1746
doi:10.1017/S1431927616009570

Microsc. Microanal. 22 (Suppl 3), 2016

Temperature Dependence of Fracture Initiation in Silicon from *In-situ* SEM

Eric Hintsala[1], Sanjit Bhowmick[1], Xie Yueyue[2], Roberto Ballarini[2], William Gerberich[3]

[1.] Hysitron, Inc., Eden Prarie MN, USA
[2.] University of Houston, Department of Civil Engineering, Houston TX, USA
[3.] University of Minnesota, Department of Chemical Engineering and Materials Science, Minneapolis MN, USA

Silicon has a rich history of technological importance, as well as serving as an ideal model material for studying mechanical behavior in semi-metallic materials. Of particular interest is the rapid transition in deformation mechanisms as a function of scale and temperature, where such concepts as dislocation character[1], nucleation/propagation control[2] and possible core structure[3] changes may contribute. The result is that under certain loading conditions, sizes, temperatures and doping, silicon can display a wide variety of response from highly brittle cleavage to over 50% plastic strain[4].

Improved understanding of silicon properties becomes increasingly important as nanotechnology continues to develop, from MEMS/NEMS devices to electronics. As these devices are scaled down and designs are pushed further in search of greater performance, the need for predictability increases. To this end, we have pursued novel microscale fracture testing as a function of temperature, which would represent one piece of what may one day become a complete view of the mechanical behavior of silicon as a function of all the relevant variables.

Silicon wedge substrates, commercially available from Hysitron, were utilized as the starting material. These wedges were fabricated using photolithography and wet etching from a B-doped wafer to produce a 1um thick minimum top thickness and 30um height. Using focused ion beam (FIB) milling through a multi-step process at 30keV, doubly-clamped, pre-notched bending beams were fabricated. The specimen shape was finalized with 2deg tilt into the side wall to reduce natural FIB tapering and possessed a nominal geometry of 10um in span, 2um in width and 1um in thickness, see Fig. 1a. The pre-notch was FIB fabricated as the final step, and targeted to traverse 33% of the bending beam width. This geometry has numerous advantages in terms of self-stabilization of the cracking process due to a decreasing stress intensity at the crack tip as it progresses across the specimen, along with high visibility of the cracking process.

Testing was performed utilizing a Hysitron PI-87 SEM PicoIndenter operating inside an FEI Versa 3D FIB/SEM operating at 20keV in secondary electron mode. Hysitron's new high temperature system was utilized which incorporates independently feedback controlled resistive probe and sample heaters at room temperature, 150C, 300C, 450C and 600C as shown in Fig. 1b. All specimens were annealed at the maximum temperature of 600C for one hour before testing and care was taken to allow stabilization with temperature changes to minimize drift. These specimens were loaded using a wedge geometry diamond tip with a 1um flat in displacement control at a loading rate of 10nm/s. Because of the self-stabilization of the cracking, the stress fields in the bending beam are of complexity required finite element analysis, which was performed to extract an applied stress intensity for initiation of cracking from the FIB pre-notch.

The results of this testing, shown in Figure 2a, demonstrate that the room temperature fracture testing corresponds well to other microscale measurements of fracture toughness in silicon. As the temperature is increased, initially the load for crack initiation decreases with an corresponding decrease in the applied stress intensity factor and is at a minimum in the vicinity of 150-300C. The load and applied stress intensity then increases at 450 and 600C, with a dramatic change in deformation mechanism at 600C. Here, it appears that instead of typical brittle crack advance, the bending beam becomes fully plastic, with significant permanently retained opening after unloading (Fig. 2b).

It seems likely that some transition in deformation mechanisms is responsible for the observed trends. More detailed investigation into this will require using of pillar compression and post-mortem TEM analysis to guide analysis.

References:

[1] S. Korte, J. S. Barnard, R. J. Stearn, and W. J. Clegg, Int. J. of Plast. **27** (2011) 1853-1866.
[2] K. Kang, and W. Cai, Int. J. of Plast. **26** (2010) 1387-1401.
[3] J. Rabier and J. L. Demenet, Scripta Mater. **45** 65 (2001) 1259-12.
[4] A. J. Wagner, E. D. Hintsala, P. Kumar, K. A. Mkhoyan and W. W. Gerberich, Acta Mater. **100** (2015) 256-265.
[5] E. D. Hintsala, C. Teresi and W. W. Gerberich, Met. Trans. A (submitted).

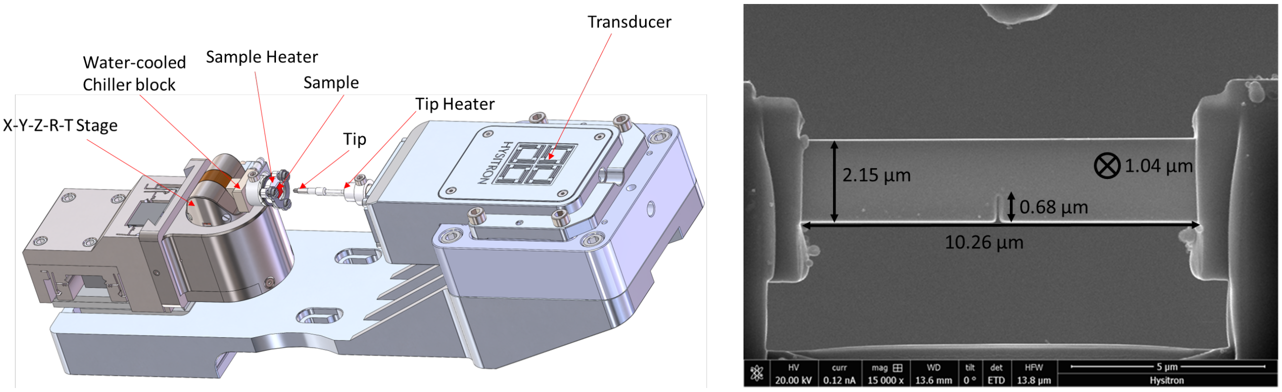

Figure 1. a) Schematic of high temperature capable Hysitron PI-87 PicoIndenter system; b) SEM micrograph of an as-fabricated microscale bending beam, with dimensions marked.

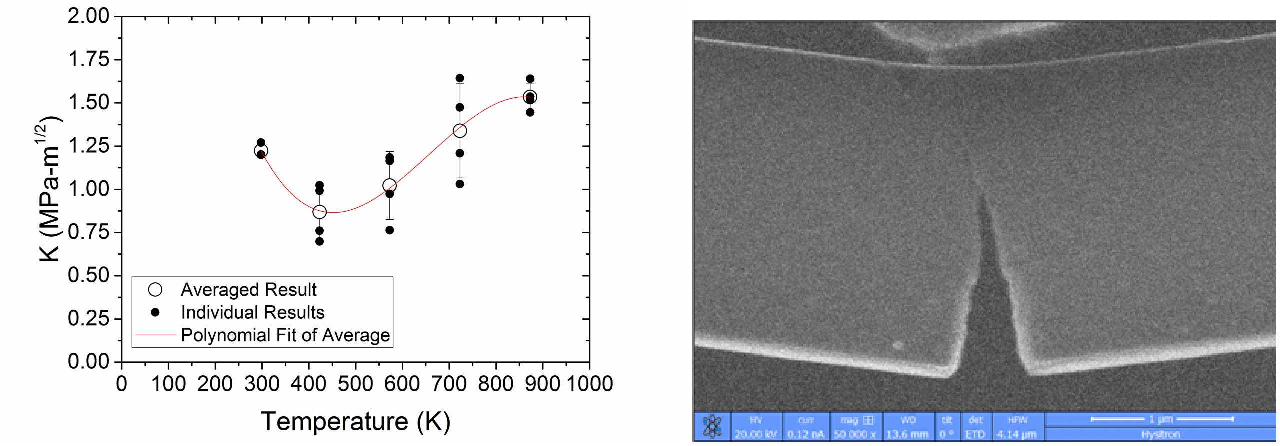

Figure 2. a) Applied stress intensity factor for crack initiation in Si versus test temperature; b) A bending beam post-testing at 600C, showing a large amount of residual bending.