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Fracture toughness measurement in fused quartz using triangular chevron-notched micro-cantilevers

Goran Žagar *, Václav Pejchal, Martin G. Mueller, Lionel Michelet, Andreas Mortensen

Laboratory of Mechanical Metallurgy, Institute of Materials, École Polytechnique Fédérale de Lausanne (EPFL), Station 12, CH-1015 Lausanne, Switzerland

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We extend to flat surfaces the fracture toughness method presented in Acta Materialia vol. 86 (2015) p. 385 and measure in this manner the fracture toughness of fused quartz. Tests give 0.67 ± 0.01 MPa $m^{1/2}$, which agrees with earlier microscopic and macroscopic test data for the fast fracture toughness of this material. Data show no signs of sub-critical crack growth; this observation is at variance with what one would expect from literature data on the phenomenon.

The lack of standards for the measurement of fracture toughness at microscopic length scales has spawned, over the last decade, an active search for reliable microscopic fracture toughness tests; a range of methods to this end have thus been proposed. Some methods are specifically adapted to materials that can be processed by lithographic or deposition techniques [1]. Along polished planes, methods for fracture toughness measurement that use radial cracks produced by nanoindentation have alternatively been used — and criticized [2–4]. More recently, modern 3-D micromachining methods, notably Focused Ion Beam (FIB) micromilling, have spawned a new generation of microscopic fracture toughness tests in which samples resembling those used in macroscopic tests are produced and loaded to measure the toughness of materials at the microscale. Microscopic methods for measuring the toughness of (mainly brittle) materials proposed and used to date include: bending micro-cantilevers with straight-through notches [5–7], samples in which cracks are nucleated and grown alongside a trough crack [8] or within a chevron notch [9], fully clamped (FC) micro-beams with a straight-through notch [10–12], double-cantilever-like (DBC) micro-specimens [13] or splitting cylindrical micro-pillars where toughness is evaluated from pillar fracture caused by nanoindenting the pillar along its axis [14].

A frequent concern with such tests is that resulting toughness data may be affected by material surface damage introduced by FIB milling and/or the use of an insufficiently sharp FIB-milled starter notch to measure critical conditions for crack propagation, both effects having the potential to cause significant fracture toughness measurement error. While the magnitude of such error apparently depends on the material being tested [7,8,12], a way to circumvent these common problems is to use configurations in which the fracture toughness is evaluated after some amount of stable crack growth; these include FC, DBC and chevron-notched micro-specimens. Among those, the chevron-notched method has the practical advantage that it does not require in situ crack length measurements to be conducted during the test, such that testing can be performed ex-situ using a conventional nanoindentation instrument [9,12].

The chevron-notched sample method for measuring the Mode I fracture toughness, originally proposed by Barker [15] and consigned for macroscopic scale tests within ASTM standards [16,17], generally uses samples that contain a thin notch surrounding a V-shaped ligament, with the notch normal pointing towards the direction of tensile stress in the loaded specimen. This method was mainly developed for (quasi) brittle materials that exhibit no significant *R*-curve behavior. In a previous contribution [9], we demonstrated its applicability at microscopic scales by measuring the fracture toughness of amorphous fused quartz and nanocrystalline alumina, using FIB-machined microscopic cantilever beams having a rectangular cross-section. These samples had to be prepared along a sharp ~90° edge of the material specimen. In this short communication we demonstrate a different chevron-notched microsample fracture test sample geometry, which can be produced by micromilling along a single (polished) surface; this eases sample production significantly and extends the field of application of the chevron-notched sample microtoughness test.

We prepared triangular chevron-notched micro-cantilevers, Fig. 1, by FIB milling the flat surface of the same fused quartz prism as used in Ref. [9]. To avoid charging effects during milling, the fused quartz surface was first coated by an ~10 nm carbon layer using a Cressington™ 208 Carbon Coater (Watford, UK). Shaping of triangular cantilevers

* Corresponding author.
E-mail address: goran.zagar@epfl.ch (G. Žagar).

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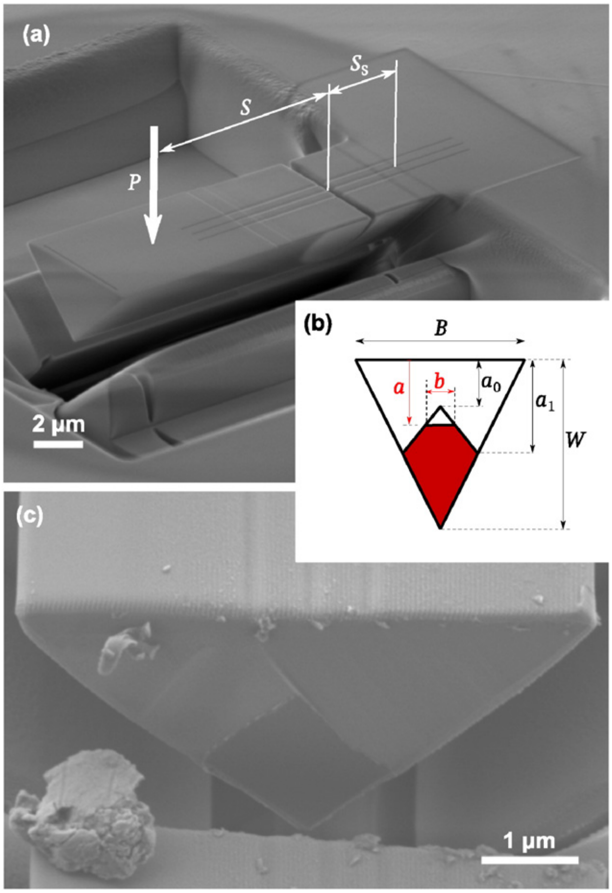

Fig. 1. (a) Triangular micro-cantilever beam with the chevron notch, as prepared on a flat fused quartz surface by FIB milling. (b) Sketch of chevron notch geometry with characteristic ligament dimensions. The fractured surface of a chevron ligament is assumed to be triangular. Shaded region (in red) represents the unfractured portion of the ligament. (c) Fracture surface of a chevron-notch ligament.

was done with a 30 kV Ga^+ source in a Zeiss™ NVision™ 40 (Oberkochen, Germany) dual beam instrument with currents of ~0.3 nA in final machining stages. The most important milling step is the final stage, in which the chevron-notch and its ligament are produced. To make the notch as thin as possible, milling was done using a low ion current, 10 pA, and in a way such that the ligament apex is located in the plane of cantilever symmetry roughly at one-half the total height of the triangular cross-section.

As in our previous work [9], each sample was modeled using finite element simulation to extract its compliance calibration curve. For convenience and efficiency of finite element analysis, the chevron notch was generally offset at some distance (S_S) away from the cantilevers' fixed end.

Machined chevron-notched cantilevers were fractured using a nanoindentation instrument (TriboIndenter TI950, Hysitron Corporation, Minneapolis, MN, USA) equipped with a cube corner probe (tip radius of ~100 nm). Vertical force P was applied in the center of the top cantilever surface at distance S from the notch, Fig. 1a. All tests were done at room temperature and 20–50% relative humidity in load-controlled mode at loading rates around 2 to 3 μN/s. Before and after each test, the cantilever specimen was analyzed by Scanning Electron Microscopy (SEM) in order to: measure all relevant specimen dimensions (Table 1), confirm that the ligament was properly fabricated and ensure that fracture took place within the notch, Fig. 1c. The actual load application point in each test was determined by observation of broken cantilevers SEM micrographs, using the imprint left by the nanoindenter probe. Six specimens were tested in this way; of these, two were dismissed as being invalid because of an irregularly machined ligament, and one due to a lack of detectable stable crack propagation in the load-displacement curve. Thus, results presented here are from the three remaining successful tests.

Table 1
Geometrical dimensions (as defined in Fig. 1), experimental loading rates $\dot{P}$, critical (peak) loads P_c, minimum of the geometrical function $F_v(\tilde{a}_c)$ at normalized critical crack length $\tilde{a}_c$ and calculated fracture toughness K_{Ivb} for triangular chevron-notched micro-cantilevers prepared from fused quartz.

Sample no.	W μm	B μm	a_0 μm	a_1 μm	S μm	S_S μm	$\dot{P}$ μN/s	P_c μN	$\tilde{a}_c$ –	$F_v(\tilde{a}_c)$ –	K_{Ivb} MPa $m^{1/2}$
1	3.6	5.9	2.0	2.8	8.2	2.2	2	16.7	0.635	435.9	0.658
2	2.7	4.6	1.2	1.9	8.7	2.0	3	15.7	0.546	315.7	0.660
3	3.5	6.1	1.3	2.5	10.3	3.2	3	31.7	0.481	244.3	0.685

Load–displacement responses of those three microfracture tests are shown in Fig. 2. Responses in Fig. 2a are corrected for the effect of indentation, by assuming that the displacement measured directly in the test is the sum of the cantilever vertical deflection and the average of two to three measured displacements at load P in conventional nanoindentation tests that were conducted within the (FIB-affected) region of the bulk fused quartz prism close to the fixed end of each cantilever.

Each successful test response featured three different regions. The first is a linear region that represents the elastic cantilever downward deflection free of crack growth. This region extends up to the point where the concentrated tensile stress normal to the notch plane at the apex of the chevron ligament initiates a crack. Crack initiation in fused quartz specimens can be a smooth process; this was the case for two specimens, Fig. 2a (squares and circles). The linear response then continuously transits into a second, nonlinear, region, in which stable crack propagation occurs, downwards through the ligament. It was also found that crack initiation can be accompanied by a "pop-in" event; this was clearly visible for the third specimen (triangle in Fig. 2a).

Once initiated, the crack traverses the notch ligament; we define the crack length a using the top of the cantilever beam as the origin, Fig. 1b. Assuming that the crack front remains straight and symmetrically situated at all times up to position $a = a_1$ defined in Fig. 1b, the crack front width b is simply given by $b(a) = B(1 - a_1/W)(a - a_0)/(a_1 - a_0)$, where B, W, a_0 and a_1 are defined in Fig. 1b. As is well known, this increasing front width b serves to stabilize crack growth because it causes the elastic strain energy release rate: $G = P^2/(2b) \times dC/da$ where dC/da is the change of the specimen compliance C with crack length a, to initially decrease with increasing a.

For brittle, linear elastic materials and under the condition of plane strain (which is commonly assumed to hold for cracks in chevron notched specimens), the stress intensity factor is $K_I = \sqrt{GE'}$, with $E' = E/(1 - \nu^2)$ and E and ν the Young's modulus and the Poisson's ratio of the material, respectively. The stress intensity factor can thus be expressed as $K_I = P/(B\sqrt{W}) \times F_v(\tilde{a})$, where the dimensionless geometrical function is defined in terms of the normalized crack length $\tilde{a} = a/W$ as:

$$F_v(\tilde{a}) = \sqrt{\frac{1}{2\left(1-\tilde{a}_1\right)} \frac{\tilde{a}_1 - \tilde{a}_0}{\tilde{a} - \tilde{a}_0} \frac{dC_v}{d\tilde{a}}}. \quad (1)$$

In Eq. (1) the normalized chevron notch geometrical parameters are $\tilde{a}_0 = a_0/W$ and $\tilde{a}_1 = a_1/W$, while the dimensionless compliance of the specimen is $C_v = CBE'$.

As in our previous work [9], the compliance calibration curve, $C_v(\tilde{a})$, of each sample was calculated by bespoke linear elastic finite element (FE) analysis of the sample knowing its (measured) characteristic dimensions, given in Table 1. For each test specimen, a series of thirty FE models was generated in a such a way that the crack length over the series is progressively incremented over the range $a_0 \le a < a_1$, i.e.