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To cite this article: Frank W. DelRio, Scott J. Grutzik, William M. Mook, Sara M. Dickens, Paul G. Kotula, Eric D. Hintsala, Douglas D. Stauffer & Brad L. Boyce (2022) Eliciting stable nanoscale fracture in single-crystal silicon, Materials Research Letters, 10:11, 728-735, DOI: [10.1080/21663831.2022.2088251](https://doi.org/10.1080/21663831.2022.2088251)

To link to this article: <https://doi.org/10.1080/21663831.2022.2088251>



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Published online: 29 Jun 2022.



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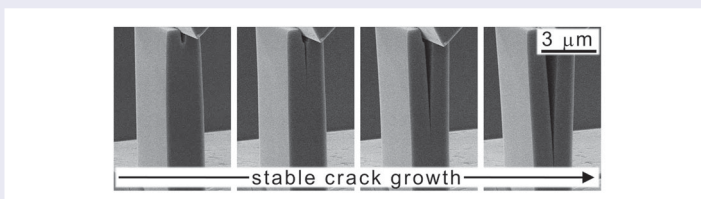
Eliciting stable nanoscale fracture in single-crystal silicon

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ABSTRACT

In this letter, we demonstrated stable nanoscale fracture in single-crystal silicon using an in-situ wedge-loaded double cantilever beam (DCB) specimen. The fracture toughness K_{IC} was calculated directly from instrumented measurement of force and displacement via finite element analysis with frictional corrections. Measurements on multiple test specimens were used to show $K_{IC} = 0.72 \pm 0.07 \text{ MPa m}^{1/2}$ on {111} planes and observe the crack-growth resistance curve in < 500 nm increments. The exquisite stability of crack growth, instrumented measurement of material response, and direct visual access to observe nanoscale fracture processes in an ideally brittle material differentiate this approach from prior DCB methods.



IMPACT STATEMENT

In-situ fracture toughness experiments on single-crystal silicon revealed stable nanoscale fracture and identified the shape of the crack-growth resistance curve, role of fabrication-induced artifacts, and origin of fracture surface features.

ARTICLE HISTORY

Received 24 March 2022

KEYWORDS

toughness; single-crystal silicon; double cantilever beam; finite element analysis; fractography

Introduction

Silicon microelectromechanical systems are often fabricated via bulk and surface micromachining techniques, and as such, possess a number of flaws of varying scale, geometry, and residual stress state, depending on the fabrication method and mechanical environment [1]. In the presence of a mechanical load, these preexisting flaws become sites for stress concentrations [2–4], thus making fracture toughness a key metric for component reliability. Historically, fracture toughness has been measured via ex-situ methods such as scratch [5], indentation [6], bulge [7], and stressed overlayer [8] tests. More recently, toughness has been evaluated in-situ in a scanning electron microscope (SEM) to visualize the fracture path and extract multiple values from each specimen [9]; the test geometries and loading types have included cantilever beam bending [10], clamped

beam bending [11], double cantilever beam (DCB) compression [12], micropillar splitting [13], DCB wedging [14], chevron-notch triangular cantilever beam bending [15], and bow-tie clamped beam bending [16]. A recent case study on silicon revealed that the first four in-situ methods resulted in similar fracture toughness values ($K_{IC} \approx 0.8 \text{ MPa m}^{1/2}$) for Si(100) and highlighted the advantages and limitations of each geometry [17]. In this letter, we conduct a similar study on single-crystal silicon with the fifth in-situ geometry, the wedge-loaded DCB specimen. The rationale of this scheme is two-fold: (1) to improve the accuracy of the approach through the inclusion of contact friction and material anisotropy and the assessment of K_{IC} from force-displacement data (previous studies have neglected these effects and calculated K_{IC} based on crack lengths or beam deflections from SEM images [14,18]) and (2) to provide further

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 Supplemental data for this article can be accessed here. <https://doi.org/10.1080/21663831.2022.2088251>

insight into the small-scale fracture behavior of silicon through an appraisal of the crack-growth resistance curve and fracture surfaces during stable crack growth (previous work has recognized the challenges associated with stable fracture in Si and thus an assessment of such properties [15]).

Materials and methods

DCB specimens were fabricated from Si(110) wafers along the {111} planes via focused ion beam (FIB) milling. The milling was performed in a ThermoFisher Helios G5 FIB at 30 kV in four main steps: (1) bulk trenching, (2) notch formation, (3) shaping, and (4) polishing. In (1), the bulk trench was milled at 20 nA to a depth of 10 μm , leaving a 5 μm $\times$ 10 μm pillar. In (2), the ‘dull’ notch for wedge loading and ‘sharp’ notch for crack propagation were milled at 90 and 26 pA, respectively. In (3), the specimens were shaped to 2 μm $\times$ 5 μm at 400 pA with each sidewall rotated 1.5° into the ion beam to minimize taper. In (4), the final polish was conducted at 26 pA. Nominally, the process resulted in rectangular prismatic specimens with lengths $l = 10 \mu\text{m}$, widths $w = 2 \mu\text{m}$, thicknesses $t = 5 \mu\text{m}$, notch lengths $l_n = 0.8 \mu\text{m}$, notch widths $w_n = 0.5 \mu\text{m}$, and initial crack lengths $a_0 = 0.3 \mu\text{m}$. The specimen size was large enough to facilitate tip-specimen engagement and crack tip imaging, but small enough to minimize FIB time and fracture loads. Electron backscatter diffraction (EBSD) was conducted on a Zeiss Supra 55-VP field-emission SEM at an accelerating voltage of 20 keV using a 120- μm aperture and 70° stage tilt. Scanning transmission electron microscopy (STEM) was performed on an FEI Company (now Thermo Fisher Scientific) Titan G2 80–200 operated at 200 keV and equipped with a spherical aberration corrector on the probe-forming optics and 4 silicon-drift X-ray detectors. High-angle annular dark-field (HAADF) STEM images were calibrated from the Si-lattice spacings (sample oriented on the [112] zone axis) to show amorphous and crystalline Si. X-ray maps were collected from the specimen to delineate Si, Ga, and Pt. The in-situ measurements were achieved on a Hysitron PI-89 PicoIndenter equipped with a sample tilt and rotation stage for tip-to-specimen alignment inside a ThermoFisher Versa 3D SEM. DCB testing was performed in displacement control at a rate of 1–2 nm/s (displacement rate effects are minimal in this range [19]) 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 using the system piezoelectric actuator and force transducer, while beam displacement

δ and crack length a were evaluated as a function of time t .

The F and d data for each DCB specimen were used to calculate K_{IC} through finite element analysis (FEA). Three software packages were utilized: (1) Sandia CUBIT [20] to generate the DCB geometry and mesh, (2) Fracture Analysis Consultants FRANC3D [21] to alter the mesh to insert a crack, and (3) Sandia Sierra/SolidMechanics [22] to perform the calculations. The model used an idealized geometry of sharp corners and 90° angles, but with dimensions matching the average measured dimensions from several real specimens. Since in this idealized geometry, contact between the indenter wedge and DCB specimen occurred uniformly along linear contact regions, the indenter tip and machine compliance were not explicitly included in the FEA model. Instead, the indenter force F was decomposed into a compressive force $F_{comp} = F/2$ in the axial direction and a crack opening force $F_{open} = (F/2)[(\cos\alpha - \mu\sin\alpha)/(\sin\alpha + \mu\cos\alpha)]$, where α is the wedge half angle (here, $\alpha = 35^\circ$) and μ is the friction coefficient. Coulomb friction was assumed between the indenter tip and specimen, with $\mu = 0.12$ [23]. F_{comp} and F_{open} were applied along the contact lines at the ends of each DCB leg. Model symmetry in the transverse direction was not assumed, as in general the material may not be symmetric in that direction. Fixed displacement conditions were assumed at the opposite end of the specimen, approximating the attachment of the specimen to the bulk Si material. The elastic properties of Si were taken as cubic anisotropic with three elastic coefficients $c_{11} = 165.8 \text{ GPa}$, $c_{12} = 63.9 \text{ GPa}$, and $c_{44} = 79.6 \text{ GPa}$ [24], with a crystal orientation matching the nominal specimen design.

Results

The wedge-loaded DCB method is depicted schematically in Figure 1(a). Briefly, the wedge-shaped tip was oriented parallel to the notch using fiducial marks in the adjacent Si and then centered over the notch in the horizontal and vertical directions. Then, d was applied and F was measured through specimen failure; the intermediate F - d data consisted of several linear F - d traces and displacement-controlled decreases in F , the latter of which correspond to increases in crack length from a_0 to a_1 to a_2 and thus translate to individual measurements of K_{IC} . High-resolution SEM images were used to characterize the specimen geometries resulting from FIB fabrication as shown in Figure 1(b). The mean values and standard deviations for l , w , t , l_n , w_n , and a_0 were found to be $9.9 \pm 0.3 \mu\text{m}$, $2.3 \pm 0.1 \mu\text{m}$, $5.0 \pm 0.1 \mu\text{m}$, $0.85 \pm 0.01 \mu\text{m}$, $0.45 \pm 0.01 \mu\text{m}$, and $0.25 \pm 0.01 \mu\text{m}$; the

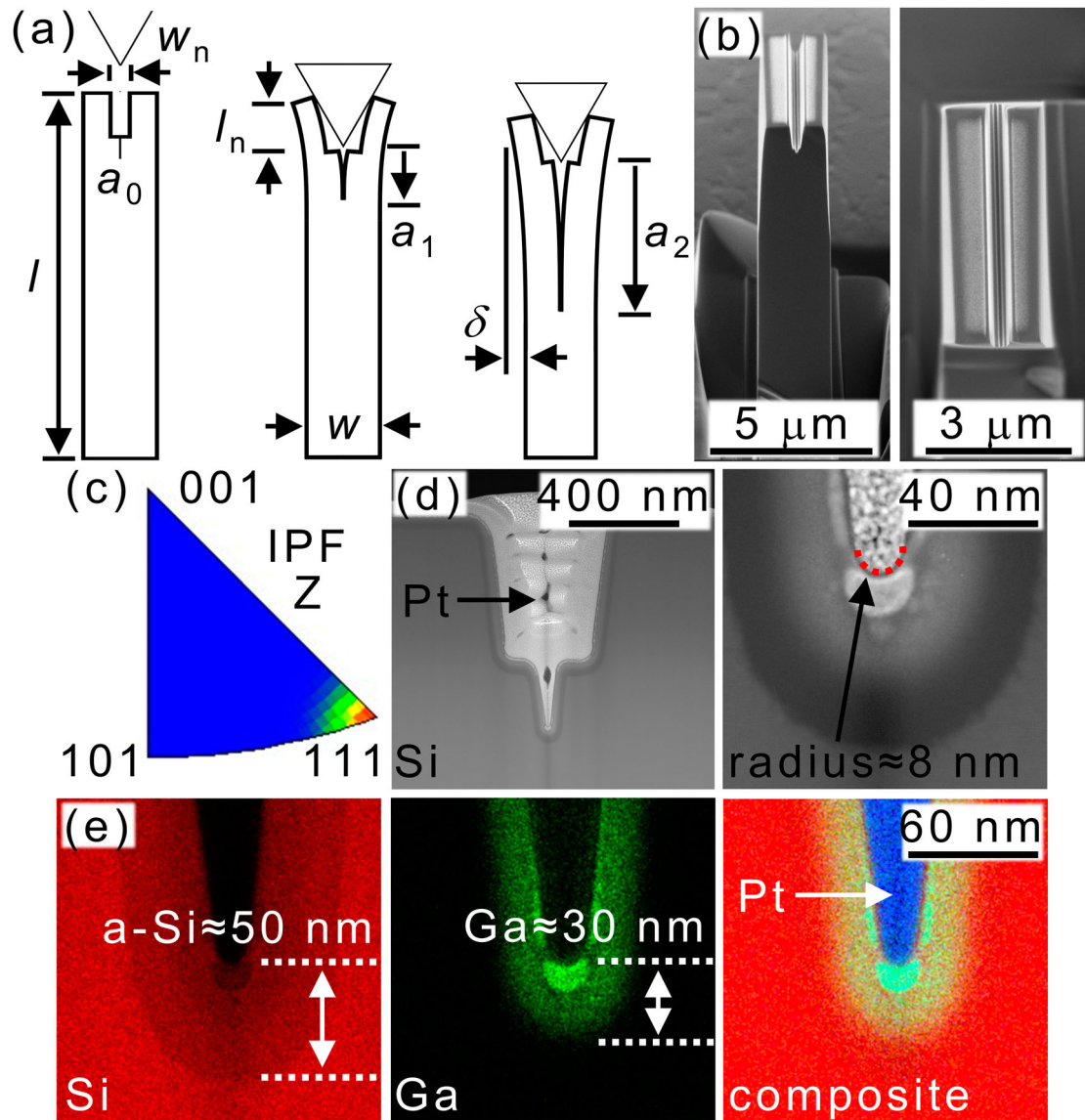


Figure 1. (a) Schematic diagrams and (b) SEM images of a wedge-loaded DCB specimen. (c) EBSD IPF maps were consistent with single-crystal Si and suggested negligible misorientation from the $\{111\}$ fracture planes. (d) STEM images and (e) X-ray maps of a notch region. The images showed a 'sharp' notch radius of ≈ 8 nm and a damaged, amorphized Si layer (a-Si) at the notch base (Ga-rich depth of ≈ 30 nm and total depth of ≈ 50 nm).

mean values were used in the FEA models and the standard deviations suggested minimal specimen-to-specimen variability. EBSD inverse pole figure (IPF) maps were generated to confirm the crystallographic orientation of each specimen set; an IPF map is portrayed in Figure 1(c). IPF maps were consistent with a single crystal, namely uniform orientation with respect to direction and no grain boundaries. Moreover, IPFs for the normal direction were within 6° of the expected $\langle 111 \rangle$ direction, suggesting negligible misorientation from the intended fracture planes. STEM images and X-ray maps of a notch region are shown in Figures 1(d) and 1(e). The images illustrate the two main artifacts of the FIB process. First, the small beam currents

used to create the 'sharp' notch for crack propagation resulted in a notch radius of ≈ 8 nm. Second, the FIB process resulted in a damaged, amorphized layer at the notch base (with a Ga-rich depth of ≈ 30 nm and total depth of ≈ 50 nm) due to Ga implantation at the milled surfaces [25].

SEM images and F - d data for four DCB specimens (denoted tests #1 through #4) are shown in Figure 2. From the SEM images, a were measured as a function of F and d and failure locations and planes were monitored. From the F - d data, failure force F_f and specimen compliance C for each fracture event were calculated for each specimen. A reference force-displacement origin was determined through the intersection of the initial

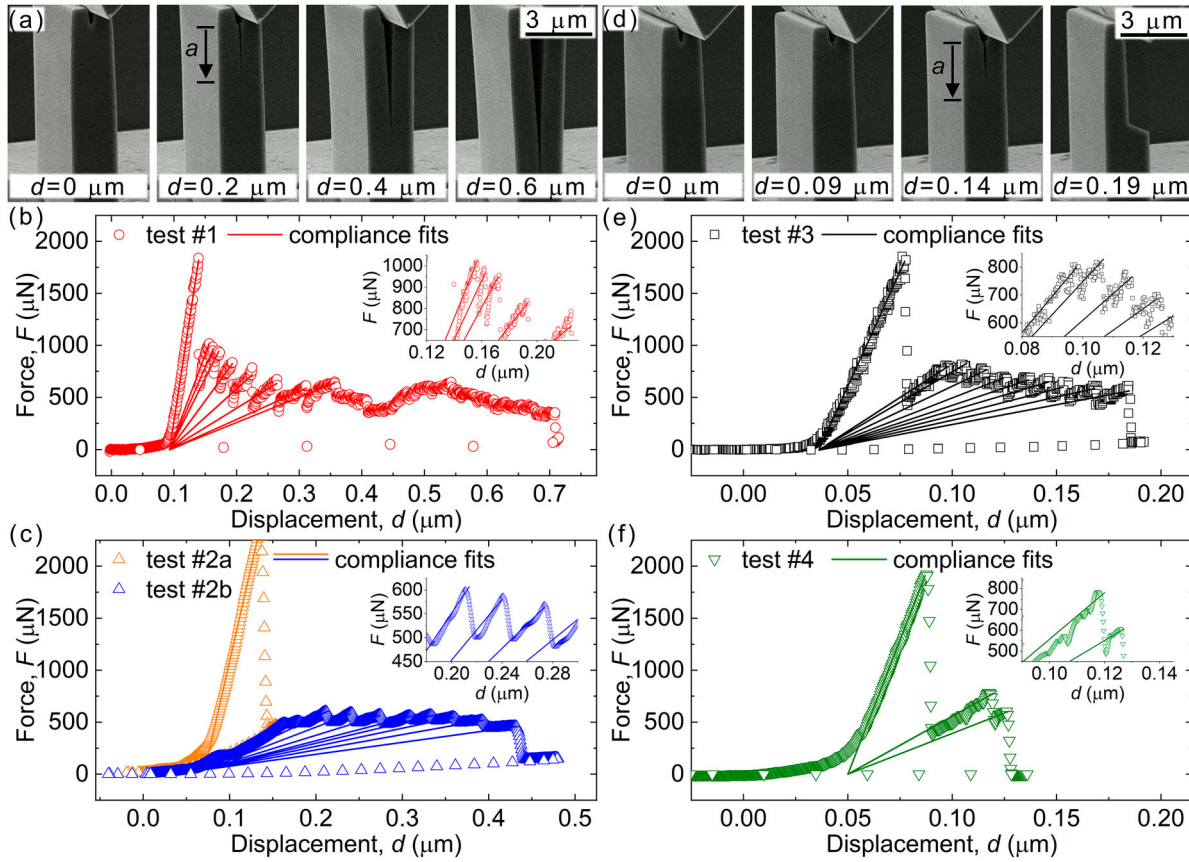


Figure 2. SEM images and F - d data for four DCB specimens (denoted tests #1 through #4). In all tests, the crack started at the FIB notch and exhibited stable growth on a $\{111\}$ plane. Moreover, F_f decreased and C increased with a . However, the SEM images demonstrated that (a, b) test #1 and (c) test #2 ended near the bottom of the samples, while (d, e) test #3 and (f) test #4 ended early. Test #2 included an unload-load cycle after the first fracture event to assess the reversibility of the loading process.

loading slope with the force-free data during specimen unloading. A linear fit was made to the data immediately before each fracture event, with the fit forced through the calculated origin. Note that immediately after each fracture event, F was found to drop beyond the linear stiffness of the next fracture event. It was concluded that the drops in F were due to a temporary loss in contact stiffness as the crack advanced, and as such, were not included in the C calculations. In all tests, the crack started at the FIB notch and exhibited stable nanoscale growth (via small propagation bursts) on $\{111\}$ planes. Two new surfaces were generated, with no evident deviation from a linear path. The initial F_f were large, but monotonically decreased with subsequent fracture events as shown in the Figure 2 insets. Moreover, it was found that C increased with a in agreement with beam theory intuition. However, it is also important to note the differences between the various tests. In particular, the SEM images demonstrate that tests #1 and #2 ended at the bottom of the samples, while tests #3 and #4 ended early. This could imply that the latter tests were subjected to minor mode II loading via tip misalignment, thus

resulting in premature failure on an unintended fracture plane.

FEA contour maps of stress in the loading direction σ_{xx} as a function of a are shown in Figure 3(a). In the model, a starting crack of length $0.5 \mu\text{m}$ was inserted and K_{IC} was calculated along the crack front with a unit indenter load, $F = 1$. Each point on the crack was advanced by a distance proportional to K_{IC}^2 until the crack reached the end of the specimen, resulting in a slightly curved crack front with an approximately-constant K_{IC} at each step. At each step, K_{IC} was assessed from the model and the specimen compliance C was calculated as d/F , where $d = d_{\text{open}}/\tan\alpha$ is the indenter displacement and d_{open} is the opening displacement at the point of force application. The computed set of K_{IC}/F_f and C results were then interpolated with a cubic spline to assign K_{IC}/F_f for all C at a unit indenter load $F = 1$ as shown in Figure 3(b). The calculated K_{IC}/F_f and a were also interpolated to allow comparison to experiments. K_{IC} were then obtained by multiplying the K_{IC}/F_f values by the respective F_f for each fracture event and plotted as a function of the a from FEA as shown in Figure 3(c).

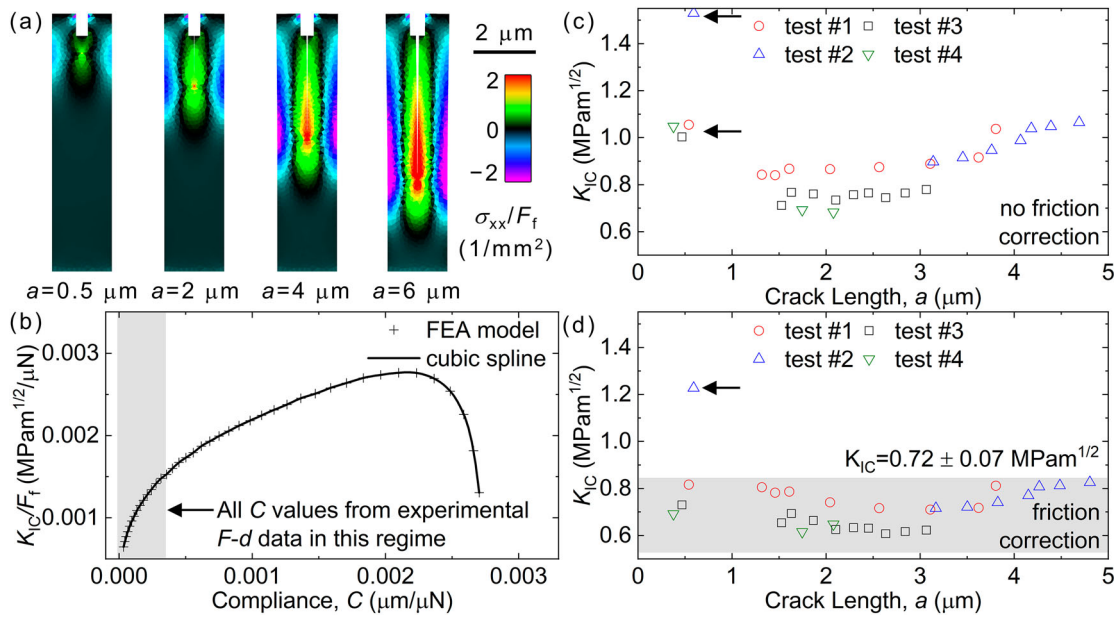


Figure 3. (a) FEA contour maps of σ_{xx}/F_f as a function of a ($a = 0.5, 2, 4$, and $6 \mu\text{m}$). (b) The computed set of K_{IC}/F_f and C were generated using FEA (symbols) and interpolated with a cubic spline (solid line) to assign K_{IC}/F_f for all C . (c) Without friction effects in the analysis, K_{IC} remained constant at $0.7 \text{ MPa m}^{1/2}$ – $0.9 \text{ MPa m}^{1/2}$, but increased at small and large a due to FIB-effects and friction-effects, respectively. (d) After considering friction, the accuracy and precision improved greatly, resulting in a value of $K_{IC} = 0.72 \pm 0.07 \text{ MPa m}^{1/2}$. The arrows in (c) and (d) indicate values that were likely invalid due to proximity to the notch root.

At small a , all four specimens exhibited larger K_{IC} values, likely from geometric and material effects from the FIB. At intermediate a , K_{IC} decreased and remained constant. This is interpreted as a valid, intrinsic K_{IC} with measured values ($0.7 \text{ MPa m}^{1/2}$ – $0.9 \text{ MPa m}^{1/2}$) in good agreement with the oft-quoted range from previous work ($0.7 \text{ MPa m}^{1/2}$ – $1.3 \text{ MPa m}^{1/2}$) [17,19, 26–28]. At large a , K_{IC} for tests #1 and #2 increased beyond this intrinsic value, seemingly due to friction or other constraints at the tip-specimen interface.

An SEM image and schematic diagram of a fracture surface from test #1 are shown in Figure 4(a) and 4(b), respectively. Overall, the SEM image demonstrates that fracture initiated at the FIB notch and generated two mirror-like surfaces along a linear path. The fracture surface was parallel to the larger face of the DCB specimen, suggesting that fracture occurred along the intended $\{111\}$ planes, as measured with EBSD in Figure 1(c). Only two types of features were visible: hackle lines and arrest lines [29]. The hackle lines started at discrete points along the notch (implying that the fracture origins were likely small defects from the FIB process) and traversed the specimen length (implying that the fracture direction was top to bottom). In contrast, the arrest lines indicated crack length changes of $\approx 500 \text{ nm}$ between fracture events (in good agreement with those in Figure 3) and uniform tensile loading (based on the symmetry of the lines about the specimen centerline).

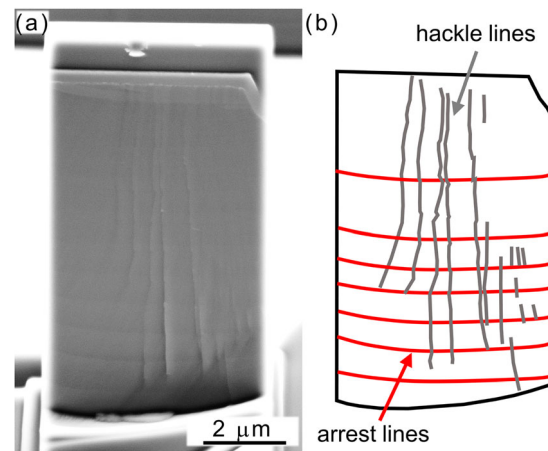


Figure 4. (a) SEM image and (b) schematic diagram of a fracture surface from test #1. The fracture generated two mirror-like surfaces along a linear path and occurred on the intended $\{111\}$ planes. Hackle lines started at discrete points on the notch and traversed the specimen length. Arrest lines indicated crack length changes of $\approx 500 \text{ nm}$ and uniform tensile loading.

Discussion

As noted earlier, the motivation for wedge-loaded DCB tests on an ideally-brittle material such as single-crystal silicon was two-fold: (1) to improve both the precision and accuracy of the approach through the inclusion of friction and anisotropy in the FEA model and (2) to provide insight into the small-scale fracture behavior of Si

through an appraisal of the crack-growth resistance curve and fracture surfaces during stable crack growth.

On point (1), the accuracy of the method was further advanced through full consideration of contact friction. The full details for including contact friction in the analyses are given in the Supporting Information. In summary, the energy release rate was defined as $G_{\text{mat}} = G_{\text{calc}} + G_{\text{fric}}$, where the internal (or calculated) release rate $G_{\text{calc}} = -\partial W_{\text{calc}}/\partial a$ is described by the internal energy W_{calc} and the friction release rate $G_{\text{fric}} = -\partial W_{\text{fric}}/\partial a$ is described by the friction energy W_{fric} . To assess W_{fric} (and thus G_{fric} and G_{mat}), the interaction between the wedge and one half of the DCB specimen was considered, which resulted in expressions for both the frictional force F_{fric} and sliding distance δ_{fric} as a function of F , d , μ , and α . After accounting for both sides of the DCB specimen and noting that $d = CF$, G_{fric} was found to be $G_{\text{fric}} = -(\mu \cos \alpha / (\mu \cos \alpha + \sin \alpha)) (F^2/2) (\partial C/\partial a)$, thus allowing G_{mat} (from the equation above) and K_{IC} (based on $K_{\text{IC}} = (G_{\text{mat}} E_{\text{eff}})^{1/2}$, where E_{eff} is an effective modulus from the FEA) to be calculated as shown in Figure 3(d). Overall, the friction corrections decreased the extracted K_{IC} and were more prominent at large a . The toughness was largely invariant with a , with an average value of $K_{\text{IC}} = 0.72 \pm 0.07 \text{ MPa m}^{1/2}$. The subsequent accuracy of the model (with material anisotropy and contact friction) was assessed through a comparison to K_{IC} from the literature and nanoindentation fracture tests. For Si {111}, Chen *et al.* [26] and Tanaka *et al.* [28] reported K_{IC} of 0.82 and 0.69 MPa m^{1/2} using four-point bend and indentation tests, respectively. The nanoindentation data in the Supporting Information revealed a toughness of $K_{\text{IC}} = 0.74 \pm 0.05 \text{ MPa m}^{1/2}$. Finally, a recent review paper established a K_{IC} value of 0.72 MPa m^{1/2} via a calculation based on surface energy and elastic fracture factor [1]. The agreement between the results suggest good accuracy, which validates the use of the F - d data in determining the a and K_{IC} values.

On point (2), stable crack growth allowed for an assessment of the crack-growth resistance curve (R-curve); from the friction-corrected data in Figure 3(d), it was deduced that K_{IC} was invariant with a , suggesting flat R-curve behavior. In ceramic materials, three main mechanisms lead to a rising R-curve: crack face interactions, microcracking and branching, and phase transformations [30]. Evidence for the first two mechanisms is absent here, seeing as crack face interactions were not observed in the F - d unloading-loading cycle in test #2 (Figure 2) and microcracking/branching were not found on the fracture surfaces (Figure 4). The last mechanism is also unlikely here, given that phase changes only occur

in Si at hydrostatic pressures of 10–13 GPa [31]. The lack of rising R-curve features and constant K_{IC} values thus provide clear evidence for a flat R-curve in single-crystal Si. However, at small a , deviations in the constant- K_{IC} value (denoted by the arrow in Figure 3(d)) may be associated with notch-shape and/or FIB-damage effects. Iqbal *et al.* [32] showed that geometric effects were negligible for notch radii between 70 and 120 nm (the notch radius here was much smaller at ≈ 8 nm), but Lauener *et al.* [19] illustrated that material effects led to increases in K_{IC} . The increase in the latter case was from the formation of an amorphous Si layer [25], which has a larger K_{IC} (1.0 MPa m^{1/2}) [33]. The X-ray maps in Figure 1 showed evidence for amorphous Si and Ga damage at the notch root, but also highlighted that the effects of implantation were limited to the first fracture event given the total depth of ≈ 50 nm. After the first event, the crack tip became sharp and extended beyond this zone, resulting in valid, intrinsic K_{IC} values as shown in Figure 3(d). Finally, it is possible to provide further commentary on the fracture surfaces in Figure 4(a). As noted earlier, stable crack growth produced mirror-like fracture surfaces, with hackle lines emanating from the FIB-notch region parallel to the crack propagation direction and arrest lines occurring periodically along the crack front perpendicular to the crack propagation direction. Perhaps more interestingly, however, is the absence of additional features such as crack front kinks and surface corrugations, which indicates subcritical steady-state crack speeds in this configuration [34].

Conclusions

In summary, we elicit stable nanoscale fracture in single-crystal Si wedge-loaded DCB specimens. For the tests, specimens were fabricated using FIB milling, tested in an SEM, and interpreted with FEA. On loading, the crack initiated at the FIB notch and exhibited stable growth along a {111} plane, such that two new surfaces were generated along a linear path. From a metrology standpoint, the results showed that the method possesses good precision and accuracy (both < 10%), but only after the effects of contact friction are incorporated. From a materials science standpoint, the results generated insight into the nanoscale fracture behavior of Si, namely the presence of flat R-curve behavior, notch-shape and FIB-damage effects, and mirror-like fracture surfaces with hackle and arrest lines during stable crack growth. Future work will further validate the method on silicon via systematic studies on the effects of specimen aspect ratio (to assess the plane-stress to plane-strain transition), crystallographic orientation (to assess fracture events on higher-toughness planes such as {110} and {100}), and

loading rate (to assess strain-rate effects at higher temperatures). Perhaps more importantly, future work will utilize the DCB method to examine complex material systems where K_{IC} values are less well known and toughening mechanisms less well understood.

Acknowledgements

F.W.D. and B.L.B. were supported by the Center for Integrated Nanotechnologies, a Department of Energy office of Basic Energy Sciences user facility. Sandia National Laboratories is a multitechnology 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 National Nuclear Security Administration under contract DE-NA0003525. 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.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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