MICROSTRUCTURAL CHARACTERIZATION OF CARBURIZED STEELS

The carburizing process must be controlled and results verified according to specifications, which usually involves case-depth measurement, surface hardness measurement, and microstructural characterization.

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Carburizing has been a popular method for obtaining a unique balance of properties in steels for many years. Steels with low carbon contents are characterized by good toughness and good ductility, but low strength and low wear resistance. By locally increasing the carbon content at the surface, followed by heat treatment to produce a very high hardness in this layer while maintaining a lower hardness in the core, the "best of both worlds" can be obtained—a hard, wear-resistant surface with good fatigue strength and a tough, ductile core to inhibit the growth of any cracks that might form at the surface. Such a steel will be tougher than a through-hardened medium carbon content steel with the same surface hardness.

Naturally, the process must be controlled and the results verified according to specifications, which usually involves case-depth measurement, surface hardness, and microstructural characterization. Depending on the application, retained austenite may be either desired to some amount or may be considered undesirable. Grain boundary carbide networks are universally undesirable. Surface decarburization is also undesirable. This paper gives examples of the use of metallography to assess the quality of carburized specimens.

Carburizing Process

Carburizing, sometimes called cementation, is a process where carbon is diffused into the surface of a steel (usually a low-carbon steel) at a temperature well above the A_{C3} where austenite is present and the diffusion rate of carbon in steel is reasonably high. The carbon content in the "case" increases according to the diffusion rate at the chosen temperature (generally 1700 to 1750°F, or 930 to 955°C) and the nature of the carbonaceous media used. However, the carbon content in the case will not exceed the carbon content of austenite defined by the A_{cm} line. However, for alloy steels, particularly tool steels, the carbon content at the surface can easily exceed 3% after carburizing.

Years ago, when the primary author worked for Bethlehem Steel, data sheets for AISI S7 (Bearcat) tool steel, invented by Bethlehem's Jack Riedel, stated that one could increase the wear resistance of Bearcat (nominally 0.5% C) by lightly carburizing parts to a depth of 0.010 in. The writer investigated numerous Bearcat die failures where this had been attempted, but failed. The failed dies exhibited very coarse prior-austenite grain sizes in carburized cases that contained very coarse plate martensite (many containing microcracks) and vast amounts of unstable retained austenite. Incremental turnings from the outer 0.005 in. of the case generally revealed carbon contents well above 3%. The 3.25% Cr and 1.40% Mo alloy additions in Bearcat gave the steel a very high equilibrium carbon content at the carburizing temperature.

In a majority of cases, however, it is not desirable to have the maximum case carbon content exceed 0.8%, or perhaps 1.0% in a few situations. The depth of the carburized case is mainly a function of the carburizing time. The earliest carburizing process was called pack carburizing, dating from antiquity. Basically, the steel part was encased in a bed of charcoal (the carbon source), heated to the desired temperature, and held for about 8 hours. This process is still used to prepare McQuaid-Ehn grain size test specimens (per ASTM E 112). Gas carburizing is a more recent development, and affords much better control of the maximum carbon content. The region of the surface containing a higher carbon content from carburizing is called the case, or carburized case.

In assessing the case depth, metallographers are usually requested to measure the total case depth or the effective case depth. These are interrelated, but they are not the same. The

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Table 1 — Typical procedure for metallographic specimen preparation for carburized steels

Surface	Abrasive	Load per specimen, lb (N)	Platen speed, rpm (direction)	Time, min
CarbiMet SiC	120 or 180 grit (water cooled)	6 (27)	240-300 (contra)	Until plane
UltraPol silk cloth	9-µm diamond(a)	6 (27) MetaDi®	120-150 (contra)	5
TriDent® polyester cloth	3-µm MetaDi Diamond(a)	6 (27)	120-150 (contra)	5
MicroCloth® synthetic suede cloth	0.05-µm MasterPrep® alumina	6 (27)	120-150 (contra)	3

(a) *Charge cloth with diamond paste, then add MetaDi fluid lubricant before commencing polishing. During the cycle, add a small amount of MetaDi suspension of the same diamond size to the cloth to maintain a high cutting rate.*

Because we must examine the surface, and generally make measurements of case depth below the surface, specimens must be prepared to retain the edge precisely. Consequently, all such specimens must be mounted in a compound that yields high edge retention.

total case depth is the depth from the surface to where the carbon content or the hardness becomes constant. The effective case depth is the distance from the surface to where the hardness reaches a specified value, usually the hardness for 50% martensite for a 0.8% maximum case carbon content, which generally is 550 HV (~580 HK).

The metallographer could also estimate the depth below the surface to where the microstructure contains 50% martensite and the balance is a 50% mixture of ferrite and bainite, or possibly a mixture of 50% high-carbon martensite and 50% low-carbon martensite (this is more difficult to determine). At the same time, the metallographer should check for any surface decarburization that may have occurred during heat treatment after carburizing, or the presence of very harmful grain-boundary carbide films. If the steel is not inherently fine grained (i.e., not aluminum killed to increase the grain coarsening temperature to well above the carburizing temperature), the metallographer should note the presence of prior-austenite grain boundaries indicating a grain size less than (coarser than) ASTM 5. It has been well known for nearly 100 years that carburized coarse-grained steels fracture intergranularly quite readily in service.

Metallographic Examination

Because we must examine the surface, and generally make measurements of case depth below the surface, specimens must be prepared to retain the edge precisely. Consequently, all such specimens must be mounted in a compound that yields high edge retention. It is impossible to make accurate measurements with polished, unmounted specimens, as the edges will be beveled. To get good results, the mount must not have gaps due to shrinkage between the specimen and the polymeric mounting compound.

Buehler's EpoMet® thermosetting epoxy mounting compound contains silica as a filler to control shrinkage and the grinding/polishing rate. It produces minimal or no shrinkage gaps, particularly when the specimen is cooled to near ambient after polymerization under pressure. Phenolic resins and diallyl phthalate resins always have shrinkage gaps, and these promote edge rounding. Cast epoxy resins, particularly those that cure more slowly, or at slightly elevated temperatures, physically adhere to the specimen and can provide good edge retention, particularly when the grinding and polishing is performed properly. Cast acrylic resins are noted for producing large shrinkage gaps and are not recommended for this work.

In preparing a metallographic sample, always cut specimens using the least damaging device—an abrasive cutoff saw or a precision saw. Use the correct blade developed for metallography work and for high-hardness steels, with the proper coolant flow. Mount specimens as discussed above and prepare with an automated grinder/polisher for best flatness and control. Central-force loading yields better flatness and edge retention than individual force. Commence grinding using the finest possible SiC abrasive paper. This is a compromise between the greater damage depth created by coarser grit papers and the slower removal rate of finer grit papers. In general, as the case hardness will be equivalent to 60 HRC (~700 HV, or ~732 HK), 120- or 180-grit SiC paper is usually chosen to get all of the specimens in the holder at the same plane and to remove the cutting damage in a reasonable amount of time. After one grinding step, start polishing using 9-µm diamond on a flat woven cloth, such as UltraPol® silk cloth. A typical procedure used at Buehler is listed in Table 1.

Other cloths can be used in place of MicroCloth, as desired. For example, ChemoMet® pads are made of polyurethane and work well, but it is necessary to increase the load per specimen to 8 lb. One can also perform the final polish using MasterMet® Colloidal Silica. Some metallographers use both abrasives: 90 s with MasterPrep Alumina and then 90 s with Mas-

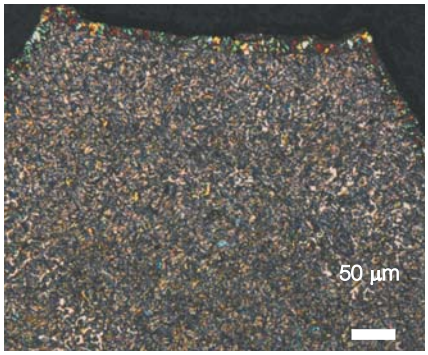

Fig. 1 — Tooth of an 8620 alloy steel gear that failed in service. The edge of the tooth was decarburized in the heat treatment following carburizing. A 15-18 µm deep rim along the tooth edge consists of equiaxed ferrite and pearlite. Also note that there are patches of grain boundary carbide films in a number of locations. The specimen was etched with aqueous 10% sodium metabisulfite, and the image was captured in polarized light plus sensitive tint.

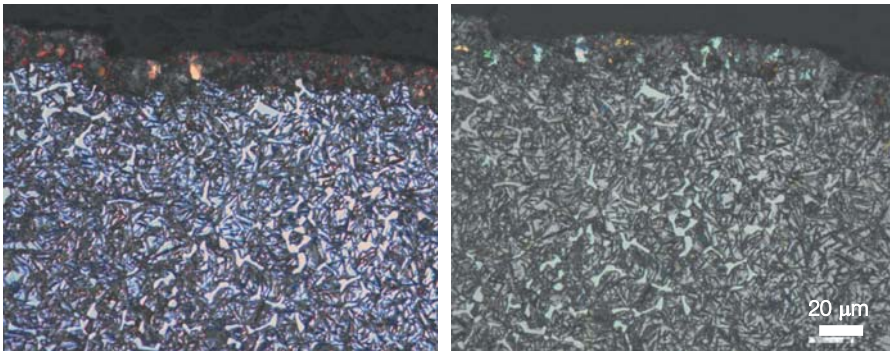

Fig. 2 — Higher magnification views of the surface of the failed 8620 gear showing the surface with Nomarski DIC (left) and with polarized light plus sensitive tint (right). Note the decarburized pearlitic surface layer with patches of ferrite and the grain-boundary cementite networks. The matrix is coarse plate martensite with substantial retained austenite.

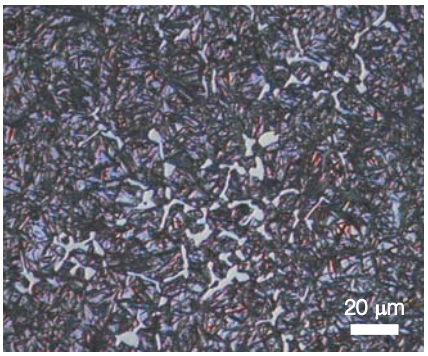

Fig. 3 — Microstructure of the case below the surface showing the grain-boundary carbide network patches and the matrix of retained austenite and plate martensite. The image was taken using Nomarski DIC after etching with aqueous 10% sodium metabisulfite.

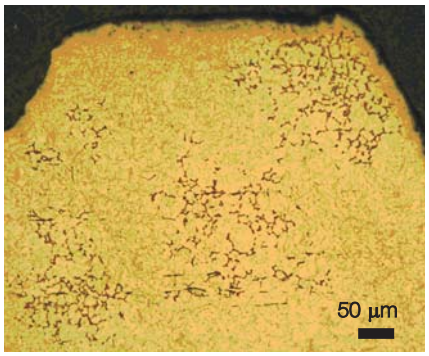

Fig. 4 — Grain boundary carbide networks revealed by etching with alkaline sodium picrate at 90°C for 90 s. This etch will color cementite (M_3C) and also will color M_6C, but that carbide type cannot form in carburized 8620.

terMet Colloidal Silica. If the core microstructure is mostly ferrite where scratch removal is more difficult, this cycle could be followed by a brief (20 min) final polish with the vibratory polisher.

There are many etchants that have been developed for steels[1]. Of the commonly used general-purpose etchants, nital (usually at 2 or 3% concentration in ethanol) is by far the most widely used. Nital is excellent for revealing martensitic microstructures, and also reveals most of the ferrite grain boundaries. Picral (4 g picric acid in 100 ml ethanol) reveals the structure of martensite only if it is tempered, and it is excellent for revealing residual carbides and for pearlitic or bainitic microstructures. There are also several highly useful color tint etchants for steels. Klemm's I (1 g potassium metabisulfite added to 50 ml saturated aqueous sodium thiosulfate) colors ferrite very strongly and will color martensite, bainite, and pearlite, but not carbides. Beraha's sulfamic acid etch #1 (100 ml water, 3 g potassium metabisulfite, 1 g sulfamic acid) is similar in action to Klemm's I, but does not color ferrite quite as strongly. Aqueous 10% sodium metabisulfite also works similarly, but is the weakest of the three at coloring ferrite. None of the above etchants will color retained austenite when present in amounts typical for carburized steels. There are etchants that will color specific types of carbides in steel[2], which may be useful.

Examples of Carburized Microstructures

Example 1: The first example is a poorly carburized and heat treated gear made of 8620 alloy steel that failed early in service. Figure 1 shows that the gear teeth surfaces were decarburized during heat treatment after carburizing, resulting in a ~15-18 µm thick layer of pearlite with patches of ferrite. Below this layer the structure consists of plate martensite and substantial retained austenite with irregularly dispersed patches containing intergranular carbide networks. None of these patches were present within the decarburized surface layer and they were not observed deeper within the case. Figure 2 shows two views of the decarburized surface layer at higher magnification. Note that the carbide networks are more easily observed using Nomarski DIC illumination. Figure 3 shows a high magnification view of the carbide networks below the surface within the case. Figure 4 shows a tooth after etching with alkaline sodium picrate (100 ml water, 25 g sodium hydroxide, 2 g picric acid; immerse at 80-100°C for 45 s or more) revealing that the carbides are cementite (as would be expected for this grade). It reveals the erratic presence of these networks, which is rather unusual.

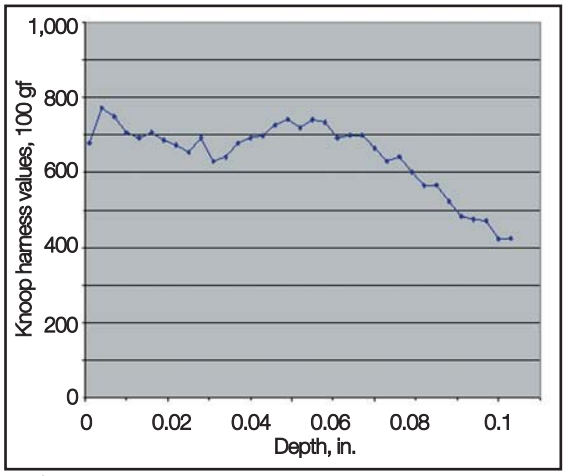

Fig. 5 — Knoop hardness traverse of poorly carburized and heat treated 8620 specimen showing erratic hardness readings in the region to a depth of about 0.03 in. (~ 800 µm) due to the high amount of retained austenite and the grain boundary carbide networks in this area.

Figure 5 shows the influence of the erratic subsurface microstructure on the Knoop hardness profile. The first data point is slightly below the decarburized surface layer. The matrix contained substantial retained austenite starting below the decarburized layer

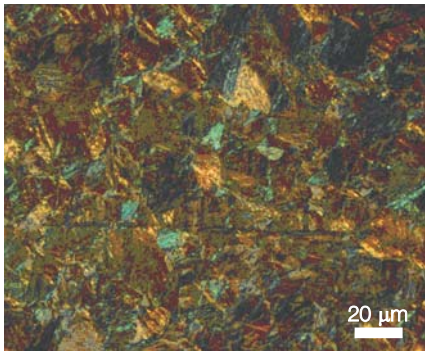

Fig. 6 — Core microstructure consists of lath martensite and a minor amount of bainite. The specimen was etched with aqueous 10% sodium metabisulfite and was photographed with polarized light and sensitive tint.

Fig. 7 — Montage showing case-to-core transition in a carburized EX-15 alloy steel component.

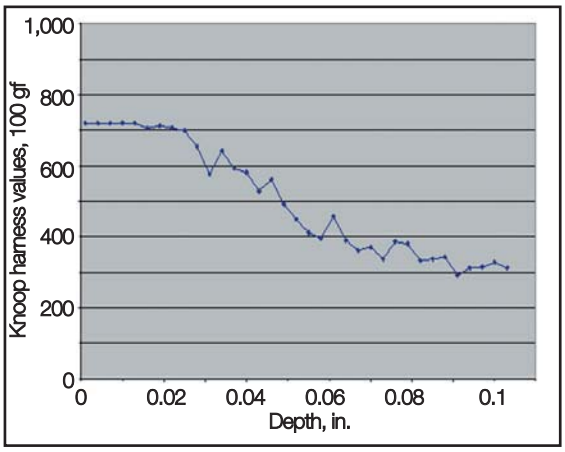

Fig. 9 — Knoop hardness traverse for the EX-15 carburized specimen showing a relatively uniform hardness in the case to a depth of about 0.025 in.

and ending at a depth of ~800 µm (~0.031 in.), which lowered the case hardness locally. The profile is a bit erratic due to the variation in retained austenite and martensite content and whether or not the indent hit the erratically distributed grain boundary carbides. The effective case depth based on the Knoop equivalent to 550 HV is at a depth of ~0.088 in. (~2,250 µm, or 2.25 mm).

Example 2: Figure 7 shows a montage covering the case-to-core transition in a carburized EX-15 alloy steel (similar in composition to AISI 8620, but without a Ni addition). Here, the microstructure is much more uniform and consistent than in the previous example, and the component performed well in service. The montage (taken at 200×) shows microstructural variations from segregation, which is most apparent in the hardened case. Figure 8 shows higher magnification views of the microstructure at the surface, in the transition zone, and in the core. At higher magnification, it is more difficult to observe the chemical segregation effect. Color etching always reveals this chemical segregation induced microstructural variation far better than standard black and white etching using nital or pi-cral. Figure 9 shows the Knoop hardness traverse for this specimen, which is much more consistent than that shown in Fig. 5. The effective case depth is ~0.048 in. (~1,230 µm, or 1.23 mm).

Example 3: It is not uncommon to see highly alloyed steels, such as hot work tool steels and stainless steels, being carburized to obtain a better balance of properties. Figure 10 shows a montage from the surface to the transition zone, but not fully into the core, of a carburized AISI 430 ferritic stainless steel specimen. This steel was given a standard carburizing treatment, not the proprietary low-temperature technique[3, 4], which avoids carbide precipitation. As Fig. 10 (and Fig. 11 at higher magnification) show, the carbide precipitation is extensive. This will provide excellent wear resistance, but the steel will be quite brittle. Figure 12 shows a montage similar to that in Figure 10, but the specimen was etched using Murakami's reagent (100 ml water, 10 g sodium hydroxide, 10 g potassium ferricyanide; immerse at room temperature to color alloy carbides) to only color the Cr_7C_3 carbides. It does not color $Cr_{23}C_6$ carbides. Near the center of the montage, there is a region with a much lower carbide concentration than above or below. Figure 13 shows the Knoop hardness traverse for this specimen. The surface hardness is approaching 1,000 HK, and the effective case depth is at ~0.058 in. (~1,485 µm, or 1.49 mm).

Example 4: The case and core microstructures of carburized (0.95% C potential) 8620 alloy steel are shown. It was carburized at 1750°F (955°C), quenched into a 50/50 mix of sodium nitrite and potassium nitrate at 480°F

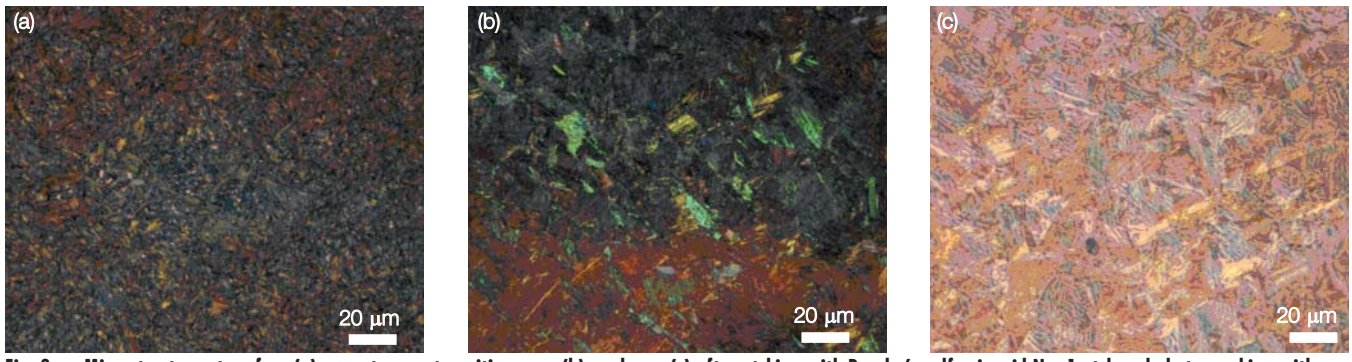

Fig. 8 — Microstructure at surface (a), case-to-core transition zone (b), and core (c) after etching with Beraha's sulfamic acid No. 1 etch and photographing with polarized light plus sensitive tint.

Fig. 10 — Montage of a carburized 430 stainless steel specimen showing the case-to-core transition after etching with Beraha's sulfamic acid etch # 4 and photographing under polarized light plus sensitive tint.

(250°C), and held 120 minutes to form lower bainite in the case. It was air cooled, forming lath martensite in the core, and tempered at 480°F for 240 minutes to an aim case hardness of 52-60 HRC. The reason for this treatment is that a lower bainitic case performs better under low-cycle fatigue conditions than a plate martensitic case.

Figure 14 shows the lower bainitic case and the lath martensitic core after etching using 2% nital. Compare the images with Figs. 15 and 16, which show the case and core microstructures, respectively, etched using aqueous 10% sodium metabisulfite in bright field and polarized light, plus sensitive tint. The color etch reveals these structures much better than nital. Figure 17 shows the Knoop hardness traverse from the surface to the core, revealing a flatter hardness profile than if the surface was fully martensitic.

Because the case structure is not plate martensite, but lower bainite, and the hardness gradient is based

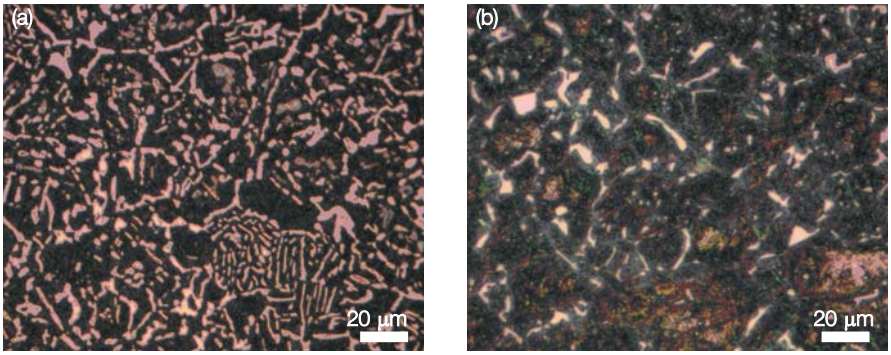

Fig. 11 — Very heavy carbide concentration at the surface (a) and in the transition zone (b) of the carburized 430 ferritic stainless steel specimen etched with Beraha's sulfamic acid reagent No. 4. The matrix is tempered martensite.

Fig. 12 — Montage of the case-to-core transition zone on the carburized 430 stainless steel specimen etched with Murakami's reagent (20°C for 40 s) showing a region near the center relatively free of Cr_7C_3 carbides.

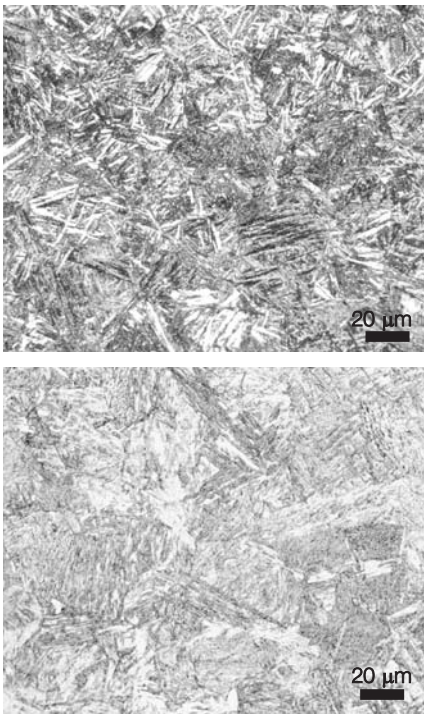

Fig. 14 — Case (top and core (bottom) of carburized 8620 that was isothermally heat treated to form lower bainite in the case; the core is lath martensite (2% nital etch).

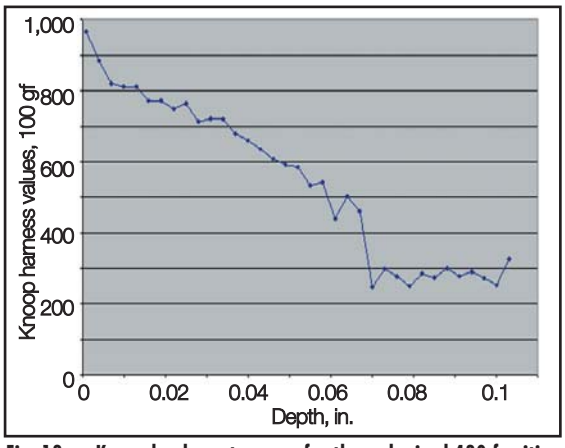

Fig. 13 — Knoop hardness traverse for the carburized 430 ferritic stainless steel specimen. The effective case depth is ~0.058 in. (~1,485 µm, or 1.49 mm).

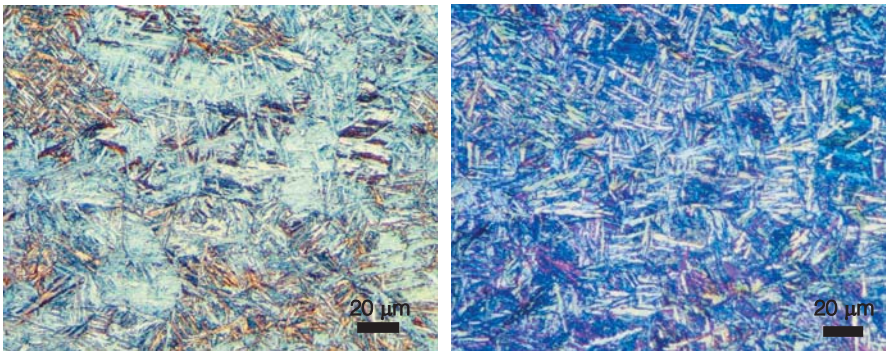

Fig. 15 — Lower bainite structure of the case revealed by etching with aqueous 10% sodium metabisulfite: bright field (left); polarized light and sensitive tint (right).

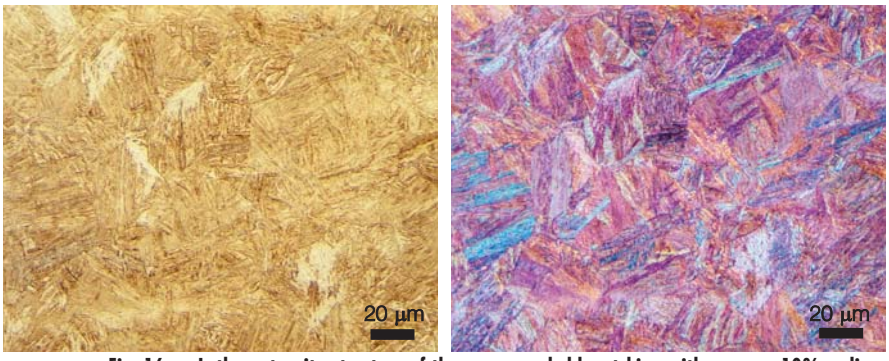

Fig. 16 — Lath martensite structure of the core revealed by etching with aqueous 10% sodium metabisulfite: bright field (left); polarized light and sensitive tint (right).

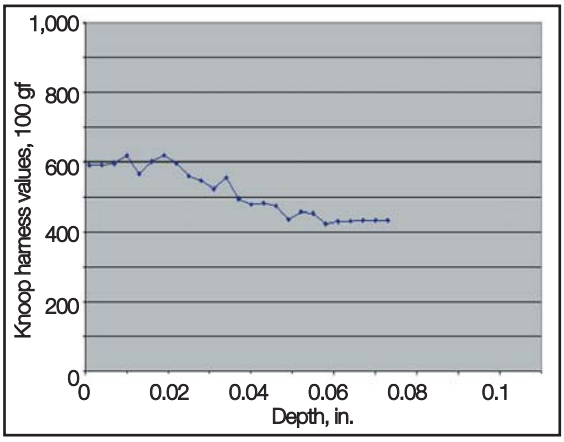

Fig. 17 — Knoop hardness traverse for carburized 8620 where the case was isothermally transformed to lower bainite and the core is lath martensite.

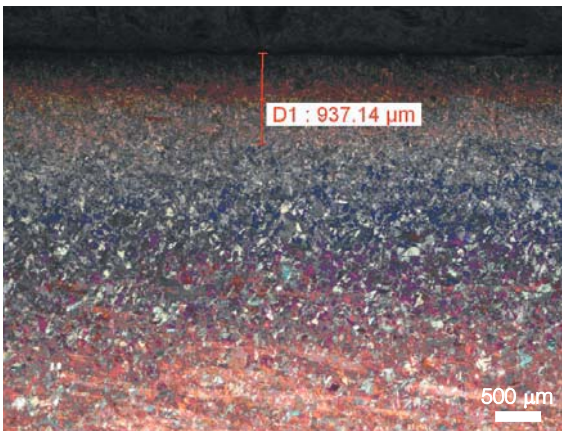

Fig. 18 — Visual estimate of the case-to-core transition depth, 937 µm (~0.0365 in.), which corresponds to a hardness of 556 HK; specimen was etched with Beraha's sulfamic acid No. 1 reagent and photographed in polarized light plus sensitive tint at 50×.

on lower bainite transitioning to lath martensite, the 550 HV criteria for the effective case depth (based on the hardness of 50% martensite/50% diffusion-controlled transformation products) is not really applicable. We can, however, base the effective case depth measurement on a visual estimate of where the structure is 50% lower bainite and 50% lath martensite. Of the various etchants tried, Beraha's sulfamic acid No. 1 (composition given above) gave the best discrimination between the constituents (Fig. 18). The visual case depth estimate of 937 µm (0.0365 in.) correlates to a hardness of 556 HK (Fig. 17), which corresponds to ~528 HV (slightly below the 550 HV predicted for 50% martensite and 50% diffusion-controlled transformation products), but what appears to be a reasonable effective case depth estimate for this specimen with the case transformed to lower bainite.

Conclusions

Metallographic examination is a powerful tool for the study of carburized steels and invaluable for diagnosing failures. To get correct results, sample sectioning (cutting) must be performed to minimize induced damage. If it is believed that the part may not have been tempered, mounting must be done using a low-viscosity cast epoxy that generates a very small exotherm during polymerization. Grinding using one SiC step is adequate. Polishing should be performed using flat woven cloths with a proper amount of diamond abrasive and adequate lubrication. Final polishing with abrasives smaller than 1 µm in size, such as MasterPrep Alumina suspension or colloidal silica, is always best to obtain scratch-free surfaces with minimal damage to properly reveal and correctly identify the microstructural constituents. Use of color etchants does require removal of the preparation-induced damage, but this is easily achieved with modern equipment and consumables. Knoop hardness traverses are usually preferred over Vickers traverses, as the Vickers half-diagonals in the hardness gradient direction may become non-symmetrical to the point of being invalid indents. **HTP**

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