SHORT COMMUNICATION

Etching and Image Analysis of Prior Austenite Grain Boundaries in Hardened Steels

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Many etchants have been used to reveal prior austenite grain boundaries in hardened steels [1, 2]. The etched grain boundary networks are often not sharply differentiated from the etched tempered martensite, and therefore measurements of prior austenitic grain size are difficult to perform. One of the most effective etchants consists of an aqueous solution saturated with picric acid containing sodium tridecylbenzene sulfonate [1, 3, 4]. The addition of small amounts of HCl improves the effectiveness of the etch [1, 4] (C. A. Apple, private communication, Bethlehem Steel Corp., Bethlehem, PA, 1980), and repolishing and re-etching accentuate the grain boundaries relative to the tempered martensite. This grain boundary etch has worked well in low-carbon core regions of carburized steels [5], CrMoV rotor steels [4], medium-carbon steels such as 4340 [3], and high-carbon roll steels (C. A. Apple, private communication, 1980).

This note has two purposes: (a) to review an etching procedure that effectively reveals austenite grain boundaries in hardened steels, and (b) to describe etching and imaging conditions that result in reliable grain size measurements by image analysis. These objectives are consistent with the current availability of powerful image analysis systems that can rapidly process large amounts of microstructural information [6], but which require properly prepared metallographic surfaces for reliable results.

EXPERIMENTAL PROCEDURE

Etching was performed on the tempered martensite core regions of hardenable carburizing steels containing 0.22% C, 0.88% Mn, 0.010% P, 0.020% S, 0.55% Cr, 0.53% Ni, and 0.20% Mo. The specimens were carburized at 927°C, and oil quenched and tempered at 150°C for 1 h. Metallographic specimens were ground through 600-grit paper and polished with diamond and alumina powders. However, it should be noted that this etch is not effective unless phosphorus is present in the steel.

The etchant is prepared as follows (C. A. Apple, private communication, 1980):

1. Prepare a standard saturated solution of picric acid in distilled water; filter out residue (note: dry, crystalline picric acid may be explosive).
2. Add 5–10 g (25–30 ml) of sodium tridecylbenze sulfonate per 500 ml to the saturated picric acid solution; this solution is stable and may be stored for an extended period of time.
3. Just before etching, add from 1–10 drops of hydrochloric acid (HCl) per 100 ml of solution. But note that: (a) the addition of HCl is particularly important for the higher-alloy grades, especially those high in Cr; and (b) the greater the carbon content in the alloy, the smaller the HCl addition that is required.

Etching to delineate austenite grain boundaries is performed as follows:

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1. Surfaces to be etched must be freshly polished.
2. Etch by immersion in a beaker containing etchant; place beaker in an ultrasonic cleaner to provide agitation.
3. Remove specimen, wash, and dry.
4. Observe etched surface with microscope. Outlines of austenite grain boundaries should be visible.
5. Lightly polish specimen on 0.05-μm alumina until etched matrix is removed, leaving only prior austenite grain boundaries visible.
6. Repeat steps 2–5 until boundaries are sufficiently delineated for measurement by manual or image analysis techniques as discussed below.

Prior austenite grain size was measured manually by a lineal intercept technique according to ASTM 112 [7]. Grain boundary intercepts with a 500-mm line were counted in 20 fields per specimen, at a magnification of ×320. Grain size was also measured by image analysis with a Leco 2001 Image Analysis System programmed to measure grain size according to ASTM E 112. Grey level detection was set to a level that retained the most lightly etched boundaries. The program also provides for reconstruction of incomplete grain boundaries.

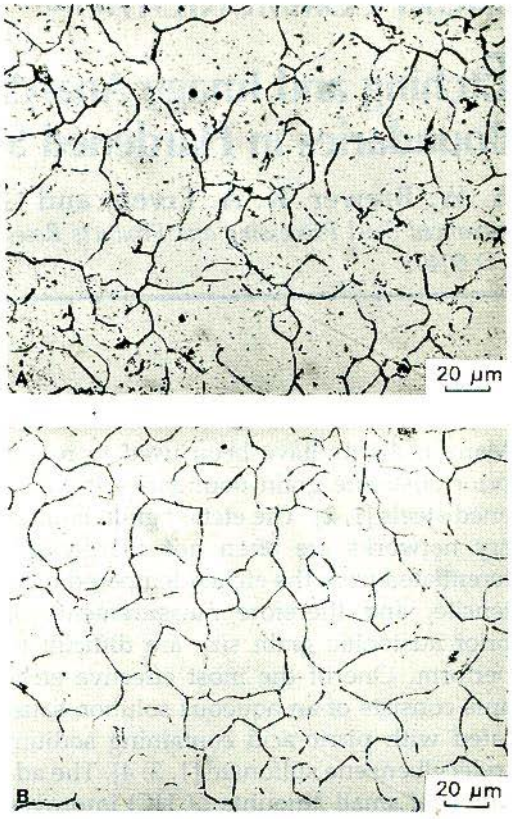

FIG. 1. Prior austenite grain boundaries in the core of a carburized steel: (a) etched and partially repolished, leaving remnants of intragranular structure; (b) etched and repolished to remove all intragranular structure. Light micrograph.

RESULTS

Figure 1 shows examples of prior austenite grain boundaries in the core microstructures of carburized specimens etched according to the procedures described above. Figure 1(a) shows a section as etched and slightly repolished so that part of the intragranular detail remains. Figure 1(b) shows only grain boundaries after considerably longer repolishing. However, many of these boundaries are discontinuous. Both conditions might influence the results of image analysis, with that of Fig. 1(a) leading to a reported grain size finer than the true grain size. But, based on the judgment of an experienced operator, both microstructures can be analyzed. If one uses the manual intercept counting technique, a good estimate of the prior austenite grain size can be made.

Two operating conditions were found to minimize the effect of incomplete etching of the grain boundary networks on the results of image analysis. The one condition consisted of defocusing the metallographic image. Figure 2 compares the image reconstruction of a focused image, with the reconstructed image of an overfocused surface. The latter image is much more complete because of increased contrast and width given lightly etched grain boundaries by the defocusing. The other operating condition that improved the results of image analysis consisted of the use of lower magnification. Figure 2(c, d) com-

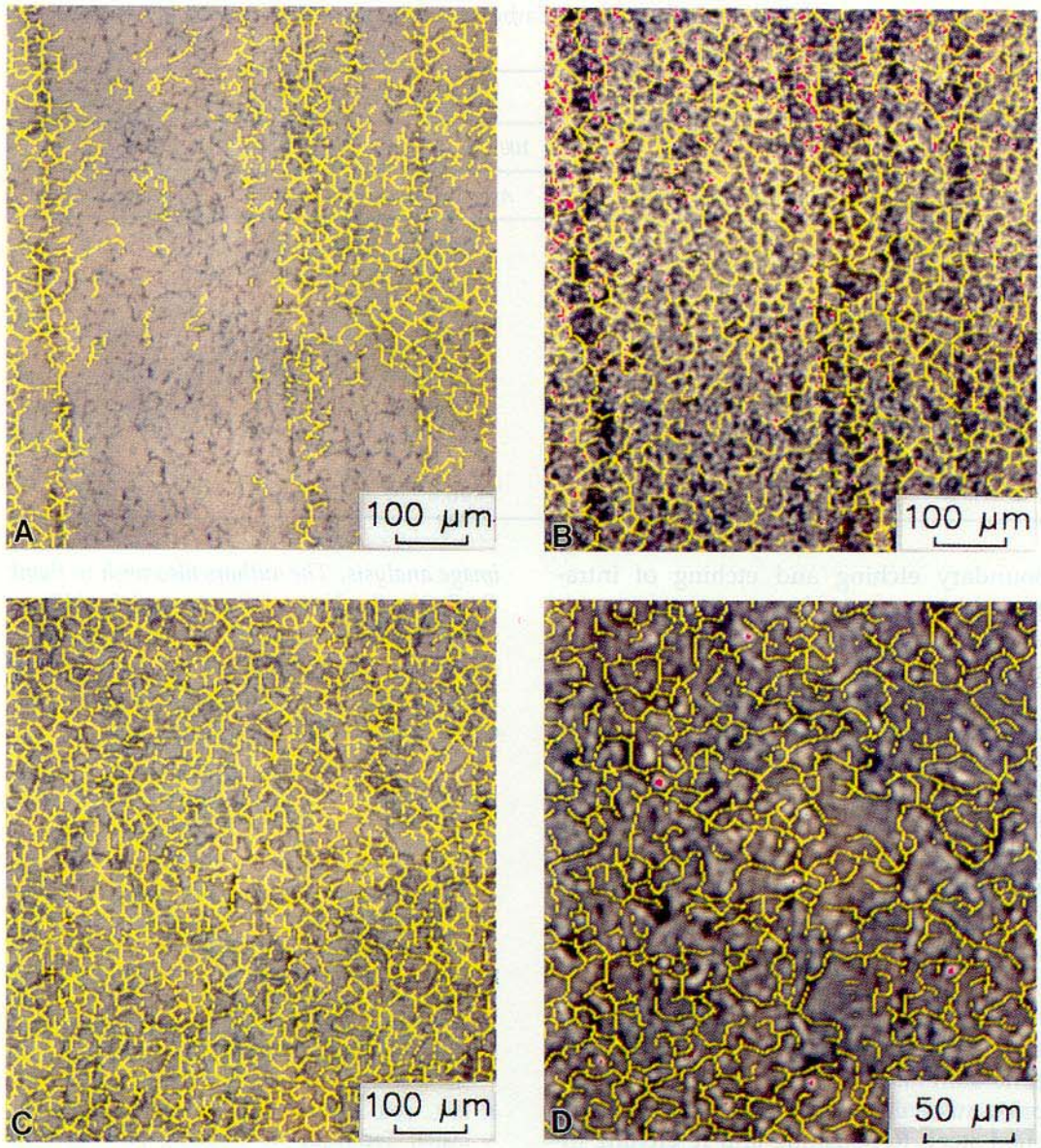

FIG. 2. Reconstructed images of prior austenite grain structures in carburized steels: (a) original structure in focus, (b) original structure overfocused, (c) low magnification of overfocused original structure, and (d) high-magnification reconstruction of same structure as shown in (c).

pares reconstructed images at ×100 and ×250. The lower magnification brings the unetched boundary gaps closer together and permits more extensive boundary reconstruction by the image analyzer. Higher-magnification examination may also emphasize the effect of intragranular etching remanents.

Table 1 compares grain sizes determined by manual lineal intercept analysis and image analysis for three specimens. The intercept counts are considered to be reliable because of the ability of the operator to discard the effect of etching artifacts. The grain sizes measured by image analysis of surfaces at ×100 agree well with those measured by intercept counting. Overfocusing and boundary reconstruction at the low magnification overcome some of the difficulties associated with incomplete

Table 1 Austenite Grain Sizes from Three Carburized Specimens Determined by Intercept Counting and Image Analysis

Intercept counting		Image analyzer			
		× 100		× 250	
ASTM G.S.	Avg. dia. (μm)	ASTM G.S.	Avg. dia. (μm)	ASTM G.S.	Avg. dia. (μm)
8.5	16.5 ± 1.0	8.5	16.7	10.2	9.4
		8.5	16.6	10.2	9.2
		8.5	16.8	10.2	9.4
8.4	17.5 ± 0.8	8.4	17.1	9.9	10.3
		8.6	16.4	10.2	9.4
		8.5	16.8	10.1	9.7
8.6	16.5 ± 0.8	8.4	17.1	9.9	10.4
		8.5	17.0	10.1	9.7
		8.5	16.8	10.0	10.0

boundary etching and etching of intragranular features. However, image analysis of surfaces at ×250 indicate a finer grain size than measured by intercept counting. This result was attributed to the interpretation of intragranular features as grain boundaries by the image analysis system. Also, incomplete grain boundaries, causing grains to be intercepted by the guard frame, were not measured, thus biasing the results toward smaller grains.

SUMMARY

Aqueous solutions saturated with picric acid and containing sodium trideylbenzene sulfonate and HCl effectively reveal prior austenite grain boundaries in hardened steel. Repolishing and re-etching increase grain boundary contrast relative to the matrix, and provide grain boundary networks that can be readily measured by an operator. However, not all boundaries are continuous, and, depending on the care and time available for etching, not all intragranular features can be removed. The latter features cause difficulty in image analysis, but we have found that they can be minimized by defocussing and examining the microstructure at low magnification.

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References

1. G. F. Vander Voort, *Metallography Principles and Practice*, McGraw-Hill Book Company, New York (1984), pp. 219–223.
2. G. Krauss, *Steels: Heat Treatment and Processing Principles*, ASM International, Materials Park, OH (1990), pp. 188–201.
3. G. A. Dreyer, D. E. Austin, and W. D. Smith, New etchant brings out grain boundaries in martensitic steels, *Met. Prog.* 86:116–117 (1964).
4. R. L. Bodnar, V. E. McGraw, and A. V. Brandemarte, Technique for revealing prior austenite grain boundaries in CrMoV turbine rotor steel, *Metallography* 17:109–114 (1984).
5. J. L. Pacheco and G. Krauss, Microstructure and high bending fatigue strength in carburized steel, in *Carburizing: Processing and Performance* (G. Krauss, ed.), ASM International, Materials Park, OH (1989), pp. 227–237.
6. P. J. Kenny, G. S. Cole, D. O. Northwood, J. Wylie, and G. F. Vander Voort, eds. Microstructural Science, *Image Analysis and Metallography*, Vol. 17, The International Metallographic Society, Columbus, OH and ASM International, Materials Park, OH (1989).
7. ASTM E 112-85, *1987 Annual Book of ASTM Standards*, Section 3, Volume 3.01, pp. 403–436.

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