Metallographic Characterization of the Microstructure of Tool Steels

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Abstract: Examination of selectively etched tool steel microstructures by light microscopy provides more information than standard etchants, such as nital, picral or Vilella's reagent. Further, the images are more suitable for quantitative measurements, especially by image analysis. Specimens must be properly prepared, damage free, if selective etchants are to be applied successfully. A number of etchants have been claimed to selectively etch certain carbides in tool steels. The response of these etchants has been evaluated using a variety of well-characterized tool steel compositions. While many are selective, they are often selective to more than one type of carbide. Furthermore, their use in image analysis must be evaluated carefully as measurements showed that the amount and size of the carbides are often greater after selective etching as many of these reagents outline and color or attack the carbides. Selective etching of the matrix, leaving the carbides unaffected works well, but no one etchant will cover the broad spectrum of tool steel compositions. No etchant has been found that will color retained austenite in tool steels and image analysis of retained austenite in tool steels are always much lower than by x-ray diffraction unless retained austenite is the dominant phase present in grossly over-austenitized steels.

Key words: carbides, etchant, ferrite, image analysis, martensite, retained austenite

Tool steels are important materials and cover a wide range of compositions from simple plastic molding die steels or water-hardening carbon steels to very highly alloyed high-speed steels. Even more exotic compositions can be achieved by the powder metallurgy route. But, there are some common features. First, all are iron-based. Metallographers deal with relatively soft annealed tool steels, as-rolled or as-forged steels with a wide range of microstructural constituents and hardness, to heat-treated microstructures generally consisting of rather high strength martensite and a variety of carbide types. In tool and die failure analysis work, an even wider range of microstructures, some undesirable, can be encountered.

In generally, tool steels are not exceptionally difficult to prepare for microstructural examination but there are a number of problems to deal with. First, sectioning can be rather difficult requiring softly bonded abrasive blades to avoid burning. In preparing specimens, edge retention is often a requirement, for example, in the rating of decarburization or in the examination of heat-treated specimens, particularly those in failures. Inclusion retention may also be important, especially if the inclusion content must be rated. For graphitic tool steels, the graphite must be properly retained. Staining problems may be encountered, particularly in high silicon grades. Carbides may be cracked or there can be voids associated with carbides, or in the center of sections in high alloy grades. The metallographer must be able to determine if these voids are real, or produced by the preparation process.

2. PREPARATION PROCEDURE

2.1 SECTIONING

Relatively soft specimens (less than 35 HRC) can be cut using band saws or hacksaws. However, such operations produce a substantial zone of deformation beneath the cut and rather rough surfaces. Thus, the initial rough grinding with a coarse abrasive (80- to 120-grit silicon carbide, for example) must remove this damage.

Higher-hardness specimens must be cut using water-cooled abrasive cutoff wheels. The blade should have a soft bonding for effective cutting and avoidance of burning. Submerged cutting limits heat generation, which is most severe when cutting as-quenched or quenched and lightly tempered tools steels. Heat generated by improper technique can produce a highly tempered appearance in the martensite and, if heating is excessive, can re-austenitize the surface. Subsequent grinding steps cannot easily remove this damage.

When working with as-quenched high-alloy tool steels, it may be helpful to fracture the specimen. This will produce a flat, damage-free surface due to the extreme brittleness of such steels. The fractured surface can then be ground and polished for examination. For high-hardness, high-alloy steels, sectioning with a low-speed diamond or cubic boron nitride wheel saw can provide high-quality surfaces with minimum cutting rate is low, such surfaces are smooth, and grinding can begin with rather fine grits (320- to 400-grit silicon carbide, for example).

2.2 MOUNTING

Bulk samples frequently can be polished without mounting. Although most modern automatic polishing devices can handle unmounted specimens, some cannot. If edge retention is important, mounting may be desired. Plating the surface prior to mounting produces optimum results, but is rarely necessary. The compression-mounting epoxy resins, like Epomet®, provide excellent edge retention even with unplated specimens. Automatic polishing devices rather than hand polishing yield better edge retention.

For small or oddly shaped specimens, mounting is preferred. If the edge is not of particular interest, most mounting mediums are satisfactory. However, some mounts have poor resistance to solvents such as alcohol, and many are badly degraded if heated etchants are required. The compression-mounting epoxies prevent these problems. If a transparent mount is required to control grinding to a specific feature, transparent methyl methacrylate compression-mounting material can be used, and many cold-mounting epoxies are also satisfactory. Cold-mounting epoxies are the only materials that produce true adhesive bonding to the sample. They also produce the lowest heat during curing and are useful when the sample cannot tolerate the higher heat used in compression mounting. When edge retention is not required and heat degradation is not anticipated, low-cost phenolic compression-molding materials can be used.

2.3 THE TRADITIONAL GRINDING AND POLISHING APPROACH

In the traditional approach, either manual (hand polishing) or automated devices are used. Water-cooled silicon carbide paper (220- to 300-mm, or 8- to 12-in. diam) is employed for the grinding stage; the initial grit size selected depends on the technique used to generate the cut surface. The usual grit sequence is 120, 240, 320, 400, and 600-grit. Finer grit sizes may be used for highly alloyed tools steels in which carbide pullout is a problem. Grinding pressure should be moderate to heavy, and grinding times of 1 to 2 min are typical to remove the scratches and deformation from the previous step. Fresh paper should be used; worn or loaded paper will produce deformation.

In the traditional approach, polishing is commonly performed using one of more diamond abrasive stages followed by one or more final abrasive stages, generally with alumina abrasives. For routine work, polishing with 6- and 1-μm diamond is generally adequate. The diamond abrasive may be applied to the polishing cloth in paste, slurry or aerosol form. For the coarser diamond abrasives, low-nap or napless cloths are performed; a medium-nap cloth is generally used with the finer diamond abrasives. A lubricant, or "extender", compatible with the diamond abrasive should be added to moisten the cloth and minimize drag. Wheel speeds of 150 to 300 rpm and moderate pressure should be used. Polishing times of 1 to 2 min are usually adequate.

Final polishing can also be conducted manually or automatically using various devices. Alumina abrasives, generally 0.3-μm α-alumina (Al_2O_3) and 0.05-μm y-Al_2O_3, are widely employed with medium-nap cloths for final polishing. Colloidal silica (SiO_2), with a particle size range of 0.04- to 0.06-μm, is also very effective. Wheel speeds, pressure, and times are the same as for rough polishing with diamond abrasives. In general, tool steels are relatively easy to polish to scratch-free and artifact-free condition due to their relatively high hardness.

2.4 THE CONTEMPORARY APPROACH

The modern procedure utilizes automated equipment for grinding and polishing. The specimens are placed in a holder designed to accommodate a number of specimens of relatively similar size, mounted or unmounted. Either 200, 250 or 300mm (8, 10 or 12 inch) diameter formats may be employed. Unmounted or mounted specimens may be prepared. Newly developed surfaces and abrasives permit achievement of surface qualities more than adequate for research work with as few as three steps.

Table 1 lists a four-step procedure that yields surfaces of a sufficient quality for any needs. For production work, step 4 could be omitted, yet the results will be quite satisfactory for routine examination. If step 4 is utilized, results are better and photographic work of publication quality is obtained.

Table 2 illustrates a simpler three-step procedure that also yields superb surfaces and research/publication quality micrographs. An Ultra-Pad cloth may also be used for step 2, although the Ultra-Pol silk cloth produces the best results. With either cloth, edge flatness is superb, as will be demonstrated in the examples that follow.

Table 1. Four-step method for preparing tool steels

Abrasive and Surface	Lubricant	rpm	Head/Platen Directions	Load per Specimen	Time (minutes)
120- to 240-grit (P120 to P280) SiC waterproof abrasive paper	water	240-300	Complementary	6 lbs (27N)	Until Plane
9-µm diamond on an Ultra-Pol™ silk cloth (or Ultra-Pad™ polyester cloth or Hercules™ H rigid grinding disk)	Metadi Fluid	120-150	Contra	6 lbs (27N)	5
3-µm diamond on a Trident™ cloth (or Texmet® 1000 chemotextile pad)	Metadi Fluid	120-150	Contra	6 lbs (27N)	3
MasterPrep™ Alumina Suspension on a Microcloth® pad	No other lubricant is needed	120-150	contra	6 lbs (27N)	1-3

Complementary rotation means that the sample holder (head) is rotating in the same direction as the platen (normally counter clockwise) while contra means that they rotate in opposite directions. This produces a somewhat greater removal rate. If the sample holder rotates at <100 rpm, the slurries will stay on the surface longer using contra. In complementary mode, centrifugal forces throw the liquids off the platen surface as quickly as it is added. However, if the head rotates at a speed >100 rpm, contra will throw the liquids all over the room. If relief patterns are observed around oxides or sulfides after step 4, simply repeat step 4 using complementary rotation and it will be removed. This happens rarely and is usually specimen specific in nature.

When charging the cloth with diamond, use paste as cutting is started faster. Apply a generous amount of diamond, and then spread the diamond with your clean fingertip. Apply the lubricant and start polishing. During the cycle, you can squirt on a diamond suspension, such as Metadi Supreme, with the same particle size as the paste to keep the cutting rate high. The slurries have the lubricant included, so you do not need to add additional Metadi Fluid lubricant, although some people do add small amounts occasionally even when using diamond in slurry form. MasterPrep alumina suspension has a 0.05-µm particle size alumina made by the sol-gel process, rather than the traditional calcinations process, and is agglomerate free. If all these steps are followed, from cutting to polishing, the final step can be 3 minutes without introducing any relief or edge-rounding problems. Avoiding excessive cutting damage, mounting with Epomet resin to avoid shrinkage gaps, starting grinding with the finest possible silicon carbide abrasive (or an equivalent sized abrasive in a different form, such as the DGD disks) – these are the key steps to obtaining perfect renderings of the true microstructure.

Table 2. Three-step method for preparing tool steels

Abrasive and Surface	Lubricant	rpm	Head/Platen Directions	Load per Specimen	Time (minutes)
120- to 240-grit (P120 to P280) SiC waterproof abrasive paper	water	240-300	Complimentary	6 lbs (27N)	Until Plane
9-µm diamond on an Ultra-Pol silk cloth cloth (or Ultra-Pad™ polyester cloth or Hercules™ H rigid grinding disk)	Metadi Fluid	120-150	Contra	6 lbs (27N)	5
MasterPrep Alumina Suspension on a Microcloth pad	No other lubricant is needed	120-150	Contra	6 lbs (27N)	5

3. ETCHING

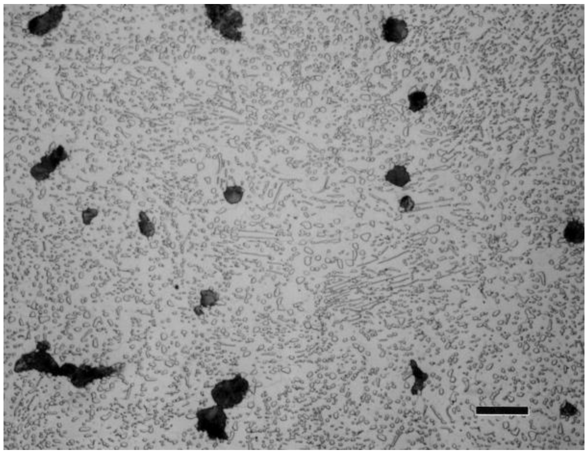
The etchant most widely used for tool steels is nital. Concentrations from 2 to 10% have been used. Generally, 2 or 3% nital is adequate for most tool steels while a 10% concentration is required for highly alloyed tool steels, such as the D types. Stock solutions exceeding 3% HNO_3 in ethanol should not be stored in pressure-tight bottles. If higher concentrations are desired as a stock reagent, a bottle with a pressure-relief valve should be used, or methanol should be substituted for ethanol. Methanol is a cumulative poison and its use should be minimized.

Nital is generally used for tool steels regardless of the anticipated microstructural constituents. Although nital is superior to picral (4% picric acid in ethanol) for etching martensitic structures, picral produces better results for examining annealed samples.

When examining spheroidize-annealed tool steels (the most common annealed condition), picral reveals only the interfaces between carbide and ferrite. Nital also reveals the ferrite grain boundaries that generally obscure the carbide shape. Also, because nital is orientation sensitive, carbides within some of the ferrite grains will be poorly delineated, making spheroidization ratings more difficult.

A 2% nital solution is usually preferred. Stronger concentrations increase the speed of etching, making it more difficult to control. Etching of martensitic high-alloy tool steels, such as the high-speed steels, may require a 5% concentration, while the D types may require a 10% solution. Etching with nital or picral is usually performed by immersion. If swabbing is used, pressures should be light to avoid smearing problems. Etching times are difficult to generalize, because of the wide range of tool steel compositions and because heat treatment can markedly alter etch response. Trial and error will determine the degree of surface dulling necessary to obtain the correct degree of etching.

Other etchants, although infrequently used, can be of greater value. Table 3 lists compositions of a number of specialized reagents for achieving selective etching or enhancing contrast among microconstituents. Figure 1 illustrates the use of the three- and four-step preparation methods with annealed O6 graphitic tool steel as an example. Note that the graphite has been fully retained regardless of the method used, and that there is no residual deformation or scratches in the ferrite and the cementite is clearly revealed. Figure 2 shows the etched microstructure of spheroidize annealed W1 water hardening tool steel etched with 4% picral, with Klemm's I reagent, and with alkaline sodium picrate. Picral uniformly dissolves the ferrite, thus appearing to outline the cementite particles. Klemm's I colors the ferrite matrix, but not the cementite, permitting easy discrimination of the cementite by image analysis. Alkaline sodium picrate colors the cementite uniformly, and does not attack or enlarge the particles. Hence, measurements of the cementite will be statistically equivalent using Klemm's or alkaline sodium picrate.



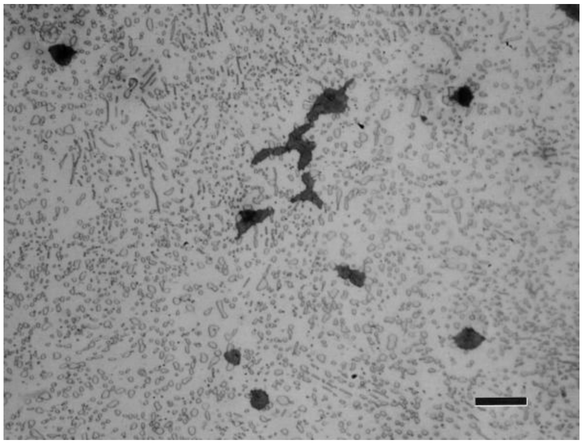
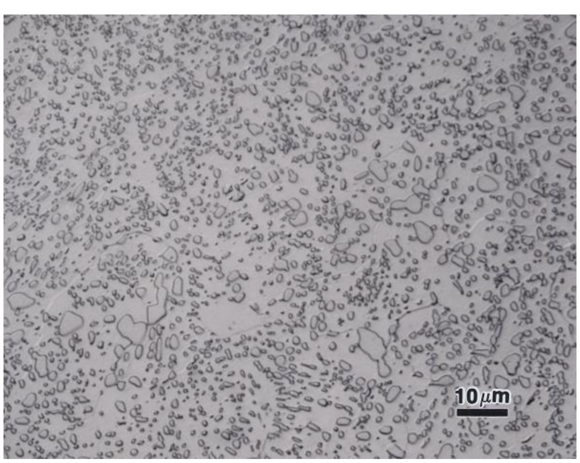
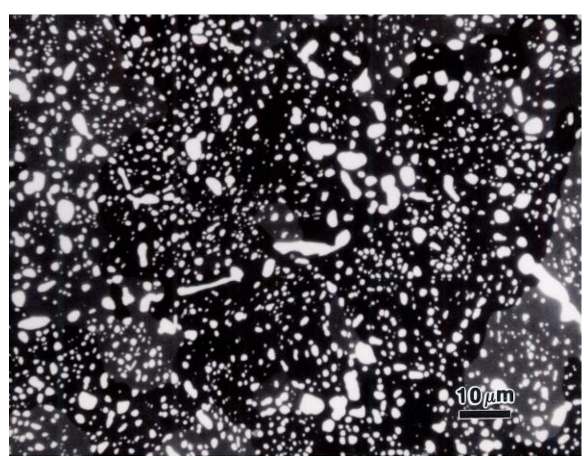


Figure 1. Annealed microstructure of type O6 graphitic tool steel prepared (left) with the three-step method using the Ultra-Pol silk cloth for step 2; and (right) using the BuehlerHercules H rigid grinding disk for step 2 (magnification bars are 10-µm long; 4% picral etch).

Table 3. Etchants for Tool Steels

Composition	Comments
1-10 mL HNO_3 99-90 mL Ethanol	Nital. Most commonly used reagent. Reveals ferrite grain boundaries and ferrite-carbide interfaces. Excellent for martensite.
4 g Picric acid 100 mL Ethanol	Picral. Recommended for annealed structures or those containing pearlite or bainite. Does not reveal ferrite grain boundaries. Addition of a few drops of zephiran chloride increases etch rate. Add 1-5 mL HCl to improve etch response for annealed higher alloy tool steels.
1 g Picric acid 5 mL HCl 95 mL Ethanol	Vilella's reagent. Reveals structure of higher alloyed tool steels.
50 mL sat. Aq. Sodium thiosulfate 1 g Potassium metabisulfite	Klemm's I tint etch. Immerse specimen until the surface is colored violet. Colors ferrite blue and red while martensite is brown. Carbides are unaffected. Works well only on low alloy and carbon tool steels.
0.6 mL HCl 0.5-1.0 g Potassium metabisulfite 100 mL water	Beraha's reagent. Immerse specimen until the surface is colored. Colors ferrite and martensite, carbides are not affected. Good for most tool steels.
3 g Potassium metabisulfite 2 g sulfamic acid 0.5-1.0 g Ammonium bifluoride 100 mL water	Beraha's sulfamic acid reagent No. 4. For carbon and low-alloy tool steels, leave out the $NH_4F{\cdot}HF$. Immerse until the surface is colored. Ferrite and martensite are colored; carbides are not affected.
2 g Picric acid 25 g NaOH 100 mL water	Alkaline sodium picrate. Colors cementite and M_6C carbides. Immerse specimen in solution at 80-100 ºC for up to 15 minutes.
10 g $K_3Fe(CN)_6$ 10 g NaOH or KOH 100 mL water	Murakami's reagent. Use at 20 ºC to outline and darken M_7C_3 and M_6C, and to outline M_2C. $M_{23}C_6$ is faintly colored.
4 g KMnO4 4 g NaOH 100 mL water	Groesbeck's reagent. Use at 20 ºC to outline M_2C and to outline and darken M_6C. M_7C_3 is faintly colored.
1 g CrO3 100 mL water	Blickwede and Cohen's etch. Use at 2-3 V dc, 20 ºC, for 30 seconds with a stainless steel cathode. Outlines $M_{23}C_6$, Outlines and colors M_7C_3, colors MC and attacks M_2C.





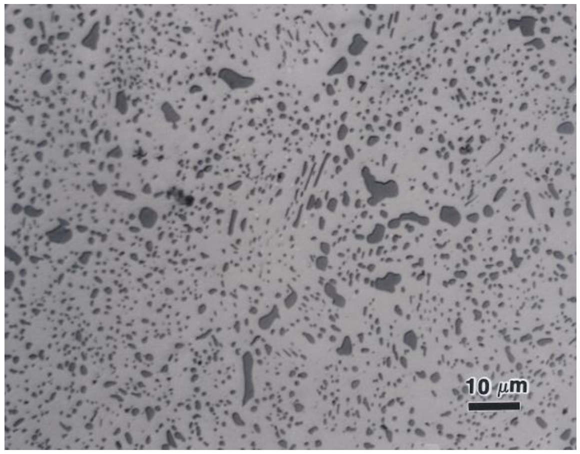


Figure 2. Spheroidize annealed W1 water-hardened carbon tool steel etched with (left) 4% picral to reveal the general structure, with (right) Klemm's I to color the ferritic matrix, and with (center, below) alkaline sodium picrate at 90 ºC for 60 seconds to color the cementite.

Experiments with a number of tool grades in either the annealed or quenched and tempered condition, or both, were conducted to assess the selectivity of the etchants listed in Table 3 that are claimed to outline, color, outline and color, or attack specific carbide types. The carbides were first characterized by electron-backscattered diffraction (EBSD). Before each etching was used the specimens were completely re-prepared. Results are given in Table 4.

Table 4. Results of the Etching Experiments

Etchant	M_3C	$M_{23}C_6$	M_7C_3	M_6C	MC	M_2C
Alk.Na Pic.	Colors	NA	NA	Colors	NA	NA
Murakami	NA	Faint	Out/Col	Out/Col	NA	Outlined
Groesbeck	NA	NA	Faint	Out/Col	NA	Outlined
1% CrO_3	NA	Outlined	Out/Col	NA	Colors	Attacks

NA – no affect Out/Col – outlined and colored

3. Conclusions

Specimen preparation must be properly performed if the true structure is to be observed and interpreted correctly. With modern semi-automated equipment, tool steel specimens can be prepared quickly and with perfect results every time. Simple three- and four-step procedures have been described. Key factors in preparing specimens were defined. First, sectioning of the specimen requires an abrasive blade designed for metallographic work and for the hardness of the particular specimen to avoid introducing excessive damage. Second, if an edge is to be examined, mount the specimen in the best possible resin. Third, commence grinding with the finest possible abrasive. Fourth, used enough abrasive in polishing to produce effective cutting. Fifth, use napless, woven or pressed cloths, except in the final step. Finally, select the best etchant to reveal the structure clearly and with good contrast.

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