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[1] Östlund, F., P. R. Howie, R. Ghisleni, S. Korte, K. Leifer, W. J. Clegg and J. Michler (2011). Philosophical Magazine, 91(7-9): 1190-1199 [2] Di Maio, D. and S. G. Roberts (2005). Journal of Materials Research 20(02): 299-302 [3] Armstrong, D. E. J., A. J. Wilkinson and S. G. Roberts (2011). Philosophical Magazine Letters, 91(6): 394-400 [4] Liu, S., J. M. Wheeler, P. R. Howie, X. T. Zeng, J. Michler and W. J. Clegg (2013). Applied Physics Letters 102(17): 171907 [5] Lawn, B. R. (1993). Cambridge, Cambridge University Press, 36-38.

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IN SITU FRACTURE TESTS OF BRITTLE MATERIALS AT THE MICROSCALE

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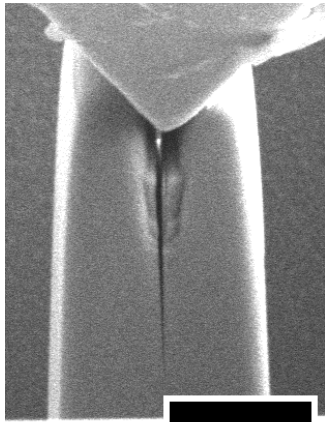
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The fracture toughness of ceramics is often dominated by the structure of their grain boundaries. Our ability to improve life of ceramic components depends on our ability to investigate properties of individual grain boundaries.

This requires development of new fracture testing methods allowing high spatial resolution and high control over the area to test. Further benefits of these 'small scale' approaches will enable testing of specimens for which big volumes are not available (e.g. thin films, coating, or simply samples of dimensions limited by production process).



2 μm

Figure 1 – Frame extracted from a video recorded by an Auriga SEM during the in situ fracture testing of a single crystal SiC DCB

Recently, several techniques have been developed using small scaled mechanical testing, based within a nanoindenter, changing tip and sample geometries, including: micropillar compression [1]; microcantilever bending [2,3]; and double-cantilever compression [4]. However, the majority of the published works utilises complex geometries resulting into complex analysis of force distribution and stress intensity factor and rely on load-displacement curves for the identification of crack initiation, with the added complication of friction.

Our approach builds upon the work of Lawn [5], who showed that a practical test geometry to obtain stable crack growth and calculate the fracture energy G is that of a double-cantilever beam (DCB) under constant wedging displacement. We replicate this configuration in our tests fabricating double-cantilever beams of micrometric dimensions by focused ion beam (FIB) milling and loading them in-situ in an SEM using a nanoindenter with a wedge-shaped tip. This has two benefits: the sample is well aligned for a controlled test; images are recorded during the test for later analysis. This allows us to use beam deflection and crack length rather than critical load to measure fracture toughness. Our tests have

proved it is possible to initiate and stably grow a crack in a controlled manner in ceramic materials (fig. 1) and our fracture energy results have been validated against prior macro-scale fracture data. This approach is being extended to multi-phase materials with unknown materials properties and extends our arsenal of small-scale characterisation techniques required to generate new processing strategies for the next generation of materials design.

References

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