

Extreme Ductility at the Nanoscale in Fe-based Alloys

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A novel method for preparing and testing bending specimens *in-situ* the TEM has been developed. This method provides high visibility of the crack front and its associated plastic zone. This is significant because energy release rates can now be calculated at many points throughout the test based on crack tip opening displacement (CTOD), crack opening angle (COA), and crack advance. Further refinement of testing methods to do dark field imaging of dislocations would allow estimation of dislocation content vs. time and may provide insights into dislocation behavior.

Much previous work has been done on *in-situ* fracture testing, though largely in the SEM. These tests are typically performed on cantilever specimens as in [1] amongst others, but the test specimens presented here simulate a three-point bend test, a scheme originally presented by Jaya et al [2]. Though this requires end clamping which invalidates the ASTM E 399 analysis method, energy based methods for determining fracture toughness are viable. FEM modeling of this specimen geometry has shown enhanced stability of the stress intensity level [2], and the increased positional stability aids in achieving high resolution imaging of dislocation processes.

Materials under investigation include Nitronic 50, an austenitic stainless steel and single crystal Fe-3%Si, which represent different typical steel constituents. Samples were prepared using the method outlined by Moser et al. [3], but the FIB milling step was modified to produce clamped beams instead of pillars. Mechanical testing was performed using a wedge-geometry tip in a Hysitron PI-95 Picoindenter holder, inside a JEOL 2100F TEM operating at 200kV. Notches were produced using a novel method, by utilizing a converged electron beam in the TEM, producing extremely sharp notches with an average root radius of curvature of 20nm.

Qualitatively it can be observed in Fig. 1 that the Nitronic 50 beams underwent severe plastic deformation in the central notched region of the beam, with no evidence of brittle behavior. Instead, the crack front advances only minutely by a ductile tearing mode. The COA and CTOD are constantly increasing throughout the test, which means critical fracture toughness values are not achieved. Though this is unfortunate from an analysis standpoint, it highlights the extreme ductility of FCC iron.

An example load-displacement curve for a Nitronic 50 beam is shown in Fig. 2a. Four separate loading cycles were performed on this specimen. An energy based method was adopted from [4] for determining K_{II} at the peak load for each cycle. This was used to produce Fig. 2b, which presents K_{II} vs. crack length a for several beams at different strain rates. It can be seen that there is good agreement between all tests, which suggests that no significant strain rate effects existed in the range investigated.

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References:

- [1] Wurster S, C Motz, and R Pippan. "Characterization of the fracture toughness of micro-sized tungsten single crystal notched specimens." *Philosophical Magazine* 92.14 (2012): 1803-1825.
- [2] Jaya B, Nagamani, Vikram Jayaram, and Sanjay Kumar Biswas. "A new method for fracture toughness determination of graded (Pt, Ni) Al bond coats by microbeam bend tests." *Philosophical Magazine* 92.25-27 (2012): 3326-3345.
- [3] Moser, G., et al. "Sample preparation by metallography and focused ion beam for nanomechanical testing." *Practical Metallography* 49.6 (2012): 343-355.
- [4] Zhu, Xian-Kui, and James A. Joyce. "Review of fracture toughness (G, K, J, CTOD, CTOA) testing and standardization." *Engineering Fracture Mechanics* 85 (2012): 1-46.

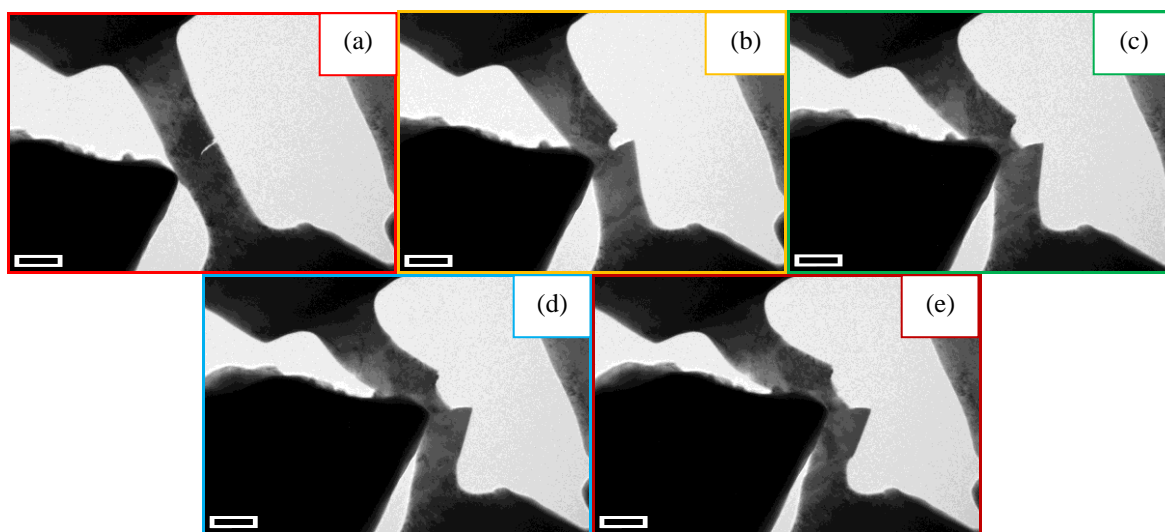


Figure 1. Bright field images taken from test video with letters corresponding to their position in the load-displacement data shown in Fig. 2. Scale bar indicated in each image is 200nm.

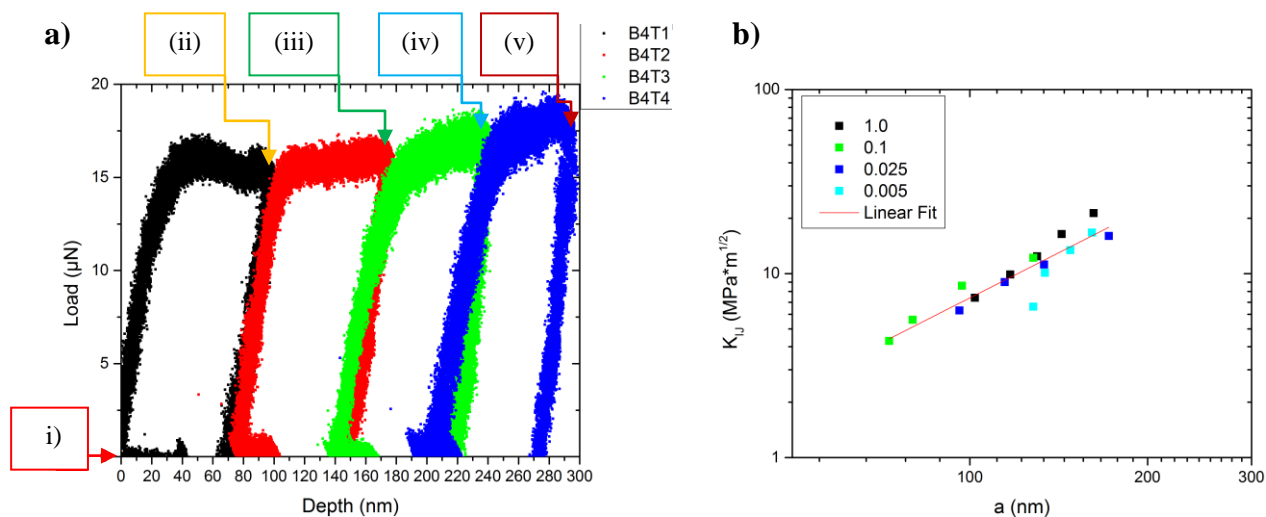


Figure 2. a) Load-Displacement data for an example beam with four separate loadings. b) K_{IJ} vs. a for several bending beams, strain rate is indicated in the legend.