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Fracture toughness testing of nanocrystalline alumina and fused quartz using chevron-notched microbeams

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Abstract—We show that chevron-notched samples offer an attractive approach to the measurement of fracture toughness in micron-scale samples of brittle materials and use the method to characterize quartz and nanocrystalline alumina. Focused ion beam milling is used to carve bend bars of rectangular cross-section a few micrometres wide and containing a notch with a triangular ligament. Load-controlled testing is conducted using a nanoindentation apparatus. If the notch is appropriately machined, cracks nucleate and propagate in a stable fashion before becoming unstable. Sample dimensions are measured using a scanning electron microscope, and are used as input in finite element simulations of the bars' elastic deformation for various crack lengths. The calculated compliance calibration curve and the measured peak load then give the local fracture toughness of the material. Advantages of the method include a low sensitivity to environmental subcritical crack growth, and the fact that it measures toughness at the tip of a sharp crack situated in material unaffected by ion-milling. The approach is demonstrated on two materials, namely, monolithic fused quartz and nanocrystalline alumina Nextel™ 610 fibres; results for the latter give the intrinsic grain boundary toughness of alumina, free of grain bridging effects.

Keywords: Toughness; Focused ion beam (FIB); Finite element analysis (FEA); Alumina (α-Al_2O_3); Fused quartz

1. Introduction

Testing for fracture toughness is inherently difficult. Test samples must be produced with a sharp, weakly preloaded crack of well-defined shape and length. Various complications can also arise: subcritical crack growth processes can cause premature failure, crack tip plasticity can throw data beyond the range of linear elastic fracture mechanics and *R*-curve behaviour can imply that the material's resistance to crack propagation cannot be characterized by a single-valued fracture toughness. When the test must be conducted on very small samples, several of those difficulties are exacerbated. Satisfying the requirements for small-scale plasticity is generally more of a challenge, even though the yield stress of very small metal samples is often higher than in the bulk. Precracking is also more difficult: machining a sufficiently sharp starting notch in small samples is not trivial, while propagating such a notch in fatigue is also a challenge. Nevertheless, knowing the toughness of small-scale samples is important because it governs the link between their strength and their structure. Extensive work and significant progress have therefore been accomplished toward quantifying the fracture toughness of materials at the micron scale in microelectromechanical system components [1], thin film materials [2], and individual phases in alloys and composites [3].

The most common approach for the direct determination of fracture toughness at small scales has been the nanoindentation-toughness technique [4]. The method, although widely applied because of its experimental simplicity, has been subject to criticism [5]; also, producing appropriate indentation cracks in thin films may be difficult [6], and cracking patterns can be too irregular for interpretation [3]. Other approaches that use samples free of initial cracks or notches include experiments in which cracks appear in small samples of simple shape (spheres or cylinders) under uniaxial compression [7–9] or observations of tunnelling cracks in stacked and bonded thin films subjected to in-plane tensile deformation [6,10].

Micrometric toughness test samples can alternatively be produced using selective microetching or focused ion beam (FIB) micromilling techniques. Testing such samples comes much closer to conventional macroscopic fracture toughness testing practice: here, miniature precracked beams are produced and loaded, often using a nanoindentation

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apparatus, and the fracture toughness is computed from a measurement of the applied load at the onset of rapid crack propagation. Miniature fracture toughness tests come in a variety of configurations; most often, small-scale cantilever beams or tensile samples are produced along a polished surface of the material to be tested. If the material to be tested is a coating or a thin film, photolithography-based selective (plasma or chemical) etching can be used to machine sidewalls of the beam, which is then freed from its substrate by etching the latter selectively [2]. If the material is not a thin film or a coating, toughness test samples can be carved out of equiaxed material samples entirely by FIB milling. Microscopic FIB-notched cantilever [11] or double cantilever beam samples [12] have been produced in this way.

The greatest challenge is most often to create a precrack in such samples. Early attempts (reviewed in the introduction of Ref. [13]) used samples having relatively wide prenotches, roughly 1 μm or so wide, instead of precracks. This led to grossly exaggerated K_c values. Nowadays, prenotching is often done by FIB milling, using a low-intensity beam in the final stages of the process so that the tip of what is, in fact, a milled notch will be made as small as possible. The radii of the resulting notch roots range from a few tens to several hundreds of nanometres (e.g. [11,14–31]). Beyond the need to produce a notch of sufficient sharpness, another difficulty with ion milling lies in producing a uniform notch depth and/or width: for this reason, in Refs. [16,32] the prenotch was machined straight down in the central part of the sample only, leaving two side walls that formed a precrack when bend-testing thin film samples of silicon oxide, nitride or oxynitride. Testing of small-scale beams containing FIB-premachined notches has been shown in several studies to give K_c values near those found for macroscopic samples [15–17,26,27]; however, in many other studies, different results, ranging from values slightly to much higher [11,18,19,21–24,30,33], or in some cases lower [20,34], than the toughness data from tests on macroscopic specimens of the same material were obtained with FIB-notched specimens.

The obvious disadvantage of this method is thus that, failing a post-test comparison of notched microsample test data with results from valid tests conducted on macrosamples, there is little way of knowing a priori that test data were not biased by the initial bluntness or other defects of the micromachined prenotch. Another important disadvantage, which is also shared with earlier etch-based notching methods [13], is that the nature and morphology of the notch surface, which will often play an important role in fracture initiation, may be affected by the notch machining process. FIB milling is indeed well known to cause significant implantation and irradiation damage, and also to redeposit removed material along the periphery of the beam trajectory.

These pitfalls of notched vs. precracked toughness samples have motivated the development of other approaches. In one, the microsample precrack is made by a fracture process that produces, before the microsample is machined, a precrack of relatively well-controlled depth. In Ref. [35], such precracks were produced by machining microsamples into one fracture surface of a larger previously fractured specimen, using sidecracks as precracks. In Ref. [36], internal defects, the size of which was deduced by post-test fractography, were used as precracks. Use has also been made at times of the presence of internal planes of lowered fracture energy (interfaces or embrittled grain boundaries) to nucleate and guide the crack [35]. Probably the most elegant method in this vein is that demonstrated initially by Kahn et al. [13] and subsequently used by several other laboratories, in which thin films are precracked using a hardness indenter before being etched and separated from their underlying substrate, with a portion of the precrack remaining in the etched thin-film test specimen. In this way, tensile or bend specimens amenable to testing could be produced. Once the method was perfected, these often gave data consistent with data from macroscopic tests of the same material (Si notably) [2,13,19,37–40]. Finally, some authors have used fatigue of notched microspecimens to create precracks in metallic specimens (prone to large-scale yielding, however) [41–43], and also in silicon [44].

Chevron-notched samples, which have a triangular ligament across a thin notch in a bending beam, are an interesting alternative to precracked fracture specimens. The tip of the triangle is the point of maximum tensile stress across the loaded specimen. If, at sufficiently low load, a crack initiates at this tip, since the crack front width increases as the crack advances through the triangular ligament, initial phases of crack growth are mechanically promoted to occur in stable fashion, also under increasing controlled load. This continues until a point is reached where the relative rate of increase in the crack front width can no longer compensate for the increase in the global elastic energy release rate G caused by the increasing average crack length. At this point the crack propagation becomes unstable and the sample breaks suddenly in two. In the absence of significant plastic deformation, and with a relatively constant toughness (meaning with no R-curve behaviour), the point at which fracture becomes unstable is entirely determined by the sample geometry, such that the fracture toughness can simply be computed from the peak load that is measured. The method is also often practised on millimetre-scale specimens (e.g. [45–47]), and it is consigned in ASTM standards [48,49].

We show here that chevron-notched specimens provide an attractive strategy for the measurement of fracture toughness in micron-scale samples of brittle materials. By definition, the method obviates the need for precracking, yet it measures toughness using a real crack. Furthermore, with the fracture toughness being computed after a finite amount of crack growth has occurred, the potential influence of milling-induced irradiation, redeposition or implantation damage is absent, since most of the crack front is located far from the machined surface in such specimens. In what follows, we show how the chevron-notch fracture test method can be scaled down to the micron scale and that it gives reproducible fracture toughness measurements in both fused quartz and nanocrystalline alumina.

2. Materials and methods

2.1. Methodology

The chevron-notched test bar of this study is a rectangular cantilever beam, of cross-section $W \times B$ and length L. It has a thin notch with a triangular ligament, the apex of which is nominally situated in the middle of the cross-section. Depending on the notch parameters a_1 and b_1, the notch is overcut if $b_1 = B$ or undercut if $a_1 = W$ (see Fig. 1). In macroscopic samples, these geometrical differences are easily controlled; the standard for measuring