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Ablative processing of fine features in brittle materials with ultrashort laser pulses

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Abstract

Laser processing of transparent brittle materials has proven to be an attractive alternative to conventional mechanical methods due to increases in quality and yield, while also allowing the processing of difficult feature geometries. Here we present a summary of recent experiments aimed at optimizing laser ablation drilling of relatively high aspect ratio (>1:1) features in brittle materials, focusing on sapphire. We present results for laser drilling of holes in sapphire wafers with both 1030nm and 515nm sub-picosecond laser sources. We determine the optimum process window as a function of wavelength, pulse energy, repetition rate, overlap, and beam waist / z-axis translation speed. In ambient air, we find that ~400µm diameter holes in 430µm thick sapphire wafers (aspect ratio depth:diameter of ~1:1) can be drilled with average taper angles of ~4° in <3 seconds, and ~2° in ~12 seconds. We determine broad ranges of process parameters that yield high-quality holes for both 1030nm and 515nm, while also identifying important process limitations. We identify the recast of sticky particulates generated during the ablation process as the main detriment towards the drilling of zero-taper holes in sapphire. We present results for reducing recast with liquid-assisted drilling, which we observe to efficiently reduce redeposition of ablated material along the hole sidewall, allowing for the drilling of holes with <2° taper in ~5 seconds. Lastly, we explore the ability to minimize drilled hole diameters in sapphire by applying the totality of the general knowledge learned throughout these studies, demonstrating the ability to drill holes with aspect ratios of up to ~10:1.

Keywords: laser ablation; through holes; femtosecond laser; transparent media; aluminum oxide

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1. Introduction

Laser-based processing of transparent brittle materials such as sapphire and glass has remained a hot topic throughout a myriad of industries in recent years. Lasers enable non-contact processing of these brittle materials, simultaneously allowing high throughput and excellent quality [Ashkenasi et al., 1997; Baier, 2014]. Ablative methods are a very attractive option for drilling of transparent brittle materials, but have been supplanted for cutting and dicing by a number of state-of-the-art spatial and/or temporal beam shaping techniques such as filamentation [Butkus et al., 2015], Bessel beams [Courvoisier et al., 2013], and controlled crack propagation [Matylitsky et al., 2016]. These techniques have demonstrated the ability to facilitate single-pass, zero-kerf cutting and dicing of materials with up to millimeter or greater thickness. However, the generation of fine internal features, including those on the order of tens of microns to a millimeter that are common in recent smart phone and smart watch devices, is often best-served-served by ablative processing. In contrast to some other impressive multi-step processes that have recently been reported for generating fine features in transparent brittle materials [Bellouard et al., 2012; Gottmann et al., 2013], this one-step laser ablation process is compliant with the REACH regulations of the European commission for avoiding hazardous materials and reducing chemical waste.

Here, we summarize recent studies and new results exploring ablative drilling of transparent materials with sub-picosecond laser pulses, primarily focused on sapphire. Sapphire is of particular interest as a cover glass in consumer electronics and luxury watch markets due to its exceptional hardness (9.0 on Mohs scale, third hardest known natural substance), scratch resistance, and optical transparency [Dobrovinskaya et al., 2009].

2. Methodology

2.1. Experimental apparatus and procedure

Experiments presented here have been performed with lasers from the Chinook product line by Eolite Lasers. The Chinook products are sub-picosecond lasers (typical specifications of <800fs at 1030nm and <700fs and 515nm) that output linearly polarized beams with Gaussian power distribution and $M^2 < 1.2$.

Previous results are available in greater detail in Lott et al., 2015 and Lott et al., 2016. New studies in this communication at 1030nm (IR) were performed with a Chinook HE (high energy) laser, with a maximum on-sample pulse energy of 80 μ J (specification of 100 μ J at laser output) and a maximum repetition rate of 500kHz. Experiments at 515nm (GR) were performed with the same laser frequency doubled through an external conversion module, resulting in a maximum on-sample pulse energy of 33 μ J (specification of 50 μ J out of Chinook HE GR laser). The beam is delivered to the work surface by scanning galvanometers (Scanlabs hurrySCAN 20 for IR, Scanlabs hurrySCAN III 14 for GR) and 100mm focal length f-theta lenses to generate beam waists of 18.5 μ m and 12.5 μ m for IR and GR, respectively. With these values, the maximum peak fluence values are similar at 55.8J/cm² (IR) and 53.8J/cm² (GR). The beam is converted to circular polarization with a $\lambda/4$ waveplate prior to the galvanometer in all experiments.

We employ a bottom-up processing method in our efforts to generate (near) zero-taper holes in sapphire. This method has been demonstrated successfully on a variety of transparent materials by other research groups [Du, 2003; Zhao et al., 2011]. Our implementation of this technique, the processing pattern and strategy utilized, and experimental limitations are described in full in Lott et al., 2015 and Lott et al., 2016. Briefly, we drill \sim 400 μ m diameter holes in sapphire as a function of repetition rate, overlap, z-axis translation speed, and pulse energy. For experiments with a water bath, the bottom surface of the sapphire wafer is placed in direct contact with the water reservoir and bottom-up processing studies are performed in an

identical manner as experiments in ambient air. With the general knowledge obtained from these results, we demonstrate the capability of the process to be modified for drilling much smaller diameter holes (down to <50 μm).

2.2. Materials

Dual-polished, 400-430 μm thick, 50.8mm diameter, c-plane sapphire wafers were used in all tests. Note that the effective thickness of these wafers is $\sim 240\mu\text{m}$, which is the distance that the laser beam waist must be translated along the z-axis to move from the bottom surface of the wafer to the top surface, or vice versa. This distance is equal to physical thickness of the wafer divided by the index of refraction of sapphire ($n = \sim 1.77$ at 515nm, ~ 1.75 at 1030nm).

2.3. Analysis

The drilled holes are analyzed along two orthogonal lines with a laser scanning microscope (Keyence VK-9700 & VK9710). The maximum (outer) and minimum (inner) diameters along each line are determined, and these values are used to calculate the average taper angle, θ , of each hole with the equation:

$$\theta = \tan^{-1} \left(\frac{D_M - D_m}{2h} \right), \quad (1)$$

Where D_M , D_m , and h are the maximum hole diameter, minimum hole diameter, and wafer thickness, respectively. Error bars are determined from the differences in taper calculated from the two orthogonal measurements. Holes are also qualitatively analyzed for cracking and chipping.

Table 1. Summary of laser and process parameters.

	Chinook IR [Lott et al., 2015]	Chinook HE GR [Lott et al., 2016]	Chinook HE IR
Max on-sample pulse energy used	26.4 μJ	33.0 μJ	75.0 μJ
Beam waist	18 μm	12.5	18.5 μm
Maximum peak fluence	20.7J/cm ²	53.8J/cm ²	55.8J/cm ²
Repetition rates tested	21, 104, 260, 521, 1042kHz	20, 100, 250, 500kHz	100, 250, 500kHz
Overlap (not all values available for all repetition rates)	70 – 98%	50 – 98%	70 – 98%
z-axis translation speed	10 to $\geq 100\mu\text{m/s}$	10 to $\geq 100\mu\text{m/s}$	10 to $\geq 100\mu\text{m/s}$

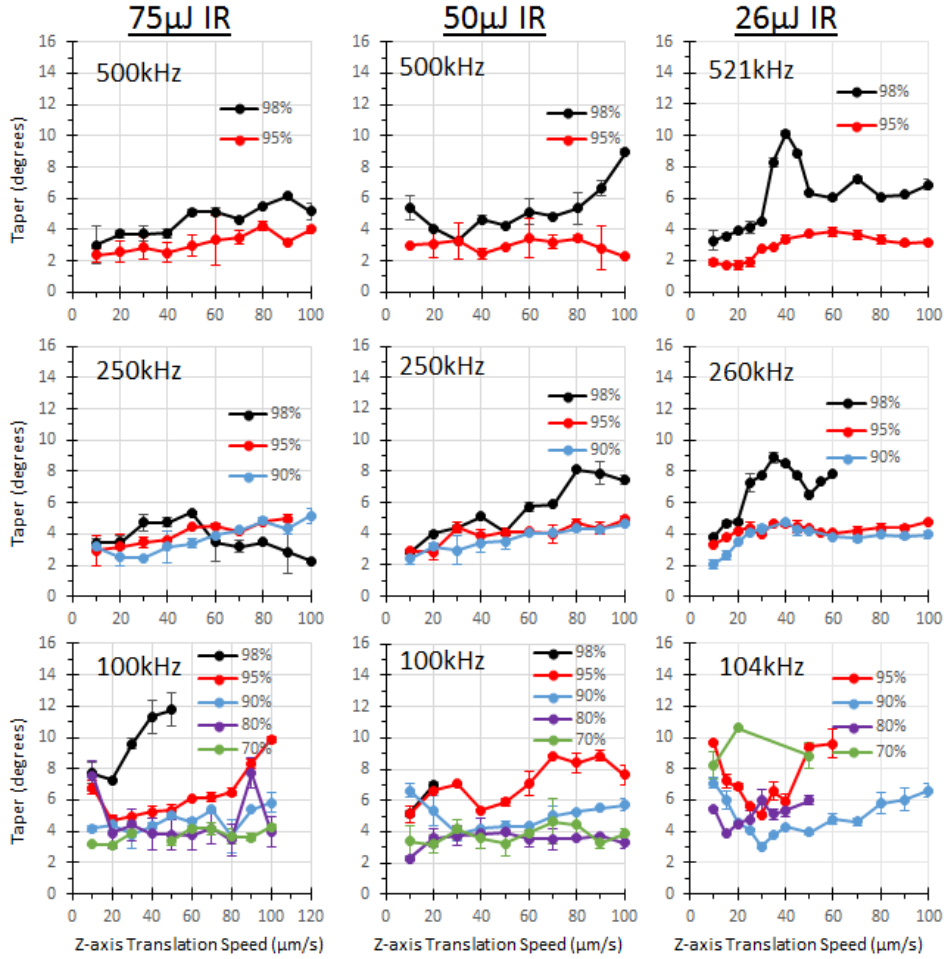


Fig. 1. Average taper vs. z-axis translation speed for 400 μm diameter holes in 430 μm thick sapphire wafers, drilled with sub-picosecond IR pulses. Pulse energies correspond to fluences of 55.8J/cm² (left), 37.2J/cm² (center) and 19.6J/cm² (right). Overlap conditions are restricted by maximum galvo scanning speed for the 400 μm pattern; all available overlaps are shown in each individual plot.

3. Results and discussion

3.1. Summary of previous studies in air

Here and in previous work, we have performed studies to optimize the drilling process for the generation of $\sim 400\mu\text{m}$ diameter holes in 430 μm sapphire wafers with a standard Chinook IR laser [Lott et al., 2015], with a Chinook HE GR laser [Lott et al., 2016], and with a Chinook HE IR laser. Holes are drilled as functions of wavelength, laser repetition rate, overlap, and z-axis translation speed. A summary of these laser and process parameters is contained in Table 1. We choose to drill holes with an aspect ratio of approximately 1:1 in order to observe a parameter space that is neither extremely narrow nor extremely broad, as are to be

expected for holes with significantly higher or lower aspect ratios, respectively. It is important to note that while the scanning galvanometers used in these studies have straight line processing speeds of $>10\text{m/s}$, their maximum speeds are greatly restricted by the small dimensions of the $400\mu\text{m}$ diameter pattern ($<800\text{mm/s}$ IR, $<1000\text{mm/s}$ GR). As consequences of this limitation, as the repetition rate is increased the number of available overlap conditions decreases, and as the pattern diameter is decreased the maximum scanning speed is further restricted.

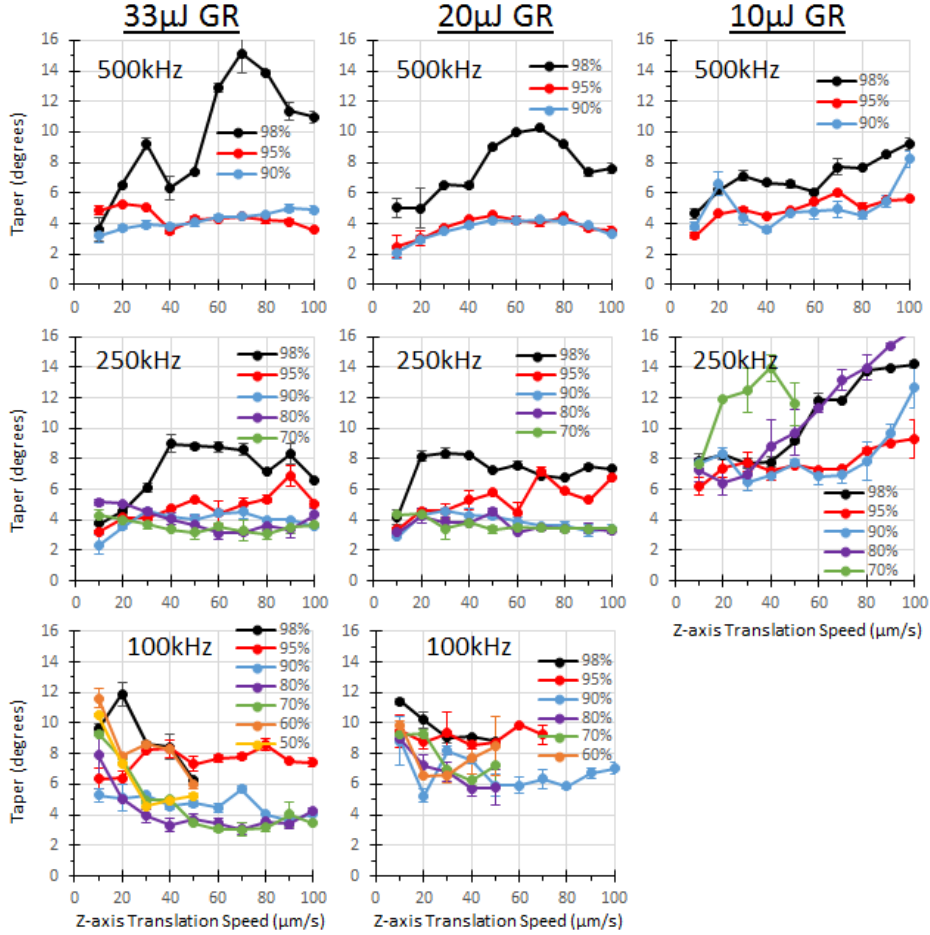


Fig. 2. Average taper vs. z-axis translation speed for $400\mu\text{m}$ diameter holes in $430\mu\text{m}$ thick sapphire wafers, drilled with sub-picosecond GR pulses. Pulse energies correspond to fluences of $53.8\text{J}/\text{cm}^2$ (left), $32.6\text{J}/\text{cm}^2$ (center) and $16.3\text{J}/\text{cm}^2$ (right). Overlap conditions are restricted by maximum galvo scanning speed for the $400\mu\text{m}$ pattern; all available overlaps are shown in each individual plot.

At each available overlap for each repetition rate, we translate the beam waist along the z-axis at speeds from $10\mu\text{m/s}$ to $100\mu\text{m/s}$ or greater unless significant and regular damage is observed at lower processing speeds, in which case the data set is truncated. Taper values for these data series are calculated with Equation 1. In Figure 1 and Figure 2 we present summary plots of IR and GR experiments, respectively. Each individual plot in the array shows the measured taper as a function of z-axis translation speed (up to $100\mu\text{m/s}$) for the overlap conditions available at specific pulse energy and repetition rate combinations.

We are able to draw a number of general conclusions from these results. It is readily apparent that no sets of process parameters for either IR or GR demonstrate the capability to drill true zero-taper holes. While the bottom-up ablation process has demonstrated the capability to generate zero-taper or near zero-taper holes in other transparent materials, the generation and redeposition of dense, molten particulates along the inner sidewall during processing inhibits this for sapphire. Post-processing by ultrasonication and potassium hydroxide baths were ineffective at removing the vast majority of the recast material. For both IR and GR we find that the lowest taper values, typically around 2° , are observed towards the slower end of the z-axis translation speeds. It is reasonable to expect that even lower taper values are achievable at slower z-axis translation speeds than $10\mu\text{m/s}$, but the throughput quickly becomes unreasonable for industrial applications since the cycle time per hole is inversely proportional to the z-axis translation speed.

One of the most important conclusions for the ablative processing of sapphire is that thermal accumulation plays a critical role in the generation of high-quality holes. Under the wrong conditions, hole quality is poor, cracking is prevalent, and the progression of taper as a function of z-axis translation speed is erratic and relatively unstructured, as is observed for all results at 100kHz (IR and GR) and at 250kHz with $10\mu\text{J}$ GR pulse energy. In these plots, only 34% of all holes drilled do not exhibit unacceptable levels of damage. We can attribute these poor results to the decreased dose (total pulse energy per unit length) per unit time in these data sets. This is evident in the decreased pulse energy for $10\mu\text{J}$ GR, and also at lower repetition rates (20kHz, results not shown; 100kHz) where both slower scanning speeds required by the reduced repetition rates and the reduced interpulse separation itself contribute to the effect of an 'underdosed' process.

In contrast, with the right selection of process parameters heat accumulation generates a smooth spatiotemporal thermal gradient that stabilizes the laser ablation process. This is true, for example, for the majority of results for all overlap conditions at 250kHz and 500kHz for both $50\mu\text{J}$ and $75\mu\text{J}$ of IR pulse energy. In these data sets, we observe a general progression of taper values that is very similar between all four data sets, with taper values increasing from $\sim 2\text{-}3^\circ$ at $10\mu\text{m/s}$ z-axis translation speed to $\sim 5\text{-}6^\circ$ at $100\mu\text{m/s}$ z-axis translation speed. Nearly all (98%) of the holes drilled in these conditions were drilled without resulting in any visible cracks or any chips greater than a few microns in size. Similar trends are often present for overlap values of 95% and/or 90% in numerous conditions (e.g. $26\mu\text{J}$ IR at 521kHz and 260kHz, $33\mu\text{J}$ GR at 500kHz and 250kHz, etc.). In these same pulse energy and repetition rate combinations, the 98% overlap results often deviate significantly from the 95% and 90% overlap results, demonstrating higher average taper values and an increased likelihood of cracking or chipping, possibly due to processes that approach the threshold for 'overdosing' the sample.



Fig. 3. Examples of damage ring (left hand side) and splash ring (right hand side) on bottom side of sapphire wafers after processing.

All holes are drilled with the bottom-up experimental configuration, but not all holes end up being drilled in a purely bottom-up manner. As the z-axis translation speed is increased there eventually comes a value for each particular set of process parameters for which the beam waist is translated faster than it can remove material or maintain sufficient incubation and heat accumulation effects. When this occurs, the process switches from purely bottom-up drilling to a hybrid bottom-up/top-down drilling process. As the z-axis translation speed continues to increase above this value, a larger and larger portion of the hole is drilled with a top-down process. This transition from bottom-up to hybrid drilling process is visible in some of the data sets, such as the 26 μ J IR results in Figure 1 for 95% and 90% overlap at 521kHz and 260kHz. As a general rule, we have observed that both drilling speed and resulting quality benefit from a purely bottom-up process, which will be discussed in greater detail in the following paragraph. In these data sets, we observe increasing taper as the z-axis translation speed is increased from 10 μ m/s to an inflection point (40 μ m/s-60 μ m/s in these examples), at which the average taper decreases before increasing roughly linearly as the z-axis translation speed is increased further (this is more clearly visible in extended plots available in Lott et al., 2015). This is visible in profilometry measurements, where the differentiation between purely bottom-up, purely top-down, and hybrid processes can be determined by the curvature of the sidewalls – purely bottom-up processes have convex sidewalls due to the recast, purely top-down processes are concave, and hybrid processes combine concave and convex to varying degrees. The switch to a hybrid process is also visible by eye during experimentation when the plasma becomes visible on the top surface of the wafer. In the examples above, we can see that this transition occurs at a higher z-axis translation speed for the 521kHz results than the 260kHz results due to an enhancement of the thermal accumulation effects during processing at higher repetition rate. This enhancement is also evident as a function of pulse energy. The pulse energy dependency is observed by eye for 500kHz GR (521kHz & 500kHz IR) results, where the transition from purely bottom-up to hybrid processing increases from \sim 60 μ m/s at 10 μ J (26 μ J) to \sim 80 μ m/s at 20 μ J (50 μ J) and to \sim 100 μ m/s at 33 μ J (75 μ J).

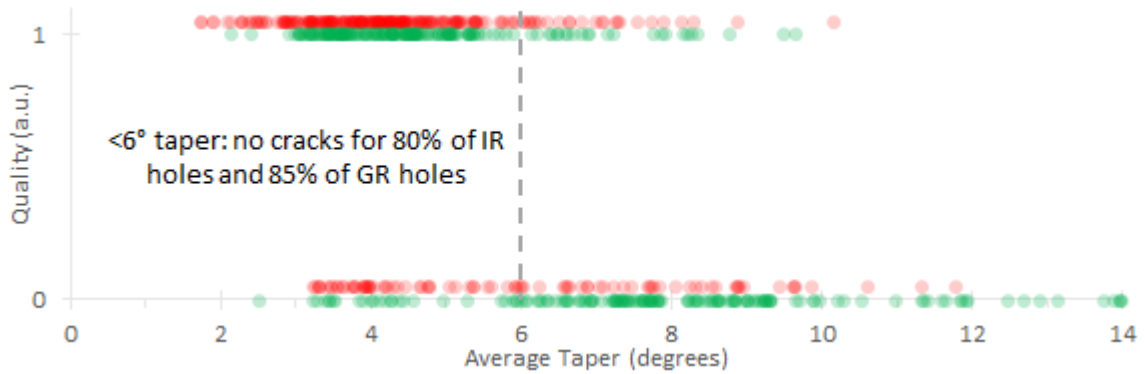


Fig. 4. Hole quality vs. taper angle for all IR and GR holes from Fig. 2 & Fig. 3. Holes are attributed a Quality value of “1” if they do not exhibit cracking or significant chipping, and a value of “0” if there is any noticeable cracking or chipping.

Top-down drilling, and therefore the hybrid drilling process, usually leads to larger sidewall taper angle, increased likelihood of cracking, and the possibility of backside damage rings. These damage rings, an example of which is shown in the left hand side of Figure 3, are caused by filaments generated at the top edge of a hole drilled in transparent material [Schulz, 2014]. These filaments propagate through the entire

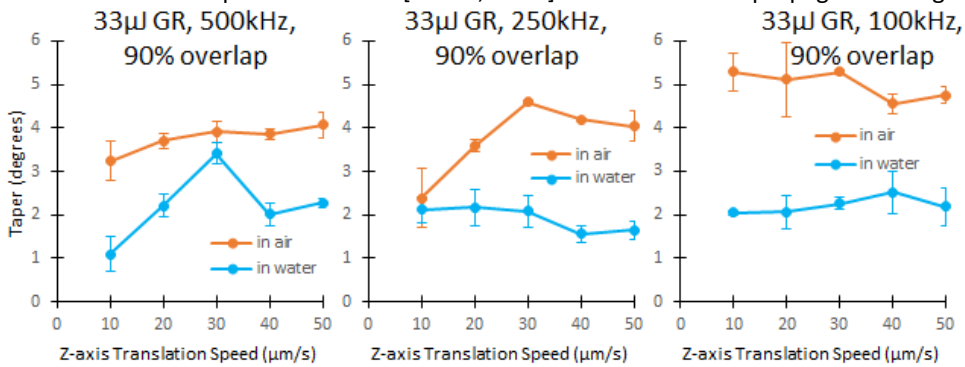


Fig. 5. Comparison of average taper vs. z-axis translation speed for holes drilled with Chinook HE GR laser in air (orange) and in water (blue) for selected processes. Taper angle is decreased with water-assisted drilling for all processes shown.

thickness of the bulk material before damaging the bottom surface of the substrate. Therefore, the damage rings are not only a qualitative issue, they are also an indicator of bulk material modification throughout the sapphire wafer. We have observed that these damage rings occur significantly more often for holes drilled with IR than with GR, and that the likelihood of damage ring formation actually decreases as the pulse energy / fluence is increased.

In addition to these damage rings caused by filamentation at the top surface, we observe a similar qualitative phenomenon on holes drilled at the highest fluence, repetition rate, and overlap combinations in IR, shown in the right hand side of Figure 3. Interestingly, this ‘splash ring’ effect is only observed at the slowest z-axis translation speeds, $\leq 30 \mu\text{m/s}$. We hypothesize that the debris generated in these conditions may be hotter and more liquid-like than the debris generated with less aggressive process parameters,

cooling more slowly and damaging the bottom surface of the wafer when it's ejected from the hole. We have not observed this phenomenon with GR processes at similar fluences.

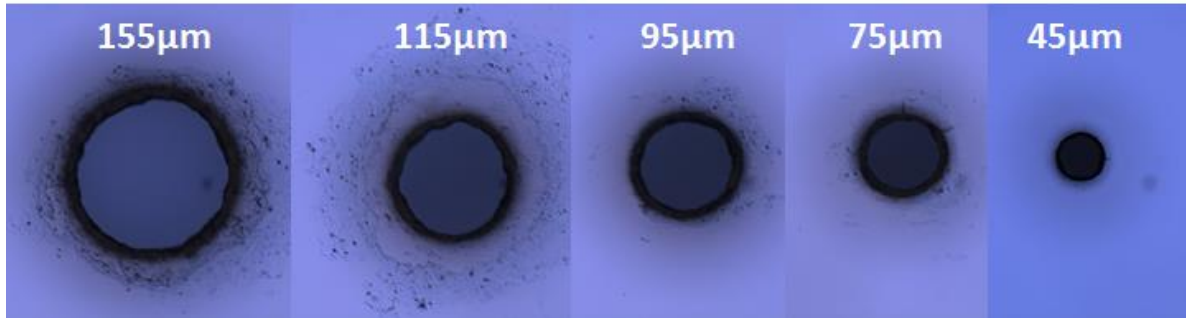


Fig. 6. Sequence of holes with reducing diameter / increasing aspect ratio, using water-assisted drilling with IR laser.

To summarize, we have determined that there is a broad, but well-defined process window for drilling high-quality 400µm diameter holes in 430µm thick sapphire wafers for both IR and GR sources. The process windows are bounded on both sides by processes which are either too aggressive or not aggressive enough. There is no significant advantage to either wavelength in terms of cycle time for holes drilled with similar fluences. Qualitatively, we observe fewer instances of damage and splash rings for holes drilled with GR than with IR, but we do not see appreciable differences in minimum achievable taper values as a function of z-axis translation speed (and therefore cycle time). We have determined that we can drill high-quality 400µm diameter holes in 430µm thick sapphire wafers with $\sim 4^\circ$ of taper in ~ 2.5 seconds (z-axis translation speed of 100µm/s and a purely bottom-up process), and holes with taper of $\sim 2-2.5^\circ$ in 12-25 seconds (z-axis translation speeds of 10-20µm/s). Finally, we do not find a reduction in hole taper by increasing pulse energy or fluence – the hole taper is minimized at $\sim 2^\circ$, and in fact new damage mechanisms are observed when the dose per unit time becomes too large, limiting the utility of very high pulse energy for this application. Increasing the pulse energy may result in decreased cycle time by maintaining the bottom-up process to a higher z-axis translation speed, but the average taper value appears to be increasing rather independent of pulse energy and therefore higher taper values are likely at lower cycle times. We are able to determine a natural separation between holes with and without cracking or chipping as a function of taper angle, as shown in Figure 4. Holes without cracking or significant chipping are given a Quality value of “1”, and holes with cracking or chipping are given a Quality value of “0”. We can see that if we separate holes at a taper value of 6° , there is a clear separation between “good” and “bad” processes – holes that are drilled with a taper angle of less than 6° are without cracking or chipping for 80% of IR holes and for 85% of GR holes. These ‘good’ holes with $<6^\circ$ taper correspond to 89% of all good IR processes and 86% of all good GR processes. Accordingly, the vast majority of processes that result in holes with $>6^\circ$ taper result in significant chipping and/or cracking.

3.2. Utilizing water-assisted processing for taper reduction

We have identified the recast of molten sapphire particulates along the inner sidewall of drilled holes as the main inhibiting factor towards the generation of zero-taper holes. Previous work by other research groups has demonstrated that liquid-assisted drilling enables the significant enhancement of microchannel drilling depth by placing the bottom surface of transparent media in contact with a water bath [Zhao et al., 2011]. Microchannel depth and sidewall quality were both improved by this technique due to water drawn

into the hole during processing via the capillary effect, facilitating more efficient removal of potential recast material during processing.

We tested the suitability of this technique for reducing hole taper in the 400 μm diameter holes by placing the bottom surface of the sapphire wafers in direct contact with a water reservoir for laser processing. We performed comprehensive tests with 33 μJ GR and 90% overlap at 500kHz, 250kHz, and 100kHz. In air, these parameter sets yield high-quality holes that are generally free of significant cracking, and demonstrate the standard progression of increasing taper angle as the z-axis translation speed is increased. As shown in Figure 5, this technique is extremely effective at reducing recast during processing. Taper is decreased across the entire range from 10 $\mu\text{m/s}$ to 50 $\mu\text{m/s}$, and the taper becomes relatively independent of the z-axis translation speed; in other words, there is no longer a tradeoff between cycle time and taper in this process window. At z-axis translation speeds above 50 $\mu\text{m/s}$, the process becomes unstable and a notable decrease in quality is observed. The water bath also acts as a heat sink, altering the thermal accumulation in comparison to in air and reducing the maximum translation speed for which the purely bottom-up process can be maintained. The plots in Figure 5 show that we are able to drill holes with $\sim 2^\circ$ of taper in ~ 5 seconds by placing the sapphire wafer in contact with a water bath, a cycle time reduction of $>50\%$ in comparison to in air. We have performed similar tests with IR processes with the wafer in contact with the water bath and the results are identical.

3.3. Increasing aspect ratio towards 10:1

Having established a set of general guidelines for drilling high-quality holes with an aspect ratio of $\sim 1:1$ in 430 μm thick sapphire wafers, we have applied this knowledge towards generating holes with higher aspect ratios. We have modified the pattern dimensions and parameters to drill holes with successively smaller diameters, all of which are drilled with the back surface of the sapphire wafer in contact with a water bath. A selection of these results are presented in Figure 6. These holes were all drilled with IR, and process parameters were scaled to account for different pattern sizes and galvanometer restrictions. The top surface of the holes are shown here; the bottom surfaces exhibit nearly identical quality. There is residue visible around the edges of these holes that is generated in the short time period between completion of the hole drilling and stopping the drilling process. This residue is easily removed with post processing, unlike molten sapphire particulates that occur during processing in air. As the hole diameter is reduced to smaller and smaller sizes, it becomes more difficult to drill holes without generating small cracks, as are most visible in the 75 μm diameter result, but are also present but less visible in the 95 μm and 45 μm diameter holes. Results from tests performed with GR are nearly identical. With these holes being drilled while in contact with a water bath, they face similar restrictions in maximum z-axis translation speeds as in the previous section. The best results, such as those shown in Figure 6, have been observed for z-axis translation speeds of 10-30 $\mu\text{m/s}$, and up to 50 $\mu\text{m/s}$ for the larger diameter holes in the sequence.

4. Conclusions

We have demonstrated the ability to drill high-quality holes with an aspect ratio of $\sim 1:1$ in 430 μm thick sapphire wafers across a broad spectrum of process parameters in IR and GR. Minimum taper in air is limited to 2° for both IR and GR due to the recast of ablated material along the inner sidewall during processing. We find that there is no appreciable difference in quality achievable between IR and GR, and increased pulse energy does not decrease the taper of an otherwise suitable process with lower pulse energy.

Utilization of a water bath during processing allows for a reduction of taper, as well as lower taper at higher z-axis translation speeds, effectively uncoupling taper from drilling time per hole (limited by cracking

at higher speeds). By combining the general rules from comprehensive studies in air with the liquid-assisted processing results, we increase the aspect ratio of drilling holes in sapphire to ~10:1 while retaining very good quality.

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