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Micromachining of quartz with ultrashort laser pulses

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Abstract. Well-defined and highly reproducible channels of a few micrometres diameter and lengths of over 1 mm have been produced in quartz with laser pulses of 790 nm wavelength (Ti:sapphire) and pulse lengths of 100–200 fs. The channel depth can be controlled by the laser fluence and number of laser shots. Comparisons were made with laser pulse lengths of 1.3 ps, 2.8 ps, 30 ps, and 5 ns. The micromachining produced by the fs laser pulses was found to be much more controllable and reproducible than the longer pulses and produced much less damage in the material.

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Material removal caused by high-intensity laser pulses, otherwise known as laser ablation, has been demonstrated to be a powerful tool in surface micropatterning and structuring of a range of materials [1]. There are a number of advantages in using lasers for micromachining: single-step processing; high flexibility; direct writing of structures by moving the laser beam at speeds much greater than can be obtained with mechanical tools; no contamination of the material being processed; sterility (important for medical and biological applications) and the precise focusing which is possible leading to highly localised treatment of materials with a spatial resolution better than 1 μm. However, this high spatial resolution normally cannot be achieved using standard ns lasers due to strong thermal effects which occur in the material and the destructive influence of the plasma which is formed above the surface. There are two main ways of minimising these destructive effects if structures with dimensions on the order of micrometres are required. UV laser light can be used which can, in principle, be focused on to smaller areas, although this is usually a problem with the most commonly used excimer lasers due to their relatively poor beam quality. Another advantage of UV light is that materials generally have a much higher absorption in the UV thus concentrating the absorbed energy in a relatively small volume leading to controllable material removal. Laser plasma interactions are less pronounced compared with visible or IR wavelengths and, finally, non-thermal excitations and photochemical bond breaking can become more important. This approach has led to a reasonable damage suppression in structures on the order of 10–100 μm dimensions or less for many materials such as organic polymers [2], biological tissue [3] and some ceramic materials such as high-temperature superconductors [4]. The accuracy and reproducibility that can be obtained with other materials of practical interest such as metals or many transparent materials is, however, still limited. The second approach to minimising the destructive effects is to use ultra-short laser pulses with pulse widths in the ps and fs range [5–10]. This is the approach used in the present paper. In addition to reducing the destructive thermal effects due to the much shorter time scale for the energy coupling into the material and eliminating the laser-plasma interaction above the surface, the use of ultrashort laser pulses can induce multiphoton absorption and other non-linear optical effects. The former leads to a much smaller absorption volume than for single-photon absorption which can give a much more controllable removal of material and also serves to reduce further the thermal stress in the bulk. Other non-linear optical effects are e.g. self-focusing of the laser beam in the transparent material due to the optical Kerr effect. Under certain conditions, this effect can be manipulated to produce precise microstructures [11].

Quartz is one of the most important materials in the fields of optoelectronics, micro-optics and fibre technology. It has a high transmission from the UV to IR and excellent thermal and electrical properties and high chemical resistivity. These properties also make it a particularly challenging material for micro-machining applications. In this paper we show that it is possible to ablate highly reproducible microchannels in quartz with diameters on the order of 20 μm or less and lengths of over 1 mm by using visible laser light from a commercial Ti:sapphire fs laser system directly focused onto the surface of the material. The results are compared with channels ablated using ps and ns pulses with wavelengths in the visible range.

1 Experimental setup

A Ti:sapphire oscillator-amplifier system based on the chirped pulse amplification technique was used for ablation with pulse lengths of 120 fs to 2.8 ps. The laser light was focused onto the sample surface by a quartz lens (focal length $f = 75$ mm) giving an $1/e^2$ irradiated area of 680 μm^2 at $\lambda = 790$ nm. The experiments were carried out in N_2 (1 atm.) and under vacuum conditions (1 mbar and $< 10^{-4}$ mbar). The repetition rate was varied between 10 Hz and 1 kHz. The experiments at 30 ps and 5 ns were carried out with the second harmonic of two Nd:YAG lasers (532 nm) at a repetition rate of 10 Hz. The same lens was used to focus the laser beams giving a slightly smaller focus area than for the Ti:sapphire system. Nd:YAG lasers were chosen for comparison since the laser beams were of comparable quality to the Ti:sapphire laser, which is important for experiments in which the laser beam is focused directly onto the surface of the material, the wavelength lies in the visible range and the intensities which could be obtained were large enough to be able to compare equivalent amounts of material removal for different laser pulse lengths. The quartz samples were 1 and 2 mm thick and polished on both the upper and lower surfaces and on the side edges thus allowing us to analyse the ablated channels with a Nomarski optical microscope where polarised light was used to increase the image contrast. The samples were supported on a metal target holder such that the laser processed area was free-standing.

2 Results and discussion

Figure 1 shows a series of channels ablated in quartz using 120 fs pulses at 790 nm for different numbers of laser shots. The experiments were carried out in a vacuum of $< 10^{-4}$ mbar with a laser fluence of 42 Jcm^{-2} (280 μJ pulse energy). One can clearly observe a characteristic narrowing of the holes from the diameter produced at the front surface of the sample to a diameter significantly smaller than the laser focus diameter (30 μm at $1/e^2$ intensity). In the examples given in Fig. 1 the channel diameter was 21 μm after approximately 200–300 μm. This diameter then remains constant until the bottom of the hole is reached. There is some evidence of damage to the surrounding material at the top of the hole, where the narrowing occurs, as evidenced by the small amount of cracking that can be observed around the hole. In this region a band of darker colouring can also be seen which indicates some changes to the material, probably in the form of mechanical stress. This damage can no longer be seen once the narrow channel has been formed. The width and length of the channels produced for a given number of shots are highly reproducible for given vacuum and laser focus conditions. The width of the channel can be adjusted by changing the focusing conditions. This is the subject of further ongoing quantitative investigations.

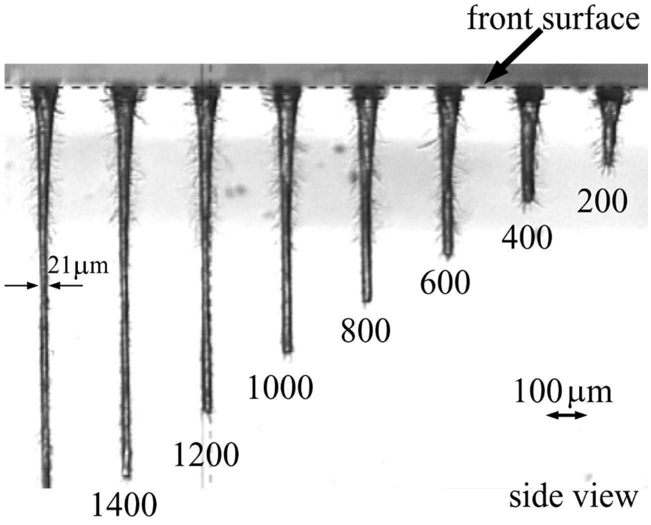

Fig. 1. Channels produced in quartz with 120 fs pulses at 790 nm, 16 Hz, 42 Jcm^{-2} (280 μJ) for different numbers of laser shots as indicated on the figure. The experiments were carried out in a vacuum of $< 10^{-4}$ mbar

Figure 2 shows an example of a 5 × 5 matrix of holes produced in a 1 mm thick quartz plate. The holes were produced with 1600 shots at 120 fs, 330 μJ pulse energy and a repetition rate of 160 Hz under vacuum conditions. The holes are highly reproducible and although they are narrowly spaced (150 μm) the bulk material remains stable after processing and does not show any tendency to crack or split apart. There is some evidence of small cracks and material removal around the entrance (Fig. 2a) and, more apparently, the exit (Fig. 2b) holes similar to the cracking observed at the top of the structures shown in Fig. 1. However, this is greatly reduced in the bulk of the material as can be seen from the side view of some of the channels shown in Fig. 3.

In the series of experiments shown in Fig. 1 the maximum hole depth reached was approximately 1.8 mm for 3000 laser shots or more. This saturation depth is smaller with narrower

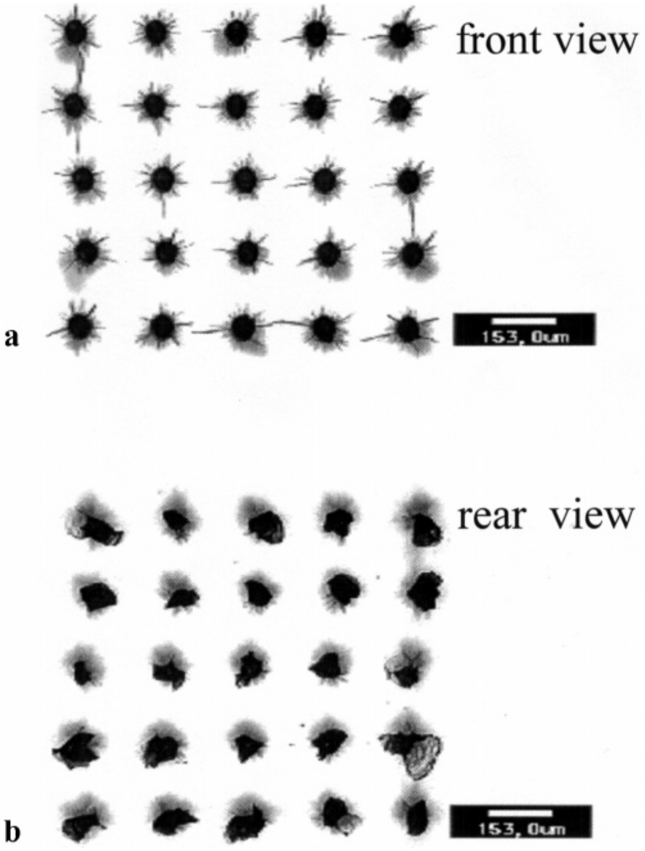

Fig. 2a,b. Channels bored through a 1 mm thick quartz plate, 120 fs, 790 nm, 330 μJ pulse energy, 160 Hz, 1600 pulses under vacuum conditions, **a** entrance holes **b** exit holes

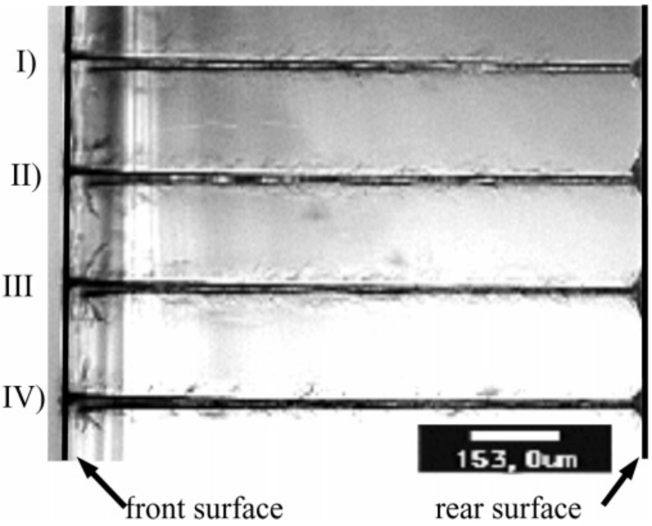

Fig. 3. Side view of channels shown in Fig. 2. There is considerably less evidence of damage in the bulk material than around the entrance and exit holes. I,II: 1120 pulses, III, IV: 1600 pulses

channels which is presumably a consequence of the difficulty of removing the material from the narrow channels. Figure 4 illustrates the dependence of the hole depth on the number of laser shots for the conditions used in Fig. 1. The dependence is nicely linear up to approximately 2500 laser shots where saturation occurs for a hole depth of 1.8 mm. The dependence of the maximum depth that can be reached on the surrounding pressure is also indicated in the figure. The saturation depth that can be reached is very similar for pressures of $< 10^{-4}$ mbar and 1 mbar but much smaller for ablation under atmospheric pressure (N_2). The saturation depth can be varied by varying the laser pulse energy. This is illustrated in Fig. 5 for different pressure conditions where one can again see the large difference between ablation under vacuum conditions and under N_2. There is also a slight indication of saturation beginning to occur in this plot at high pulse energies. (Note that the laser focus conditions in this series of experiments were slightly different than for those in Fig. 4 leading to channels with diameters of approx. 10 μm. This gave maximum hole depths of ca. a factor two smaller than for the 21 μm wide channels.) We were unable to observe the formation of the long, narrow channels for ablation in N_2. Some examples of the holes produced under vacuum and in N_2 for the series of experiments analysed to obtain the results given in Fig. 5 are shown in Fig. 6.

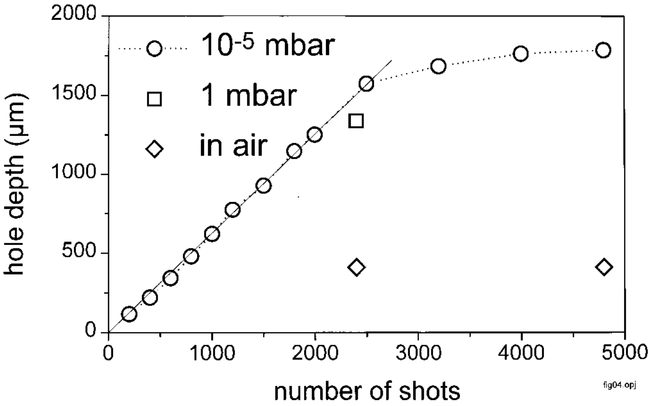

Fig. 4. Hole depth as a function of number of laser shots for ablation with 790 nm, 120 fs, 280 μJ (focus conditions as in Fig. 1) for different pressure conditions. Saturation is reached for 2500 laser shots

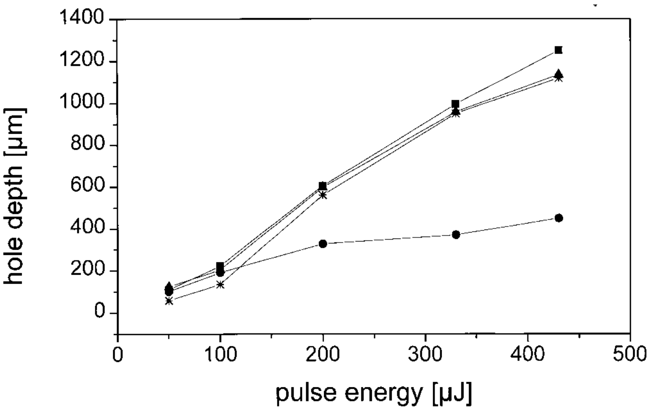

Fig. 5. Saturated hole depth as a function of laser pulse energy energy for different pressure conditions (790 nm, 120 fs, 1 kHz). The channel diameter was approx. half the size of those in Figs. 1 and 4 leading to saturated hole depths which are a factor of two smaller. *Squares*: $< 10^{-4}$ mbar; *triangles*: $< 10^{-4}$ mbar, 5×2000 pulses; *asterix*: rough vacuum (10^{-3} mbar); *circles*: 1 atm. N_2

The maximum depth that can be reached does not depend to any significant extent on the laser pulse length in this range, where the threshold values for ablation do not show

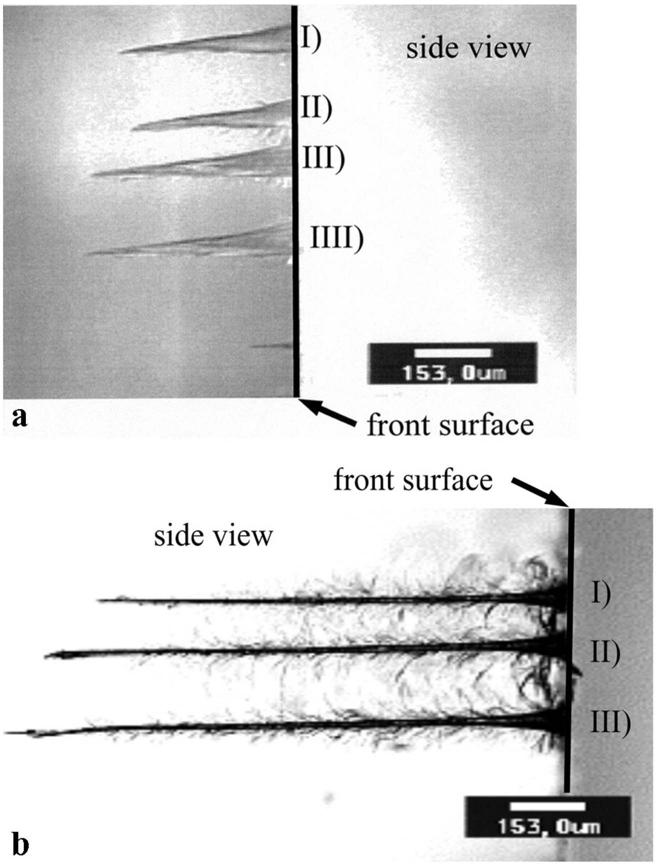

Fig. 6a,b. Channels produced at 790 nm, 1 kHz, 10 000 pulses. **a** in N_2 (I,II) 330 μJ, (III, IV) 430 μJ **b** vacuum conditions (I) 330 μJ (II,III) 430 μJ a large dependence on this parameter [12], but is predominantly a consequence of the laser pulse energy (or fluence). This is illustrated in Fig. 7 for ablation under vacuum conditions with three different laser pulse lengths (200 fs, 1.3 ps and 2.3 ps) and two different values of the laser fluence (the focus was identical in all cases). We have found a very similar behaviour for the laser ablation of sapphire with ultrashort laser pulses [11].

The channels produced with 200 fs and 2.8 ps laser pulses are very similar to the 120 fs results discussed above. Some examples are shown in Fig. 8 for irradiation under $< 10^{-4}$ mbar pressure with a laser fluence of approximately 15 Jcm^{-2} (pulse energy of 100 μJ) for a large number of laser pulses where saturation has been reached. The maximum depth that could be obtained (ca. 500 μm) is considerably smaller than for the example given in Fig. 1 due to the much smaller pulse energy used. However, one can still see the narrowing of the channel at the beginning of the structure with the constant diameter channel being reached at a distance of approximately 200 μm from the surface as was the case above. The main difference between the 200 fs and 2.3 ps results is the slightly increased cracking around the channel for 2.3 ps. The increased damage obtained with the longer pulses can also be seen very clearly in Fig. 9 where the entrance holes are compared for a series of pulses with 200 fs pulse length (upper trace) and 2.8 ps (lower trace) both for a pressure of $< 10^{-4}$ mbar, 100 μJ pulse energy and with increasing number of laser shots going from left to right. The entrance holes are significantly smaller and better formed for the shorter pulses and show much less evidence of damage outside the irradiated area than the holes produced with 2.8 ps pulses. In general, the peripheral damage is seen to increase with increasing laser pulse energy but, for a given pulse energy, the damage is less for shorter pulses.

A comparison was carried out with pulse durations of 30 ps and 5 ns (10 Hz repetition rate) using frequency doubled Nd:YAG lasers (532 nm). We do not expect any significant differences in the ablation due to the difference in wavelength. Both wavelengths used lie in the visible region of the spectrum and are far away in energy from any resonant absorptions or direct excitation mechanisms that could occur in quartz. Support for this comes from measurements of the threshold fluences for damage in this material which were found to be the same for 780 nm [13] and 1053 nm [14] in the pulse length range of 10 ps to 10 ns (for different kinds of experiments carried out in different groups). There is however a very significant difference in the threshold fluences as the pulse length is increased from a few ps to 5 ns [12, 13]. For this reason we chose to compare laser fluences for the longer pulses which lay approximately a factor of 5 above the threshold fluence, as was the case for the 200 fs and 2.8 ps pulses which gave the results shown in Figs. 8 and 9. The results for 30 ps pulses at a pressure of $< 10^{-4}$ mbar are shown in Fig. 10 for different numbers of laser shots. The reproducibility in the depth of the hole produced with a given number of laser shots is very good, as was the case for the shorter pulses, however, the lateral damage and cracking has become much more pronounced. This was also apparent in the regularity with which the irradiated samples splintered and broke, both during the laser irradiation and afterwards. The damage around the irradiated areas was seen to increase with increasing number of laser shots and it was normal to find large cracks connecting the holes.

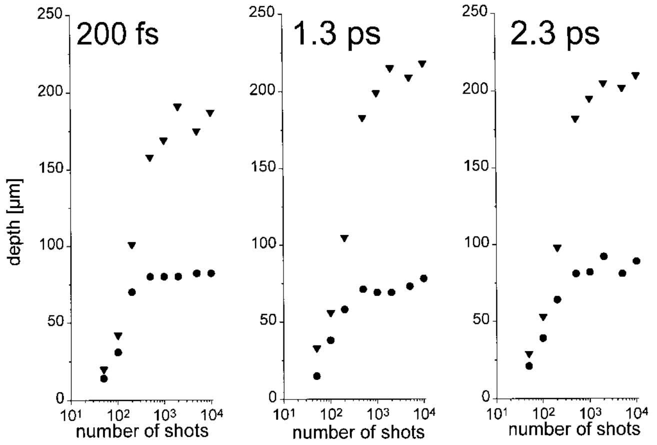

Fig. 7. Hole depth as a function of number of laser shots for ablation with 790 nm (10 Hz) under vacuum conditions for three different laser pulse lengths, 200 fs, 1.3 ps and 2.3 ps. *Triangle*: 12 Jcm^{-2}, *circle*: 6 Jcm^{-2}

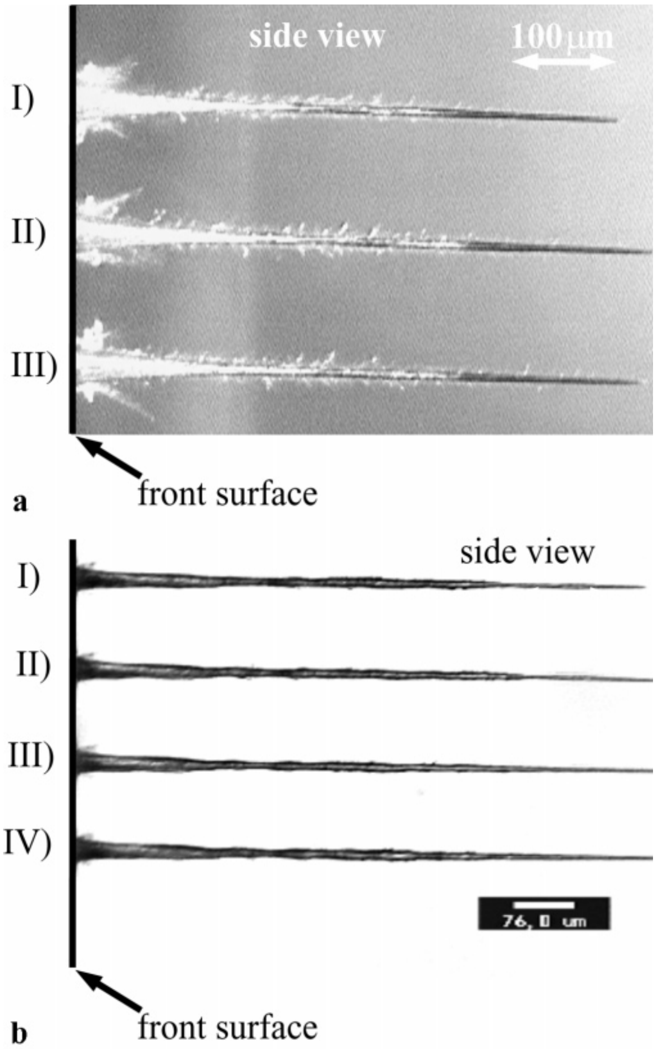

Fig. 8. Side view of channels produced in quartz with 790 nm, 100 μJ under vacuum conditions for different numbers of laser shots. *lower*: 200 fs, I: 10 000, II: 20 000, III: 30 000 shots; *upper*: 2.8 ps I,II: 10 000, III,IV: 20 000 shots

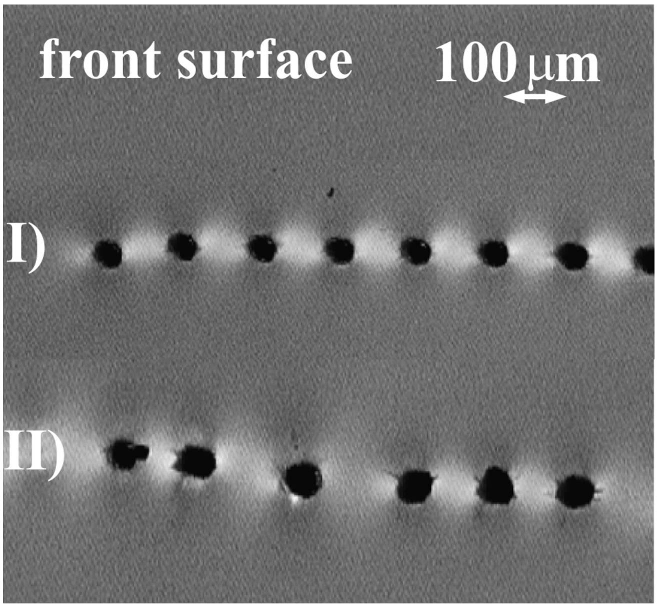

Fig. 9. Top view of entrance holes produced with 790 nm, 100 µJ pulse energy for increasing number of laser shots moving from left to right under vacuum conditions. I: 200 fs pulses, II: 2.8 ps pulses

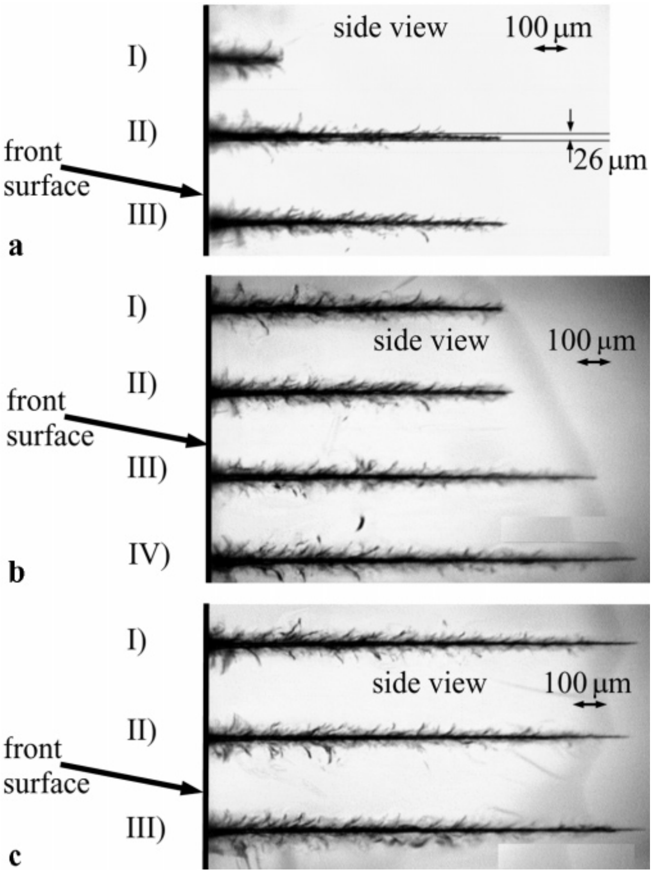

Fig. 10a–c. Channels produced in quartz with 532 nm, 30 ps laser pulses, pulse energy 450 µJ, under vacuum conditions for different numbers of laser shots. **a** I: 100, II and III: 500, **b** I and II: 500, III and IV: 1000, **c** I: 5000, II and III: 10000

When the laser pulse length is increased to 5 ns the holes produced are much less reproducible for given laser and pressure conditions. Most irradiated samples broke apart rather spectacularly during the experiments so that only a very few channels could be analysed in the optical microscope. Some examples can be seen in Fig. 11. It is possible to produce channels in quartz with visible ns pulses but these are much wider than those produced with fs and ps pulses even although the focus area is smaller than for the experiments with the Ti:sapphire laser. It should also be stressed here again that these examples shown in Fig. 11 are the rare exception to the rule that the sample splinters and breaks up with ns ablation at 532 nm. The pictures in Fig. 11a and b suggest that at high laser pulse energies (3 mJ) one first produces an irregular path through the material which is then “smoothed out” by subsequent pulses. Unfortunately these two channels were the only ones of many to survive for this high pulse energy so that we cannot presently prove this supposition. The example for ab-

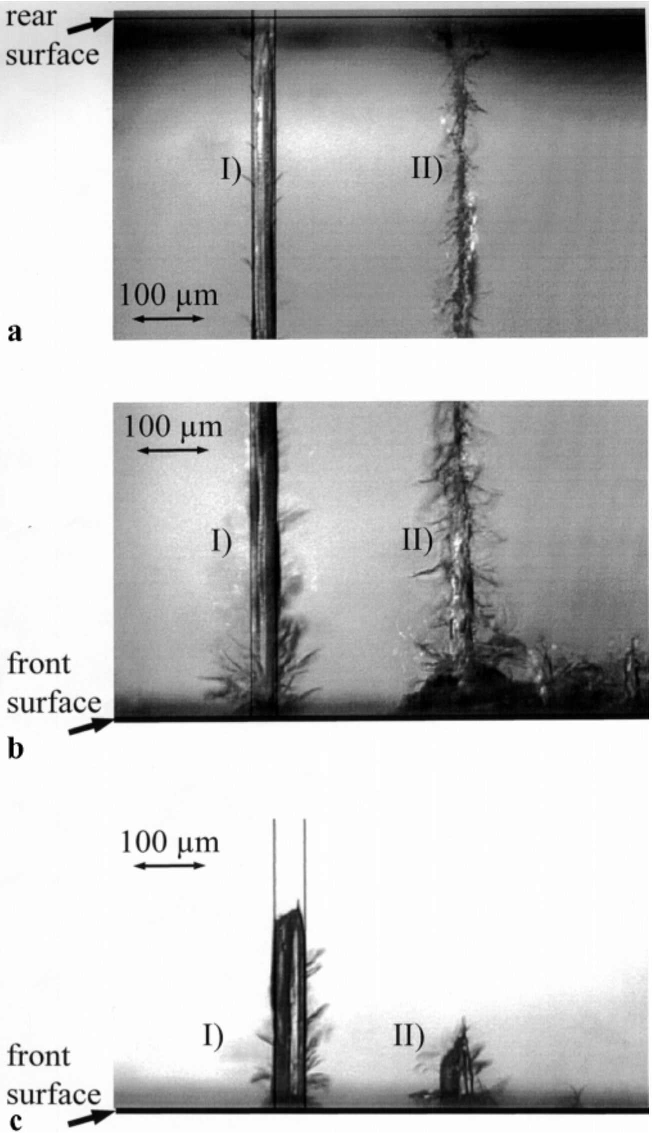

Fig. 11a–c. Channels produced in quartz with 532 nm, 5 ns laser pulses, vacuum conditions. **a** and **b** 3 mJ pulse energy, I: 10 000 shots, II: 5000 shots; **c** 2 mJ pulse energy, I: 10 000 shots, II: 5000 shots

lation with a lower pulse energy of 2 mJ (Fig. 11c) does not seem to show this effect.

There are some earlier studies of comparison of laser ablation of quartz with ns and 500 fs pulses at UV wavelengths (excimer lasers) available in the literature [15, 16]. For these studies the laser beam was not focused directly onto the sample surface but mask imaging was used to produce shallow holes with diameters on the order of 100–400 μm, depending on the wavelength. The quality of the structuring was seen to improve on going from 308 nm to 193 nm with ns pulses. Considerable damage around the hole could be seen with 308 nm and 248 nm pulses with the best results being obtained for ablation at the rear side of the sample with low intensity 193 nm pulses. The production of deep holes was unfortunately only shown for 308 nm [16] where the quality of ablation was considerably worse than that reported in the present paper for fs and ps pulses. Only very shallow “pockets” were shown for ablation with 500 fs pulses at 248 nm. More recent work has shown that it is possible to produce long narrow channels with 500 fs, 248 nm ablation in quartz [17]. However, the work we present in this paper shows that it is not necessary to resort to UV lasers with all the problems associated with working at these wavelengths and with the inherently poor quality of excimer laser beams. The important parameters for determining the quality of the ablation of quartz are the laser pulse length and the laser fluence. The former allows for multiphoton excitation of electrons into the conduction band followed by extensive heating and material removal (see below) and also helps avoid the build up of thermal stress and cracking which occurs with ns pulses. The latter determines the amount of material that can be removed under given laser focus and pressure conditions.

The production of long narrow channels with high aspect ratio has been reported for a number of other materials. For example, it is possible to obtain such structures in ductile materials such as metals by using a high-intensity copper vapour laser (511/578 nm) with 50 ns pulses [18] or a Ti:sapphire laser with 200 fs pulses [19]. A deep narrow hole can be generated when the laser fluence is sufficiently high to ablate by evaporation over the cross section of the beam. The constant diameter of the hole can be attributed to multiple reflection of the light at the channel walls. Detailed electron microscope studies of the holes produced in both cases [20] have shown the deposition of material along the walls of the channel and provided evidence for strong mechanically and thermally induced changes in the material around the channel extending for a distance of a few μm into the bulk. Similar channelling effects have also been observed in silicon and germanium with 100 fs at 248 nm [10] although in this case the effects were attributed to self-structuring (as evidenced by the formation of columnar structures for a small number of laser shots) and non-thermal ejection of material.

The ablation of channels in brittle materials like quartz using high intensity copper vapour ns lasers, which can give reasonably good results with ductile materials as discussed above, fails due to the large amount of cracking or spallation which occurs [18], as we also observed in our ns experiments (Fig. 11). There is, thus, a very large advantage to be gained in using ultrashort laser pulses to ablate brittle materials since the cracking, or spallation, effects can be reduced considerably under these conditions and the material behaves almost as if it were ductile.

We interpret our results as being due to multiple reflection of the light at the channel walls, as invoked to explain the results with metals [18, 19]. Multiphoton absorption at the high intensities used to produce the channels will lead to an extremely rapid build-up of electrons in the conduction band of the material during the rise time of the ultrashort pulses. The material will thus show metal-like behaviour such as a high reflectivity for the subsequent part of the laser pulse. However, the strong dependence of the channel diameter on the exact focus conditions still remains to be understood. The independence of the channel diameter on laser pulse length in the range 120 fs - 2.8 ps suggests that we do not have to invoke non-linear self-channelling effects to explain the experimental results. The dependence of the maximum obtainable hole depth on laser fluence and independence on laser pulse length (Fig. 7) is at first sight rather surprising but may simply indicate that the important factor is the energy input into the material. This will determine the temperature rise and thus the kinetic energy of the ablated particles. The particles will require a certain energy in order to escape from the channel which will increase with increasing channel length.

3 Conclusion

We have shown that it is possible to produce well-defined and highly reproducible channels of a few micrometres diameter and lengths of over 1 mm in quartz with laser pulses of 790 nm wavelength (Ti:sapphire) and pulse lengths of 100–200 fs. The channel depth can be controlled by the laser fluence and number of laser shots. Comparisons were made with laser pulse lengths of 1.3 ps, 2.8 ps, 30 ps, and 5 ns. The main parameter influencing the quality of the ablation is the laser pulse length, which should be sufficiently short in order to produce a multiphoton excitation of electrons into the conduction band during the rise time of the laser pulse. This induces a metal-like behaviour in the quartz and the formation of narrow channels with constant diameter due to multiple reflections at the channel walls. An additional advantage of ultrashort laser pulses is that the laser pulse length is short compared to the time scale for thermal diffusion into the material. In this way the build-up of thermal and mechanical stresses in the material, and thus cracking and breaking which normally occurs for brittle substances like quartz, can be avoided to a large extent.

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