

***In-situ* Fracture Toughness of Single Crystal Silicon Double-Cantilever Beams**

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Recent studies have utilized scanning electron microscopy (SEM) to quantify fracture toughness on Si(100), [1, 2] because SEM is capable of imaging the fracture path with excellent resolution. Additionally, these studies found that of four different in-situ methods tested (cantilever beam bending, clamped beam bending, double cantilever beam (DCB) compression, and micropillar splitting) all produced similar plain-strain fracture toughness values ($K_{IC} \approx 0.8 \text{ MPa m}^{1/2}$). Here we report on a fifth in-situ geometry, the DCB wedging method, which allowed us to also examine the impacts of stress corrosion, notch geometry, and ion damage on the precision of toughness measurements.

The DCB samples were fabricated utilizing a Thermo Fisher Helios G5 FIB using a 30kV accelerating voltage and Si (110) and (100) wafers, resulting in approximate DCB dimensions of length $l=10 \mu\text{m}$, width $w=2 \mu\text{m}$, thickness $t=5 \mu\text{m}$, notch length $l_n=0.8 \mu\text{m}$, notch widths $w_n=0.5 \mu\text{m}$, and initial crack lengths $a_0=0.3 \mu\text{m}$.

Electron Backscatter Diffraction (EBSD) was used to verify the crystallographic orientation of the fabricated DCBs. This measurement was acquired using an Oxford Symmetry Detector in a Zeiss Supra 55-VP Field Emission SEM. All EBSD was performed using an accelerating voltage of 20 keV and a 120 μm aperture, with high current mode enabled on samples tilted 70° with respect to the electron beam. Images of two of the DCBs examined, on a (100) and (110) wafer, are presented in Figure 1. Confirmation of desired orientation is demonstrated by the pole figures, shown in Figure 2.

In-situ toughness measurements were performed using a Hysitron PI 89 PicoIndenter equipped with sample tilt and rotation stage for tip-to-specimen alignment within a JEOL JSM-IT500HR SEM and on a Thermo Fisher (formerly FEI) Versa 3D. DCB testing was performed at a displacement rate of 1-2 nm/s using an electrically conductive 70° diamond wedge tip with a 10 μm nominal length. The tip displacement d and force F were controlled and measured through the system piezoelectric actuator and force transducer, while beam displacement d and crack length a were evaluated as a function of time t .

In both orientations of DCB tests, the crack started at the FIB notch and exhibited stable growth (which was demonstrated via small displacement bursts) along the {111} plane. Two new smooth surfaces were produced; no evidence of deviation from a linear path was observed. These efforts, in combination with finite element analysis (FEA) modeling, resulted in computation of $K_{IC}=0.7\text{--}0.9 \text{ MPa m}^{1/2}$ and $\psi=0.017\pm 0.001$ for Si, both of which are in excellent agreement with previous work [3].

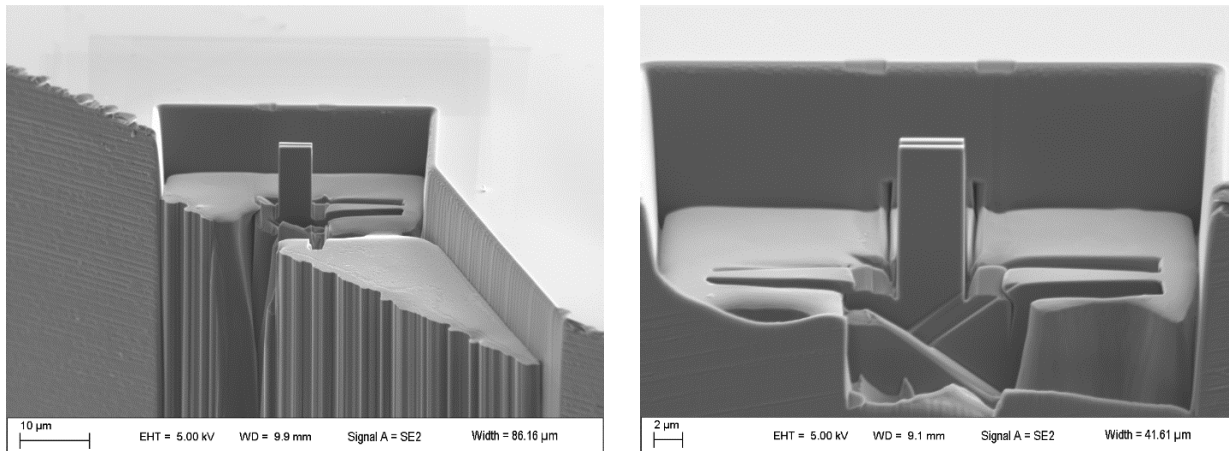


Figure 1. Fabricated DCBs (left) on a (100) wafer and (right) on a (110) wafer.

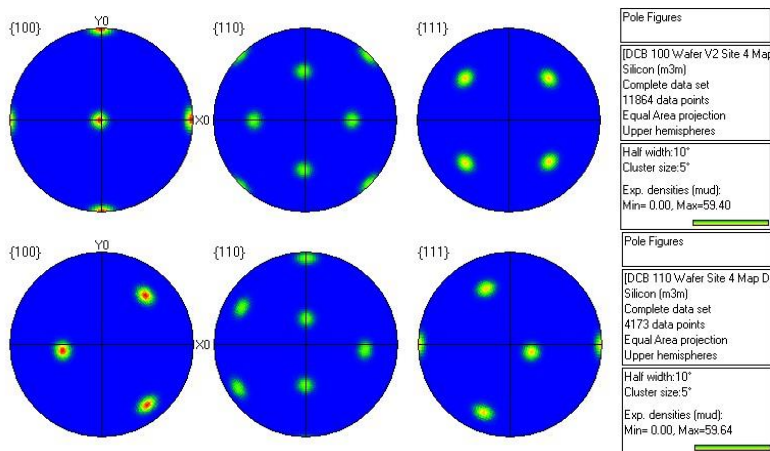


Figure 2. Orientation of DCBs presented in Figure 1, where the top pole figure corresponds to the DCB on the (100) wafer and bottom pole figure corresponds to the DCB on the (110) wafer.

References:

- [1] BN Jaya, C Kirchlechner and G Dehm, *J. Mater. Res.* **30** (2015), p. 686.
- [2] ED Hintsala et al., *Scr. Mater.* **130** (2017), p. 78.
- [3] This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525 SAND2022-2060 A