FURTHER EXAMINATION OF CRACK TIP MICROSTRUCTURES IN F82H ON THE LOWER SHELF -

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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 further 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 to produce electron transparency just ahead of the crack tip. Further ion milling has allowed improved examination of the microstructure immediately ahead of the crack tip. It is found that subgrain structure is relatively unaffected near the crack tip whereas 3 μm from the crack tip, dislocation loop structure was identified.

PROGRESS AND STATUS

Introduction

In a previous report [1] procedures were described to allow observation of dislocation structures at the crack tip of a compact tension specimen. The specimen of F82H was fatigue precracked and then loaded to 25.6 MPa $m^{1/2}$ at –196ºC. It was then prepared for TEM by sectioning, dimple grinding and ion milling in order to produce electron transparency just behind the crack tip. The present effort is a continuation of that effort following further milling so that the structure ahead of the crack tip could be examined in greater detail.

Experimental Procedure

As described previously [1] a 0.4T compact tension specimen of F82H (0.35x0.84x0.89 $in.^3$) in the fully tempered precracked condition was loaded in order to produce crack tip deformation. The specimen, identified as P4-10, was fatigue precracked about half way through and then loaded to 25.6 MPa $m^{1/2}$ in a liquid nitrogen bath.

The specimen was then sectioned using a slow speed diamond-impregnated-bladed saw 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. The present effort included three further milling steps in order to allow improved examination of microstructure ahead of the crack tip.

Microscopy was performed using a Tecnai 30 Transmission Electron Microscope (TEM) operating at 300 KeV. All images were digitized from scanned negatives. Image processing included electronic dodging to compensate for negatives with very high contrast.

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Results

The thinned cross section sample of P4-10 was remilled and examined to determine the location of the region of electron transparency relative to the crack tip. After three attempts, only ~200 nm of the tip remained. The milling stages are shown in Figure 1, with the original transparent region reported previously labeled "1st", and subsequent milling stages labeled "2nd" through "4th". Following the 4th milling stage, the region that was electron transparent included more than 3 μm ahead of the crack tip.

The region in the vicinity of the crack tip was tilted repeatedly to provide g=200 dislocation contrast for most of the subgrain structure. The g=200 contrast will show all (a/2)<111> dislocations in the field of view. The images have been combined to provide the montage in Figure 2 intended to show as much

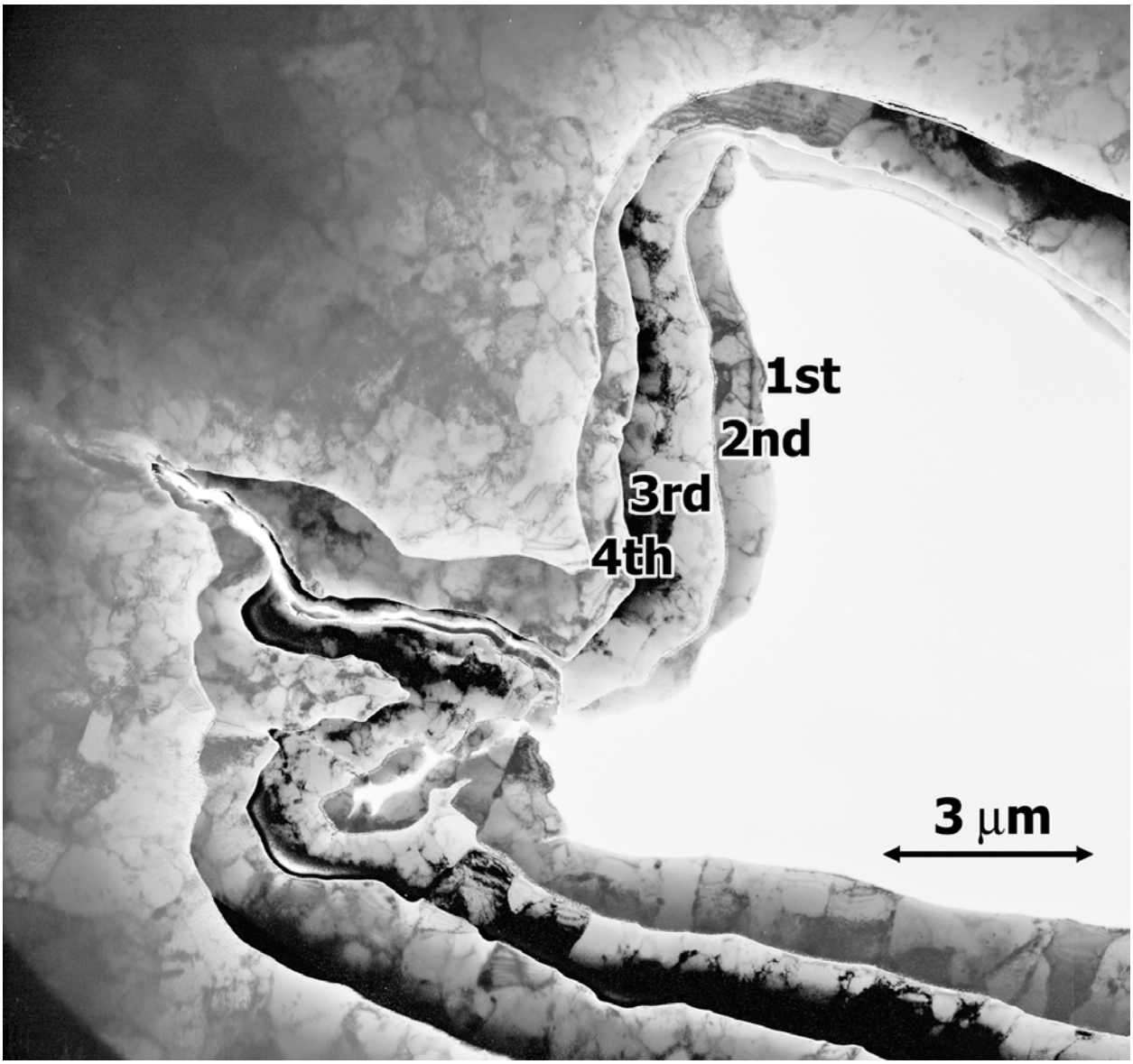

Figure 1. Montage showing the regions of electron transparency as initially prepared and after a further three remilling procedures.

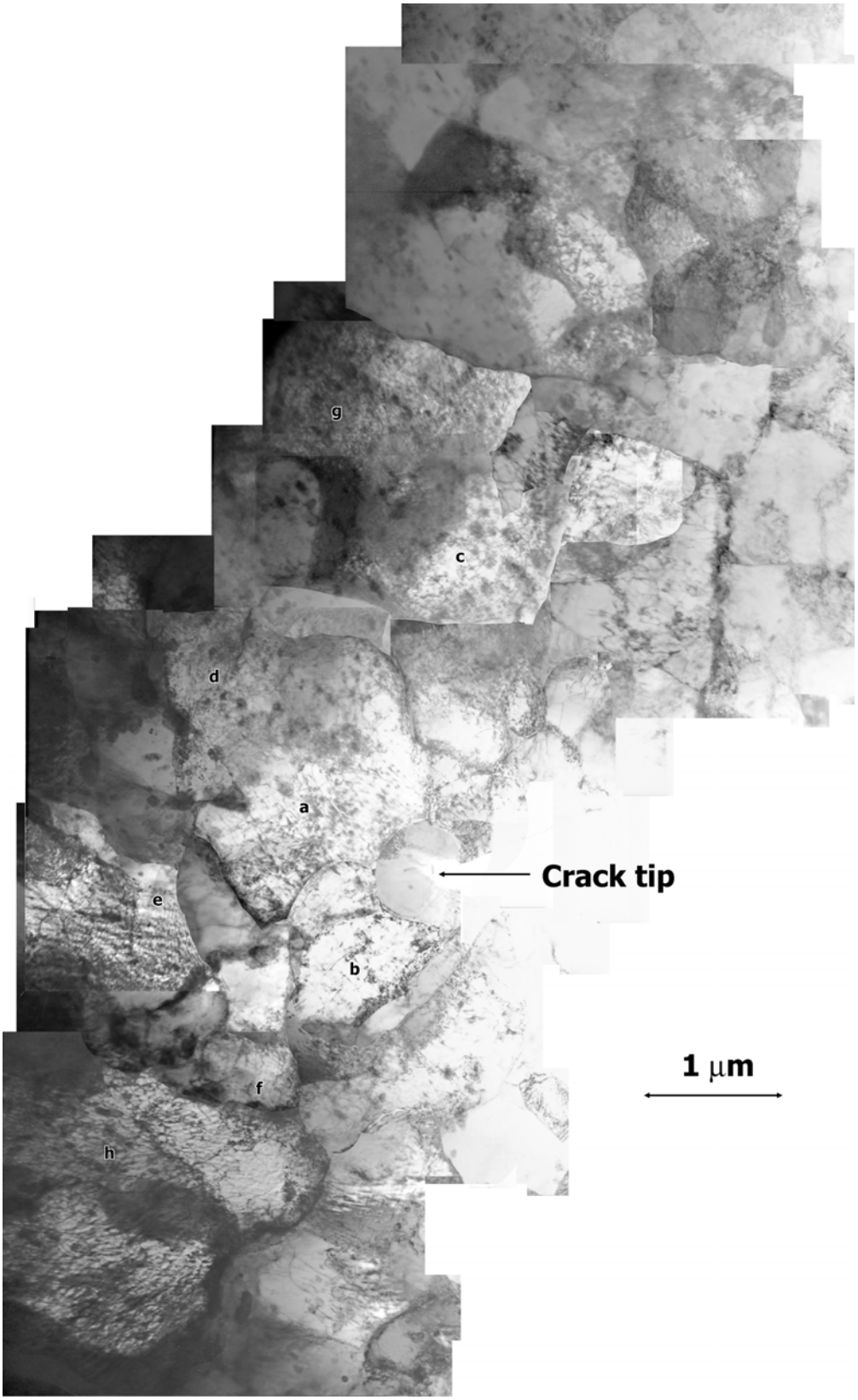

Figure 2. Montage showing dislocation structure ahead of the crack tip in g=200 contrast.

dislocation structure as possible in the region. Separation between images was generally chosen along subgrain boundaries, so that boundary configurations have not been conserved.

The microstructure in the vicinity of the crack tip consisted of intergranular carbides, carbides decorating martensite lath (sub-grain) boundaries, sub-grain boundary dislocation arrays and dislocations within the laths. The dislocation density within 1 μm of the crack tip was very low, consisting predominantly of subgrain boundary structure. (In fact, no attempt was made to image the dislocations within 0.5 μm of the crack tip.) Further from the crack tip, the dislocation spacing within subgrains decreased, but no attempt has been made to measure foil thickness and quantify the dislocation density.

Higher magnification images of dislocation structures 1, 2 and 3 μm from the crack tip are provided in Figure 3. Figure 3b) shows very few dislocations within a small 1 μm subgrain and Figure 3a) shows a larger subgrain with a crosshatched dislocation array at about half the spacing. Both regions are ~1 μm from the crack tip. Figures 3c) through 3f) show similar structures but containing tighter dislocation spacings for regions 2 μm from the crack tip. Figures 3g) and 3h) are for regions 3 μm from the crack tip, and the dislocation structures appear somewhat different, showing still tighter spacing, but no longer simply as crosshatch arrays. Instead, examples of elongated loop structures can be identified. These regions can be located on Figure 2 by the small letters a through h. Therefore, figures 3d) and 3e) are located almost directly ahead of the crack tip whereas the remainder tend to be above or below it.

Discussion

The results obtained for dislocation structures in the vicinity of a crack tip demonstrate the difficulty of such efforts in martensitic steels. The microstructure of undeformed fully heat treated F82H consists of subgrain structure as small as 1 μm in diameter, with well defined arrays of dislocations defining subgrain boundaries. Deformation can be expected to promote dislocation motion through the structure, enhancing dislocation densities. The difficulties arise when imaging the dislocations from one subgrain region to the next, and differentiating the deformation dislocations from dislocations present prior to deformation, including subgrain boundary structure. Further difficulties can be noted. After completing a large number of specimen tilts to record dislocation structures, it was found that the crack tip had advanced and the dislocation density at the tip had increased. Therefore, the electromagnetic forces on the thin foil were sufficient to cause further deformation. Apparently, crack tip specimens can have a limited lifetime for imaging experiments, and concern must also be given regarding dislocation rearrangements due to surface interactions, as well.

Several observations made are noteworthy. Dislocation densities ~1 μm from the crack tip tend to be quite low with the original subgrain boundary dislocation structure relatively intact. The process of dislocation generation at the tip (and returning to the tip upon unloading) is hard to envision. Such motion would be expected to destroy the subgrain boundary dislocation structure or at least put in steps, which did not appear to be the case. An example showing a subgrain in g=200 contrast very near the crack tip is given in Figure 4. Note that the subgrain boundaries are step-free at this magnification (so steps ~10 nm should be visible), and rearrangements of the dislocations in the subgrain boundary are similar to those reported by us previously well away from the crack tip and attributed to fatigue damage. In fact, no evidence for slip band formation was identified, either close to the crack tip or more than 3 μm away from it. Observations of dislocation loops within subgrains ~3 μm from the crack tip suggest that deformation is arising from small carbide particles distributed through the microstructure. The loops were found in regions containing carbide particles that were smaller than the loops. Away from the carbides, dislocations were more crosshatched in nature. Therefore, it can be suggested that dislocation nucleation may arise from interfacial dislocations breaking away from these small carbides. Larger carbides should be more greatly affected.

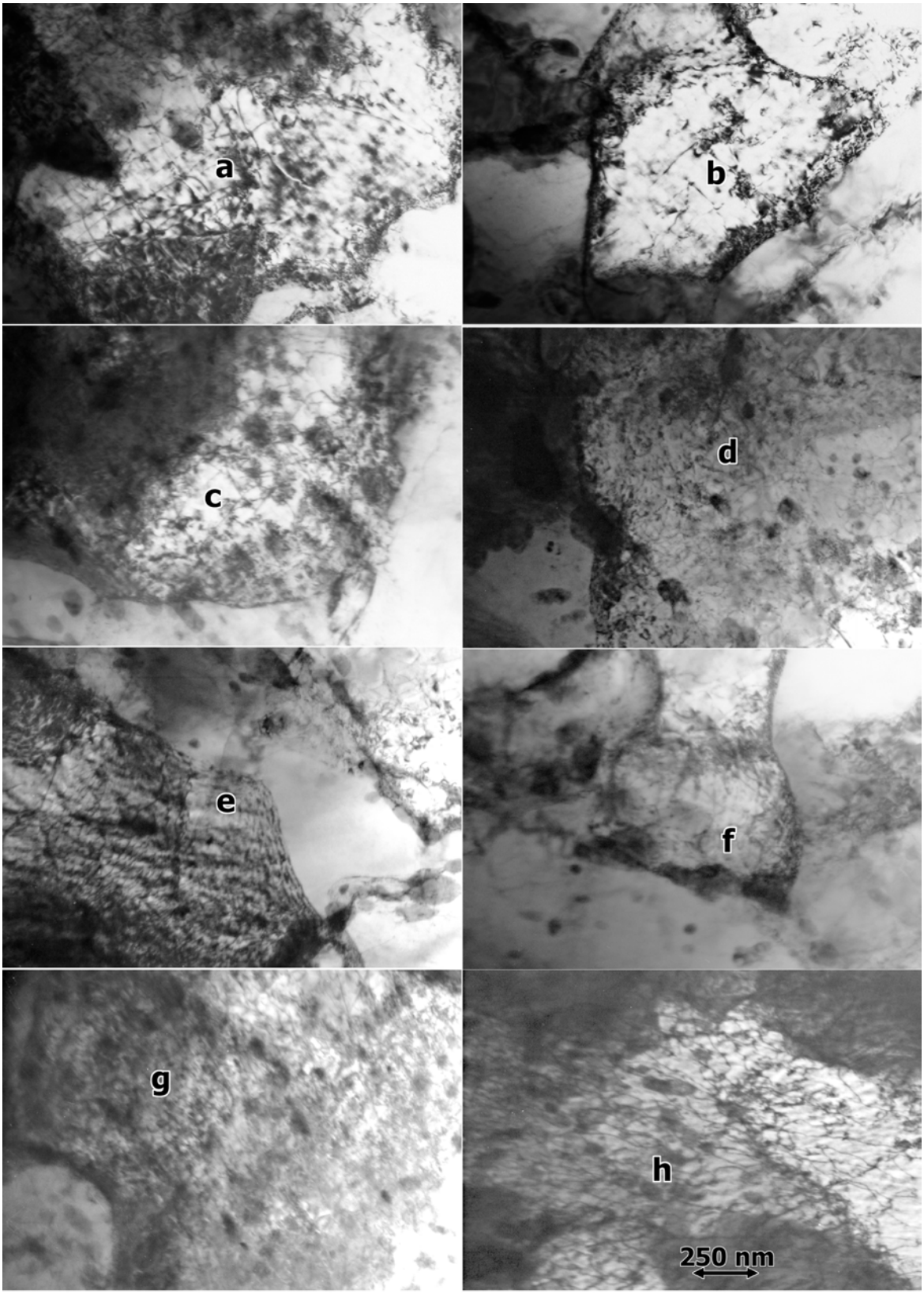

Figure 3. Selected regions of Figure 2 shown at higher magnification.

It can also be noted that recently published work by Tanigawa and coworkers [2] is based on similar goals with similar results regarding fatigue precracking. They studied microstructures at crack tips associated with prior fatigue deformation in both unirradiated and irradiated F82H. Specimen preparation employed a focused ion beam microsampling system so that small thin specimens suitable for microscopy were cut by an ion milling process perpendicular to an available surface. They showed that fatigue crack propagation often followed prior austenite grain boundaries, similar to conclusions drawn in our previous report. Also, they noted a breakdown of the lath structure during cyclic fatigue but prior to crack propagation into more equiaxed subgrain structure. It can be argued that the structure found here is similarly controlled by the fatigue precracking. Further studies of crack tip microstructures in precracked specimens may be required.

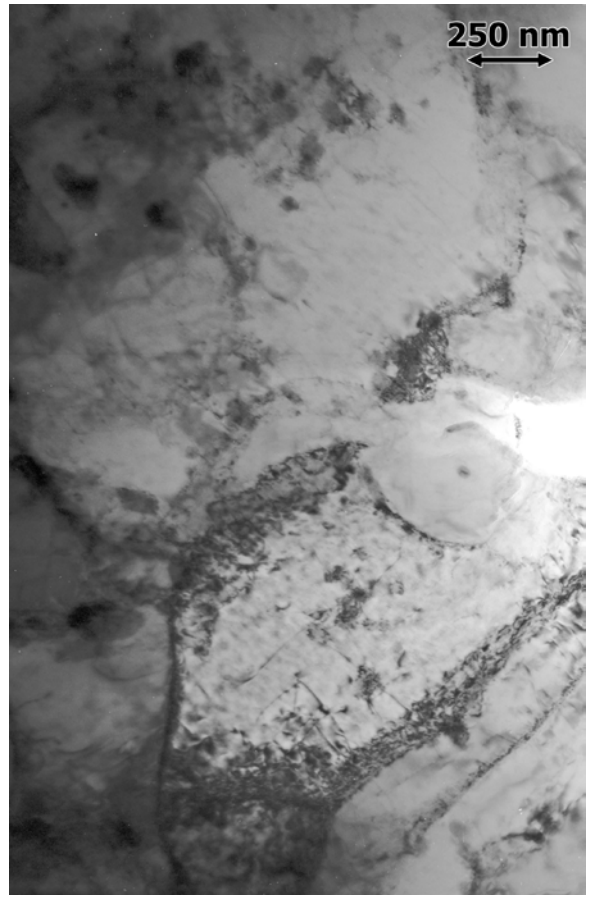

Figure 4. Dislocation microstructure near the crack tip.

Finally, it is worthwhile putting these results into perspective. The load of 25.6 MPa $m^{1/2}$ applied at –196°C to specimen P4-10 corresponds to approximately half the fracture toughness for F82H plate at that temperature as estimated by Master Curve analysis [3] but several fracture toughness data points exist at ~25 MPa $m^{1/2}$ [4]. Therefore, the load applied created plasticity at the crack tip, as demonstrated by a crack tip width of 200 nm, and could have exceeded K_{JC} if the microstructure had been appropriate. The stress distribution on the plane ahead of a blunting crack tip can be expected to exceed the tensile stress σ_0 by more than a factor of 3 at a distance ~ (J/σ_0) [5]. As $J=K_J^2/E$, with σ_0 ~ 1000 MPa [6] and E ~ 230 Gpa, [7] both estimated for -196°C, the distance ahead of the crack where the maximum stress is found is estimated to be 2.7 μm. Therefore, the scale of the experimental observations appears to make sense in terms of the expected fracture toughness parameters for F82H.

Conclusions

Dislocation microstructures have been examined ahead of the crack tip of a fatigue pre-cracked compact tension specimen of F82H loaded at -196°C to 25.6 MPa $m^{1/2}$. It is found that dislocation densities within subgrains are moderate in regions 1 to 2 μm from the crack tip, and slip band behavior is not observed either within subgrains or by its effect on subgrain boundary structure. In regions ~3 μm from the crack tip containing fine carbide particles, dislocation loops were found.

Future Work

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

References

[1] D. S. Gelles, G. R. Odette and P. Spätig, in DOE/ER-0313/33, July-December 2002, 79.

[2] H. Hanigawa, T. Hirose, M. Ando, S. Jitsukawa, Y. Katoh and A. Kohyama, J. Nucl. Mater., V. 307-311 (2002) 293.

[3] M. A. Sokolov, this progress report.

[4] K. Wallin, A. Laukkanen and S. Tähtinen in Small Specimen Test Techniques: Fourth Volume, ASTM STP 1418, M. A. Sokolov, J. D. Landes and G. E. Lucas, Eds., (ASTM International, West Conshohocken, PA, 2002) 33.

[5] R. M. McMeeking and D. M. Parks in Elastic-Plastic Fracture, ASTM STP 668, J. D. Landes, J. A. Bergley and G. A. Clarke, Eds., American Society for Testing and Materials, 1979, 175.

[6] P. S, G. E. Odette, G. E. Lucas and M. Victoria, DOE/ER-0313/26 (1999) 83.

[7] IEA Data Base for F82H.