Final Report

FEASIBILITY OF THIN-FILM ELECTRON MICROSCOPY FOR STUDY OF FINE DEFECT STRUCTURE IN STEEL AFTER LOW CYCLE FATIGUE

by

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Abstract

Application of recent developments in thin-film transmission electron microscopy to specimens of ABS Class-C steel fractured by low cycle fatigue demonstrates that changes in fine defect structure can be clearly observed. We conclude that this method is capable of revealing much information about the extent, magnitude and nature of low cycle fatigue damage in mild steel.

Objective

This study was directed toward determining the feasibility of thin-film transmission electron microscopy as a measuring probe for the study of low cycle fatigue in ship steel.

Material and Methods

The material used for this study was an ABS Class-C fully killed steel, specially machined to 3/4-in. thickness, which had been failed in low cycle fatigue with an initial central notch. This prior work was carried out in a project sponsored by the Ship Structure Committee at the University of Illinois (Project SR-149), under Professor W. H. Munse, and the specimen used here is fully indentified as RC-21 (1). The portion studied, near where the crack length was about 0.6 in., had been exposed to 20,000 cycles of $\pm$27 ksi when the crack tip reached this region. We studied two areas in this region: 0.02 in. below the fracture surface, designated as "fracture"; and 1 in. below the fracture surface, designated "non-fracture."

If the fine defect structure is altered during the preparation of a very thin specimen, the observed configuration cannot be unequivocally attributed to the known history of the bulk specimen. Aside from alterations in dislocation position that are inherent in reducing the thickness, the chief sources of structural change are high temperature and plastic strain during thinning and handling of the very small specimens.

We have avoided the effects of high temperature during specimen preparation by using very low cutting speeds during the

mechanical machining portion of the thinning. A slice about 1/16 in. thick x 1/2 in. wide x 3/4 in. long is first removed by a saw at low speed, then cemented to a large steel block containing a recess 0.005 in. deep. After careful hand filing to about 0.005 in. thickness, the specimen is removed from the block by soaking in a solvent over night and placed in an electro-thinning apparatus of the Fisher-Szirmae type. We used two cathodes with central holes, as adapted by Glenn and Raley (2). Approximately 10 hr of thinning in a chromic-acetic acid solution with current in the 50 - 100 ma range is required to reduce the thickness to the order of 0.001 in. During this time, the principal effects of the hand filing should be eliminated, since the very large number of passes required for filing suggests effective depths of cut of the order of 0.0001 in. or less.

In order to eliminate plastic distortion resulting from handling the specimen when it is very thin, we next utilized the technique evolved by Despres (3). Several specimens of 3.5 mm diameter, the size of specimen holder in the electron microscopes, are cut out with a punch and die set, and then mounted between stainless steel washers of the same outside diameter and 1.5 mm inside diameter. This sandwich is spot-welded together at two points (which does not heat the central test portion of the specimen, according to Despres) then further thinned in the electro-thinning apparatus until holes appear. The washers apparently cause the current density to be quite uniform over the specimen surface, with more even thinning than in un-mounted specimens.

Oxidation of the surfaces is controlled by immersion in methyl alcohol from immediately after the completion of thinning until the specimen is put into the high vacuum of the microscope.

We observed these specimens in a JEM-6A electron microscope, at both 80 and 100 kv. Use of a tilting stage permitted clear discrimination between extinction contours, an interference effect

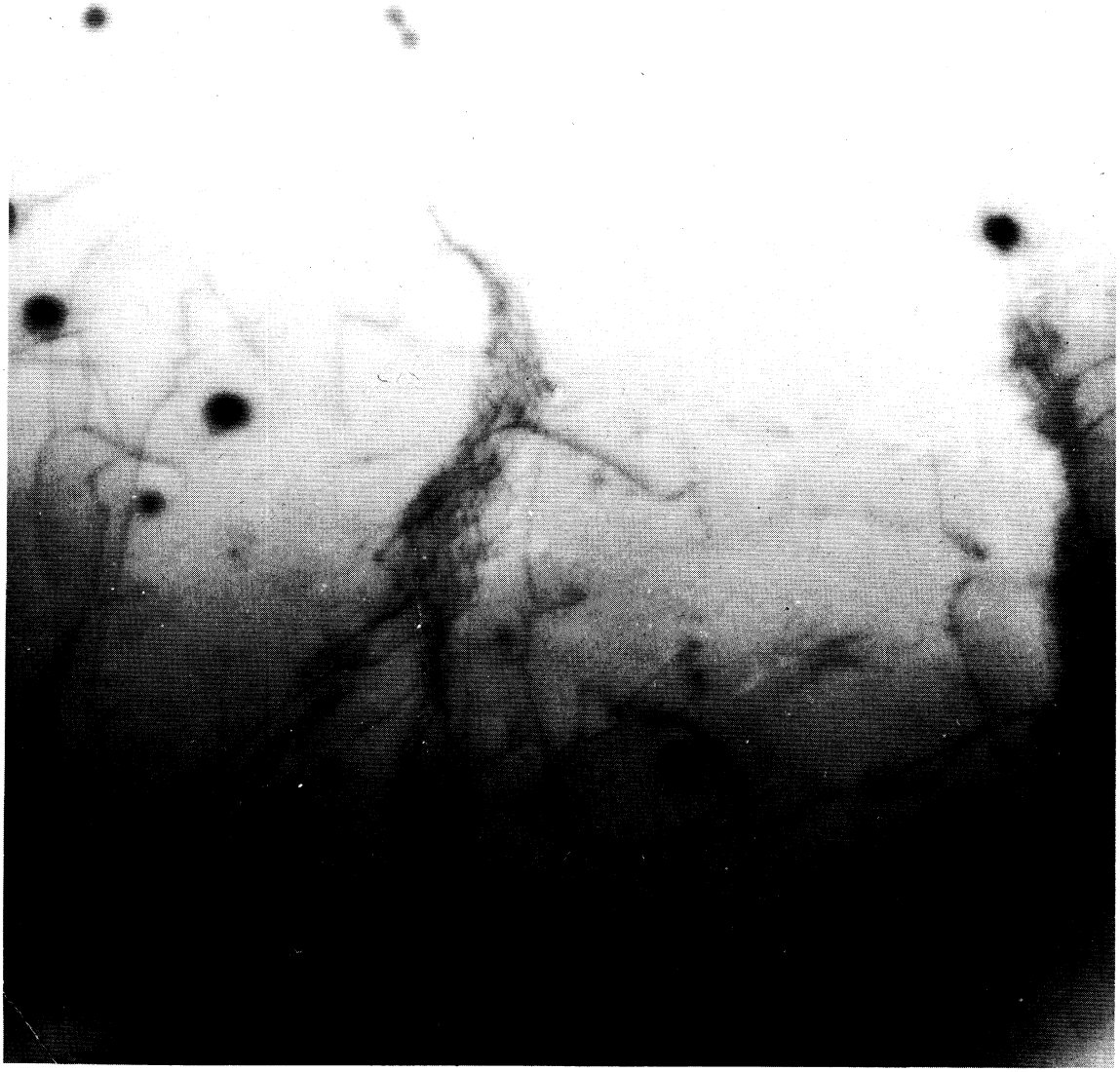

Fig. 1. Specimen RC-21, 1 in. from fracture surface. 48,000x.

from non-parallel surfaces, and fixed defect structure. Photographs were made on Kodak fine grain positive film, using exposure times from 5 to 60 sec.

Results

An example of fine defect structure can be seen in the dislocation network in ferrite, Fig. 1. This specimen was taken from the non-fracture region, about 1 in. from the low cycle fatigue surface of ABS Class-C specimen RC-21. Although this part of the specimen doubtless shows some effects of the cyclic loading, and thus would probably differ from the as-received material, the local stresses were much less than in the region of the central notch and its subsequent slowly propagating crack. The relatively low density of dislocations in this region suggests that little strain hardening of the type often associated with high-stress low cycle fatigue had occurred.

Figures 2 to 4 show several regions, all from the area close to the fracture region, about 0.02 in. from the fracture surface of specimen RC-21. The aggregation of imperfections into a kind of coarse network is shown in Fig. 2. A very fine, dense network of dislocations has developed in the region of Fig. 3. Figure 4 shows a fine network of dislocations, more dense than in the non-fracture specimen of Fig. 1. In addition, Fig. 4 contains a sub-grain boundary which is apparently the result of localized dislocation tangles of much higher density than in either Fig. 1 or 3.

Fig. 2. Specimen RC-21, 0.02 in. from fracture surface. 48,000x.

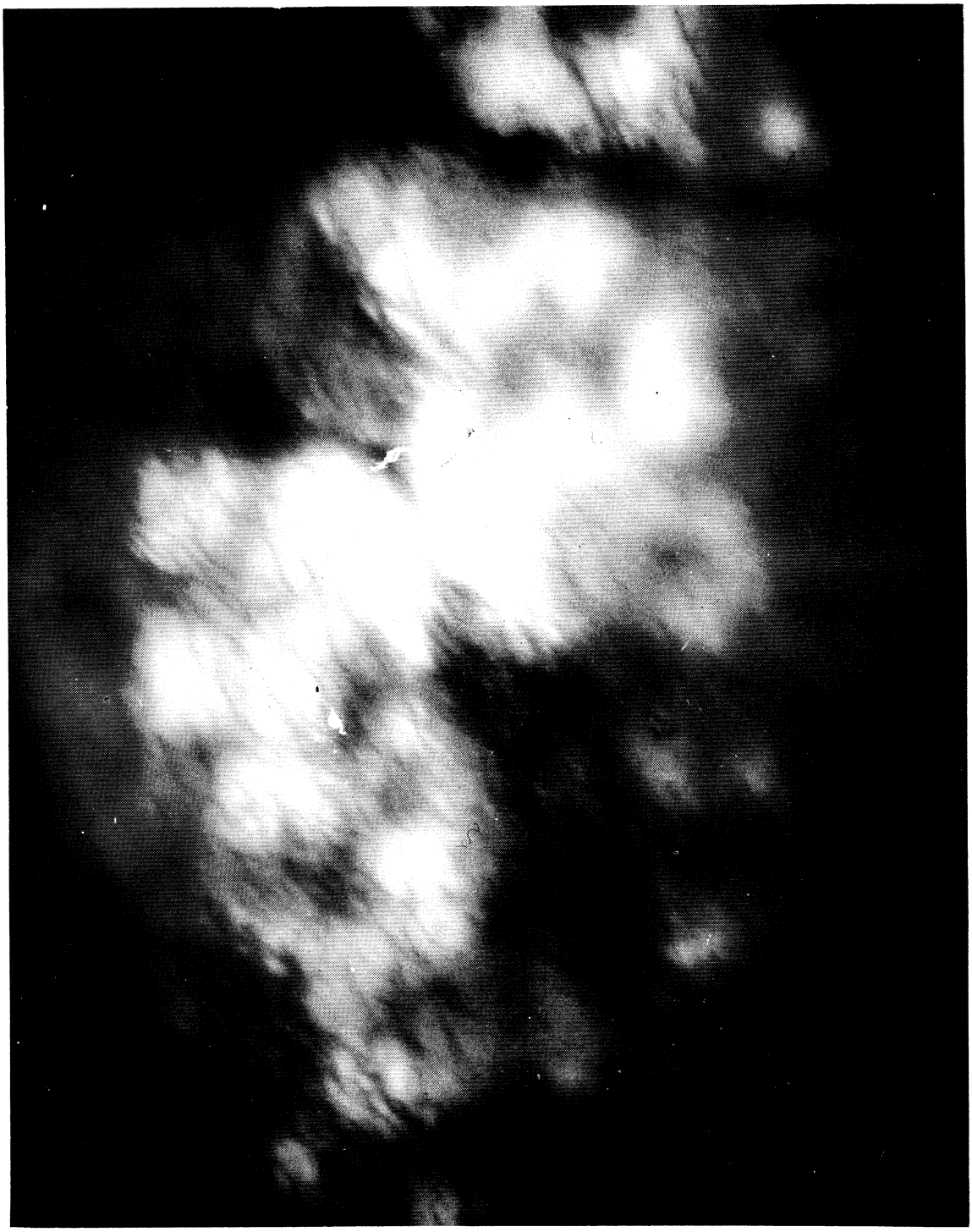

Fig. 3. Specimen RC-21, 0.02 in. from fracture surface. 60,000x.

Fig. 4. Specimen RC-21, 0.02 in. from fracture surface. 60,000x.

Conclusions

The sample micrographs show that the defect structure can be observed and that the differences noted in a limited number of specimens are of a kind that can be supported by theory. Furthermore, the aggregation of dislocations into coarse networks has also been observed in single-phase metals after fatigue, so we should expect the possibility of the same sort of behavior in a two-phase material such as pearlitic steel.

The amount of information contained in these micrographs is greater than we can interpret today, but careful study of representative electron micrographs from a graded series of fatigued specimens (e.g., cut from specimens loaded to the same stress but for different numbers of cycles) holds promise of revealing meaningful trends. Thus we conclude that this method of thin-film transmission electron microscopy can be applied as a potentially powerful probe for showing the changes in fine structure that accompany low cycle fatigue in steel.

References

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3. T. A. Despres, "Application of a Conducting Mask for Thinning Metallic Foils for Electron Transmission Microscopy," presented at ASTM Annual Meeting, June 1963.

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