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Direct Observations of the Substructure Network in Iron

The existence of substructure within high-energy grain boundaries has been proven for different materials.¹⁻⁴ These subgrains are actually low-angle boundaries in which the orientation differences across the boundary are very small. Since this orientation difference is so slight, the procedures to show the presence of substructure are involved and often difficult.

This Letter describes a new technique and offers some metallographic results on substructure in high-purity iron. Another paper to be released at a later date will discuss the theoretical aspects of this research problem and the results that were obtained with pure iron and ingot iron.

Material and methods

The metallographic techniques discussed in this paper were applied to vacuum-melted iron that was obtained from the Vacuum Metals Corporation of Massachusetts. The exact percentage composition of this material by weight is as follows: carbon 0.007, phosphorus 0.003, sulphur 0.006, silicon 0.010-0.015, oxygen 0.0016, nitrogen 0.00018, copper 0.001-0.03, aluminum 0.01-0.03 and manganese 0.004-0.01.

After fabrication into standard tensile blanks, the material was vacuum annealed for 4 hours at 850°C to remove all effects of cold working and any dislocation network that may have existed in the specimens. Both x-ray and metallographic evaluations of annealed samples revealed only the presence of equiaxed grains devoid of substructure. Polygonization was produced in these samples by application of tensile strains between 2-15% at a constant strain rate prior to short vacuum anneals at 200°-700°C. This strain-anneal treatment allowed the dislocations to rearrange into low-angle boundaries of different orientation values. All metallographic samples were sectioned from the center of tensile specimens through the use of a jeweler's saw. Encapsulation techniques were standard in that Bakelite, room temperature resins, or metal jigs were used consistent with the

requirements of each sample. After mounting, the specimens were wet processed through silicon carbide papers of 180-600 grit.

Electrolytic polishing proved to be the only procedure to yield completely scratch-free samples. The electrolyte was adequate as either a polish or an etchant when used with the following composition and conditions.

Table 1 Electrolytic polishing data.

Solution	850 cc Ethyl alcohol (abs) 140 cc Distilled water 39 cc Perchloric acid ($\delta = 1.48$)
Cell	Disa Electropol type 53A
Current density	4.0 amps/cm ² (polish) 0.13 amps/cm ² (etch)
Time	17 seconds (polish) 7 seconds (etch)
Temperature	17°C
Flow rate	13 cm/sec
Cathode	Stainless steel

A water cell of special design was constructed to cool the bath reservoir to 17°C and to combat the potential hazards of using perchloric acid in the Disa unit. Etching electropolished samples was accomplished to a comparable degree by using either 0.1% Nital or the electrolyte at reduced amperage.

For electron microscopy a plastic replica solution was used which contained 0.5% collodion in ether and dilute additions of 1% amyl acetate for strengthening purposes. The best resolution of substructure was achieved through low angle shadowing of the replica with chromium at an angle of 15° to the replica surface. Small amounts of aluminum were also deposited from a position normal to the replica surface for strengthening purposes.

A Bausch and Lomb metallograph was used successfully to study this substructure; however, in all cases the phase-contrast accessories were needed to intensify

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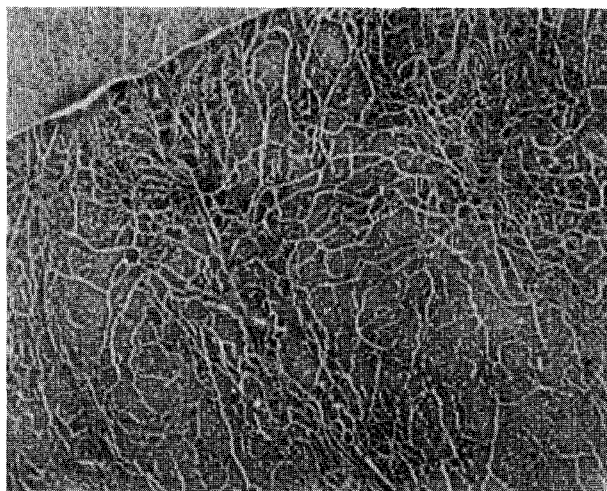


Figure 1 Dislocation network at 950 $\times$.

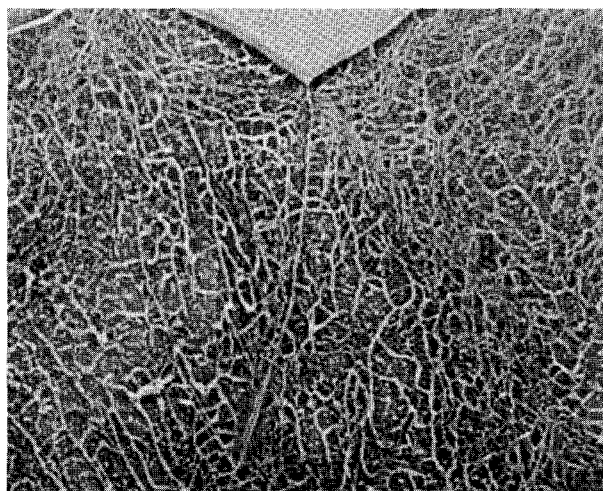


Figure 2 Dislocation network at 950 $\times$.

the contrast to a level suitable for good photomicrographs. The recommended procedure for direct observation of the substructure network in iron is, then, 1) electropolish in a perchloric-alcohol bath, and 2) observe, using phase-contrast microscopy. This new technique allows the investigator to study a polygonized ferrous sample within five minutes after encapsulation.

Results

Figures 1-3 show the characteristics of substructure in iron when illustrated to the best degree of resolution possible. The results of this new technique (i.e., electropolishing in a perchloranol bath prior to phase-contrast microscopy) allow direct observation of the dislocation network in iron to a degree that permits any precision study or quantitative determination that might be desired. The advantages of a good surface preparation and the use of phase-contrast accessories combine to obviate the slightest surface irregularity. Without the utilization of both electropolishing and phase contrast, substructure is not nearly as well resolved and the apparent continuity of low-angle boundaries is sporadic.

Figures 1 and 2 are examples of the degree of polish and etch necessary to show a dislocation network at low magnifications. In both photomicrographs the sub-boundaries are predominantly continuous, which indicates that the tilt angles are large or near 1° maximum. In certain areas small pits are clearly resolved and similar in appearance to etch pits which occur at the sites of individual edge dislocations. Such a possibility is unlikely since the resolution of the optical microscope is insufficient to show the sites of single dislocations at this original magnification of only 950 $\times$. Instead, these small pits are considered to be slight surface imperfections grossly exaggerated

by the sensitivity of the phase-contrast optics and the selectivity of the electrolyte for ferritic versus carbon-rich zones.

With any polygonization study on polycrystalline bulk materials, the effect of heterogeneous stresses on different grain orientations can never be completely avoided. The grain in the top center portion of Fig. 2 shows the competitive effect of grain misorientation. In this area of the photomicrograph, substructure is obvious but not too clearly resolved due to a slight disregistry between the above-mentioned grain and its neighboring grains.

When the strain-anneal method is used for the generation of substructure, an increase in either strain or temperature causes the sub-boundaries to become more pronounced until some optimum strain-anneal relationship is achieved. At this point sub-boundaries may be readily seen using the techniques reported herein. For iron of the composition used in this study, samples strained 9% appeared to have the most pronounced subgrain formation when heated in the 600° to 700°C range. Figure 3 (500 $\times$) illustrates the appearance of a sample strained 9% and vacuum annealed at 700°C for one hour. This sample was processed quite near the recrystallization temperature for the strain used. In fact, Lauegrams of this sample yielded some evidence of partial recrystallization. However, one will observe that the sample still exhibits a substructure over its entire surface. Polygonization in the recrystallized areas may well have been generated from some slight stress at the high annealing temperature just after recrystallization occurred. Such an occurrence has been observed previously by Boswell.⁵

Electron microscopy was an integral part of this study for two reasons: 1) to substantiate the validity of the different microstructures observed with the many metallographic procedures that were attempted

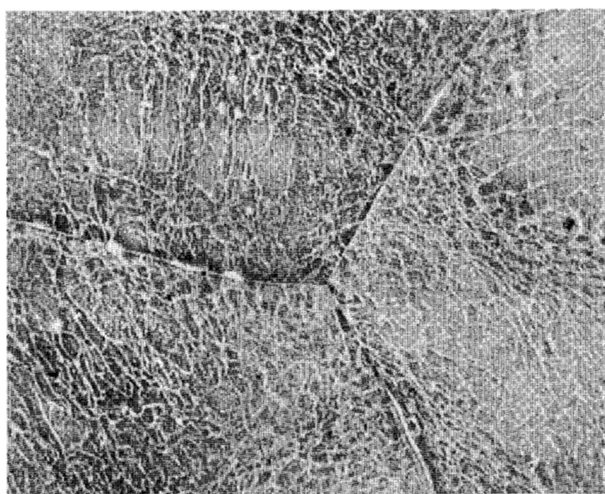


Figure 3 Dislocation network at 500 $\times$.

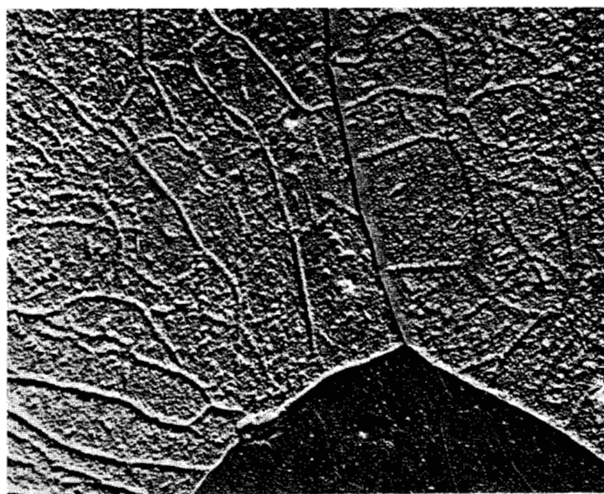


Figure 4 Dislocation network at 16,500 $\times$.

before a general procedure finally was evolved, and 2) to serve as a support tool for some minute measurements which are possible only at the higher magnifications. Figure 4 shows the appearance of this dislocation network at 16,500 $\times$ when metallographically prepared by electropolishing in the perchloric-alcohol bath. When properly etched, this electron micrograph exhibits a distinct similarity to Figs. 1-3. Such agreement in the structures serves to support the evidence that phase-contrast optics can be reliably used for direct observation of iron subgrains without requiring the involved techniques of either x-ray, electron microscopy, or etch pitting. Detailed measurements on other electron micrographs showed that the dislocation spacing in these iron samples corresponded to boundary tilts of between 15 minutes and 1 degree maximum, which is in agreement with the literature.⁶ These measurements were, of course, based on the assumption that the low-angle tilt boundaries were an

array of edge dislocations. No pronounced amounts of repetitive irregularities in the dislocation spacings were observed to support concrete indications of the presence of screw dislocations. The few repeated interruptions in the standard spacings that were occasionally observed were attributed to the result of different etching reaction rates between dislocations in the boundaries.

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