

# ETCHING ISOTHERMALLY TREATED STEELS

*Relative advantages of using nital, picral, and 10% sodium metabisulfite, an underutilized tint etchant, to explore the microstructures of three low-alloy steels.*

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**T**he microstructure of iron-based alloys is very complicated, being influenced by composition, homogeneity, processing, and section size. Microstructures of coarse-grained steels are much easier to observe than those of fine-grained steels. Of course, steels are normally made with a fine grain size to optimize their mechanical properties.

In general, it is easiest to identify heat treated structures after transformation and before tempering. However, in most applications, hardened steels must be tempered and are usually examined in this condition. If a mixed microstructure of bainite and martensite is formed during quenching, these constituents will become more difficult to identify reliably as the tempering temperature increases toward the lower critical temperature ( $A_{c1}$ ).

These factors make it more difficult to identify phases and constituents in steels. Further, while ferrous metallographers tend to use nital almost exclusively for etching, nital is not always the best reagent for properly revealing all microstructures. It is unfortunate that some companies prohibit use of picral for the reason that picric acid can be made to detonate under certain conditions. Picral is an excellent etchant for revealing certain microstructural constituents in steel and, frankly, accidents have been less common with it than with nital.

This article compares the advantages of using nital, picral, and 10% aqueous sodium metabisulfite (SMB), a tint etchant, as aids to phase/con-

stituent identification in the isothermally transformed microstructures of low-alloy steels containing 0.2, 0.4, and 0.6% carbon (SAE 8620, 4140, and 5160, respectively).

SMB deserves to be more widely used. It behaves much like nital but is more uniform in its action. And unlike picral, it will reveal the structure of as-quenched martensite. It also is excellent for revealing the diffusion-controlled products ferrite, pearlite, and bainite, as well as the diffusionless product, martensite. In addition, 10% SMB often gives the best contrast, and is safer to use than either nital or picral.

## **Specimen preparation basics**

To observe the microstructure of ferrous metals, they must be properly prepared. Many view this task as a trivial exercise, yet its proper execution is critical to successful interpretation.

**Sampling:** The first step is to select the test locations to be sampled. This is critical if the interpretation is to be valid for the part or lot being evaluated. Specimens must be representative of the lot. Note that the plane of polish may be oriented in different directions relative to the piece being sampled.

Sectioning is almost always required to obtain a piece of the proper size and orientation for subsequent metallographic examination. The abrasive cut-off saw is the most commonly used sectioning device. It produces good surfaces with minimal damage, when the proper blade is used with adequate coolant. More aggressive sectioning methods are often used in production operations. These tend to cause greater damage, which must be removed if the true structure is to be

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examined.

**Mounting:** The specimen is then mounted in a polymeric material to facilitate handling, simplify preparation, enhance edge retention, and/or make it easy to identify the specimen. Mounting may be done in a press using a thermosetting or thermoplastic resin, or with a castable resin that does not require external heat and pressure for polymerization.

**Grinding and polishing:** The use of automation in specimen preparation has grown enormously over the past 25 years. Automatic grinder/polisher machines produce better results than can be achieved manually. They yield more consistent results, better flatness, and better edge retention, with greater productivity.

There is no one correct procedure for successfully preparing ferrous metal specimens. Many procedures and many different products can be used, although some methods may favor certain types of specimens or be better suited to address specific problems. The procedures given in the table (last page of article) were used to prepare the specimens discussed here, and are appropriate for most steel specimens. They give consistent results with good edge retention.

Other variations are possible depending upon needs and specimens. The first step, often called planar

grinding, can be done using several products. The traditional silicon carbide paper is always satisfactory, but has a short life. Aluminum oxide paper may also be used. Always start with the finest possible abrasive that can remove the damage from cutting and get all of the specimens in the holder co-planar in a reasonable time. Note that continuing to grind after the paper has lost its cutting efficiency will generate heat and damage.

Resin- or metal-bonded grinding disks are excellent for obtaining flatness and edge retention and yield high stock removal rates. Their surface is covered by small, diamond-containing pads. Diamond-free regions surrounding the pads reduce surface tension and increase cutting efficiency. These disks have a long life. Use metal-bonded disks for harder ferrous alloys and resin-bonded disks for the softest steels.

Rigid grinding discs (RGDs) can be used for the second step, and even for the first (planar) grinding step with coarse diamond (30 to 45  $\mu\text{m}$ ). They also yield very flat surfaces and are recommended when edge retention is critical. These disks do not contain embedded abrasive; rather, diamond is added periodically to the surface, usually as a suspension. "Universal" RGDs suitable for preparing most steels, and ones designed for the softest grades are available.

There are cloth alternatives for the second step that work well, but they don't last as long as an RGD.

**Etching:** Etchants for steels are listed in many standard textbooks<sup>1</sup> and handbooks<sup>2</sup>, and in ASTM E 407-99, "Standard Practice for Mi-

croetching Metals and Alloys."

Nital, usually 2%, is the most commonly used etchant for steel specimens. It is excellent for revealing the structure of martensite. Nital is also very good for revealing ferrite in a martensitic matrix and for bringing up ferrite grain boundaries in low-carbon steels. Recommended nital composition: 1 to 5 mL nitric acid ( $\text{HNO}_3$ ), 100 mL pure ethanol. (Avoid using methanol as it is a cumulative poison.)

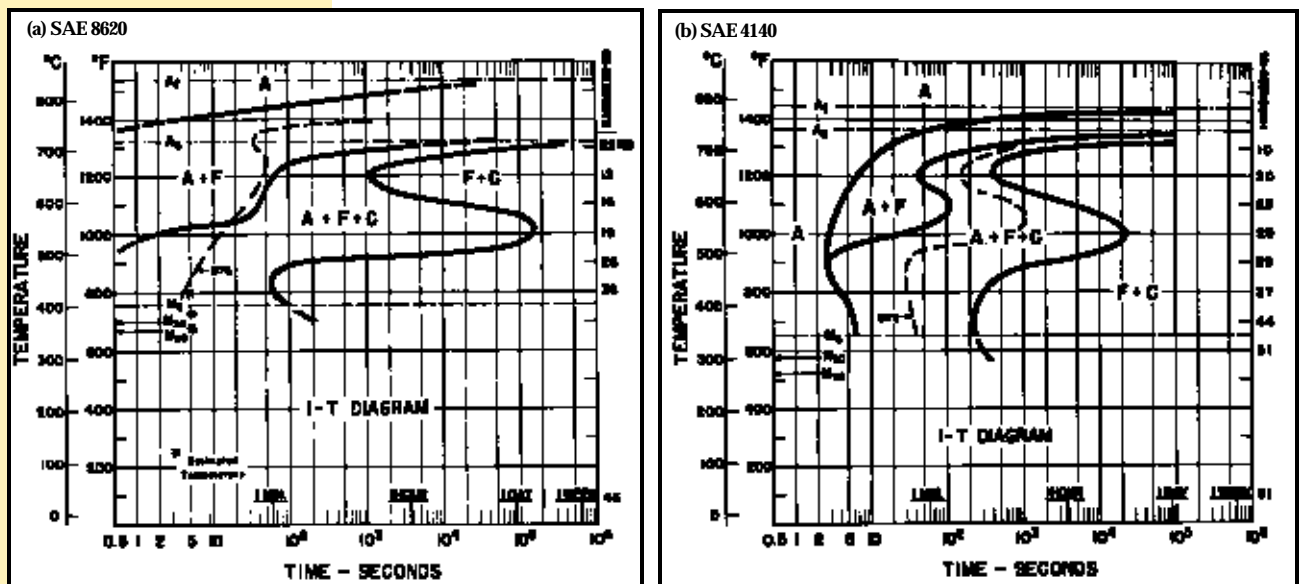
Picral is better for revealing the cementite in ferritic alloys and the structure of the ferrite-cementite constituents pearlite and bainite. Recommended picral composition: 4 g picric acid (trinitrophenol,  $(\text{NO}_2)_3\text{C}_6\text{H}_2\text{OH}$ ), 100 mL pure ethanol.

Nital and picral both dissolve ferrite, but nital's dissolution rate is a function of crystal orientation while picral's is uniform. Other reagents have their uses, especially when dealing with higher alloy grades or when trying to selectively reveal certain constituents or prior-austenite grain boundaries.

**Tint etchant:** Because some laboratories are prohibited from using picral, the specimens prepared for this article were also etched with 10% aqueous sodium metabisulfite (abbreviated SMB, and also called sodium pyrosulfite), which has many of the virtues of both nital and picral. It is prepared by adding 10 g sodium metabisulfite ( $\text{Na}_2\text{S}_2\text{O}_5$ ) to 100 mL of water. Compared with other tint etchants, it is relatively simple to use. Etching of these specimens was by immersion for 5 to 15 seconds. Never swab.

SMB will etch a wide range of com-

Fig. 1 — Isothermal transformation (IT) or time-temperature-transformation (TTT) diagrams for three low-alloy steels: (a) SAE 8620, (b) SAE 4140, and (c) SAE 5160.  $A_f$  and  $A_s$  are equivalent to the upper and lower critical temperatures  $A_{c3}$  and  $A_{c1}$ , respectively. A = austenite, F = ferrite, C = cementite or carbide. (Ref. 5)



positions and does produce coloration, which is not strong, but can be enhanced using partially crossed polarizing filters with a sensitive-tint plate. It will lightly color ferrite (like nital or picral) and reveal ferrite grain boundaries (like nital) but with greater uniformity. It will also reveal pearlite and bainite as well as picral, and will reveal as-quenched martensite.

The etchant is quite safe to use. Of course, it should not be ingested and direct contact should be avoided.

### Isothermal transformations

Microstructures, even in steels, were not well understood and heat treatment was more of an art than a science until Davenport and Bain published their landmark paper on the isothermal transformation of austenite.<sup>3</sup> This led to the development of isothermal transformation (IT) or time-temperature-transformation (TTT) diagrams for each steel composition.<sup>4,5</sup> They were developed for many compositions using several approaches.<sup>6</sup> Isothermally transformed specimens are highly useful in teaching interpretation of microstructures.

Austenite transformation also can be studied using controlled cooling rates and can be plotted on continuous cooling-transformation (CCT) diagrams. In this article, only isothermally transformed structures are illustrated (although the untransformed austenite is converted to martensite during the quench after the isothermal hold).

The carbon content of a steel is a major factor influencing the amount and appearance of phases and constituents. Three common alloy steels of different carbon content were se-

lected for this work: SAE 8620, 4140, and 5160. They were austenitized at the recommended temperatures and then held isothermally at different subcritical (below  $A_{c1}$ ) temperatures to convert some or all of the austenite to other phases and constituents. The TTT diagrams for these steels are reproduced in Fig. 1.

**Terminology:** There is confusion about and misuse of certain microstructural terms. Sorbite and troostite, for example, were dropped from the metallographic lexicon in 1937 because they referred to microstructural constituents inaccurately. However, they are still occasionally used.

"Phase" is often used incorrectly in reference to mixtures of two phases, such as pearlite or bainite, which are more properly called "constituents." A phase is a homogeneous, physically distinct substance. Martensite is a phase when formed by quenching but becomes a constituent after tempering when it decomposes from body-centered tetragonal (bct) martensite to body-centered cubic (bcc) ferrite and orthorhombic cementite.

### Proeutectoid ferrite

Alpha iron, strictly speaking, refers only to the bcc form of pure iron that is stable below 912°C (1674°F), while ferrite is a solid solution of one or more elements in bcc iron. Often these terms are incorrectly used as synonyms. Ferrite may precipitate from austenite in acicular form (Widmanstätten ferrite) under certain cooling conditions. Sheet steels are ferritic, and there are ferritic silicon electrical steels and stainless steels. Ferrite is a very soft, ductile phase, although it loses its toughness below some critical temperature.

If the isothermal transformation temperature is close to the lower critical temperature ( $A_{c1}$ ) and the steel is hypoeutectoid (<0.8% C), ferrite will precipitate first before pearlite is formed. This ferrite is called proeutectoid ferrite.

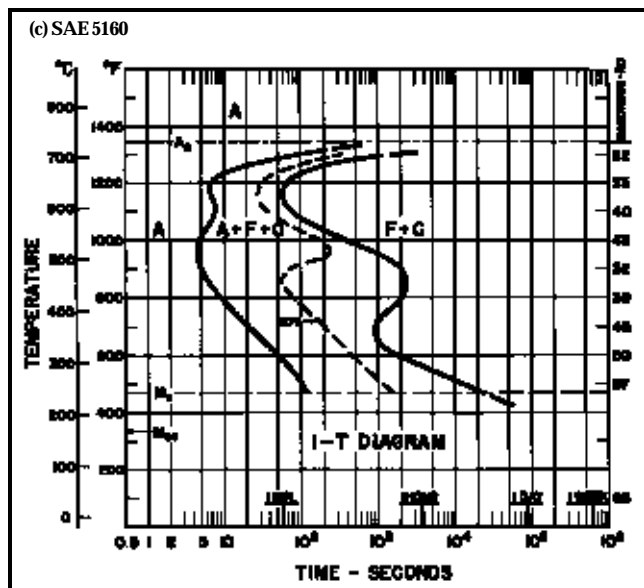
Figures 2, 3, and 4 show proeutectoid ferrite and martensite in 8620, 4140 and 5160, respectively, etched with 2% nital, 4% picral and 10% SMB. For the 8620 and 4140 specimens, the

holding times were short enough so that only ferrite formed isothermally before the specimens were water quenched (which transformed the remaining austenite to martensite). However, proeutectoid ferrite is much harder to form in 5160, due to its high carbon content. Note that there is no separate region on the 5160 TTT diagram (Fig. 1) indicating formation of proeutectoid ferrite. So, for the 5160 specimen, we observe a few tiny patches of proeutectoid ferrite (much less than 1% by volume), pearlite, and as-quenched martensite.

**SAE 8620:** Figure 2 shows that nital and 10% SMB revealed the martensite (m), SMB slightly colored some of the ferrite grains (f), and both etchants revealed the ferrite grain boundaries, while 4% picral revealed nothing. (The slight relief between the ferrite and pearlite is due to hardness differences, and was revealed as the aperture diaphragm was stopped down.) The 10% SMB etch colored some of the ferrite grains, darkened the martensite more than nital, and revealed more of the ferrite-ferrite grain boundaries than nital.

**SAE 4140:** Figure 3 shows proeutectoid ferrite (arrows) and as-quenched martensite in 4140 with similar results. Of course, due to its higher carbon content, less proeutectoid ferrite can form. The picral etch produced only the slightest degree of etching of the martensite, which has been enhanced by stopping down the aperture diaphragm. Again, the 10% SMB etch gives the best contrast.

**SAE 5160:** Results for the 5160 specimens, Fig. 4, are somewhat different. Each field was selected to show a very small amount of proeutectoid ferrite (arrows). These are not typical fields, as most fields contained no ferrite. The picral etch shows darkly etched pearlite (p) and large white patches that are as-quenched martensite (m). It might be hard to say that the small patches of proeutectoid ferrite are, or are not, as-quenched martensite based upon this etch. Nital is not much more help. The large, light patches appear to have some faint structure in them, but it is unclear if this is as-quenched martensite, ferrite, or lightly etched pearlite. Viewed through the green filter in black and white, Fig. 4(c), the 10% SMB etch reveals all of the constituents, and as-quenched martensite can be distinguished from fine pearlite, but this is not simple. (Note: The green filter was required for black





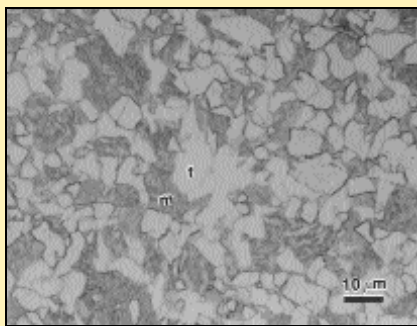
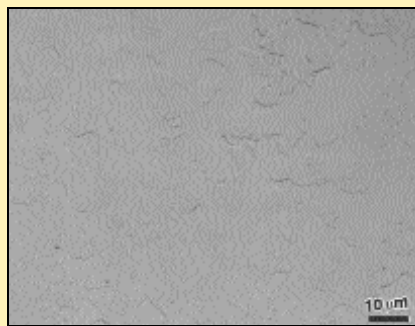
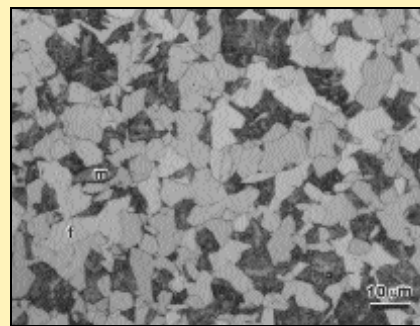


Fig. 2 — (a) SAE 8620, 2% nital, 1000X.



(b) SAE 8620, 4% picral, 1000X.



(c) SAE 8620, 10% SMB, 1000X.

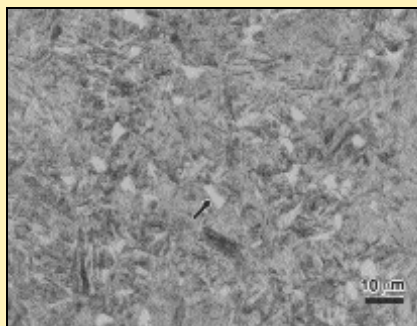
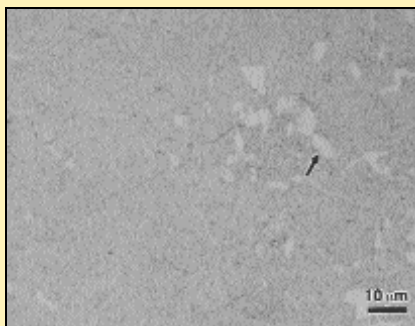
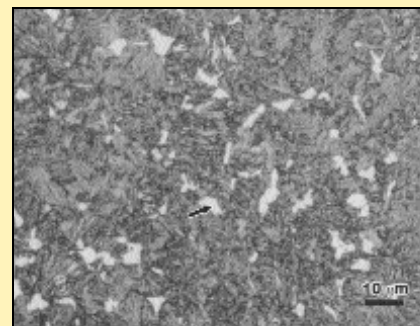


Fig. 3 — (a) SAE 4140, 2% nital, 1000X.



(b) SAE 4140, 4% picral, 1000X.



(c) SAE 4140, 10% SMB, 1000X.

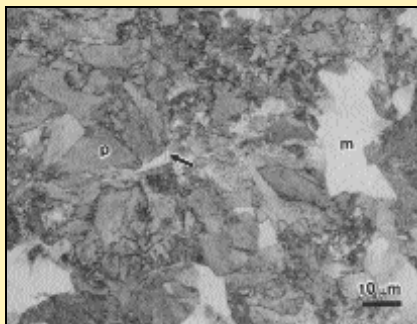
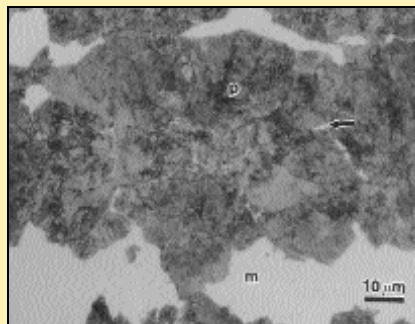
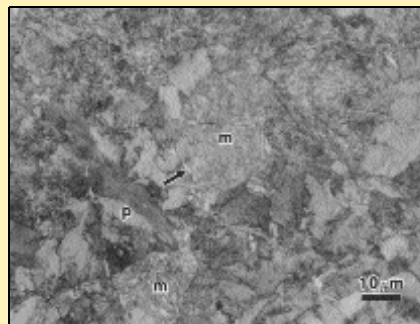


Fig. 4 — (a) SAE 5160, 2% nital, 1000X.

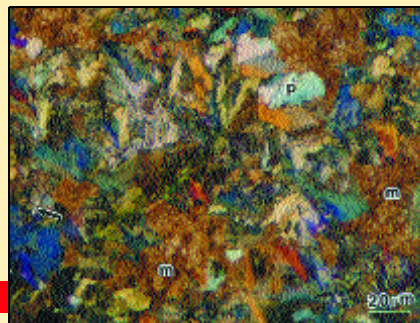


(b) SAE 5160, 4% picral, 1000X.



(c) SAE 5160, 10% SMB, green filter, 1000X.

**Proeutectoid ferrite** (*f* for arrows) and as-quenched martensite (*m*) formed in isothermally transformed low-alloy steels. Fig. 2: SAE 8620; austenitized at 925°C (1700°F) for 30 minutes, isothermally transformed at 675°C (1250°F) for 1 minute, water quenched. Fig. 3: SAE 4140; 845°C (1550°F) for 30 minutes, 675°C (1250°F) for 30 seconds, water quenched. Fig. 4: SAE 5160; 830°C (1525°F) for 30 minutes, 675°C (1250°F) for 30 seconds, water quenched. *p* = pearlite. SMB = sodium metabisulfite. Part. XP+ST = partially crossed polarizing filters plus sensitive-tint plate.



(d) SAE 5160, 10% SMB, part. XP+ST, 500X.

and white photography.)

Viewed in color, Fig. 4(d), the distinction between as-quenched martensite and fine pearlite is quite clear. The colors were enhanced by using partially crossed polarizing filters and a sensitive-tint plate.

#### Austenite: parent phase

Gamma iron, as with alpha iron, pertains to only the face-centered cubic (fcc) form of pure iron that is stable between 912 and 1394°C (1674 and

2541°F), while austenite is a solid solution of one or more elements in fcc iron. Again, these terms are often used interchangeably, which is not correct. For heat treatable steels, austenite is the parent phase for all transformation products that make ferrous alloys so versatile and useful commercially.

Austenite is not stable at room temperature in ordinary steels. It is, however, stable at room temperature in the important family of Cr-Ni stainless steels (the AISI 300 series). Austenite

is a soft, ductile phase that can be work hardened to high strength levels, particularly in the fully austenitic Hadfield manganese steels (~1.2% C, 13% Mn). Austenite is not treated further in this article.

#### Ferrite and pearlite

Carbon in iron exists as either graphite or as cementite. Graphite is the stable form of carbon in iron (mainly observed in cast iron), while cementite is metastable and can trans-

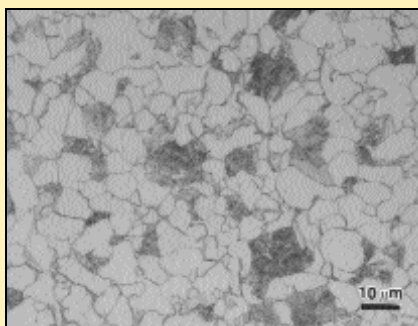
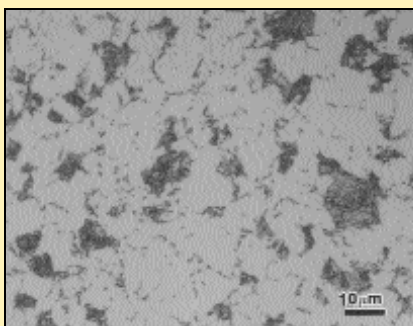
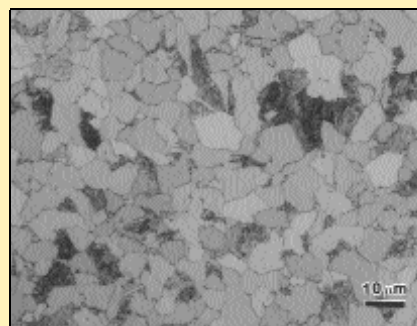


Fig. 5 — (a) SAE 8620, 2% nital, 1000X.



(b) SAE 8620, 4% picral, 1000X.



(c) SAE 8620, 10% SMB, 1000X.

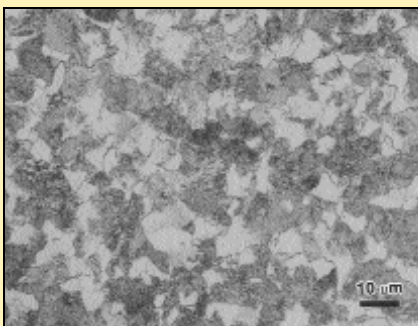
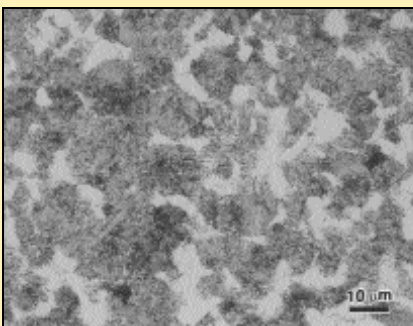
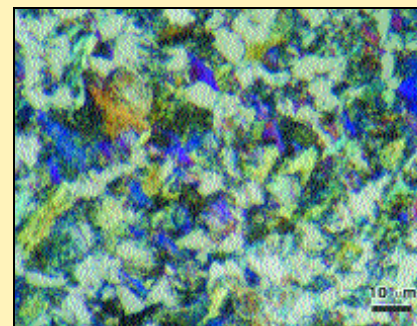


Fig. 6 — (a) SAE 4140, 2% nital, 1000X.



(b) SAE 4140, 4% picral, 1000X.



(c) SAE 4140, 10% SMB, part. XP+ST, 1000X.

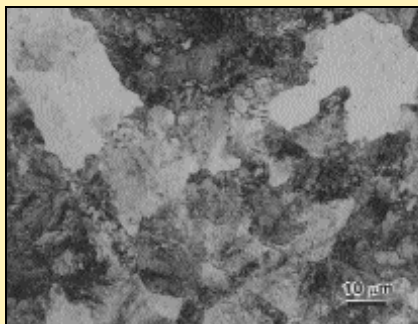
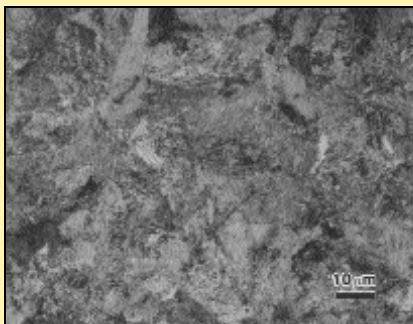
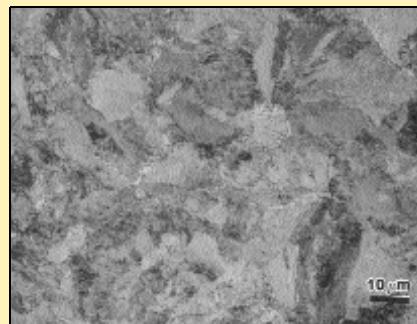


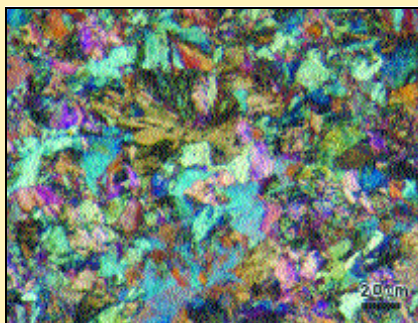
Fig. 7 — (a) SAE 5160, 2% nital, 1000X.



(b) SAE 5160, 4% picral, 1000X.



(c) SAE 5160, 10% SMB, green filter, 1000X.



(d) SAE 5160, 10% SMB, part. XP+ST, 500X.

**Proeutectoid ferrite and pearlite** formed in isothermally transformed low-alloy steels. Fig. 5: SAE 8620; austenitized at 925°C (1700°F) for 30 minutes, isothermally transformed at 675°C (1250°F) for 30 minutes, water quenched. Fig. 6: SAE 4140; 845°C (1550°F) for 30 minutes, 675°C (1250°F) for 45 minutes, water quenched. Fig. 7: SAE 5160; 830°C (1525°F) for 30 minutes, 675°C (1250°F) for 30 minutes, water quenched. SMB = sodium metabisulfite. Part. XP+ST = partially crossed polarizing filters plus sensitive-tint plate.

form to graphite under long-term, high-temperature exposure. Cementite is a compound of iron and carbon with the approximate formula  $\text{Fe}_3\text{C}$  and an orthorhombic crystal structure. Some substitution of other carbide forming elements, such as manganese and chromium, is possible. Hence, it is more general to refer to the formula as  $\text{M}_3\text{C}$ , where M stands for metal. But, note that only small amounts of the various carbide forming elements can be substituted before alloy car-

bides of other crystal structures and formulas are formed.

The carbon content of cementite is 6.67 wt%, which is usually the terminus for the Fe-C phase diagram. Cementite is hard — about 800 HV for pure  $\text{Fe}_3\text{C}$ , and up to about 1400 HV for highly alloyed  $\text{M}_3\text{C}$  — but brittle.

**Pearlite:** If a hypoeutectoid carbon or low-alloy steel is air cooled after austenitizing, or isothermally transformed just below the critical temperature, a diffusion-controlled transfor-

mation occurs where ferrite precipitates first, followed by pearlite. Pearlite is a metastable lamellar aggregate of ferrite and cementite. Pearlite forms by a eutectoid reaction. A eutectoid transformation is an isothermal, reversible reaction in which a solid solution (austenite) is converted into two intimately mixed solid phases (ferrite and cementite). All eutectoid products are lamellar, even in nonferrous systems.

Figures 5, 6, and 7 show the com-



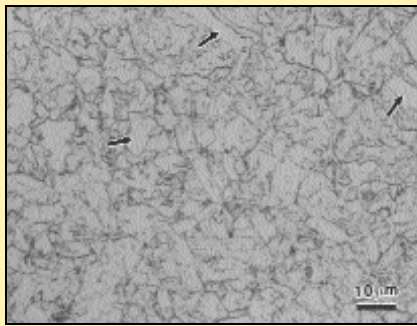
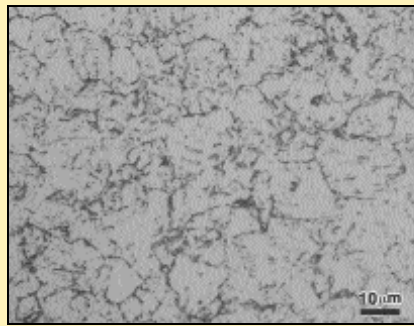
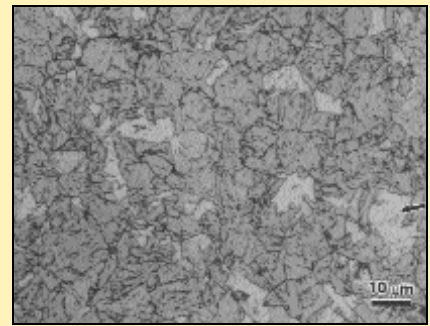


Fig. 8 — (a) SAE 8620, 2% nital, 1000X



(b) SAE 8620, 4% picral, 1000X.



(c) SAE 8620, 10% SMB, 1000X.

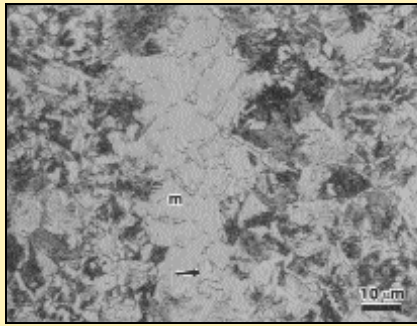
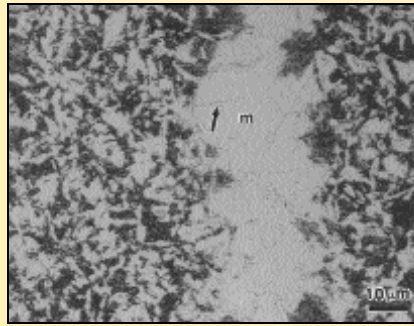
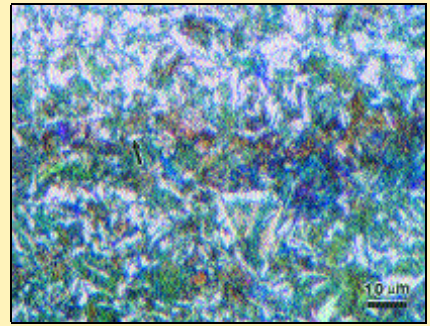


Fig. 9 — (a) SAE 4140, 2% nital, 1000X.



(b) SAE 4140, 4% picral, 1000X.



(c) SAE 4140, 10% SMB, part. XP+ST, 1000X.

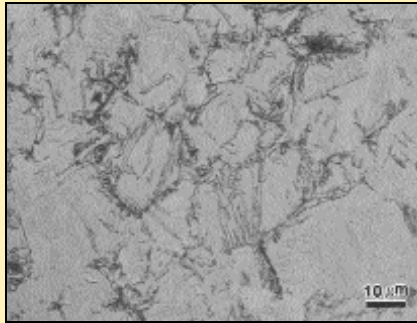
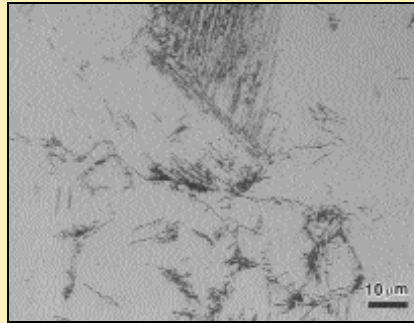
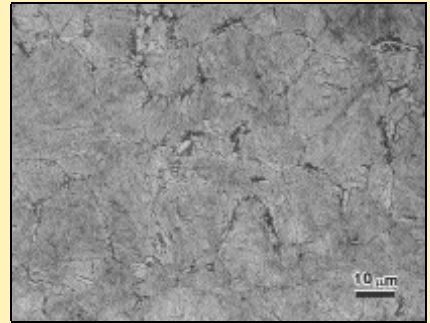


Fig. 10 — (a) SAE 5160, 2% nital, 1000X.

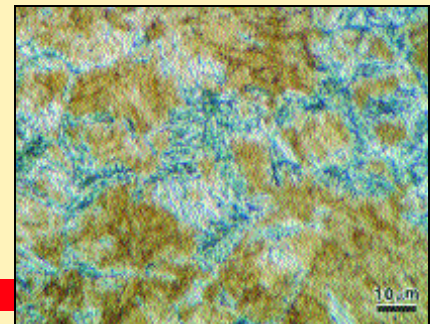


(b) SAE 5160, 4% picral, 1000X.



(c) SAE 5160, 10% SMB, 1000X.

**Upper bainite** and as-quenched martensite formed in isothermally transformed low-alloy steels. Fig. 8: SAE 8620; austenitized at 925°C (1700°F) for 30 minutes, isothermally transformed at 540°C (1000°F) for 60 minutes, water quenched. Arrows point to as-quenched martensite. Fig. 9: SAE 4140; 845°C (1550°F) for 30 minutes, 565°C (1050°F) for 15 minutes, water quenched. The arrows point to the start of bainite formation in a segregate-rich band of light-etching martensite (m). Fig. 10: SAE 5160; 830°C (1525°F) for 30 minutes, 540°C (1000°F) for 60 seconds, water quenched. SMB = sodium metabisulfite. Part. XP+ST = partially crossed polarizing filters plus sensitive-tint plate.



(d) SAE 5160, 10% SMB, 1000X.

plete transformation of austenite to ferrite and pearlite in the 8620, 4140, and 5160 steels when etched with nital, picral, and SMB. As would be expected, the amount of ferrite decreases and the amount of pearlite increases as the carbon content is increased from about 0.2 to about 0.6%.

**8620:** Figure 5 shows that nital and SMB produced similar results for ferrite and pearlite in 8620, although there is some coloring of the proeu-

tectoid ferrite grains by SMB. Picral, as usual, did not reveal the ferrite grain boundaries. As a result, one can see more easily the many small particles of cementite that are present at ferrite grain boundaries.

**4140:** For 4140 (Fig. 6), nital produced light etching of some of the pearlite, and it is hard to tell if these patches are pearlite or as-quenched martensite. 10% SMB did an excellent job of revealing the structure.

**5160:** For 5160 (Fig. 7), nital again produced lightly etched patches of pearlite that could be easily misidentified as as-quenched martensite, or maybe even ferrite. It is quite clear, using either picral or SMB, that the structure is close to 100% pearlite with a very fine interlamellar spacing.

#### Upper or 'feathery' bainite

If the isothermal transformation temperature is lowered to below the



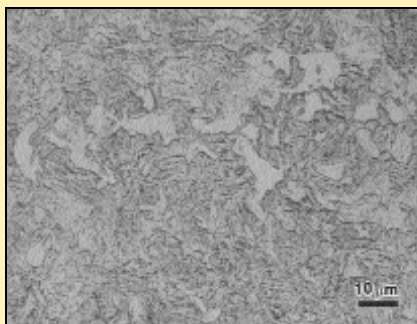
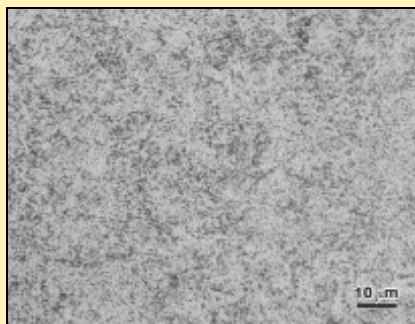
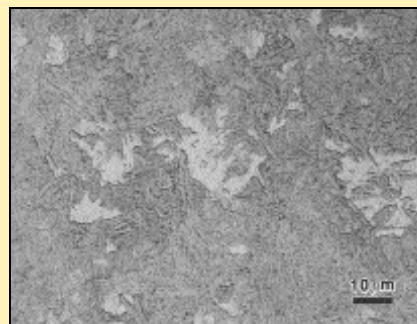


Fig. 11 — (a) SAE 8620, 2% nital, 1000X.



(b) SAE 8620, 4% picral, 1000X.



(c) SAE 8620, 10% SMB, 1000X.

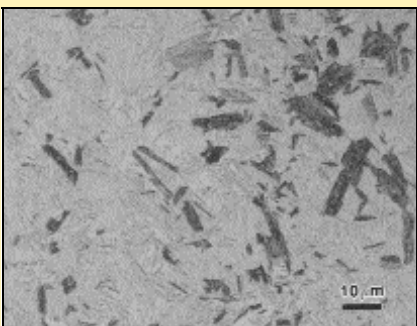
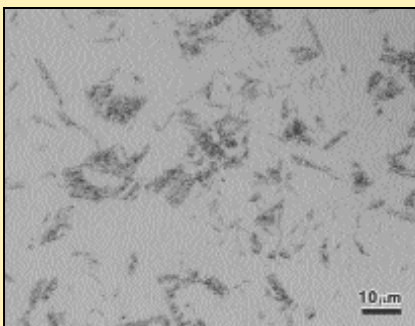
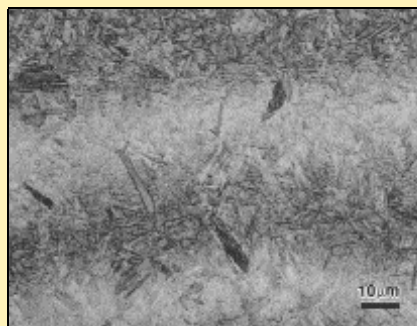


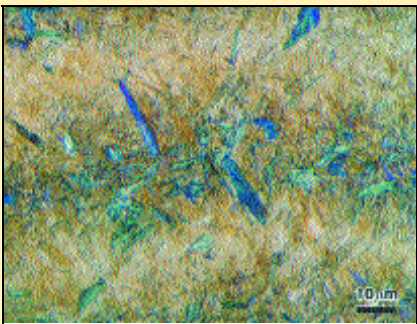
Fig. 12 — (a) SAE 4140, 2% nital, 1000X.



(b) SAE 4140, 4% picral, 1000X.



(c) SAE 4140, 10% SMB, green filter, 1000X.



(d) SAE 4140, 10% SMB, part XP+ST, 1000X.

**Fully and partially transformed lower bainite** in isothermally transformed low-alloy steels. Fig. 11: Fully transformed SAE 8620; austenitized at 925°C (1700°F) for 30 minutes, isothermally transformed at 425°C (800°F) for 5 minutes, water quenched. Fig. 12: Lower bainite and as-quenched martensite in SAE 4140; 845°C (1550°F) for 30 minutes, 415°C (775°F) for 30 seconds, water quenched. Fig. 13: Lower bainite and as-quenched martensite (m) in SAE 5160; 830°C (1525°F) for 30 minutes, 345°C (650°F) for 5 minutes, water quenched. SMB = sodium metabisulfite. Part. XP+ST = partially crossed polarizing filters plus sensitive-tint plate.

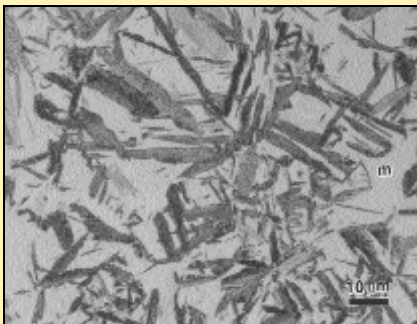
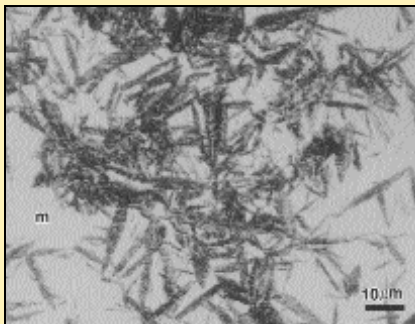
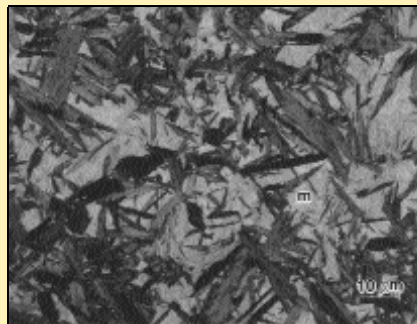


Fig. 13 — (a) SAE 5160, 2% nital, 1000X.



(b) SAE 5160, 4% picral, 1000X.



(c) SAE 5160, 10% SMB, 1000X.

“nose” of the TTT curve, but not below the temperature where martensite starts to form (the  $M_s$  temperature), a different two-phase constituent, called bainite, may be observed. This is a metastable aggregate of ferrite and cementite that forms from austenite at temperatures below where pearlite forms and above the temperature where martensite starts to form. The appearance of bainite changes with the transformation tem-

perature, being called “feathery” in appearance at high temperatures and “acicular” at low temperatures. The feathery appearance of “upper” bainite is also influenced by carbon content and is most often associated with high-carbon steels. “Acicular,” however, is not a perfect descriptor of the shape of “lower” bainite.

Figures 8, 9, and 10 show upper bainite in the three low-alloy steels, as revealed by nital, picral and SMB. Note

the substantial difference in its appearance as a function of the steel’s carbon content.

**8620:** Figure 8 shows 8620 isothermally transformed at 1000°F (540°C). Note that it has a mixture of upper bainite and as-quenched martensite (arrows). Picral is the best etchant for showing only the upper bainite, while nital and SMB reveal both constituents, but their identification is very difficult.

## Typical automated specimen preparation practices for steels<sup>1</sup>

Surface	Abrasive and size	Load, lbf (N) <sup>2</sup>	Speed, rpm, and direction <sup>3</sup>	Time, min.
Ultra-Prep metal- or resin-bonded disks	45 or 30 µm diamond, water cooled	6 (27)	240–300, Comp.	Until plane
BuehlerHercules H or S rigid grinding disks	9 µm Metadi Supreme diamond suspension	6 (27)	120–150, Comp.	5
Texmet 1000 or Trident cloths <sup>4</sup>	3 µm Metadi Supreme diamond suspension	6 (27)	120–150, Comp.	3
Microcloth, Nanocloth, or Chemomet I cloths	Masterprep 0.05 µm alumina suspension, or Mastermet colloidal silica	6 (27)	120–150, Contra	2
<b>Stainless and maraging steels</b>				
Carbimet waterproof paper	120 or 240 grit SiC, water cooled	6 (27)	240–300, Comp.	Until plane
BuehlerHercules H or S rigid grinding disks	9 µm PC Metadi Supreme diamond suspension	6 (27)	120–150, Comp.	5
Texmet 1000 or Trident cloths <sup>4</sup>	3 µm PC Metadi Supreme diamond suspension	6 (27)	120–150, Comp.	5
Microcloth, Nanocloth, or Chemomet I cloths	Masterprep 0.05 µm alumina suspension, or Mastermet colloidal silica	6 (27)	120–150, Contra	2–5

1. Trade-named products are proprietary to Buehler Ltd., Lake Bluff, Ill.

2. Load is per specimen in a holder.

3. Comp./contra means that the specimen holder and platen rotate in the same direction/opposite directions.

4. For the most difficult specimens, a 1 µm diamond step can be added after this 3 µm diamond step, using the same materials, speeds, and direction, but somewhat less time.

**4140:** Figure 9 shows very clearly how alloy segregation affects these split transformations. Picral and nital revealed the as-quenched martensite (m) in 4140 as a light-etching region containing acicular ferrite (arrows) — the start of upper bainite formation — in a segregate-enriched band (longitudinal plane). SMB, Fig. 9(c), darkened the as-quenched martensite. Color revealed the structure better than black and white (not shown).

**5160:** Partially transformed upper bainite in 5160 is shown in Fig. 10. Also note the significant difference in the martensite phase. Picral does not reveal the martensite at all. It is lightly developed by nital (overetching would darken it), and somewhat darker with SMB. Again, the SMB image reveals more in color.

### Lower or 'acicular' bainite

Specimens partially and fully transformed to lower bainite are illustrated in Figures 11, 12, and 13, again using the three etchants.

**8620:** Figure 11 shows the structure of 8620 that is fully transformed. The lower bainite is a rather indistinct structure in this alloy. Results with nital and SMB are quite similar, while use of picral produced much different results.

**4140:** Figure 12 shows SAE 4140 microstructures with a mixture of lower bainite and martensite. Picral gives the most delicate results with only the bainite visible. Nital and SMB show both constituents. SMB reveals the martensite better than nital. Again, the color image of the SMB-etched structure is much better than the black and white image.

**5160:** Finally, Fig. 13 shows lower bainite and as-quenched martensite in 5160. The same basic trends are observed. SMB produced the strongest contrast and best visibility for both constituents.

### The martensite phase

If the cooling rate from the austenitizing temperature is so rapid that the

above-mentioned phases do not form, martensite will be produced. Martensite is a generic term for the body-centered tetragonal (bct) phase that forms by diffusionless transformation — not isothermal transformation — and the parent and product phases have the same composition and a specific crystallographic relationship. (The cooling rate required to form martensite is a function of section size, hardenability, and quench medium.)

Martensite can be formed in alloys where the solute atoms occupy interstitial sites, as for carbon in iron, producing substantial hardening and a highly strained, brittle condition. However, in carbon-free alloys with high nickel contents, such as maraging steels, the solute atoms (nickel) can occupy substitutional sites, producing martensites that are soft and ductile.

In carbon-containing steels, the appearance of the martensite changes with carbon in the interstitial sites. Low-carbon steels produce lath martensites. High-carbon steels produce plate martensite, often incorrectly called "acicular" martensite, when all of the carbon is dissolved in the austenite. The martensite shown in most of the photomicrographs in this article formed because the transformation time was not long enough to convert all of the austenite to ferrite and pearlite or bainite. **HTP**

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