Journal of the European Ceramic Society xxx (2017) xxx–xxx

Contents lists available at www.sciencedirect.com

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc

Short communication

Evaluation of damage in front of starting notches induced by ultra-short pulsed laser ablation for the determination of fracture toughness in zirconia

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ARTICLE INFO

Article history:
Received 12 May 2017
Received in revised form 26 June 2017
Accepted 8 July 2017
Available online xxx

Keywords:
Ceramics
3Y-TZP
Fracture toughness
Laser damage
Notch

ABSTRACT

Machining a very sharp notch on the surface of ceramics for fracture toughness testing has been a critical issue during many years. In this work, we explore a novel method capable of inducing sharp features with negligible damage with laser pulsed ablation. We study the effect of different laser ablation parameters in the notch length and damage induced in 3 mol% yttria doped zirconia. In front of the notch it is found a very narrow microcracked zone which spreads in the notch direction, whose length only depends on the laser pulse energy; meanwhile the length of the notch is proportional to the energy deposited during the process.

1. Introduction

Fracture toughness of ceramics is usually determined by the single edge V-notch beam (SEVNB) method [1]. The notch is made with a saw cut and its tip is thinned by honing with a razor blade impregnated with a diamond slurry [2], [3]. By doing so, a relative sharp notch tip is finally achieved, and a number of microcracks, with a size typically 1–3 times the grain size, can be left in front of the notch tip by the machining process [4]. For the determination of K_{Ic} by this method, the stress intensity factor of this configuration of notch plus microcracks can be taken as that of a straight sharp crack as far as the length of the microcracked zone is longer than the notch tip radius [5]. This condition can be fulfilled in coarse grain size ceramics but it cannot be obeyed when the grain size is in the microscale or below, since then the radius of the notch tip should be smaller than is practically possible to achieve by honing. This makes machining sharp notches in sub-micrometer grain size ceramics for fracture toughness testing a critical issue.

One way of machining a very sharp notch on the surface of ceramics is ultrashort pulsed laser ablation (UPLA), which, in principle, is a technique capable of inducing very sharp features with negligible damage [6–8]. It is then an appealing technique for inducing a very sharp starting notch for measuring fracture toughness [9–12].

With UPLA the material is removed by direct vaporization and the heat affected zone is expected to be extremely small as compared to other techniques, provided the deposition of laser energy on the surface of the material occurs in a timescale which is much shorter than the typical time for energy transfer between electrons and lattice. Therefore, the material leaves the irradiated zone by either Coulomb or phase explosion mechanisms, depending on the density of energy of the laser on the surface [13], [14], and the rest of the material remains almost unaltered.

UPLA has been used for the production of high quality microstructures (grooves and pores) on the surface of cylindrical 3 mol% yttria (3Y-TZP) dental implants [15]. In a previous work [9], [10] we have determined the fracture toughness of this material from surface sharp notches induced by UPLA. Here the attention is focused on studying the change in notch length and damage in

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http://dx.doi.org/10.1016/j.jeurceramsoc.2017.07.006
0955-2219/

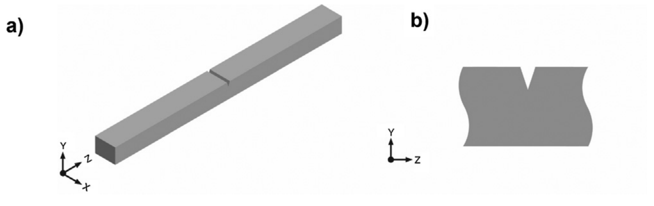

Fig. 1. Schematic view of the prismatic samples and the notch configuration: a) isometric view and b) lateral view focused on the notch.

front of notches machined under different laser ablation parameters. The change in the microstructure in front of the notch tip is here analysed by SEM-FIB showing that it consists mainly of a very narrow short microcracked band extended in the direction of the notch. The possible influence of this damage in the determination of fracture toughness is discussed.

2. Experimental procedure

Commercial powder 3Y-TZP (grade TZ-3YSB-E, Tosoh) was cold uniaxially pressed at 100 MPa and sintered at 1450 °C with heating and cooling rates of 3 °C/min and 1 h of maintenance, obtaining prismatic bars of 4 mm × 3 mm × 45 mm (directions X, Y and Z, respectively, see Fig. 1) with a density above 5.95 g/cm^3 as measured by the Archimedes method, and a grain size of about 330 nm. All specimens were chamfered, and all surfaces were ground and polished following the standard methods up to 3 μm finishing.

Small notches were laser machined on the XZ surface of the samples by UPLA, as pictured in Fig. 1. The ablation was carried out with infrared ultra-short laser pulses (120 fs, 795 nm) along the Y-direction (Fig. 1). The system used was a commercial Ti: Sapphire oscillator (Tsunami, Spectra Physics) and a regenerative amplifier system (Spitfire, Spectra Physics) based on chirped pulsed amplification. The pulses were linearly polarized and the repetition rate was 1 kHz. The focusing system used was an achromat doublet lens with 50 mm focal length. The samples were placed on a XYZ motorized stage and moved along the X-direction. The notches for the fracture toughness determination were produced using a pulse energy of 5 μJ, scanning speed of 50 μm/s and four passes. In one of the samples, several notches were produced using different laser parameters in order to investigate the damage, as summarized in Table 1.

Two notches, 7 and 13 of Table 1, were selected for deeper observation, since they correspond to the lowest and highest total energies delivered for producing the notch. Field Emission Scanning Electron Microscopy (FESEM, JEOL) was used for a visual examination of the notches. First, a few tens of microns were removed from the YZ lateral surface by grinding and polishing to observe the damage in the interior of the sample and to avoid any edge effect on the notch fabrication and the consequent damage.

The tomography study of the microcracked area in front of the two notches was made using a dual beam Scanning Electron Microscope/Focused Ion Beam (FIB/SEM, Neon40, Zeiss), using a Ga$^+$ ion column. A platinum layer was deposited on the surface prior milling to reduce the curtain effect. Slices on the XZ plane with a thickness of 12 nm were milled using a current of 500 pA in the Y direction (Fig. 1). The volumes involved in the study were 5.7 × 9.3 × 8.1 μm^3 for notch 7 and 5.7 × 25.7 × 8.1 μm^3 in the case of notch 13.

FEI Avizo software was used to stack the slices and to create a segmented surface of the microcracks. Few prior gaussian and edge-preserving filters were required in order to improve image quality and erase artifacts.

3. Results and discussion

Fig. 2 shows a general view of the notches. In front of each one there is always a small microcracked band. It can be seen in Table 2 that the lengths of the notches are between ~10 μm and 29 μm and that the microcracked bands are between ~7.3 and 24 μm in the direction of the notch. There is also a correspondence between the laser energy deposited and the dimensions of the notches and microcracked bands so that the two notches produced with the lowest and highest laser pulse energies (number 7 and 13) are also the ones with shorter and longer total damage (notch + length of microcracked band).

Taking into account laser pulse energy, scanning speed, number of passes, pulse duration, and an estimated energy loss of 8% after the laser light passes through the focalization system (see Table 1), the total radiation energy per unit of length can be obtained and plotted as a function of notch depth (Fig. 3a). As expected, the notch depth increases with the radiation energy per unit of length.

However, the size of the damage in front of the notch does not depend on the total energy, since it does not change with the number of pulses per unit of length. It varies only with the energy of the pulse, as it can be observed in Fig. 3b where the microcracked band length in front of the notch is plotted in terms of the number of pulses per unit length. Therefore, scanning speed and number of passes has no effect on the extent of damage observed. The average extension of the microcracked band length is plotted in Fig. 3c in terms of the pulse energy.

In Fig. 4a notches 7 and 13 with their microcracked bands are shown in more detail. The tomographic analysis was carried out in the slab indicated by the rectangle drawn in front of the notch with dimensions of 5.7 μm x9.3 μm in the YZ plane and with a depth along the X-direction of ~8.1 μm in the case of notch 7. In the case of notch 13 (right) the same dimensions as for notch 7 with the exception of the length in the Z direction was now ~25.7 μm since micro-cracking was detected up to ~24 μm from the notch root. Two videos on the 3D reconstruction of the microcracked zone are incorporated as supplementary material which gives light on the shape and interconnection of the cracks.

In order to compare both notches, a region of interest was defined as that which encloses all the cracked region in front of the notch. The beginning is arbitrarily defined as the location where the surfaces of the notch are opened by a gap of 2 μm, and it extends below the notch tip until no damage is detected. This was done to avoid some ambiguity in the detection of the true notch tip in some notches.

The slabs in front of the notch were sectioned along planes perpendicular to the notch (plane XZ, see Fig. 1) and the cracked area in each slice was measured. The fraction of cracked area detected

Table 1
Parameters used for the ablation of the notches (pulse duration 120 fs, wave length 795 nm, repetition rate 1 KHz, spot radius 2.8 μm).

Notch Number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Pulse energy (μJ)	5	5	5	5	5	5	3	3	3	3	3	3	8	8	8	8	8	8
Average power (mW)	5	5	5	5	5	5	3	3	3	3	3	3	8	8	8	8	8	8
Scanning speed (μm/s)	50	50	100	100	150	150	150	150	100	100	50	50	50	50	100	100	150	150
Number of passes	4	2	2	4	4	2	2	4	4	2	2	4	4	2	2	4	4	2
Pulses per linear micron	80	40	20	40	27	13	13	27	40	20	40	80	80	40	20	40	27	13
Energy per linear micron (mW)	400	200	100	200	133	67	40	80	120	60	120	240	640	320	160	320	213	107

Please cite this article in press as: M. Turon-Vinas, et al., Evaluation of damage in front of starting notches induced by ultra-short pulsed laser ablation for the determination of fracture toughness in zirconia, *J Eur Ceram Soc* (2017), http://dx.doi.org/10.1016/j.jeurceramsoc.2017.07.006