Surface morphology and fracture strength analysis of nanosecond ablated alumina

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Although the research on laser ablation advances steadily, mainly on ultra-short pulses (in the order of pico- and femtoseconds), research on the low-cost and low-power-consuming nanosecond regime (>100 ns) is relatively scarce. This process is still difficult to predict, due to the many simultaneous and interacting physical processes that take place in a relatively short time. This study provides an experimental analysis for a ytterbium pulsed fibre-laser on Al2O3, by evaluating surface morphology and fracture strength for two sets of parameters. A very well-defined difference in removal rate and resulting surface topographies was observed, suggesting a threshold point between distinct ablation mechanisms. Laser machining also caused a clear increase on the Weibull modulus, while reducing the characteristic stress.

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1. Introduction

Pulsed lasers are currently widely used, in applications that go from fundamental research, materials processing, production of nanoparticles and chemical analysis techniques to medical procedures and art restoration.1) Nanosecond infrared lasers have an economic advantage due to their low cost and maintenance requirements compared with pico- and femtosecond pulsed lasers.2),3) Laser ablation is the process by which material is removed from a surface using high-intensity laser pulses. It takes place by pyrolytic processes, by which the beam energy is absorbed by the materials electrons and converted into heat, leading to melting and vaporization; and by photolytic processes, which the photons’ energies are high enough to overcome the chemical bonds, avoiding thermal effects. The latter is usually associated with ultra-short pulses, since the photon density is much higher for the same average power output, favouring multiphoton absorption. Pulse lengths are classified as ‘ultra-short’ when the distance by which heat diffuses during the irradiation is smaller than the optical penetration depth; and as short, otherwise. For ceramics, ultra-short pulses are typically shorter than 10 ps.1)

In nanosecond ablation, the irradiated energy is absorbed by the solid as heat, that leads to melting or sublimation, and further to vaporization and/or dissociation. The vapour plume above the working spot also absorbs beam energy and becomes ionized, producing a plasma that further absorbs and scatters the beam and change the flux received by the surface.4) These processes occur only above a certain fluence (pulse energy per focus spot area) threshold, that is related to the amount of energy needed to overcome the latent heat of evaporation in the working spot.3)

Since ablation is a complex process governed by many interacting physical phenomena, that depend on properties that evolve during the process, it is difficult to directly translate process parameters into desired effects such as removal depth and surface roughness or morphology.5) Studies have been carried out regarding parameters optimization,7),10) and numerical models for thermal processes5),13),14) and plasma dynamics.3),15),17) Samant and Dahotre8) developed a comprehensive model for one-dimensional machining of ceramics, being able to predict removed depth from laser parameters and material properties; Vora et al.19) modeled the evolution of surface quality. Three-dimensional machining, however, is not fully developed and still has limited industrial use,20),21) therefore further work is required. Besides that, there are no quantitative studies on the effect of laser-ablative processes on the mechanical properties of ceramics.

In this context, the present study reports an experimental analysis of three-dimensional machining of alumina through ablation with a nanosecond pulsed laser, evaluating its practical feasibility regarding ablation rate, surface quality and loss of strength, and providing data for future numerical models.

2. Experimental

Experiments were carried out using a pulsed ytterbium fibre-laser (wavelength λ = 1,06 μm) with 200 W maximal average output power, using focal distance of 163 mm and nominal pulse width of 120 ns. Dense (3,96 g/cm³) by Archimedes method) alumina bars were fabricated by uniaxial and isostatic pressing with 100 and 400 MPa, respectively; followed by sintering at 1350°C for 1 h, with heating and cooling rates of 10°C/min, giving final dimensions of approximately 6 × 4 × 36 mm. The broader surfaces were ground to ensure parallelism, and one of them was polished down to a grit size of 0,25 μm. For the four-point bending test, samples were cut down to 2 × 2,5 × 36 mm and the side surfaces were also ground, according to.22) The main parameters in the present three-dimensional laser machining system are a) the pumping current, which is related to the pulse energy; b) the frequency or repetition rate, which together with the pumping current defines the average power output and the beam’s fluence; c) the track distance (S), which is
the spacing between each of the lines that forms the desired bi-dimensional feature on the samples surface; and d) the scan speed \(V_s\), the speed at which the beam advances to ablate each track. The number of layers also plays an important role, since they determine the final depth, but they have been kept out of the present study in order to avoid the complexity introduced by multi-pulse enhancement,\(^{33}\) which are beyond the proposed scope.

A confocal laser scanning microscope (CLSM) was used to measure the sizes and surface profile of the ablated features, and morphologies were investigated using optical- and scanning electron microscopy (SEM).

The different experiments were named as XX\(\Phi\)YY-XX is the experiment identification and YY is the fluence in J/cm\(^2\)-e.g. TDD\(\Phi\)30 refers to the set of the track distance (TD) evaluation with fluence of 30\(J/cm^2\).

### 2.1 Fluence threshold

Because the two parameters pump current and frequency define the laser’s average output power, and based on previous works that recommend fluences close to the threshold in order to minimize the deposited energy and hence the heat affected zone,\(^ {5\,1\,3,\,14}\) the following steps were used to estimate the threshold fluence. Noting that the ablated spot corresponds to the regions of the focus spot where the intensity is higher than the threshold, the beam’s Gaussian intensity profile can be rewritten as:

\[
I_0 = I_\text{th}\exp\left(-2r^2/w_0^2\right)
\]

where \(I_0\) is the intensity threshold, \(I_\text{th}\) is the maximal intensity value at the centre of the beam, \(r\) is the ablated radius and \(w_0\) is the focus spot radius. Now rearranging Eq. (1) for the ablated diameter (\(D_a\)), which can be taken as an ablated line’s width, and substituting the intensity by the equivalent peak power (\(P_\text{th}\)), one comes to:

\[
D_a^2 = 2w_0^2[\ln(P_\text{th}) - \ln(P_\text{th})]
\]

where \(P_\text{th}\) is the threshold peak power.\(^ {23}\)

Considering this, single tracks were ablated with increasing pumping currents from 30 to 80\%, while keeping the frequency (25 kHz) constant for two different scan speeds (150 and 300 mm/s). Each of the twelve combinations was repeated 5 times. The width of the tracks were measured using the CLSM, and the averages for each set of parameters were used to calculate the focal spot size and fluence threshold using Eq. (2).

### 2.2 Track distance

The optimal track distance was investigated by ablating squares on the samples’ polished surfaces, using parallel tracks with distances varying from 6 to 20\(\mu\)m. Frequency (25 kHz) and scan speed (150 mm/s) were kept constant for three different fluences, which are shown in Table 1. For the set of higher fluence, the distances were bigger to compensate the ablated diameter and allow similar overlapping.

### 2.3 Scan speed

Three parameter combinations were chosen to ablate squares with 50 layers of removal, whose depths were then measured and used to calculate the ablation rate \(A_t\) as:

\[
A_t = h_sV_sS_pn^{-1}
\]

where \(h_s\) is the ablated depth, \(V_s\) is the scan speed, \(S_p\) is the track distance and \(n\) is the number of ablation layers. Track distance was kept constant at 10\(\mu\)m, remaining parameters are listed on Table 2. Each combination was repeated 5 times and the average was considered for the calculation.

### 2.4 Fracture stress

The parameter combinations with highest ablation rate within the sets SS\(\Phi\)36 and SS\(\Phi\)42, shown on Table 3, were chosen for fracture strength evaluation; for comparison a polished, non-ablated control group was measured. Each group consisted of 36 samples.

These parameters were used to ablate a rectangle on the central region of the bending-test specimens, so that the whole surface between the rollers was processed. These samples were subjected to four-point flexure test according to\(^ {22,\,23}\) with support and loading spans of 20 and 10 mm, respectively.

Critical defect sizes (\(a_c\)) were attributed to the fracture strength values (\(\sigma\)) according to the Griffith/Irwin criterion:

\[
a_c = \pi^{-1}(K_{IC}/Y\sigma)^2
\]

by setting the fracture toughness \(K_{IC}\) to 3,4 MPa.m\(^{1/2}\)\(^ {24,\,25}\) and the defect geometric factor \(Y\) to \(\pi/2\), for penny-shaped cracks.

### 2.5 Surface morphology

Surface morphologies were assessed using SEM and CLSM. The latter’s optical profilometer has the advantage of not depending on a probe tip’s size, like other contact methods such as atomic force microscopy.

### 3. Results and discussion

#### 3.1 Parameters optimization

Figure 1 shows the ablated diameter as function of peak power. According to Eq. (2), a beam radius of 23.1 \(\mu\)m, and threshold fluences of 16.7 and 19.3 J/cm\(^2\) for scan speeds 300 and 150 mm/s, respectively, have been calculated; the fit is also indicated in Fig. 1. The inverse proportionality between scan speed and threshold fluences can be explained by the different machining temperature, which is higher for lower speeds, as pointed by Wang et al.\(^ {33}\) for nanosecond ablation of Y-TZP ceramics. However, all
tested fluences lower than 30 J/cm² presented a quite irregular removal behaviour, as can be seen in Fig. 2, which shows the profile of the track distance evaluation with fluence of 24 J/cm². This non-sharp threshold fluence is mentioned by Brown and Arnold⁴ as characteristic for the nanosecond regime, given that there is enough time for the irradiated volume to transfer the absorbed energy to its surroundings.

Comparing the surface profiles of the track distance experiment (Figs. 2–4), there seems to be a critical distance for the TDφ30 set (Fig. 3) at 14 μm, where the expelled melt from each line is thrown over the previously ablated line, impairing the process efficiency. The effect of track distance is less pronounced for the TDφ42 set (Fig. 4), what was attributed to this set's higher evaporation/melting ratio. Track distances of 10 μm and smaller gave very similar surface morphologies independent of the fluence, therefore this value was adopted for all later tests.

In Fig. 5, the dependence of ablation rate $A_t$ on scan speed $V_s$ is shown. The SSφ42 set shows a behaviour that was also

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**Fig. 1.** Groove width as function of pulse peak power, for two different scan speeds.

**Fig. 2.** Height profiles showing non-ablated (left) and ablated (right) regions for the TDφ24 set, with decreasing track distances: a) 16 μm; b) 14 μm; c) 12 μm; d) 10 μm; e) 08 μm and f) 06 μm.
reported by Wang et al. The observed peak at 100 mm/s is justified by the fact that higher speeds result in shallower tracks, hence giving smaller ablation rates, while lower speeds make the tracks too deep, so that the recoil pressure of the vapour phase is not enough to expel the molten material from their bottom. Consequently, the material resolidifies within the track, impairing the process efficiency. The other sets, of slightly lower fluences, resulted in considerably lower ablation rates, pointing that different mechanisms or phenomena are taking place. This is further discussed in the following section.

3.2 Mechanisms of removal

Besides the pronounced difference in ablation rate, sets Wφ36 and Wφ42 also showed very distinct surface morphologies, whose profiles can be seen in Fig. 6. While the Wφ36 set presented a fairly uniform surface, with distinguishable melt pool marks and evenly distributed “holes”, the Wφ42 set gave rise to a highly irregular, rough surface. The holes in the first can be related to cracks that are formed due to thermal stresses, which expand and spall as consequence of later pulses. This mechanism seems reasonable, considering the effect of multiple reflections on the holes’ walls and noting the molten appearance of the holes’ interior and their size, much larger than the pulses regular melt pool edges. The marks around these holes were also noteworthy, which resemble rather linear shock-waves, with well-defined direction changes (Fig. 7).

When looking to the SEM pictures of Wφ36, Wφ42 and Wφ00 as non-treated reference (Fig. 8), the kinds of morphologies can be related to the ablation rate behaviors. From the lower rates for Wφ36 set (Fig. 5), combined with the flat morphology of pulse marks and visible melt signs around holes, one can infer that the irradiated energy is not enough to totally overcome the latent heat of vaporization. The Wφ42 set’s irregular morphology can be credited to turbulence being caused on the melt phase by the vaporizing surface—as suggested by Harimkar and Dahotre for continuous-wave Nd:YAG treatment of alumina—followed by
very fast resolidification that freezes the non-equilibrium structures. Knowles et al.\(^6\) reported this kind of mechanism even for longer (micro- and miliseconds) pulses, and that good surface quality was achieved on alumina, without signs of melting, by using a copper vapor laser (511 and 578 nm wavelength) and 20 ns pulse-width.

### 3.3 Weibull statistics

Both tested sets, W\(\phi\)36 and W\(\phi\)42, show considerable lower strength values compared to the non-treated reference W\(\phi\)00, i.e. 242 and 144 MPa compared to 477 MPa, respectively (Table 4). On the other hand, the Weibull modulus increased from 7.4 to 19.5 and 16.4, respectively.

These results indicate that the laser machining did introduce defects larger than the original ones, and these new defects are more homogeneous in size (Fig. 9), thereby giving less scattering of the strength.
4. Conclusions

Laser ablation of alumina with a 120 ns pulsed ytterbium fiber laser has been experimentally analysed, allowing the individual optimization of each laser parameter in a sequential way. Two removal regimes were observed, with very distinct ablation rates, surface morphologies and fracture strengths. Thereby, the removal rates were found to be indirectly proportional to the samples strength, pointing that this kind of laser process cause considerable surface defects, even for moderate beam fluences.

The results confirm that the laser-material interactions are very particular to the beam and material characteristics, and these

| Set name | Wφ00 | Wφ36 | Wφ42 |
|----------|------|------|------|
| Mean strength (std. dev.)/MPa | 483(77) | 242(15) | 142(11) |
| Determination coefficient R² | 0.980 | 0.956 | 0.975 |
| Characteristic strength/MPa | 515 | 249 | 146 |
| Weibull modulus | 7.4 | 19.5 | 16.4 |

Table 4. Weibull modulus and characteristic stress results

Fig. 6. CLSM surface measurement of a) Wφ36 and b) Wφ42, showing non-ablated (top border) and ablated regions.

Fig. 7. SEM picture of a “hole” feature on the surface of Wφ36 samples, with straight shockwave-like lines.

Fig. 8. SEM pictures of Wφ00, Wφ36 and Wφ42 (first, second and third columns, respectively), at increasing magnifications from top down.
results are expected to contribute to the development of future technologies as well as provide a basis to validate numerical models to come.

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