CRACK TIP MICROSTRUCTURES IN F82H ON THE LOWER SHELF - D. S. Gelles (Pacific Northwest National Laboratory)*, G. R. Odette (University of California at Santa Barbara) and P. Spätig (École Polytechnique Fédérale de Lausanne, – Centre de Recherches en Physique des Plasma, Villigen PSI, Switzerland)

OBJECTIVE

The objective of this effort is to better understand deformation behavior in low activation ferritic steels with regard to fracture toughness testing.

SUMMARY

Dislocation microstructures have been examined near the crack tip of a compact tension specimen of unirradiated F82H loaded to 25.6 MPa $m^{1/2}$ at –196°C after fatigue precracking. A specimen was prepared by sectioning, dimple grinding and ion milling in order to produce electron transparency just behind the crack tip. The tip was found to have trifurcated with moderate dislocation densities ahead and to the side of each tip extending at least 4 to 5 μm, but regions adjacent to the fatigue crack but back from the tip displayed only minor dislocation rearrangement of lath boundaries.

PROGRESS AND STATUS

Introduction

Observation of microstructure at cracks in metals has only recently been considered viable with the development of ion milling techniques suitable for preparation of metallic specimens. Recent work at PNNL has utilized the ion milling procedure to allow observation of microchemistry and microstructure in irradiated austenitic steels undergoing stress corrosion cracking[1] and to allow dislocation and very small bubble examination in ^{3}He injected palladium foils.[2] Given recent advances in our understanding of post-irradiation deformation response in ferritic steels based on miniature tensile specimens,[3-4] the opportunity to study plane strain deformation behavior at a crack tip generated during a fracture toughness test was deemed appropriate. For example, it has not yet been demonstrated that post-irradiation deformation behavior under tensile loading, which can lead to channel deformation, will be duplicated under plane strain conditions. The present effort is intended to show that deformation at a crack tip can be studied. As procedures are in place to prepare similar specimens of irradiated austenitic steels, it is hoped that this work will be expanded to include examination of a similar sample of F82H following irradiation.

Experimental Procedure

Two 0.4T compact tension specimens of F82H (0.35x0.84x0.89 in.^{3}) in the fully tempered precracked condition were loaded in order to produce crack tip deformation. The first, P4-2, was loaded at room temperature to 100 MPa $m^{1/2}$ and the second, P4-10, was loaded to 25.6 MPa $m^{1/2}$ in a liquid nitrogen bath. Specimen P4-2 contained a remaining ligament of only ~0.03 in. with a crack tip microstructure therefore affected by the back surface, whereas P4-10 was cracked approximately half way through, so only P4-10 was prepared for examination.

Specimen P4-10 was sectioned using a slow speed diamond-impregnated-blade to provide a central longitudinal slice perpendicular to the crack surface and a 3 mm disk was cut with the crack tip located at the midpoint. One side was ground and polished to a high polish using 1-2 μm BN grinding compound and the other side was dimple ground to a thickness of ~10 μm with a similar final polish. Final thinning was performed on an ion mill using ultra high purity (99.999%) argon. Milling was centered just behind the crack tip with conditions for thinning 1 h at ±6º and 5 kV, and for final polishing 15 min at ±4º and 2 kV.

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* Pacific Northwest National Laboratory (PNNL) is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO-1830.

Both the Dimple Grinder model 656 and Precision Ion Polishing System (PIPS) model 691 were obtained from Gatan, Inc of Pleasanton, CA and the procedures used are based on those developed by Gatan.

Microscopy was performed using either a JEM-840 Scanning Electron Microscope (SEM) operating at 20 KeV or JEOL 1200EX or Tecnai 30 Transmission Electron Microscopes (TEM) operating at 120 and 300 KeV, respectively. All images are digitized, either directly or from scanned negatives. Image processing included electronic dodging to compensate for low magnification negatives with very high contrast.

Results

Metallography

In order to ensure that the 3 mm disk contained the crack tip at the center, the central slice was polished and etched to show grain boundary and lath structure. The microstructural features found are provided in Figures 1 and 2.

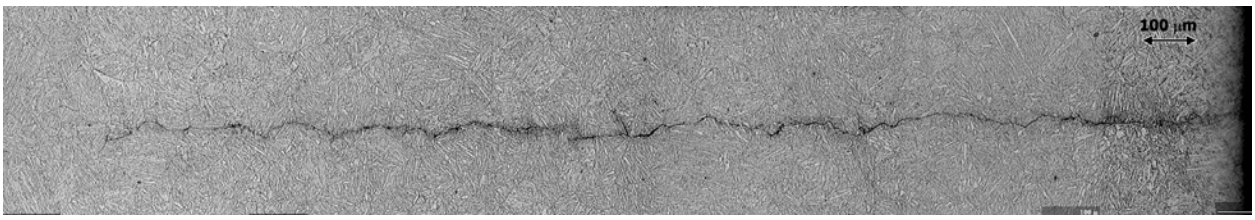

Figure 1. Etched metallographic section showing the crack in specimen P4-10.

Figure 1 shows that in cross section the fatigue crack is not straight, with deviations on the order of 50 µm. The crack follows some prior-austenite grain boundaries, but transgranular cracking tends not to produce straight sections, in contradiction to what might be expected based on fractographic examinations. A central section of the fatigue crack shows both a vertical secondary crack and a step. From the higher magnification image in Figure 2, it can be estimated that the crack stopped at a prior-austenite grain boundary. However, another crack appears to have formed ahead of the propagating crack, shown at the upper left. It is possible that the two are linked, but the crack is not revealed by etching. Note that the step at the center of Figure 1 also shows poor crack definition.

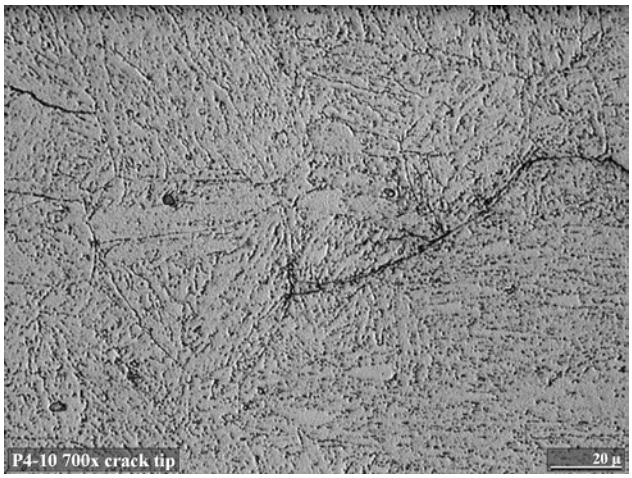

Figure 2. Crack tip of Figure 1 at higher magnification

Microscopy

Ion milling produced a perforation that was as much as 15 µm wide. An SEM montage of the crack and perforation is provided in Figure 3. In this case, several crack sections are found to be quite straight, and a secondary crack again can be noted near the center of the montage. The ion milled perforation shows a darker electron-transparent region adjacent.

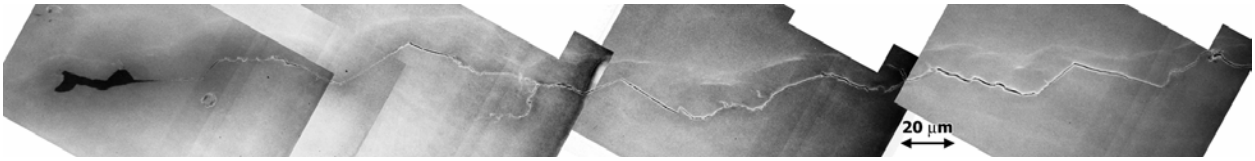

Figure 3. SEM montage of the crack tip following ion milling.

A low magnification TEM image of the perforation is provided in Figure 4. The image was developed to show the microstructure adjacent to the perforation at low magnification, and as a result the edge of the perforation is not visible. This image defines three cracks at the crack tip and a fourth secondary crack, marked 1, 2, 3, and 4 respectively. Therefore, it is possible that this crack tip developed three (or four) small cracks on loading.

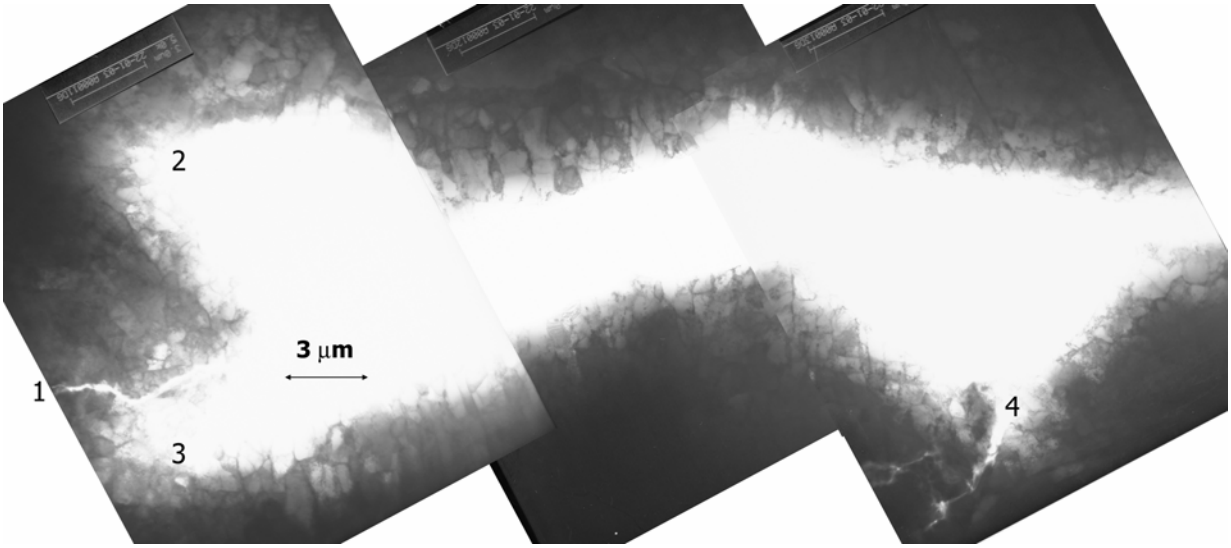

Figure 4. Low magnification montage of the perforation behind the crack tip.

Examination of the regions around the perforation but away from cracks showed microstructure characteristic of as-tempered treatments except for two features. Evidence of surface restructuring from the ion milling could be found that produced wide darker parallel wavy bands, and the dislocation structure at lath boundaries appeared to be rearranged. Three examples are provided in Figure 5 using g=200 so all (a/2)<111> dislocations are visible. The images are anaglyphs requiring colored glasses to see the structures in stereo. The ion milling artifacts are most visible in Figure 5c running from the upper left to lower right as wavy dark bands in the central sub-grain. In comparison, a dislocation slip band can be identified in a similar region in Figure 5b. However, the sub-boundary structure, most apparent towards the bottom of Figure 5a, consists of dislocations that extent a short distance from the subgrain boundary. In comparison, the as-tempered sub-boundary structure is simpler and more planar. This extended structure indicates that the dislocations in the original sub-boundary moved as a result of the stresses from the nearby fatigue crack tip.

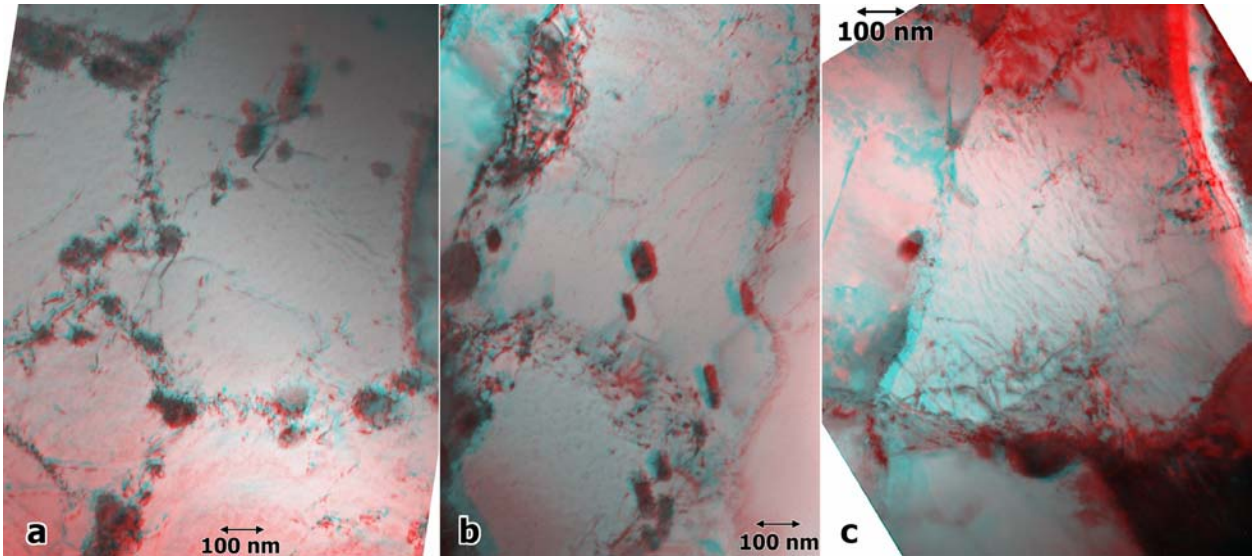

Figure 5. Anaglyph examples of the dislocation structures away from the crack tips.

In comparison, the dislocation structure ahead of the crack tip was more complex. The dislocation density was higher, but individual dislocations could be imaged clearly at higher magnifications. An example is shown as a low magnification montage in Figure 6. The dislocation density below the crack tip in Figure 6 is shown to be much higher than that found in Figure 5 but regions back from the tip are at a more moderate density. Note that local shear has occurred to allow the crack tip to open and the crack is about 200 nm wide near the tip.

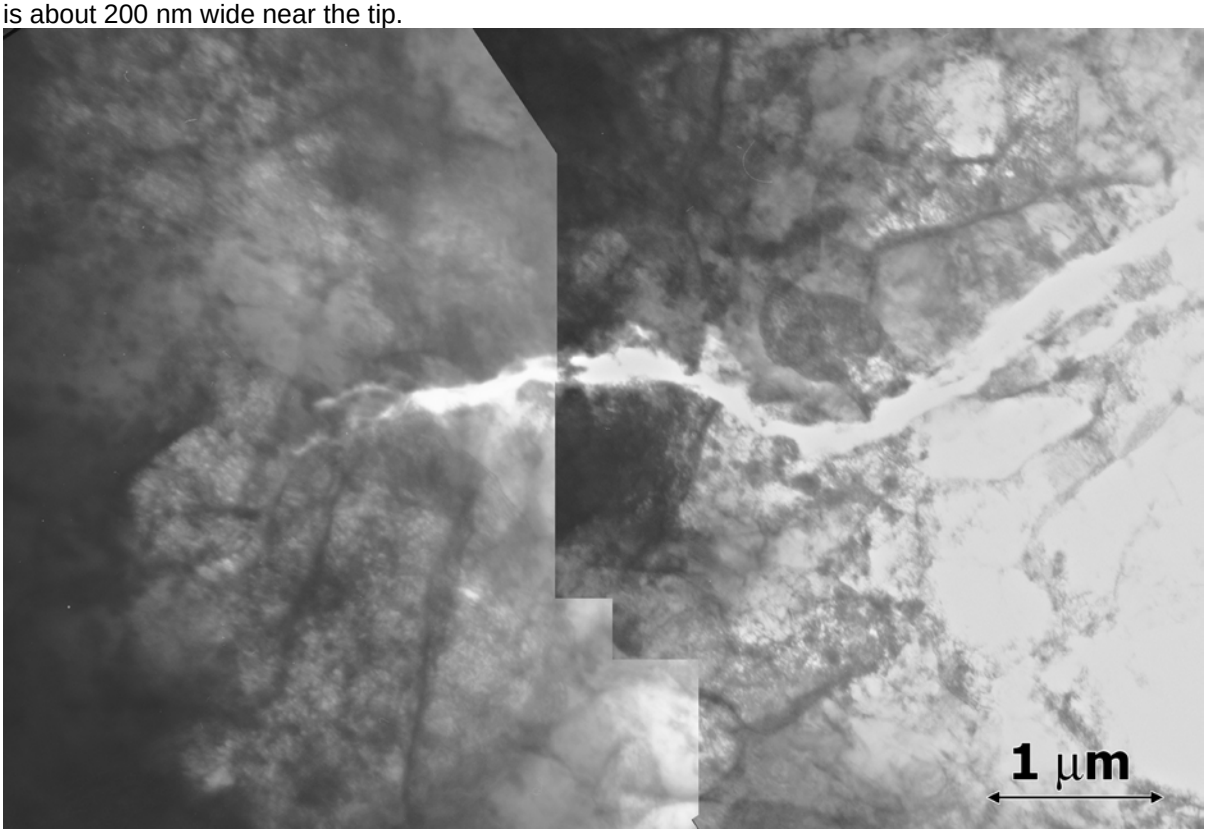

Figure 6. Dislocation structures near blunted crack tip 2.

In order to define the extent of the deformation, a low magnification montage has been prepared for this crack tip, shown in Figure 7. The crack tip appears dark on the right. Many carbide particles can be identified in lighter areas (diffracting weakly), but darker regions in dislocation contrast show fine dislocation structure. The upper portion of this figure approaches the area affected by crack 1, but similar deformation can be identified on either side of the crack. Therefore, Figure 7 demonstrates that significant dislocation damage has occurred at least 4 to 5 μm from the crack.

Discussion

The results obtained for dislocation behavior at a crack tip in a compact tension specimen of F82H tested at -196°C (on the lower shelf) demonstrate that such experiments are viable. Ion milling provides specimens that retain deformation microstructures with few artifacts. It can therefore be anticipated that similar examinations are possible on irradiated specimens. However, it should be emphasized that such experiments are quite difficult. Specimen miniaturization procedures such as 1 mm specimen geometries can simplify microstructural studies of magnetic materials due to reduced magnetic mass, but preparation of the crack tip within a 1 mm sample is expected to be very difficult. Therefore, magnetic effects complicate imaging. The nature of the lath martensite structure requires tilting of each lath in order to optimize dislocation imaging, and deformation within a lath further complicates imaging procedures. In order to understand crack tip deformation, imaging must extend over many lath distances, and therefore, a display of the dislocation structure requires montage techniques that are difficult to optimize. We expect to continue examination of specimen P4-10 after further ion mill thinning, so that the extent of deformation beyond the blunted crack tip can be better understood.

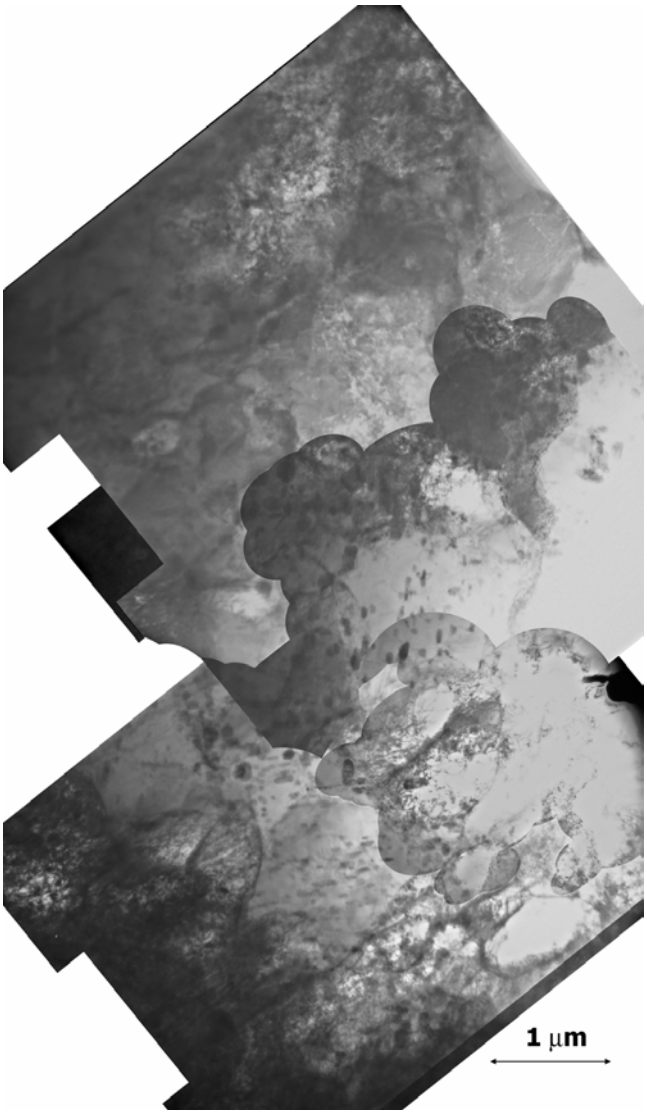

Figure 7. Low magnification montage of microstructure around crack 2.

Results obtained thus far demonstrated that fatigue pre-cracking can proceed with very minor effects on nearby dislocation structures. Only minor rearrangements of sub-grain boundary dislocations are observed. However, crack propagation from further loading of a fatigue pre-crack produces local plastic deformation. One example showed widening of the crack tip to 200 nm. The density of dislocations at a crack tip can be described as moderate; individual dislocations can be imaged in bright field. But the extent of the deformation from a crack tip is at least 4 to 5 μm, but the extent of the plastic zone size is not yet determined. Therefore, this effort must be considered a beginning.

Conclusions

Dislocation microstructures have been examined near the crack tip of a fatigue pre-cracked compact tension specimen of F82H loaded at -196°C to 25.6 MPa $m^{1/2}$. The tip was found to have trifurcated with moderate dislocation densities ahead and to the side of each tip extending at least 4 to 5 μm, but regions adjacent to the crack but back from the tip in the fatigue pre-cracked region displayed only minor dislocation rearrangement of lath boundaries.

FUTURE WORK

The effort is expected to continue and eventually shift to examination of a specimen tested following irradiation in HFR, Petten.

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