PROCESSING OF INDUSTRIALLY RELEVANT NON METALS WITH LASER PULSES IN THE RANGE BETWEEN 10PS AND 50PS

Paper (M103)

Beat Neuenschwander[1], Beat Jäggi[1], Marc Schmid[1], Urs Hunziker[1], Beat Luescher[2], Carmine Nocera[2]

[1] Bern University of Applied Sciences, Engineering and Information Technology, Institute for Applied Laser, Photonics and Surface Technology ALPS, Pestalozzistrasse 20, CH-3400 Burgdorf, Switzerland
[2] University of Applied Sciences Northwestern Switzerland, School of Engineering, Institute for Product and Production Engineering, Klosterzelgstrasse 2, CH-5210 Windisch, Switzerland

Abstract

In materials processing pulses in the ps range are an excellent tool to achieve high precision. Although for metals even shorter pulses would be desirable, the lower ps range is attractive from the point of view of the availability of industrial grade systems. In that sense we have analyzed metals and found that the increase of the pulse duration from 10ps to 50ps directly goes along with a significant drop of the maximum achievable volume ablation rate which may reduce the attractiveness of systems with pulse durations in the range of a few tens of ps. The situation changes for non metals like ceramics, polyether ether ketone (PEEK) and polycrystalline diamond (PCD). For PEEK and PCD the dependence of the ablation efficiency on the pulse duration in the investigated regime is strongly reduced. Silicon again shows a metal like behavior, which seems to be independent from the doping level. The porous form of the chosen zirconium oxide ceramic thwarted a systematic study to determine the threshold fluence and the energy penetration depth at the used laser wavelength of 1064nm. Nevertheless it was possible to machine small structures into the zirconium oxide. The obtained results show, that laser systems with pulse durations in the range of a few tens of ps, which are expected to be fiber based and cost effective, may be a very attractive alternative to machine industrially relevant non metals.

Introduction

The interest in ps-laser pulses for industrial applications has significantly increased in the last few years. Today, available ps-lasers are industrially applicable turnkey systems, set up in a MOPA arrangement with rod or disk amplifiers and mostly have pulse durations of 10ps or slightly less. Compared to ns-pulses from Q-switched systems, these ultra short pulses show less thermal effects due to the significantly reduced thermal penetration depth. The change to fiber based amplifier technologies would help to build more compact and cost effective systems but with pulse durations of several tens of picoseconds [1,2,3].

For metals the energy and heat-transfer process is described with the two temperature model [4], where the temperatures of the free electrons and the lattice are treated separately. The time for the energy transfer from the free electrons to the lattice, the electron-phonon thermalization time, amounts a few ps [5,6]. A further reduction of the pulse duration will not lead to additional benefits in terms of the threshold fluence ϕ_{th} [5,6,7]. On the other hand, ϕ_{th} increases when the pulse duration is raised from 10ps to 50ps [8,9]. Additionally the energy penetration depth δ decreases when the pulse duration is raised in the same interval. These influences lead to a reduction of the maximum achievable volume ablation rate by a factor of up to 5 when the pulse duration is raised as described above [8,9]. From this point of view the longer ps pulses are not very attractive for machining metals.

For non-metals the situation is different. For optical transparent materials shorter pulses in the fs-regime will lead to reduced threshold fluences due to the benefit of nonlinear effects [10,11]. In this case longer ps pulses will therefore not be practical. On the other hand the addressed region of pulse duration has been hardly investigated for material processing of non-metals and may be well suited for machining semiconductors and industrial relevant opaque non metals. In the present work investigation were performed for undoped and phosphor doped silicon, polycrystalline diamond (PCD), polyether ether ketone (PEEK) and zirconium oxide (ZrO_2).

Theory

For metals and ultra short pulses in the fs- and the ps-regime the ablation depth z_{abl} is a logarithmic function of the applied fluence ϕ and is given by [4,6,7,12,13,14]:

$$z_{abl} = \delta \cdot \ln\left(\frac{\phi}{\phi_{th}}\right) \qquad (1)$$

where ϕ_{th} and δ denote the threshold fluence and the energy penetration depth, respectively. For fs pulses two regimes can be observed [4]. In the first, low fluence regime δ is associated with the optical penetration depth going along with a corresponding threshold. For higher fluences a second regime is observable where δ denotes the thermal diffusion length of the electrons. The corresponding threshold fluence is higher than in the first regime. For ps pulses the lower regime is often not observed and a decrease of the slope, directly going with the penetration depth, can be observed [8,9]. This behaviour may tentatively be ascribed to changes in the optical and thermal properties of the material during the pulse. The logarithmic ablation law (1) also holds for many non metals e.g. indium phosphide [15] for ultra short pulses in the low fluence regime. For a Gaussian shaped beam as emitted by most ultra short pulsed systems the fluence as a function of the distance r to the beam centre reads:

$$\phi(r) = \phi_0 \cdot e^{-2\cdot\frac{r^2}{w_0^2}} \qquad (2)$$

Where the peak fluence ϕ_0

$$\phi_0 = \frac{2 \cdot E_p}{\pi \cdot w_0^2} \qquad (3)$$

is given by the pulse energy E_p and the spot radius w_0. The ablated volume per pulse, ΔV, can be calculated by inserting (2) and (3) into (1) and integrating over the ablated area. It reads [16,17]:

$$\Delta V = \frac{1}{4} \cdot \pi \cdot w_0^2 \cdot \delta \cdot \ln^2\left(\frac{\phi_0}{\phi_{th}}\right) \qquad (4)$$

For a laser working at a constant average power P and repetition rate f the pulse energy is given by $E_p = P/f$ and the total ablated volume per time amounts:

$$\dot{V} = f \cdot \Delta V \qquad (5)$$

Inserting (3) and (4) into (5) lead to:

$$\dot{V} = \frac{1}{4} \cdot \pi \cdot w_0^2 \cdot \delta \cdot f \cdot \ln^2\left(\frac{2 \cdot P}{f \cdot \pi \cdot w_0^2 \cdot \phi_{th}}\right) \qquad (6)$$

This expression clearly shows that for a given average power P and spot radius w_0, the volume ablation rate depends on the material parameters ϕ_{th} and δ and the repetition rate f. Fig. 1 compares this model (6) with experimental data for Cu-DHP (in US: C12 200).

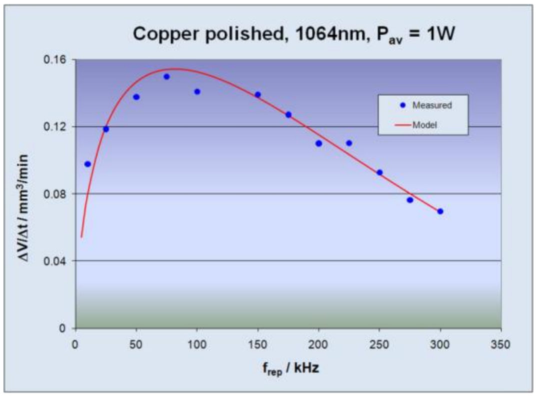

Fig. 1: Measured volume ablation rate (blue dots) compared with a least square fit to the model (red solid line) for Cu-DHP.

A very good agreement between the model (6) and the experimental data can be observed. It can also clearly be seen that the volume ablation rate shows a maximum value at an optimum repetition rate. The corresponding values per average power can be calculated from (6):

$$\frac{\dot{V}_{max}}{P_{av}} = \frac{2}{e^2} \cdot \frac{\delta}{\phi_{th}} \qquad (7a)$$

$$\frac{f_{opt}}{P_{av}} = \frac{2}{e^2} \cdot \frac{1}{\pi \cdot w_0^2 \cdot \phi_{th}} \qquad (7b)$$

The maximum volume ablation rate dV_{max}/dt as well as the optimum repetition rate f_{opt} directly scales with the average power. In addition the maximum volume ablation rate per average power only depends on the material parameter ϕ_{th} and δ, i.e. these parameters define the maximum achievable volume ablation rate for a material. Both parameters depend on the pulse duration and the number of pulses applied. The dependence of the threshold fluence on the pulse duration is well known and described e.g. in [5-7,18]. The dependence of δ of the pulse duration is mentioned in [19] and partially measured e.g. in [8,9].

Due to incubation effects the threshold fluence may strongly depend on the number of pulses applied, which is described for metals in [8,9,16,20,21], for semiconductors in [15] and for transparent materials in [11]. The most proposed model to describe the dependency of the threshold fluence on the number of pulses applied is given in [20]:

$$\phi_{th}(N) = \phi_{th,1} \cdot N^{S-1} \quad (8)$$

Where $\phi_{th,1}$ denote the threshold fluence for one pulse and the exponent S denotes the incubation coefficient. For $S = 1$ incubation is absent. But it has to be pointed out that the equation (8) predicts for an infinitive number of pulses that the threshold fluence per pulse becomes zero. From a physically point of view this is not possible. Therefore [11] propose an alternative model:

$$\phi_{th}(N) = \phi_{th,\infty} + \left(\phi_{th,1} - \phi_{th,\infty}\right) \cdot e^{-k \cdot (N-1)} \quad (9)$$

Here $\phi_{th,1}$ denotes the single pulse threshold fluene and $\phi_{th,\infty}$ the threshold fluence for an infinite number of pulses. Fig. 2 shows the difference between the two models for a pulse duration of 10 ps and Cu-DHP based on measured data from [8,9].

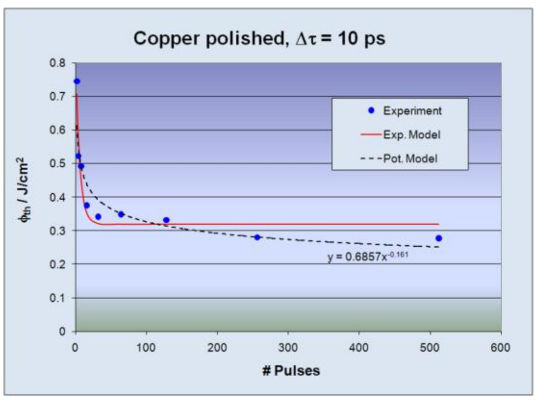

Fig. 2: Threshold fluence ϕ_{th} as function of the number of pulses applied and corresponding least square fits with the two models.

Despite the physical incorrectness in (8) this model seems to fit better the measured values. Similar models can be used for δ and again the fit with a potential function better reproduces the measured values shown in Fig. 3. Both figures clearly show, that ϕ_{th} and δ strongly varies with the number of pulses applied. This has to be considered to deduce the maximum volume ablation rate (7a) and the optimum repetition rate (7b) for a given material i.e. for reliable predictions ϕ_{th} and δ have to be measured as a function of the number of pulses applied.

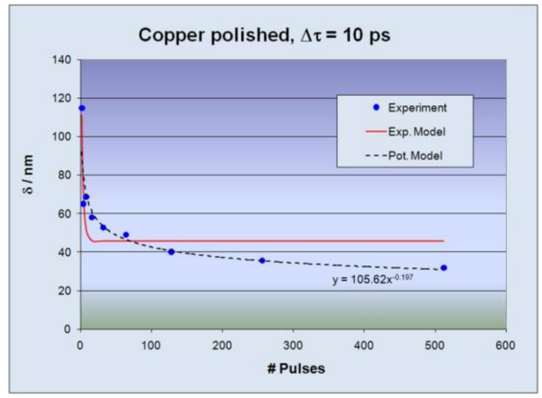

Fig.3: Penetration depth δ as function of the number of pulses applied and corresponding least square fits with the two models.

Experimental Set-Up

The experiments were performed with a DUETTO™ (Time Bandwidth Products, Switzerland) ps-laser system working at a wavelength of 1064 nm with pulse duration of about 10 ps set up in a MOPA arrangement. By introducing corresponding etalons the pulse duration could be raised to 20 ps, 30 ps and 50 ps. The pulse duration was controlled with an autcorrelator measurement whenever the etalon was changed. A pockels-cell, introduced after the last amplifier stage, served as a pulse on demand option but also limited the maximum usable repetition rate to 350 kHz. The beam is guided through a 2 times beam expander onto a turbo-scan 15 (Raylase, Germany) galvo scanner head with an f = 160 mm f-θ objective. The spot size was measured with a rotating slit beam profiler and amounts about 17.5 µm with an M^2 value of 1.3. The beam can therefore be considered as Gaussian shaped. The transmission through the whole optical path with bending mirrors and lenses amounted 83.5%. In order to deduce ϕ_{th} and δ as a function of the pulse duration and the number of pulses applied, series with single ablated craters were generated for each set of these parameters, i e. 1,2,4,...,512 pulses and 10 ps, 20 ps, 30 ps and 50 ps. For one series the pulse energy was raised from below up to multiples of the threshold. To avoid thermal accumulation effects the repetition rate was set to 50 Hz with the pockels cell. The crater depth depths in the centre were deduced with a laser scanning microscope and a digital microscope. These results were compared with the theoretical crater depth by introducing the peak fluence (3) into (1). The two parameters ϕ_{th} and δ were then deduced by a least square fit. Especially at low pulse energies i.e. low fluences or low number of pulses applied the deduction of the crater depth became extensive and could contain high measurement errors. The deduction of the threshold fluence with the squared diameter as e.g. done in [15] would be much easier in this regime but doesn't give any information about the penetration depth δ which is necessary to deduce the maximum volume ablation rate according to (7a). The volume ablation rate can also be determined by marking straight lines with the galvo scanner. Here different repetition rates, marking speeds and repeats could be chosen. The number of pulses applied can be estimated by the spot diameter, repetition rate, marking speed and number of repeats. The volume ablation rate can then be deduced by measuring the ablated volume of a line segment with the laser scanning microscope. Together with the marking speed, the length of the segment and the number of repeats the volume ablation rate can exactly be calculated. By marking lines at constant average power, repetition rate and marking speed the volume ablation rate can directly be compared for different pulse durations. But, according to Fig. 1 and (7a/b) a maximum volume ablation rate can be obtained and this is the fairest measure to compare different pulse durations and materials. On the one hand, this can be done by deducing ϕ_{th} and δ as described above or by marking lines at a constant average power but different repetition rates for constant distance from pulse to pulse and number of repeats. With the model (6) ϕ_{th} and δ can then be deduced via a least square fit.

Results

Silicon

The maximum volume ablation rate for undoped and phosphor doped crystalline silicon was deduced by (7a) based on an experimental measurement of ϕ_{th} and δ by ablating single craters as described above. For a comparison straight lines were marked with the scanner at an average power of 1.25 W and a repetition rate of 75 kHz with two different marking speeds, 40 mm/s and 320 mm/s, and number of repeats leading to an average number of 350 pulses at any position in the line. The results are summarized in Fig. 4. The difference between doped (empty markers) and undoped silicon (filled markers) is within the uncertainty of measurement, i.e. no difference between the two types of silicon could be observed.

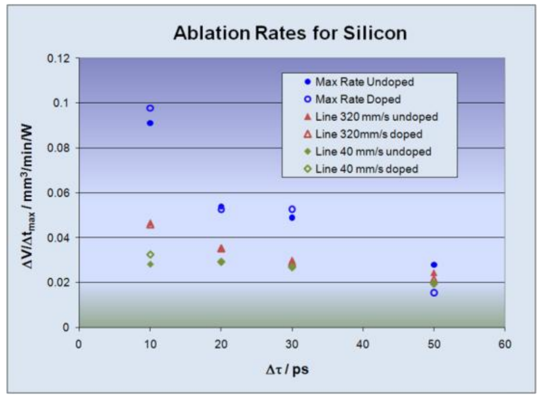

Fig. 4: Experimentally measured and deduced ablation rates for silicon.

The maximum volume ablation rate drops by about 80% when the pulse duration is raised from 10 ps to 50 ps. This strong drop is also observed for metals [8,9]. For the lines, the volume ablation rate equals the maximum rate for the pulse duration of 50 ps. As ϕ_{th} raises with the pulse duration the optimum repetition rate would become higher for lower pulse durations according to (7b) i.e. the chosen repetition rate is too low compared to the optimum. In the typical curve of the volume ablation rate as shown in Fig. 1, the working point is on the left side of the optimum where the volume ablation rate strongly drops. This fact explains the strong difference between the maximum and the measured rate in Fig. 4 for shorter pulse durations.

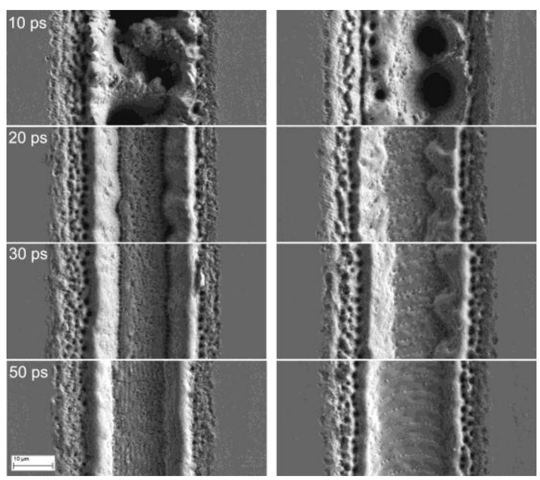

Fig. 5: SEM images of marked lines in undoped silicon with average power of 1.25W at a repetition rate of 75 kHz. Left: marking speed 40 mm/s, 8 repeats. Right: Marking speed 320 mm/s, 64 repeats. The scale bar denotes a distance of 10 µm.

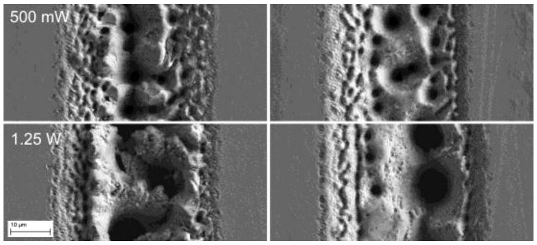

Fig. 6: SEM images of marked lines in undoped silicon with 1.25 W and 0.5 W average power. The scale bar denotes a distance of 10 µm.

Strong deviations in the line shape have been observed between the pulse duration of 10 ps and longer pulses as illustrated in Fig. 5 for undoped silicon. Here strong melting effects at the borders and on the bottom of the line for pulse durations of 20 ps and more can be observed. In contrast for 10 ps strong small craters and structures in the line can be observed. This kind of cones and deep small craters has been often observed by ablating steel with ultra short pulses, as well.

A reduction in the average power from 1.25 W down to 0.5 W leads to smaller structures but the effect does not disappear as illustrated in Fig. 6. Exactly the same behaviour was observed for the phosphor doped silicon.

Polycrystalline Diamond (PCD)

For PCD again similar to the previous experiments marked straight lines were compared with the maximum volume ablation rate obtained from a measurement of ϕ_{th} and δ as a function of the pulse duration and the number of pulses applied. The lines were marked with an average power of 1 W and a repetition rate of 75 kHz at the two marking speeds of 40 mm/s and 320 mm/s. The number of repeats was 16 and 144 leading to 700 and 788 pulses at any position in the line. These results are shown in Fig. 7 together with the maximum volume ablation rate deduced from the study with 512 pulses applied. Almost equal results were obtained for the marked lines and the maximum rate. The small differences are within the uncertainty of measurement.

It was found, that the threshold fluence raises with increasing pulse duration similar to all investigated materials, but in contrast to metals and silicon, in the case of PCD the penetration depth also becomes longer for higher pulse durations. Therefore the maximum volume ablation rate (7a) is much less affected by the pulse duration.

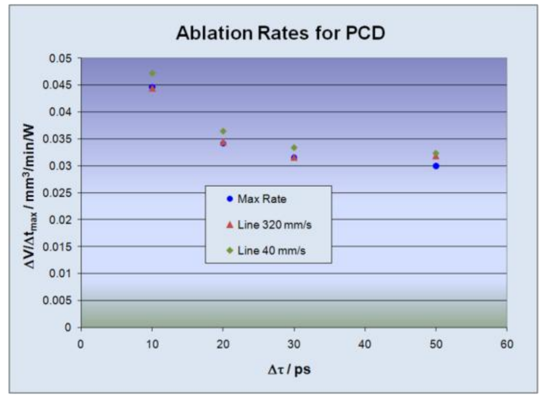

Fig. 7: Experimentally measured and deduced ablation rates for polycrystalline diamond.

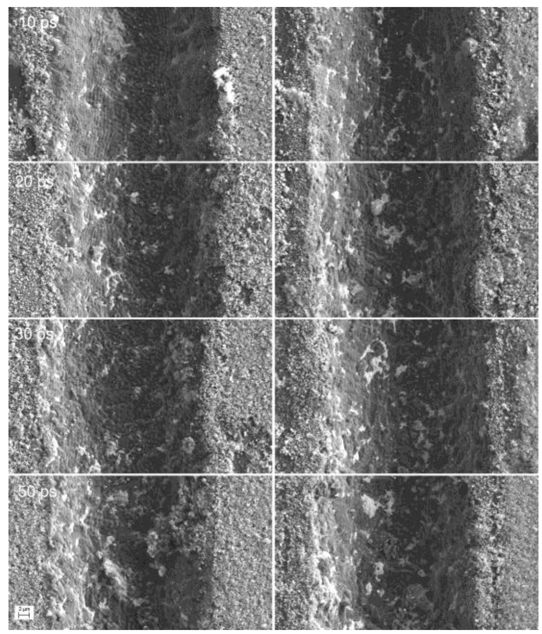

Fig. 8: SEM images of marked lines in polycrystalline diamond with average power of 1 W at a repetition rate of 75 kHz. Left: marking speed 40 mm/s, 16 repeats. Right: Marking speed 320 mm/s, 144 repeats. The scale bar denotes a distance of 2 µm.

SEM images with details of the ablated lines are shown in Fig. 8 for all pulse durations and both marking speeds. The lines were not cleaned after machining and therefore small contaminations with small particles can be observed at the border of the lines. Based on these images almost no difference between the different pulse durations and marking speeds can be observed i.e. the pulse duration for the regime between 10 ps and 50 ps only has a minor influence on the marking quality.

Polyether ether ketone (PEEK)

For PEEK ϕ_{th} as well as δ strongly drops with the number of pulses applied and, as for PCD, raises with the pulse duration. Again the contrary dependence of δ from the pulse duration has been observed compared to metals. In contrast to all other investigated materials where δ is in the order of a few 10 nm for PEEK it is at least one magnitude higher and can go up to more than 1 µm for a low number of pulses applied. The high value of δ leads to a very high maximum volume ablation rate (7a).

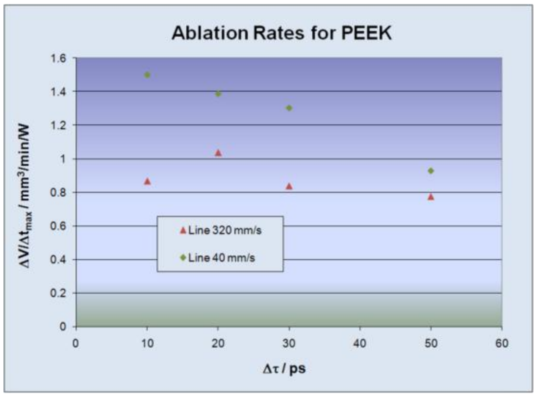

Fig. 9: Experimentally measured ablation rates for polyether ether ketone PEEK.

Straight lines were marked at an average power of 2.8 W and again with a repetition rate of 75 kHz and with marking speeds of 40 mm/s and 320 mm/s. Due to the high volume ablation rate only one repeat was done leading to an average of 65 and 8 pulses at any position in the line, respectively. For this low number of pulses applied ϕ_{th} as well as δ could not be measured accurately enough to directly compare the deduced maximum volume ablation rate with the measured ablation rates from the lines. The results for the ablated lines are summarized in Fig. 9. Compared to silicon and PCD the obtained volume ablation rates are about on order of magnitude higher. For the fast marking speed of 320 mm/s almost no change in the ablation rate can be observed where for 40 mm/s the ablation rate continuously drops by about 40% when the pulse duration is raised from 10 ps to 50 ps.

SEM images from these lines marked with 40 mm/s were shown in Fig. 10. Melting effects are observed at the border of the lines and the lines marked with 50 ps pulse duration are significantly less broad. Complete different structures were observed for a marking speed of 320 mm/s as shown in Fig. 11. Here, any kind of worms from material are formed by local melting effects. But the line width is almost unchanged when the pulse duration is raised up to 50 ps. This implies that the quite flat surfaces of the lines marked with lower marking speed are a result of strong heating effects of the material. The significant higher volume ablation rate is a sign that thermal accumulation effects may play an important role for the ablation of PEEK with ultra short laser pulses. Further investigations have to be done to clarify the influence of these effects.

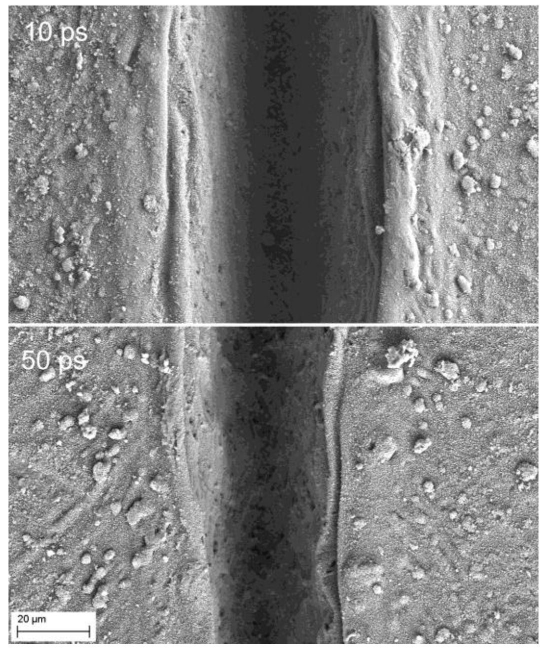

Fig. 10: SEM images of marked lines PEEK with average power of 2.8 W at a repetition rate of 75 kHz and a marking speed of 40 mm/s with one repeat.

Zirconium oxide (ZrO_2)

Zirconium oxide has served as a white and scattering ceramic. The porous form of the ZrO_2 thwarted a systematic study to determine ϕ_{th} and δ. Instead of marking straight lines, 3x3 small pyramids were machined into the material as real 3d structures. The side length of the pyramids amounted 1 mm and its spacing was 1.2 mm. The structuring was done in a conventional 2½ -process by dividing the pyramid into 98 slices each hatched with a distance of 17 µm. The hatch pattern is turned by 5° from slice to slice. The marking speed was 150 mm/s, the average power was set to 1 W and the repetition rate was 50 kHz. In Fig. 12 the pyramids machined with 10 ps and 50 ps are shown. Moiré patterns can be observed for both pulse durations but the pyramid edges seems to be more pronounced for the short pulse duration of 10 ps. The structure depth continuously decreases from 280 µm at 10 ps pulse duration down to 130 µm at 50 ps. Details of the machined surface are shown in

Fig. 13. The surfaces seem to be random structured and locally melted, but no significant difference could be observed for the different pulse durations, i.e. ZrO_2 can be machined with all investigated pulse durations. Similar detail surface structures were also observed for alumina ceramic machined with pulses of 180 fs duration [22].

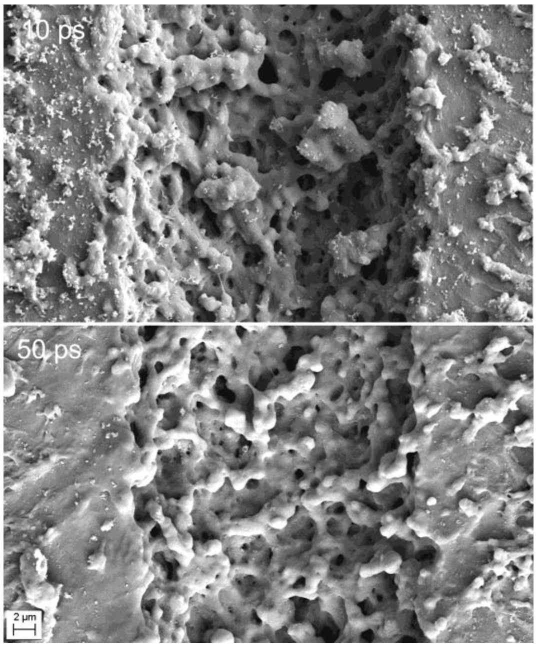

Fig. 11: SEM images of marked lines PEEK with average power of 2.8 W at a repetition rate of 75 kHz and a marking speed of 320 mm/s with one repeat.

The total time to machine all the 9 pyramids has amounted 480 s. From this and the knowledge of marking and jump speed of the scanner, the scanning strategy and the number of slices one can estimate the time the laser was on target. Together with the ablated total volume which is given by the ablation depth and the dimensions of the pyramids the volume ablation rate can be estimated. The ablation rates deduced in this way are shown in Fig. 14. A reduction of the volume ablation rate by about 55% at 50 ps compared to 10 ps pulse duration was observed. The values are between PCD and PEEK, i.e. ZrO_2 can be machined more efficient than PCD but not as good as PEEK. It has to be noted here, that these values for ZrO_2 may significantly differ from the maximum values which could be obtained by optimum laser parameters.

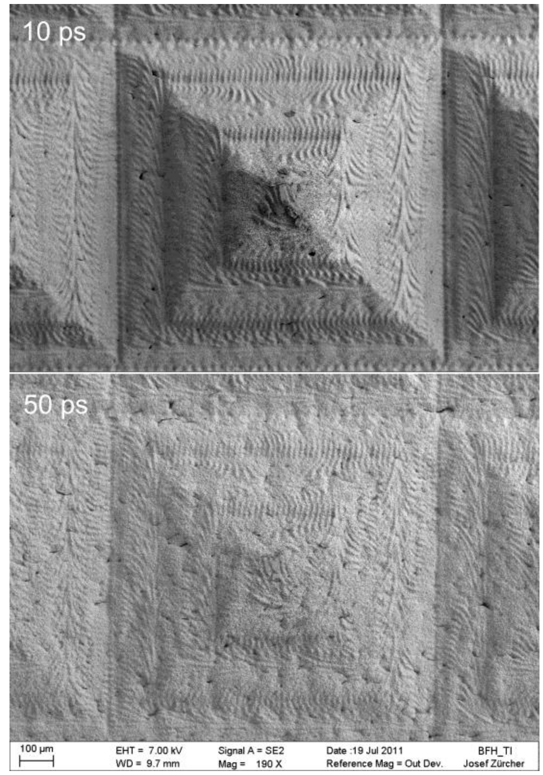

Fig. 12: SEM image of the machined pyramids with 10 ps and 50 ps.

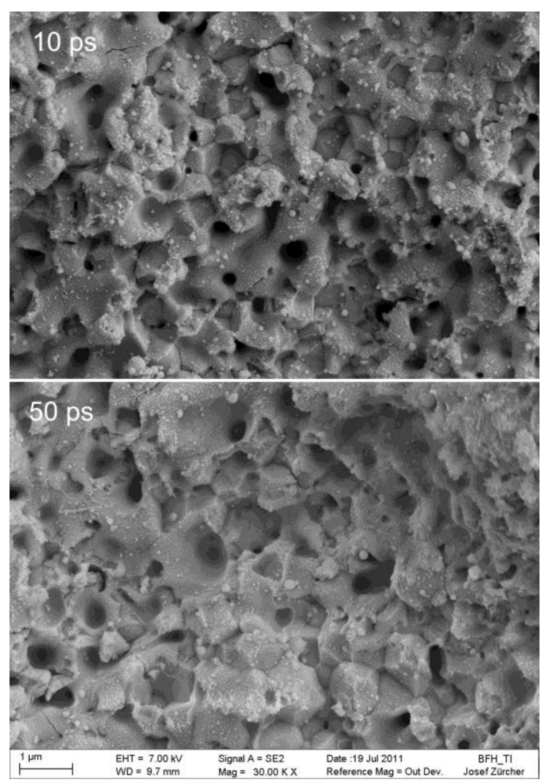

Fig 13: Detail image of the machined ZrO_2 surfaces.

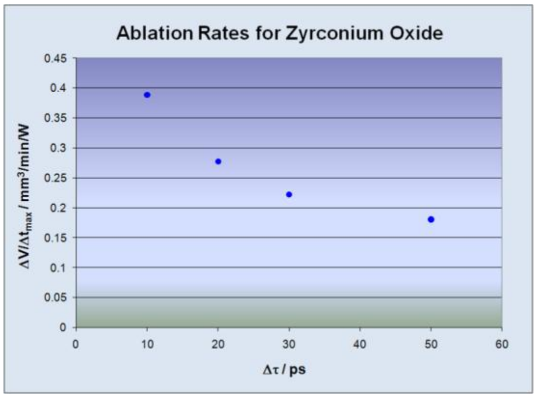

Fig. 14: Experimentally measured ablation rates for zirconium oxide (ZrO_2).

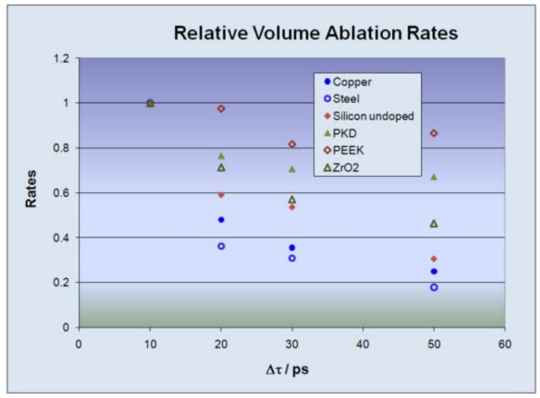

Fig. 15: Normalized volume ablation rates for all investigated materials and two additional metals.

Conclusion

All the investigated materials (silicon, PCD, PEEK and ZrO_2) can be machined with pulses in the addressed regime between 10 ps and 50 ps pulse duration. In the case of doped and undoped silicon strong differences has been found between 10 ps pulse duration and longer pulses. For the short pulses cones and craters were observed and melting effects dominate the regime of the longer pulse durations. No such difference has been found for all other investigated materials. PEEK and ZrO_2 can be machined with very high efficiency but machining of PEEK can be affected by thermal accumulation effects. The volume ablation rate will be able to reach a maximum value if the laser parameters are optimized. This maximum rate depends on the threshold fluence and the energy penetration depth. For all materials a reduction of the volume ablation rate has been observed when the pulse duration is raised from 10 ps up to 50 ps. Fig. 15 shows the volume ablation rate for the investigated materials and additionally for copper and steel. The values are normalized to the ones at 10 ps pulse duration. For the metals the reduction in the volume ablation rate amounts about 80%. Here the threshold fluence raises and the penetration depth decreases with increasing pulse duration. This enhances the reduction of the ablation rate. A similar behavior was observed for silicon (undoped and phosphor doped). For ZrO_2, PCD and PEEK the drop in the ablation rate is much less pronounced. For PCD and PEEK it has been found that the penetration depth increases with increasing pulse duration. This behaviour would be expected when the energy transport is dominated by heat conduction.

Finally the pulse duration regime from 20 ps to 50 ps may be very attractive to machine ZrO_2, PCD and PEEK due to the small drop in the volume ablation rate and the small influence onto the surface quality. For silicon the raise of the pulse duration to 20 ps prevents cone and crater formation but leads to melting effects.

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Meet the Author

Beat Neuenschwander studied physics at the University of Bern and realized 1996 his PhD at the institute of applied physics. Since 2000 he is at the Bern University of Applied Sciences where he built up the laboratory for laser micro machining. He lectures physics and applied laser technology and is currently head of the optics section of the Swiss Society for Optics and Microscopy SSOM.