Scripta Materialia 132 (2017) 53–57

Contents lists available at ScienceDirect

Scripta Materialia

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

Regular article

A chevron-notched bowtie micro-beam bend test for fracture toughness measurement of brittle materials

Fiona Yuwei Cui *, Richard P. Vinci

Department of Materials Science and Engineering, Center for Advanced Materials and Nanotechnology, Lehigh University, Bethlehem, PA 18015, United States

ARTICLE INFO

Article history:
Received 17 January 2017
Received in revised form 27 January 2017
Accepted 27 January 2017
Available online xxxx

Keywords:
Fracture
Toughness
Bending test
Glass
Zerodur

ABSTRACT

A micro-mechanical fracture testing method has been developed that uses a bowtie-shaped micro-beam specimen with a chevron notch. This clamped-clamped specimen can produce stable crack growth in brittle materials. Cyclic loading causes progressive crack extension, thereby producing multiple fracture toughness results in one experiment. The symmetric geometry eliminates the mixed mode fracture that exists in single-ended cantilevers. A 3D finite element analysis model was used to relate the crack length to the beam compliance, and then to the fracture toughness. The results of tests using fused quartz and glass-ceramic materials match very well with published fracture toughness values.

Traditional mechanical testing methods developed for bulk materials are often difficult or impossible to apply directly to small-scale specimens, due to sample handling challenges and the small loads and displacements involved. While a number of techniques now exist for measuring elastic and plastic properties of thin films and microstructural constituents, measurement of fracture toughness still presents a number of challenges. The micro-cantilever deflection test has recently emerged as a preferred method due to the simplicity of the cantilever beam configuration. It has been utilized to analyze structure-property relationships in a variety of materials such as thin films and individual phases within multiphase materials, and different phenomena including the influence of impurity segregation on grain boundary mechanical behavior [1–4].

While cantilevers have a number of advantages for micro-scale fracture testing, the inherent mixed mode fracture cannot be neglected at large deflections and in anisotropic materials, and the difficulty in producing a truly sharp crack by fatigue or other methods makes the fracture results sensitive to the radius of the pre-machined notch. Moreover, regardless of the specific specimen shape, site-specific micro-scale sample fabrication typically requires techniques that are time consuming and costly. Generating sufficient fracture data to ensure statistical significance is often not practical for any configuration that results in a single data point per test specimen. Recently, stable crack growth has been achieved in cantilevers milled with a chevron notch on the top surface; in this way there is sufficient stability to reliably produce a truly sharp crack prior to the onset of failure [5], but the mixed mode fracture toughness effect due to the asymmetric geometry of a cantilever is still present and each specimen yields only a single fracture toughness value. A symmetric clamped-clamped beam with a straight notch on the underside has been explored as a way of eliminating the mixed mode fracture, and has been shown to achieve stable crack growth under certain conditions [6]. However, the high stresses induced at the clamped ends can cause nearly simultaneous failures at the beam center and ends, complicating analysis and limiting the number of stable fracture events that can be measured [7].

To address these shortcomings, we have developed a bowtie-shaped beam configuration that is fixed at both ends, and that has a chevron notch milled into the underside at the beam center, as shown in Fig. 1. The stiff, symmetric test specimen configuration with a centralized chevron notch allows the controlled propagation of a crack advancing along a straight path. This makes it possible to collect multiple measurements of toughness with a single specimen by cyclically loading to a series of increasing peak load values, each of which causes a small, stable, and measureable increase in crack length. The specimen configuration also eliminates mixed mode fracture toughness. The milling time needed to create such a specimen is only about 25% greater than to create a standard cantilever.

The loading point is located directly above the chevron notch in the middle of the beam. When loading, the tip of the chevron notch will be the highest stress concentration, and thus the preferred site for crack initiation. As the crack advances along the chevron notch, the crack-front length will increase, as shown in Fig. 2a, thus a higher load is required for further propagation in each step [8,9]. Under appropriate testing conditions, such as the right chevron geometry and a very stiff loading instrument, the crack would advance in a stable fashion and

* Corresponding author.
E-mail address: fiona.yw.cui@gmail.com (F.Y. Cui).

http://dx.doi.org/10.1016/j.scriptamat.2017.01.031

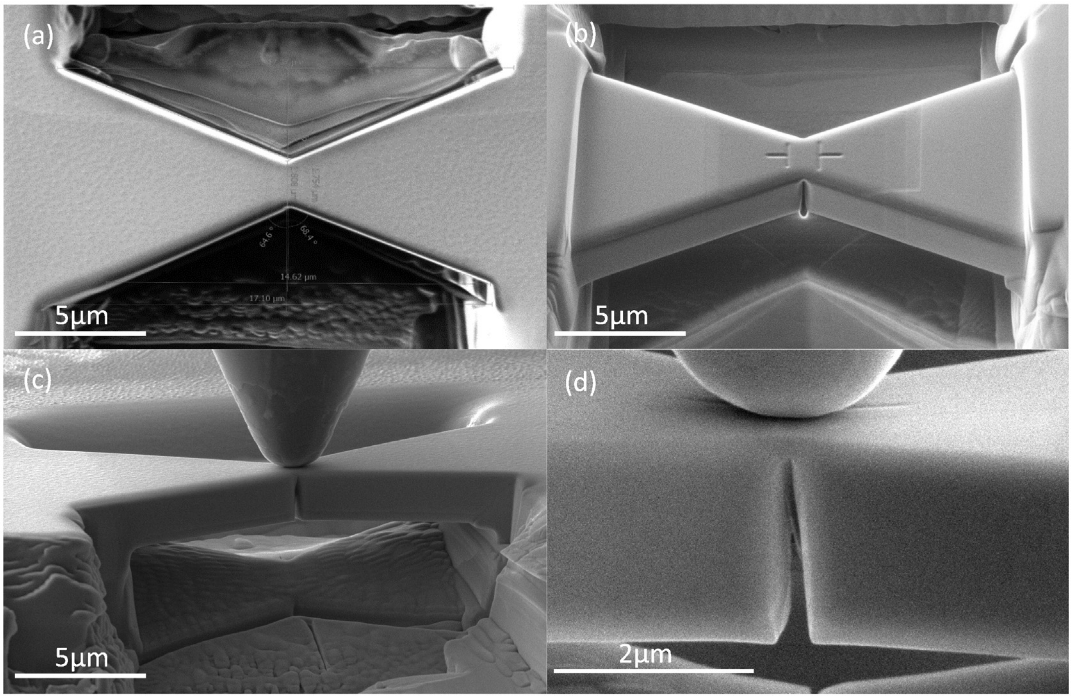

Fig. 1. Fused quartz bowtie chevron-notched specimen fabricated with Focused Ion Beam milling. (a) top surface of a bowtie chevron beam, (b) 30° view of a bowtie specimen, (c) in-situ mechanical test with indenter on top of specimen, (d) crack propagation visible under SEM observation during in-situ test.

the extent of crack propagation could be controlled by the applied maximum load. If a cyclic load-unload function is employed, the compliance change from one cycle to the next is a predictable measure of crack propagation [10]. This correlation enables the measurement of compliance as a function of crack length, the crack advance per load cycle, and the fracture energy associated with the corresponding maximum load value at each step.

A bowtie shaped specimen was selected to avoid the end-cracking often observed in rectangular clamped-clamped specimens. Experience with chevron-notched rectangular beams demonstrated that the ends of the beam would sometimes simultaneously fracture in a catastrophic fashion so that the real crack propagation at the chevron notch was hard to distinguish [7]. A triangle-shaped specimen has a reduced stress concentration at the beam ends as compared to a specimen with a uniform cross-section [11]. This eliminates or delays the end-cracking, allowing the central crack to initiate and propagate at the notch area for evaluation of the fracture toughness value.

In the examples reported here, each specimen was fabricated from a bulk fused quartz or lithium-aluminosilicate glass-ceramic (Zerodur™) sample. These two materials were selected because they both are amorphous brittle materials, so the fracture toughness measurements will not be affected by anisotropy or plasticity. Furthermore, published fracture toughness values for both materials are available for comparison.

The test specimens were made using Focused Ion Beam (FIB) milling in an FEI Scios instrument. The bulk sample was first mechanically ground and polished on two adjacent surfaces so that a 90-degree edge was exposed for FIB milling. After the basic shape was established using a high ion beam current, the test structure was ion polished using 0.1 nA at a tilt angle of $\pm 1.5°$ to ensure smooth surfaces with minimal ion damage and parallel sides. The chevron notch was fabricated by milling at an angle to the surface in three steps, starting with an ion beam current of 10 pA with a larger width milling pattern, reducing to a smaller width, and finishing with 1.5 pA and a minimum milling width. Thus, a ‘V’ shaped segment was generated in the center of the

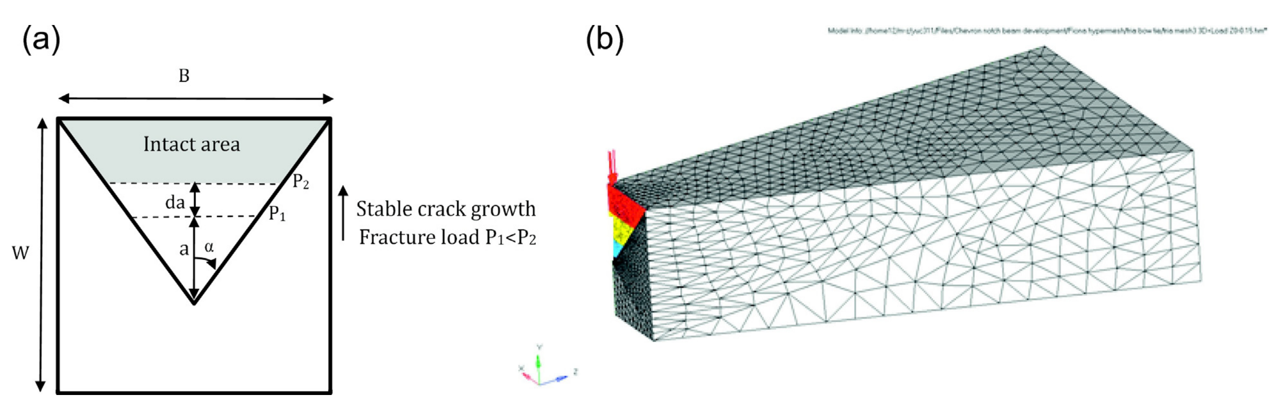

Fig. 2. (a) Schematic cross-section of the bowtie chevron-notched beam design, showing the chevron notch during crack propagation. The triangle represents the original intact region and the grey area represents the intact region after some degree of crack growth has occurred. (b) Quarter symmetry FEA model. Axial displacement constraints (z-axis) were applied at the intact area ahead of the crack front, where different color highlights represent different crack lengths. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)