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# Liquid-assisted laser ablation of advanced ceramics and glass-ceramic materials



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#### 1. Introduction

Laser technology has been developed during the last decades, introducing new processing techniques in basic and applied research, such as machining and micromachining, marking, drilling and laser deposition [1-3]. These techniques are suitable in several fields of basic and applied research, such as optics, microelectronics and medicine, due to its high accuracy and quality. Laser ablation can be applied to a wide range of materials, from metals to ceramics. Latest developments in laser technology have allowed the availability of more powerful systems with better features and lower prices, making the laser a versatile and cost-effective tool for industrial applications. In the last decade, pulsed laser ablation in liquids, PLAL, has gained an increasing interest. It has commonly been used to synthesize nanostructures and for nanoparticle fabrication. In this technique, target materials to be machined by laser are introduced in liquids. Despite the fact that a liquid may attenuate the laser energy deposited on the surface of the material, this energy density can be enough to allow the ablation process. This technique is of great interest due to the properties of the liquid and the phenomena which occur when the laser beam goes over a liquid medium [4–6].

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#### ABSTRACT

In this work, results obtained by laser ablation of advanced ceramics and glass-ceramic materials assisted by liquids are reported. A Q-switched Nd:YAG laser at its fundamental wavelength of 1064 nm with pulsewidth in the nanosecond range was used to machine the materials, which were immersed in water and ethylene glycol.

Variation in geometrical parameters, morphology, and ablation yields were studied by using the same laser working conditions. It was observed that machined depth and removed volume depended on the thermal, optical, and mechanical features of the processed materials as well as on the properties of the surrounding medium in which the laser processing was carried out. Variation in ablation yields was studied in function of the liquid used to assist the laser process and related to refractive index and viscosity. Material features and working conditions were also related to the obtained results in order to correlate ablation parameters with respect to the hardness of the processed materials.

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When a laser beam is focused on the surface of a material immersed in a liquid, laser-liquid and laser-matter interaction, laser-induced breakdown and plasma are produced. Due to plasma expansion, a shock wave is emitted, followed by a cavitation bubble formation, expansion and collapse [6-10].

Compared to the common laser ablation in air, the presence of liquids provides several advantages. High pressures and temperatures, up to 107 Pa and 10,000 K respectively, can be reached, so that a mechanical component is added to the ablation process, allowing the extraction of a higher amount of material from the target surface, thus producing a considerable increase of the ablation yield. In addition, the liquid layer cools the surface of the target material reducing thermal damages on the sample. Furthermore, debris is diffused in the surrounding liquid, cleaning machined areas and avoiding recast of material in the working zone. As vapor and debris are diffused in the liquid, emissions to the atmosphere are reduced, keeping a cleaner working area and avoiding respiratory damage on operators. Debris released in the liquid gives rise to a solution with nanoparticles which can be used in a subsequent process. Water is the most common liquid used to assist laser machining, but dissolutions or reactive liquids can also be used for photochemical processes [4-15].

Advanced ceramics such as Zirconia and Alumina are usually used in basic and applied research as structural and functional components due to their lightness, hardness, wear resistance and chemical stability at high temperatures [16–19]. However their

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#### Table 1

Mechanical, optical and thermal properties of 8YSZ, alumina and Schott Ceran Suprema® glass-ceramic.

Property	Glass-ceramic	8YSZ	Alumina
Density (g/cm <sup>3</sup> )	2.5 <sup>a</sup>	5.85 <sup>b</sup>	3.88 <sup>b</sup>
Bending strength (MPa)	110 <sup>a</sup>	265 <sup>b</sup>	500 <sup>b</sup>
Hardness (Vickers)	800	1200	1500
Toughness (MPa m <sup>1/2</sup> )	1.5	7	4
Thermal conductivity (W/mK)	1.7 <sup>a</sup>	2.5 <sup>b</sup>	25 <sup>b</sup>
Thermal diffusivity $(m^2/s \times 10^{-6})^c$	0.85	1.07	7.58
Melting temperature (K)	1498 <sup>a</sup>	2959 <sup>b</sup>	2327 <sup>b</sup>
Diffuse reflectance (1064 nm)	0.93	0.95	0.84
Optical absorption $\alpha$   1064 nm (cm <sup>-1</sup> )	3.52	9.34	10.90
Absorption length $L_{\alpha} = \alpha^{-1}   1064  \text{nm}(\text{cm})$	0.28	0.11	0.09
Thermal diffusion length $L_{th} 10  ns(\mu m)$	0.18	0.20	0.55

<sup>a</sup> Schott technical data.

<sup>b</sup> Kerafol technical data.

<sup>c</sup> Measurement carried out at the Institute of Ceramic and Glass, ICV-CSIC.

high hardness and brittleness are a drawback for being machined by traditional methods. Laser machining features provide a noncontact and micron-sized tool to machine these ceramics remaining unchanged the adjacent areas. For instance, zirconium oxide ceramic stabilized with 8 mol.% yttrium oxide (8YSZ) is used in energy applications as anode or electrolyte of solid oxide fuel cells (SOFCs). In particular, anode laser machining has been used to generate 80  $\mu$ m drills to improve gas permeation and electrolyte laser machining for reducing the membrane thickness from 150 to 50  $\mu$ m and so the Area Specific Resistance, giving rise to an operation temperature reduction of 120 °C [20–24]. Alumina is used as a bioceramic material in scaffolds and implants, owing to its biocompatibility [25,26].

Glass-ceramics have been commonly used as photonic materials due to its interesting optical properties in mirrors, waveguides, lasing systems, etc. [27–29]. Glass-ceramic materials are of great interest for some industrial applications because of their mechanical and thermal properties. These properties are good chemical inertness, excellent thermal shock resistance, low coefficient of linear expansion, impact and abrasion resistance, and high temperature stability. Laser processing of glass-ceramics is of great interest in industry for functional purposes, such as machining blind holes for inclusion of sensors and grooves for frame locks. Furthermore, surface modifications can be made in glass-ceramics by laser processing to create thermal-barriers or heat-conductor tracks [30–32].

In previous works, dependence between sample reference position and ablation yields when laser processing was carried out in air was studied. It was shown that when samples were machined by deflecting and overlapping laser pulses, the highest ablation yields were obtained machining above and below the focal plane. It was also demonstrated that in the nanosecond range this behavior was an intrinsic characteristic of this machining technique and did not depend on the laser wavelength, pulsewidth, working frequency or scanning speed. In addition, it also was observed a correlation between material hardness and ablation yield [33,34]. The aim of this work is to study the effect of the liquids used to assist the laser processing of advanced ceramics and glass-ceramic materials. We will analyze geometrical parameters, ablation yields, and their correlation to the mechanical properties of ceramic substrates.

#### 2. Experimental

#### 2.1. Laser processing

A diode-pumped Nd:YAG laser system (Rofin Sinar E-Line 20) has been used in this work. It operates at its fundamental wavelength of 1064 nm, with a maximum mean power of 11 W, Gaussian mode TEM<sub>00</sub> with a beam quality factor  $M^2$  < 1.3. An opto-acoustical Q-Switch commutator controls the cavity output in a continuous or pulsed mode. 8–25 ns pulses can be generated in a frequency range of 1–40 kHz. The laser system is equipped with a programmable galvanometer at the output of the cavity, which is controlled by a CAD software, so that the laser beam can be deflected making a bidirectional movement in such a way that any predefined pattern can be performed. The laser system is equipped with an x5 beam expander before the galvanometric mirrors. A convex lens with focal length of 100 mm is used. The laser beam is controlled by means of changing the diodes pump current *I* (related to peak power), pulse frequency *F*, linear speed  $V_L$ , and distance between adjacent lines  $\Delta$ .

As materials, glass-ceramic substrates (Schott Ceran Suprema<sup>®</sup>), dense alumina sheets with a content of 99%  $Al_2O_3$  and dense substrates of zirconium oxide ceramic stabilized with 8 mol.% yttrium oxide (both produced by Kerafol) were used. Their thermal and mechanical properties are shown in Table 1.

Plates were machined above and below the focal point, varying the reference position around it, as shown in Fig. 1. The sign convention taken is: negative when the sample is moved upwards and positive when it is moved downwards.

To attain reliable results, three grooves were machined in each position, with dimensions  $5 \text{ mm} \times 0.3 \text{ mm} \text{ length} \times \text{width}$ , and three measurements of depth and width were taken for each groove. With these measurements, average values and standard deviations of depth, width and removed volume were calculated.

Before machining in water, the process was carried out in air in order to compare obtained results with different mediums.

An aluminum base plate holding the samples was introduced into a Pyrex vessel, which was filled up with liquid until the desired thickness *l* was reached, as shown in Fig. 2. Three different thicknesses of liquid layer were used: 3, 5 and 10 mm.

When laser radiation goes through a liquid, a phenomenon of refraction occurs due to change of medium, so the variation of the focal length can be calculated according to the following equation [5]:

$$\Delta f = l \left( 1 - \frac{1}{n} \right) \tag{1}$$

where l is the thickness of the layer and n is the refractive index of the liquid.

Two different liquids, water, and ethylene glycol, were used in order to study the influence of their properties on the ablation yield. Their properties are shown in Table 2.

With the purpose of comparing the obtained results, samples were processed using the same working conditions. Pulses of 2.45 mJ of energy and a peak power of 260 kW were used. Processing parameters were 20 mm/s of scanning speed, frequency of 2 kHz, and distance between adjacent lines of 10  $\mu$ m.

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