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Revealing Prior-Austenite Grain Boundaries

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The mechanical properties of heat treated alloy steels are strongly influenced by the grain size of the parent austenite phase. But, revealing the prior-austenite grain boundaries (PγGBs or PAGBs) can be difficult, or impossible, depending upon the alloy and its microstructure. In failure analysis work, the PγGBs can only be revealed by etching as other methods, such as the McQuaid-Ehn test, require heat treatments that alter the grain size of the failed part. The first reasonably successful etchant for PγGBs was published in 1955 by Bechet and Beaujard [1] using a saturated aqueous picric acid solution 0.5% of a wetting agent, "Teepol" (sodium alkylsulfonate) at room temperature. This etchant has been the foundation of many subsequent modifications to improve its effectiveness.

The writer tried this etch [2] on specimens of 8620, 4140, and 5160 in the as-quenched condition and after tempering at 400, 800 and 1200 °F using sodium tridecylbenzene sulfonate as the wetting agent. It did not reveal grain boundaries on any of the 8620 specimens. It did reveal the PγGBs on as-quenched and tempered (400 and 800 °F) specimens of 4140 and 5160, but did not reveal them on any specimens tempered at 1200 °F. This etch does not reveal PγGBs in martensitic or bainitic steels with carbon contents below ~0.3%, or with phosphorus contents below ~0.010%, even when subjected to step-embrittlement cycles, or for steels tempered above ~1050 °F. Research has shown that phosphorus must be in the grain boundaries for this etch to reveal the PγGBs at 20 °C.

However, if a small amount of HCl is added, and the etchant is used at ~80-90 °C (results were good at 70 °C when tried on one specimen), these limitations are overcome. Also, etch time has never been more than 2 minutes, a large time savings over the previous etch time of at least 7 minutes. Filtering the solution before use reduces staining/pitting attack. Careful, low-pressure back-polishing on a stationary cloth using an alumina slurry is very effective at reducing extraneous etch detail within the grains and enhancing grain boundary visibility.

Several broken heat treated 4340 alloy steel nut inserts from the riser of an oil rig were examined. To determine if the crack patterns were intergranular, specimens were etched in the saturated aqueous picric acid filtered solution with HCl using Nacconol 90G as the wetting agent (Nacconal is a registered trademark of the Stepan Company, Northfield, Illinois). This is described as sodium alkyl benzene sulfonate. On the MSDS sheet the composition is given as 90-93% sodium dodecylbenzen sulfonate, 5% sodium sulfate, 1% sodium chloride and 1.5% water. HCl was added in the amount of 6 drops per 100 mL of the saturated aqueous picric acid solution (1 mL to 500 mL). After this was mixed, the excess picric acid was removed by filtering. Etching was conducted at room temperature for 7

minutes using the ultrasonic cleaner for agitation. However, the results were marginal. Hence, the writer heated the solution to 80-90 °C (below the boiling point to retard evaporation). Specimens were swab etched for 2 minutes and then back polished. Results were exceptionally good, as shown in Figure 1, revealing an almost fully intergranular crack path from stress-corrosion cracking (bulk hardness was well above the safe limit for high-strength steel in salt water).

References

1. S. Bechet and L. Beaujard, "New Reagent for the Micrographical Demonstration of the Austenite Grain of Hardened or Hardened-Tempered Steels," **Rev. Met**., Vol. 52, 1955, pp. 830-836.

2. G.F. Vander Voort, **Metallography: Principles and Practice**, McGraw-Hill Book Co., NY, 1984 and ASM International, Metals Park, OH, 1999, p. 222.

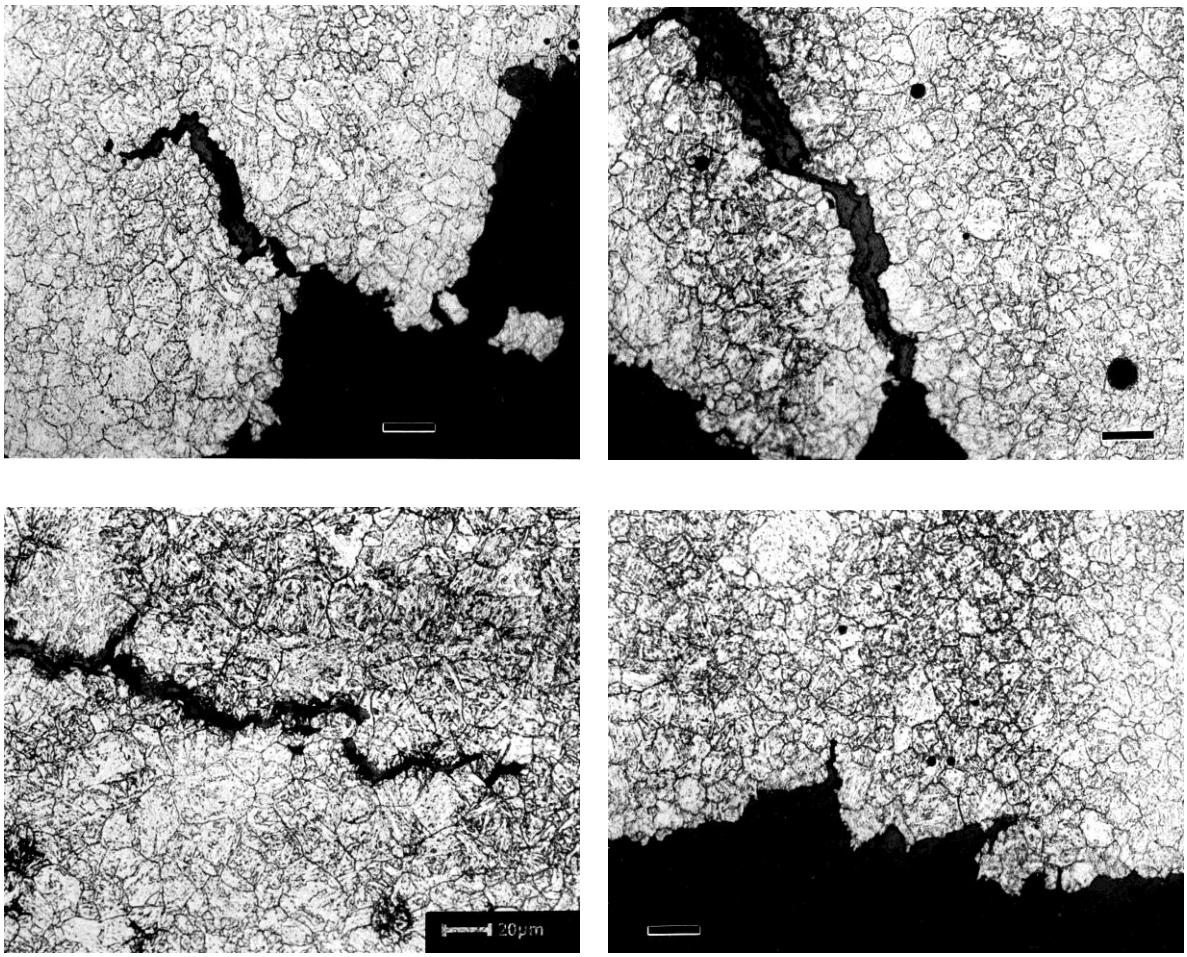

Figure 1. Examples PγGBs revealed in 4340 alloy steel nut inserts that failed due to stress corrosion cracking in sea water (all magnification bars are 20 µm in length).

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