Revealing Prior-Austenite Grain Boundaries

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Examples of Phase Decoration

Depending upon the steel composition, a variety of procedures can be used to decorate the prior-austenite grain boundaries – if the steel can be heat treated. Examples include ferrite or cementite decorating the grain boundaries after slow cooling or after carburizing, or using the oxidation method.

If the specimen can not be heat treated, as in failure studies, we can only use etchants to bring out the prior-austenite grain boundaries.
Examples of Grain Boundary Decoration by a Second Phase

Ferrite precipitated in the prior-austenite grain boundaries in an as-cast Fe-0.38% C – 0.26% Si – 0.79% Mn steel (2% nital).
Examples of Grain Boundary Decoration by a Second Phase

Ferrite decorating prior-austenite grain boundaries in a wrought medium carbon steel, 2% nital
Examples of Grain Boundary Decoration by a Second Phase

Fe – 1.31% C steel, as-rolled, cementite outlining PAGBs: 2% Nital (left), 4% picral (right). Neither clearly reveals the grain boundaries.
Examples of Grain Boundary Decoration by a Second Phase

Fe – 1.31% C as-rolled: Alkaline sodium picrate, 90C, 60 s, 500X (left); Klemm’s I (right); both revealed the grain boundaries.
Examples of Grain Boundary Decoration by a Second Phase

Microstructure of 9310 alloy steel after the McQuaid-Ehn test using alkaline sodium picrate (90 °C – 45 s) to darken the cementite precipitated in the prior-austenite grain boundaries (magnification bar is 100 µm long).
In high-alloy steels it is often possible to precipitate a second phase, such as pearlite, on the prior-austenite grain boundaries so that they can be easily examined. This experimental 5% Cr hot-work die steel specimen was austenitized at 1080 °C, cooled to 700 °C and held for 1 h, then air cooled to precipitate pearlite on the grain boundaries. It was etched with glycerregia. Magnification bar length is 25 µm.
Oxidation Technique

The specimen was ground to a 400-grit SiC finish, then austenitized, quenched, and lightly reground but not polished.
The Most Successful Etchant

Make 500 mL of the following solution using a magnetic stirring plate and an 800 – 1000 mL beaker

1. **Saturate water with picric acid**
2. **Add 1 mL HCl to 500 mL of the saturated solution**
3. **Add 1 large tablespoon of a wetting agent (I use Nacconal 90G, Stepan Co., Northfield, IL)**

Dissolve thoroughly. Filter at least 100 mL into a 240 mL beaker. Heat solution to 80-90 °C. Swab or immerse specimen for 40-60 s. Wash and dry. Examine with LOM. Light back-polishing with 0.05 µm alumina slurry on a stationary cloth to remove excess matrix etching. Etch again and back-polish until the boundaries are clearly revealed.
Prior-austenite grain boundary delineation improved by back-polishing after etching to remove excessive etch attack in A-350 (LF3) alloy steel.
Etching Examples

Specimen B1, mount T3, 2% nital (left, 50X); saturated picric at 20 °C (right, 200X)

Etching with nital reveals packets of lath martensite; the aqueous saturated picric acid solution with HCl and a wetting agent at 20 °C faintly revealed the prior-austenite grain boundaries in SAE 723, Grade 3, Class 3 pressure vessel steel (Fe – 0.33% C – 0.25% Mn – 0.13% Si – 3.55% Ni – 1.66% Cr – 0.48% Mo – 0.12% V)
Using aqueous saturated picric acid plus HCl and a wetting agent at 90 °C, however, revealed the prior-austenite grain boundaries (left: bright field illumination; right: dark field illumination); in SAE 723, Grade 3, Class 3 pressure vessel steel.
Etching Examples

Specimen B1, mount L1, 2% nital (left, 50X); saturated picric at 90°C (right, 200X)

Etching with nital reveals packets of lath martensite; the aqueous saturated picric acid solution with HCl and a wetting agent at 90 °C revealed the prior-austenite grain boundaries in SAE 723, Grade 3, Class 3 pressure vessel steel (Fe - 0.33% C – 0.25% Mn – 0.13% Si – 3.55% Ni – 1.66% Cr – 0.48% Mo – 0.12% V )
Etching Examples

Specimen B1, Mount L1, saturated picric at 90 °C viewed with DF at 200X)

Using aqueous saturated picric acid plus HCl and a wetting agent at 90 °C revealed the prior-austenite grain boundaries as shown in dark field illumination); in SAE 723, Grade 3, Class 3 pressure vessel steel.
Prior-austenite grain boundaries revealed in martensitic A-350 (LF3) alloy steel (Fe – 0.07% C – 0.74% Mn – 3.66% Ni – 0.2% Cr – 0.07% Mo (1350 °F temper) using aqueous saturated picric acid plus HCl and a wetting agent.
Etching Examples

As Quenched

Prior-austenite grain boundaries revealed in 8620 alloy steel using aqueous saturated picric acid plus HCl and a wetting agent at 80-90 ºC, 60s.

400 ºF Temper
Etching Examples

800 °F Temper

1200 °F Temper

Prior-austenite grain boundaries revealed in 8620 alloy steel using aqueous saturated picric acid plus HCl and a wetting agent at 80-90 °C, 60s.
Prior-austenite grain boundaries revealed in fully martensitic 4140 alloy steel using aqueous saturated picric acid plus HCl and a wetting agent at 80-90 °C.
Prior-austenite grain boundaries revealed in fully martensitic 4140 alloy steel using aqueous saturated picric acid plus HCl and a wetting agent at 80-90 °C.
Prior-austenite grain boundaries revealed in two alloy steels isothermally transformed to lower bainite using aqueous saturated picric acid plus HCl and a wetting agent.
Etching Examples

Prior-austenite grain boundaries in 4340 alloy steel isothermally transformed to lower bainite using aqueous saturated picric acid plus HCl and a wetting agent.
Prior-austenite grain boundaries in 4340 alloy steel isothermally transformed to lower bainite using aqueous saturated picric acid plus HCl and a wetting agent.
Prior-austenite grain boundaries revealed in fully martensitic 4340 alloy steel using aqueous saturated picric acid plus HCl and a wetting agent.
Stress-corrosion cracks (SCC) in 4340 alloy steel nut risers (39 HRC) etched with saturated aqueous picric acid, 80 °C, 500X (magnification bars are 20 um long).
Prior-austenite grain boundaries in 43B17 alloy steel (Fe – 0.17% C – 1.85% Ni – 0.75% Cr – 0.25% Mo – 0.003% B) revealed by etching with aqueous saturated picric acid plus 1% HCl and a wetting agent. The specimen at the left was given a normal heat treatment (843 °C – 30 min, oil quench, 230 °C temper) and has a fine grain size. The specimen on the right was “mock carburized” (925 °C for 8 h, oil quench and tempered at 230 °C) and rapid grain growth has begun. This specimen is not suitable for carburizing.
Prior-austenite grain boundaries revealed in fully martensitic Modified 4330V alloy steel (Fe – 0.29% C – 0.39% Mn – 3.54% Ni – 1.69% Cr -0.54% Mo – 0.11% V (1110 °F temper) using aqueous saturated picric acid plus HCl and a wetting agent.
Etching Examples

Prior-austenite grain boundaries revealed in fully martensitic 5160 alloy steel using aqueous saturated picric acid plus HCl and a wetting agent.

5160, 1525 °F – OQ – 300 °F Temper

Prior-austenite grain boundaries revealed in fully martensitic 5160 alloy steel using aqueous saturated picric acid plus HCl and a wetting agent.
Alternative Etchants
Prior-austenite grain boundaries can be revealed easily in precipitation hardenable stainless steels and in maraging steels if they are in the solution annealed condition (before aging). This specimen of 17-4PH stainless steel (Fe - <0.07% C – 17% Cr – 4% Ni – 4% Cu – 0.3% Nb) was solution annealed at 1038 °C. It was etched with aqueous 60% HNO₃, 1.1 V dc, Pt cathode, 30 s.

If they have been aged, it is more difficult but superpicral has been found to work.
Prior-austenite grain boundaries

18Ni250 solution annealed and aged maraging steel. Modified Fry’s reagent almost brings out the prior-austenite grain boundaries (magnification bar is 25 µm long).
Examples of Grain Boundary Decoration by a Second Phase