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On the Direct Observation of the Substructure Network in Iron

A Letter to the Editor in the October 1962 issue of this journal by W. L. Mitchell, C. Hays, and R. E. Swift[1] described a technique for developing and observing the substructure in iron. A study is currently being conducted in the IBM Rochester Development Laboratory on the correlation of magnetic and physical properties of iron-base soft magnetic alloys. One phase of this study investigates the effect of low-angle grain boundaries and dislocation configuration on the magnetic properties of an industrial grade of pure iron, with the following weight percent composition: carbon 0.015-0.030, phosphorus 0.02, sulphur 0.004, manganese 0.03, silicon 0.012, copper 0.07. The substructure for both conventional and electron microscopy is observed with a technique developed by Dunn and Hibbard.[2] This technique offers certain advantages over the method reported by Mitchell et al.

The data for the electrolyte, used both for polishing and etching, are given in Table 1.

Since this electrolyte does not contain perchloric acid, there is no safety hazard in specimen preparation. The main advantage of this electrolyte is that it attacks primarily the carbon-rich areas, without disturbing metallic surfaces, thereby locating individual dislocation sites. Hibbard and Dunn[3] developed a statistical method to show that a one-to-one relation can be established between dislocation sites and etch pits when this technique is used. According to Dunn and Koch,[4] 0.004% carbon is a suitable minimum for revealing the individual dislocation

Table 1 **Electrolytic polishing and etching data.**

Solution	133 ml glacial acetic acid, 7 ml distilled water 25 g CrO_3
Cell	Buehler 1721-2
Power source	Buehler 1715-1
Current density	0.25 amps/cm² (polish) 0.01 amps/cm² (etch)
Time	20 min (polish) 5 min (etch)
Cathode	copper

Figure 1 **Low-angle grain boundaries and dislocation distribution at 500× with bright field illumination.**

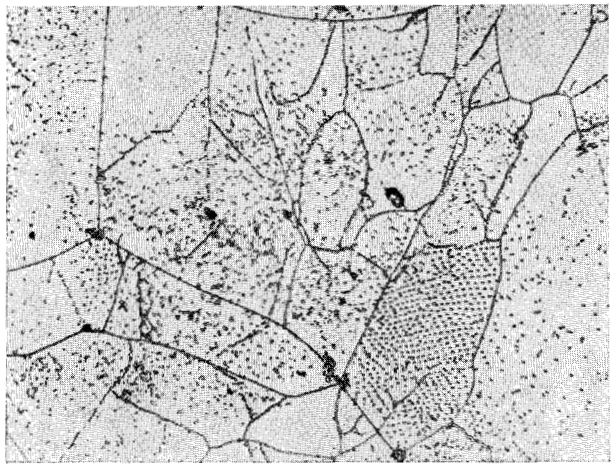
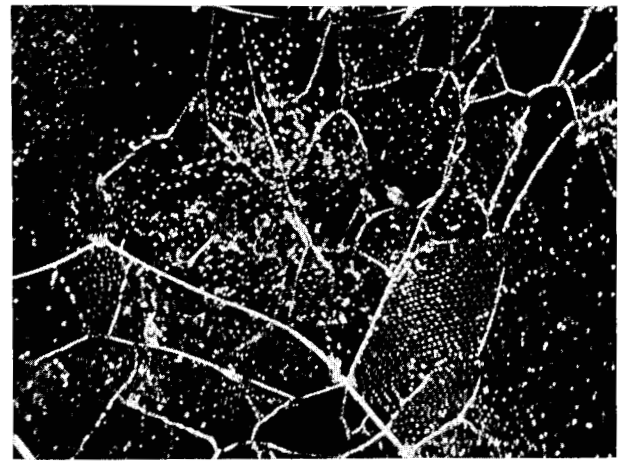


Figure 2 **Same area as in Figure 1 at 500× with polarized light illumination.**



sites by etch pits. The substructure of a cold-rolled specimen is shown in Figs. 1 and 2 in bright field and polarized light illumination, respectively. While the polarized light shows a higher apparent contrast for both the low-angle boundaries and the individual dislocation sites, the bright field illumination clearly provides the best resolution. Phase contrast illumination did not reveal any additional details in the substructure, and the resolution was inferior to that obtained with bright field illumination. This is most probably due to the open aperture diaphragm required for phase contrast illumination. The individual dislocation sites are clearly resolved at this moderate magnification. (Prior to reproduction here, magnification was 1000 ×).

Figure 3 **Detail of area in Figure 1 at 8000×.**

Figure 3 is an electron micrograph of a collodion-carbon replica, shadowed with germanium. It indicates a similarity between the individual dislocation sites and the low-angle grain boundaries, enabling calculation of the boundary tilt angle. The excellent resolution of individual dislocation sites permits good evaluation of dislocation distribution and density.

Both the light and electron micrographs show that this particular etching technique does not depend on grain orientation, thus providing equally good resolution in all grains examined.

Conclusion

The technique described offers the following advantages compared to the method proposed by Mitchell et al.:

1. It is safer because perchloric acid is eliminated from the electrolyte.

2. Individual dislocation sites are resolved at moderate magnifications by etch pits.

3. Bright field illumination, which is a simpler, more convenient method than phase contrast and provides better resolution, can be used to reveal the substructure.[5]

References

1. W. L. Mitchell, C. Hays, and R. E. Swift, *IBM Journal* **6**, No. 4, 467 (1962).
2. C. G. Dunn and W. R. Hibbard, Jr., *Acta Metallurgica,* **3**, 409 (1955).
3. W. R. Hibbard, Jr. and C. G. Dunn, *Acta Metallurgica,* **4**, 306 (1956).
4. C. G. Dunn and E. F. Koch, *Acta Metallurgica,* **5**, 548 (1957).
5. This conclusion might be modified somewhat by the observation that Mitchell et al. used somewhat purer materials, which are generally more difficult to etch.

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