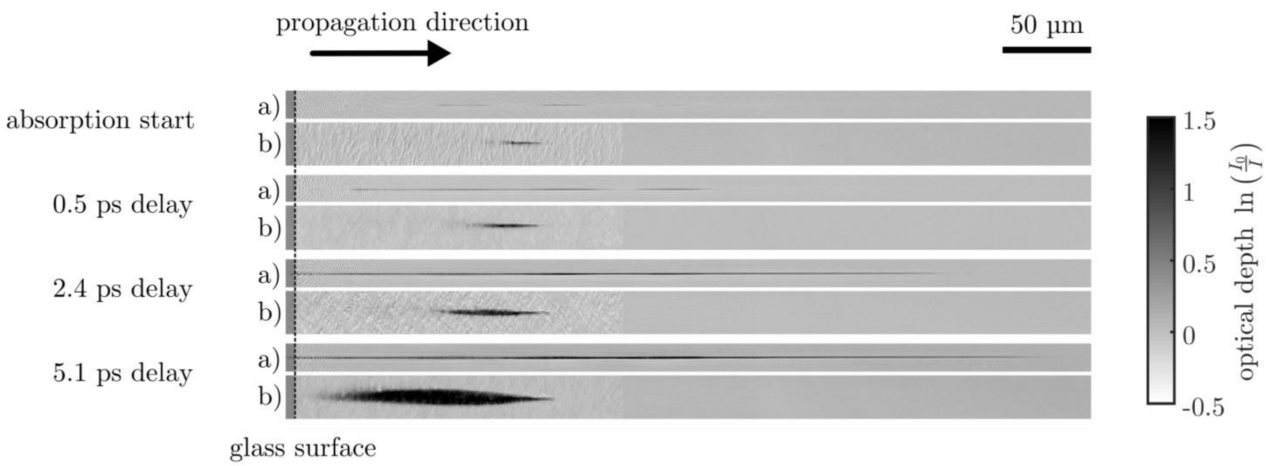


Figure 13. Pump probe images of 5 ps pulses focused inside non-strenthened glass (Coring Gorilla®) by a focal length of 10 mm for different delays of probe beam. a) Gauss-Bessel beam generated by an axicon imaged into the workpiece with a pulse energy of 10 µJ. b) Gaussian beam with a pulse energy of 30 µJ.

We identified a processing regime for selective laser etching based on adapted density of absorbed energy and high cooling rates. From the results presented above, we concluded that the high aspect ratio of the absorption region generated by a Gauss-Bessel beam and a local effective pulse duration not affected by moving breakdown effects are beneficial for this application[1,7]. This furthermore reduces detrimental accumulation effects. By adapting the temporal pulse parameters and the maximum repetition rate and further improving the absorption profile by means of diffractive beam shaping, we achieved single pass modification processing suitable for cutting and drilling by selective laser etching.

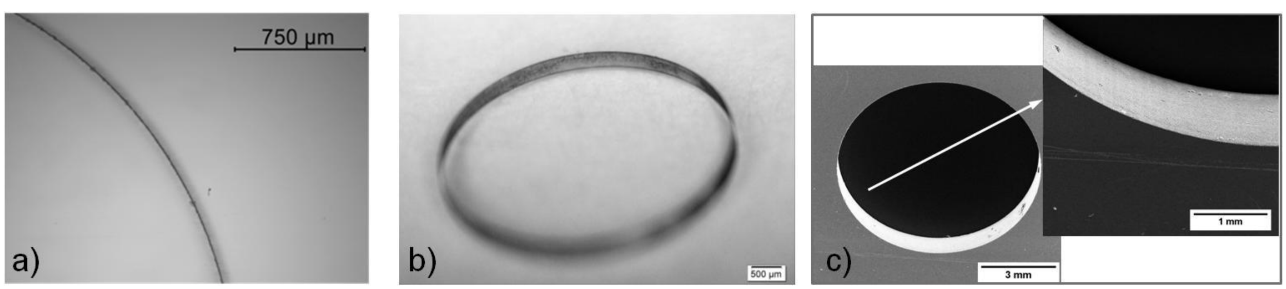


Figure 14: Hole cutting of strengthened glass (Coring Gorilla®, thickness 550 µm) by single pass laser modification followed by wet etching. Top view of inscribed modifications. b) Visualization of etched gap by ink penetration. c) SEM image of etched surface with detail.

Hole cutting of strengthened Coring Gorilla® glass is displayed in Figure 14. In Figure 14a a microscopic image of the sample from the beam entrance side shows the modification. Figure 14b is an image of the sample after etching. The gap was made visible by ink penetration. The etched surface in Figure 14b revealed a smooth surface without any visible taper, cracks or chipping.

The examples on components cut by single pass laser modification followed by wet etching given in Figure 15 illustrate the suitability of the processing methods for different materials. Typical etching rates of about 10 µm/min could be achieved for these materials applying KOH etch solutions in an ultrasonic bath.

For the development of this modification process the observation of absorption geometry, influenced by pulse energy, temporal and spatial beam shape, and the analysis of accumulation, depending furthermore on repetition rate and feed rate, were beneficial.

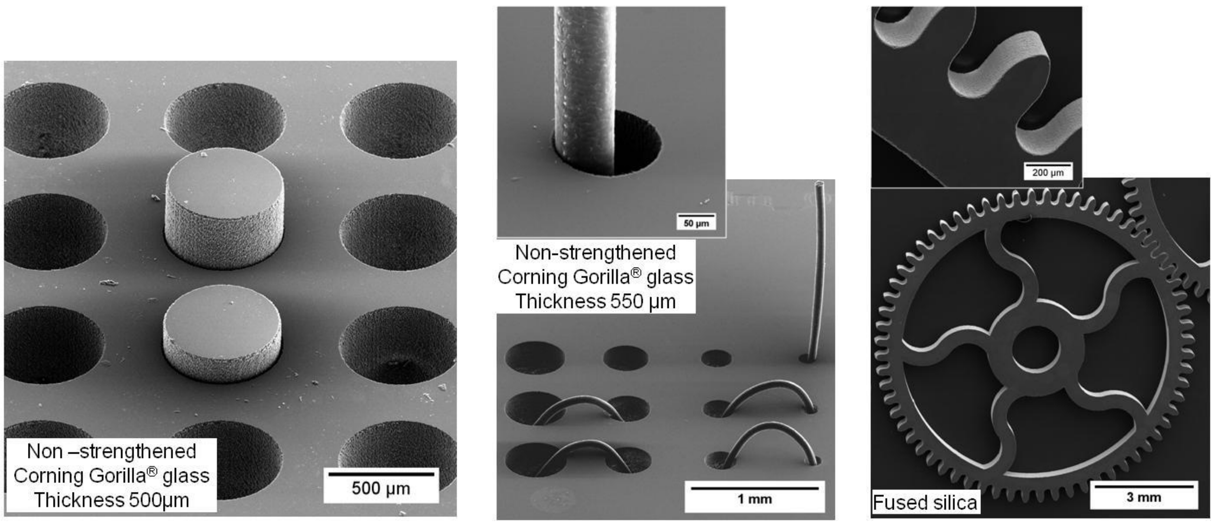


Figure 15: SEM images of samples cut by single pass laser modification followed by wet etching.

## 4. CONCLUSION

Processing of transparent materials by ultrafast lasers offers a huge potential for industrial applications. Nonlinear absorption can be tailored for a plurality of different tasks. For processing of transparent materials, optical in situ diagnostics can be applied. The present work demonstrated the benefits of such in situ diagnostics for the development of different applications. Absorption profiles, shockwaves and incubation effects have been analyzed by pump probe technology at low repetition rate. This allowed adapting the temporal and spatial pulse shape for diverse processing methods, taking into account nonlinear beam propagation. Transient stress birefringence microscopy gained understanding for processing in accumulation regime. These promising results motivate an extension of optical in situ diagnostics towards high repetition rate pump probe technology with fs-resolution for more complex temporal and spatial shaped pulses, applicable for absorption, phase and stress birefringence observation, with different observation direction relative to beam propagation and translation. We expect faster development of improved and new applications and enabling technology for industrial processing of transparent materials due to improved in situ diagnostics.

## ACKNOWLEDGEMENT

This work has been funded in part by the German Federal Ministry of Education and Research (BMBF) within the Digital Photonic Production research campus (DPP Femto), FKZ: 13N13309.

## REFERENCES


[1] Kumkar, M., Bauer, L., Russ, S., Wendel, M., Kleiner, J., Grossmann, D., Bergner, K., Nolte, S., "Comparison of different processes for separation of glass and crystals using ultrashort pulsed lasers," Proc. SPIE 8972, 8972-14 (2014).

[2] Russ, S., Siebert, C., Eppelt, U., Hartmann, C., Faisst, B., Schulz, W., “Picosecond laser ablation of transparent materials,”

[3] Eppelt, U., Russ, S., Hartmann, C., Sun, M., Siebert, C., Schulz, W., “Diagnostic and simulation of ps-laser glass cutting,” ICALEO 2012, Paper #M504, Proc LIA (2012)

[4] Gottmann, J., Hermans, M., Ortmann, J., “Microcutting and hollow 3D microstructures in glasses by In-volume Selective Laser-induced Etching (ISLE),“ JLMN Vol. 8 No. 1 (2013)

[5] Buhyan, M.K., Courvoisier, F., Lacourt P.A., Jacquot, M., Salut, R., Furfaro, L., Dudley, J.M., “High aspect ratio nanochannel machining using single shot femtosecond Bessel beams,” Appl Phys Lett 97, 081102 (2010)

[6] Kumkar, M., Kaiser, M., Kleiner, J., Grossmann, D., Flamm, D., Bergner, K., S. Nolte, S., “Cutting of Transparent Materials by Tailored Absorption” Proc. ASSL #ATh1A.2 (2014)

[7] Flamm, D., Grossmann, D., Kaiser, M., Kleiner, J., Kumkar, M., Bergner, K., Nolte, S., “Tuning the Energy Deposition of Ultrashort Pulses inside Transparent Materials for Laser Cutting Applications”, Proc. LiM, #253 (2015)

[8] Richter, S., Döring, S., Zimmermann, F., Lescieux, L., Eberhardt, R., Nolte, S., Tünnermann, A., “ Welding of transparent materials with ultrashort laser pulses“, Proc SPIE 8244, 824402 (2012)

[9] Sakakura, M., Tochio, T., Eida, M., Shimotsuma, Y., Kanehira, S., Nishi, M., Miura, K., Hirao, K., “Observation of laser-induced stress waves and mechanism of structural changes inside rock-salt crystals”, Optics Express, V19, P17780 (2011)