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Laser notching ceramics for reliable fracture toughness testing

Holly D. Carlton[a,*], John W. Elmer[a], Dennis C. Freeman[a], Ronald D. Schaeffer[b], Oleg Derkach[b], Gilbert F. Gallegos[a]

[a] Lawrence Livermore National Laboratory, Livermore, CA 94550, USA
[b] Photomachining Inc., Pelham, NH 03076, USA

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ABSTRACT

A new method for notching ceramics was developed using a picosecond laser for fracture toughness testing of alumina samples. The test geometry incorporated a single-edge-V-notch that was notched using picosecond laser micromachining. This method has been used in the past for cutting ceramics, and is known to remove material with little to no thermal effect on the surrounding material matrix. This study showed that laser-assisted-machining for fracture toughness testing of ceramics was reliable, quick, and cost effective. In order to assess the laser notched single-edge-V-notch beam method, fracture toughness results were compared to results from other more traditional methods, specifically surface-crack in flexure and the chevron notch bend tests. The results showed that picosecond laser notching produced precise notches in post-failure measurements, and that the measured fracture toughness results showed improved consistency compared to traditional fracture toughness methods.

1. Introduction

There is a wide range of methods to evaluate a ceramic's fracture toughness and method selection can be material and resource dependent. Many ceramics display brittle characteristics and are highly sensitive to flaws. For ceramics, flaws can be found at many length-scales in the form of pores, inclusions, grain boundaries, and machining defects [1,2]. However, the pre-existing defect distribution in a ceramic specimen is not always known and can be time consuming to characterize. During fracture toughness testing a known worst-case flaw is introduced into the material and then the energy required to fracture the material is determined [3]. When selecting a fracture toughness method it is imperative that the specimen geometry involve either notches or pre-cracks that are reliable, reproducible and avoid introducing unwanted damage, in order to create accurate test results.

Two common specimen geometries used for calculating fracture toughness in ceramics are shown in Fig. 1. The chevron notch (CN) method has a sharp V introduced into the width of the bend specimen, and the surface-crack in flexure (SCF) method, where a pre-crack is introduced via a micro-hardness indenter [4–6]. Recently though single-edge-V notched beam (SEVNB) has shown promise as a reliable method for calculating fracture toughness in ceramics [7–12]. The specimen geometry for a SEVNB test calls for a sharp V in a different orientation than the CN V and is based on straight-through cracks and does not require the addition of pop-in cracks [13]. A schematic showing the different geometries is presented in Fig. 1. In preparing the SEVNB specimen the notch is commonly introduced via scribing, which is often done using a razor blade and diamond suspension. However the sharp radius necessary for the fracture toughness test is limited by the achievable radius, which is constrained by the thickness of the blade used for notching. This can be problematic for fracture toughness testing in brittle materials, because when the root radius of the notch is larger than relevant microstructural features the fracture toughness results will be adversely affected by the size effect [12], where the fracture toughness values are shown to vary with the square root of the radius until a critical root radius is achieved [14,15]. Therefore, for this study we investigated an alternative for notching ceramics for fracture toughness testing utilizing laser-assisted-machining with pulses in the picosecond (ps) range.

The benefits of using the ps-laser are made possible by the fact that material removal occurs by ablation, whereby energy transferred to the surrounding lattice is insignificant. The result is that material is removed with little or no thermal effect on the surrounding material [16], and a smooth reproducible notch is produced. Another study [17] that was going on concurrently to this one, showed the benefits of using an ultra-short pulsed femtosecond laser to introduce shallow notches (20–40 μm depth) into zirconia

* Corresponding author at: 7000 East Ave, Livermore, CA, 94550, USA. Fax: +1 925 423 1572.
E-mail address: carlton4@llnl.gov (H.D. Carlton).

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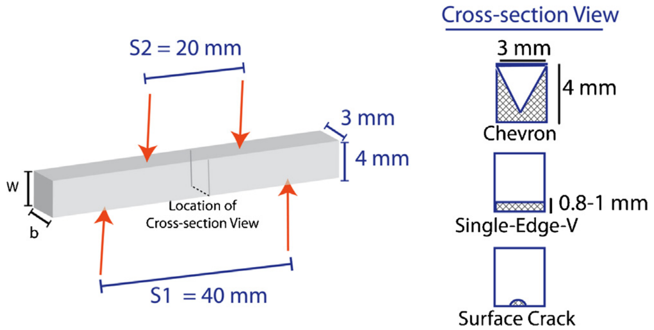

Fig. 1. Specimen geometry. A notched rectangular alumina specimen (3 × 4 × 45 mm) is taken to failure using a four-point bend configuration. Also shown here on the right is a cross section through the sample (see location) with drawings of the three sample geometries used in this study. The three methods investigated here are CN, SEVNB, and SCF. All the notching was done on the 3 mm face for consistency.

with sub-micron grain size for fracture toughness testing. However, for the study presented here, the relative crack depth used for the SEVNB tests on ceramics is much larger, ~25% versus 1%, which eliminates the need to measure any beginner micro-cracks around the notch tip.

For this study we used a ps laser to introduce a notch that tapers to a V-shape for SEVNB fracture toughness testing. This method is used to evaluate the fracture toughness of three different purities of aluminum oxide (96%, 97.5%, and 99.5%), and the results are compared with two more traditional methods. The three purities of aluminum oxide were selected to observe the effects of notching on ceramics with varying toughness values. Specifically, the three types of purities have different grain sizes, which is known to influence the fracture behavior in aluminum oxide [18]. Also, these types of aluminum oxides are known to display some toughness in the form of stable crack growth once a crack initiates.

2. Experimental methods

2.1. Laser notching

The laser notching for the SEVNB specimen was performed using a Lumera Super Rapid Q-switched DPSS laser with pulses in the picosecond range [19]. The laser notching parameters, i.e., laser wavelength, focal length, power, energy/pulse, pulse rate, and pulse length, were selected by making and assessing practice cuts to a sacrificial piece of alumina (99.5% Al_2O_3). These tests were done with pre-selected parameters that would optimize cutting speed and notch quality, as summarized in Table 1. Here, two ps laser wavelengths were tested, infrared (1064 nm) and ultraviolet (355 nm). The laser power was 12 W in the infrared and 4 W in the UV, and the laser beam diameter was kept constant at 25–30 μm using a lens focal length of 80 mm when in the infrared and 100 mm when in the ultraviolet. After inspecting the trial laser-assisted-machining cuts, we selected the UV set of parameters, which are shown in Row 2 of Table 1 for SEVNB testing reported in this study.

The laser, which was pulsed during notching, was capable of operating at pulsing rates as high as 1 MHz. However, for our study the micromachining was performed at a 100 kHz pulse rate based on prior experience with laser machining ceramics. At 100 kHz, the pulse rates of the laser allowed for fast travel speeds of 500 mm/s, which was used in this investigation to produce a pulse-to-pulse pitch of approximately 5 μm. This pitch produces approximately 80% overlap of subsequent pulses, and created a smooth bottomed cut. All of the laser micromachining was performed in air with no additional gas shielding.

Once the parameters were developed the ps-laser was used to remove material to produce a notch with a specific depth, a, through the sample thickness, B, for the SEVNB specimen, as illustrated in Fig. 1. The notch depth a was selected so that the ratio, a/W, known as the normalized crack size, fell in a valid range: 0.2–0.3 [4] (where W is the specimen's width and is defined in Fig. 1). The cutting times are proportional to the notch depths, but are very fast compared to the other methods. For the parameters used here, it took 40 passes to complete a ~1 mm deep notch in the 99.5% purity alumina. At the travel speed of 500 mm/s and a sample thickness of 3 mm, the entire notch was completed in less than 1.5 min. This includes the cutting time required to remove extra material near the original alumina surface, which allows the notch to form properly.

2.2. Traditional fracture toughness tests

The SCF and CN test specimens were machined using a high precision surface grinder, to a geometry similar to what is specified in the ASTM standard for determining fracture toughness of ceramics [4]. The SCF fracture toughness test used a Knoop indenter to introduce a semi-elliptical surface crack [5,6] into the ceramic, which acts as a starter crack when brought to failure in a four-point bend configuration. The Knoop indenter was selected for introducing the surface crack over other types of indenter tips since it produces a much shallower indentation and it has been shown to be less susceptible to cracking problems [20]. Although the ASTM standard calls for an indentation load of less than 100 N, it was determined by the operator that a load of 220 N was needed in order to pop in a pre-crack that could be resolved in post examination, as well as to make sure the indent spanned several grains. Fig. 2a shows an optical micrograph (OM) of a Knoop indented alumina specimen with visible cracks extending approximately 0.5 mm across the width of the indentation, and localized cracking or crushing near the central portion of the indentation. In future tests this crushing zone should be polished and removed per the ASTM standard 1421.

For the CN specimens a notch was introduced by machining the chevron using a diamond wafering blade attached to a surface grinder (Chevalier FSG-2A818). Custom-built fixtures were required for machining the chevron geometry into the rectangular beam specimen. After machining the chevron into the ceramic specimen, it was loaded in a four-point bend configuration. The CN sample geometry allows for the preferred crack initiation site to occur at the tip of the chevron, which allows for a sharp crack to develop during loading. This notch geometry is designed to promote stable crack growth at the tip of the triangular notch until catastrophic failure. The CN method is generally preferred over the SCF method because it produces more consistent results if the notch is properly machined. However, the precision fixtures were expensive to fabricate and the low speed saw cutting was time consuming, adding considerable cost to the test plan.

2.3. Materials, test specimen geometries, and mechanical testing

The materials studied here were 96%, 97.5%, & 99.5% pure, polycrystalline aluminum oxide. Density, grain size and porosity for each type of alumina are presented in Table 2. The grain size for the 97.5% alumina is 3–4× larger than the other two types of alumina, which is a contributing factor in fracture toughness, in addition to purity. The 96% CoorsTek alumina was procured as uncertified material, meaning that there may be more variability in the microstructure and fracture toughness than the other materials.

The bend specimens for the tests were machined from the as-received alumina plates into rectangular samples having a nominal width, W, thickness, B, and length, L, of 4, 3, and 45 mm, respectively. For the SCF specimens surface cracks on the ceramic material were created with a Knoop indenter installed on a low capacity