TECHNotes

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Metallographic Preparation of Tool Steels

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 general, tool steels are not exceptionally difficult to prepare for microstructural examination but there are a number of problems to consider. First, sectioning can be rather difficult requiring weakly bonded abrasive blades to avoid burning. In preparing specimens, edge retention (ensuring the sample and mounting media are in the same plane) 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, particularly 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.

Preparation Procedures

Sectioning

Relatively soft specimens (less than 35 HRC or 345 HV) 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 cut-off wheels. The blade should have weak 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 brittleness of such steels. The fractured surface can then be ground and polished for examination. For high-hardness, high-alloy steels, sectioning with a diamond or cubic boron nitride wheel in a precision saw can provide high-quality surfaces with minimum damage. Although the cutting rate is low, such surfaces are smooth, and grinding can begin with rather fine grits (240- to 320-grit silicon carbide, for example).

Mounting

Mounting involves encapsulating the specimen in thermoplastic or thermoset media prior to grinding and polishing. While bulk specimens can sometimes be prepared without mounting, it is almost always advantageous to mount. Mounting acts to preserve the quality of the specimen edge and provides significant benefits for handling during preparation whether performing manual, hand polishing or automating the procedure. If examination of the specimen edge is important, mounting is necessary to provide edge-retention, ensuring the mount media and sample surface are in the same plane, thus providing a crisp edge of the sample for analysis. Electroless nickel plating has been performed to further enhance edge-retention, however with top quality mounting media such as EpoMet, plating is generally not necessary.

Compression mounting in a mounting press, such as the SimpliMet® 4000, is the best option. The alternative is cast mounting using two-part epoxy or acrylic. While compression mounting requires capital investment of the mounting press, compression media is more economical than cast media, mold quality is generally superior with a mounting press, molding time is shorter and there is no mixing of liquids and clean-up associated with cast mounting. If cast mounting is used, good quality epoxy, such as EpoKwick® FC or EpoxiCure®, is in order to ensure shrinkage gaps at sample edges are not present and infiltration of voids in samples is assured. Acrylic media is ill advised due to shrinking concerns and poor resistance to solvents and some etch solutions.

EpoMet®, a silica-filled thermoset compression medium with epoxy component, provides the best quality metallurgical mold available. Far superior to Bakelite, EpoMet exhibits high abrasion-resistance, leaves no shrinkage gaps and ensures edges of the specimen are crisp and in focus for microscopic examination. It is not degraded by exposure to solvents and etch solutions. PhenoCure® is a low-cost alternative that may be used in cases where edge information of specimens is less critical.

The Traditonal Grinding and Polishing Approach

In the traditional approach, either manual (hand polishing) or automated devices are used. Water-cooled silicon carbide paper (200- to 300-mm, or 8- to 12-inch diameter) 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 minutes 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 adequate. The diamond abrasive may be applied to the polishing cloth in paste or slurry form. For the coarser diamond abrasives, low-nap or napless cloths are preferred; 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 100 to 150 rpm and moderate pressure should be used. Polishing times of ~2 minutes 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 γ- 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 a scratch-free and artifact-free condition due to their relatively high hardness.

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 various sizes, mounted or unmounted. Either 200, 250 or 300mm (8, 10 or 12 inch) diameter formats may be employed. Newly developed surfaces and abrasives permit achievement of surface qualities more than adequate for research work with as few as three steps.

Automating the polishing process with the AutoMet® or EcoMet® 30 machines provides superior surface finish than can be obtained with hand polishing. Automation also significantly increases through-put as preparation of many samples can be accomplished in minutes.

Central-force mode, where pressure is applied to a specimen holder centrally and specimens are locked into the holder throughout the preparation process, lends itself to optimal flatness of the sample surface. Single-force mode, where specimens are inserted freely into the holder and pressure is applied individually to each, is highly useful for re-polishing (to remove an etch for example) or preparation of one or two specimens at a time. The AutoMet and EcoMet 30 machines are capable of both central- and single-force operation.

Table 1 lists a four-step procedure that yields surfaces of a sufficient quality for any needs. For production work, step 6 could be omitted, yet the results will be quite satisfactory for routine examination. If step 6 is utilized, results are better and photographic work of publication quality is obtained.

In complementary rotation the sample holder (head) is rotating in the same direction as the platen (normally counter clockwise) while in contra they rotate in opposite directions. Contra produces a somewhat greater removal rate. If the sample holder rotates at <100 rpm, the abrasive and lubricant will stay on the surface longer using contra. In complementary mode, centrifugal forces throw the liquids off the platen surface almost as quickly as it is added. If relief patterns are observed around oxides or sulfides after step 6, simply repeat step 6 using complementary rotation and it will be removed. This happens rarely and is usually specimen specific in nature.

MasterPrep® Alumina Suspension has a 0.05-µm alumina particle size and is made by the sol-gel process, rather than by the traditional calcination process, and is agglomerate free. If all these steps are followed, from cutting to polishing, the final step can be less than 5 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 discs), and using contra rotation with a low head speed – these are the key steps to obtaining perfect renderings of the true microstructure.

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.

Table 1. SumMet Method for Iron

<table>
<tr><th>Step</th><th>Surface</th><th colspan="6"></th></tr>
<tr><td>1</td><td>Sectioning</td><td colspan="6">Abrasive Cutter with a wheel recommended for use on ferrous materials HRC15 -35</td></tr>
<tr><td>2</td><td>Mounting</td><td colspan="6">Compression or Castable, typically with EpoMet or VariDur 200</td></tr>
<tr><th>Step</th><th>Surface</th><th>Abrasive</th><th>Lubricant</th><th>Load - lbs [N]</th><th>Time (min:sec)</th><th>Platen Speed (rpm)</th><th>Rotation</th></tr>
<tr><td>3</td><td>CarbiMet</td><td>320 [P400] grit</td><td>Water</td><td>6 [27]</td><td>Until Plane</td><td>300</td><td>Comp</td></tr>
<tr><td>4</td><td>UltraPad</td><td>9µm MetaDi Diamond</td><td>NA</td><td>6 [27]</td><td>5:00</td><td>150</td><td>Contra</td></tr>
<tr><td>5</td><td>TriDent</td><td>3µm MetaDi Diamond</td><td>NA</td><td>6 [27]</td><td>3:00</td><td>150</td><td>Comp</td></tr>
<tr><td>6</td><td>MicroCloth</td><td>0.05µm MasterPrep Alumina</td><td>NA</td><td>6 [27]</td><td>2:00</td><td>150</td><td>Contra</td></tr>
</table>

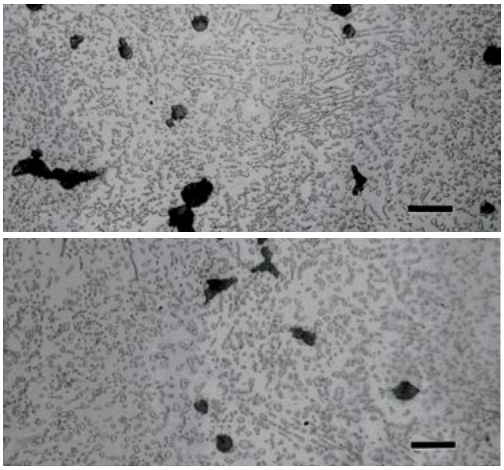

Figure 1. Annealed microstructure of type O6 graphitic tool steel prepared (top) with the three-step method using the UltraPol® silk cloth for step 2; and (bottom) using the ApexHercules® H rigid grinding disc for step 2 (magnification bars are 10-µm long; 4% picral etch).

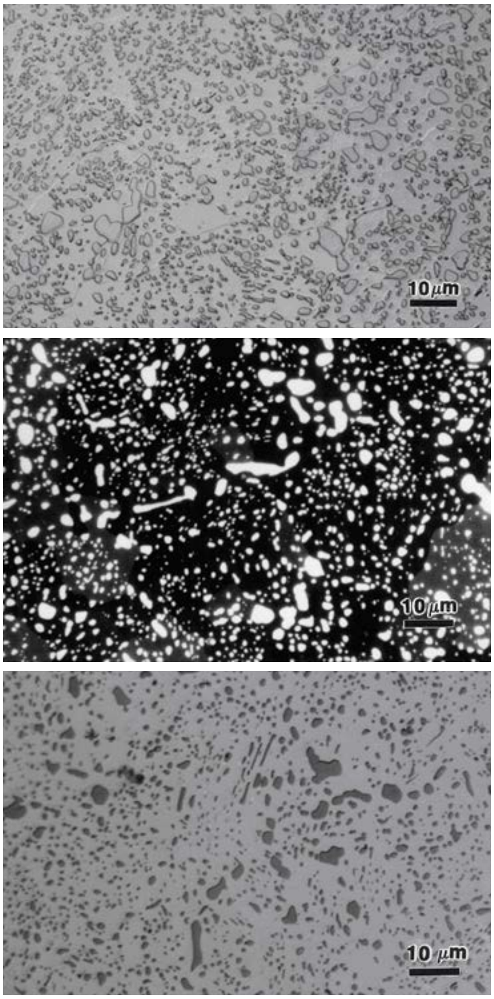

Figure 2. Spheroidize annealed W1 water-hardened carbon tool steel etched with (top) 4% picral to reveal the general structure, with (middle) Klemm's I to color the ferritic matrix, and with (bottom) alkaline sodium picrate at 90 °C for 60 seconds to color the cementite.

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 which generally obscurs 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 for martensitic steels. 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 less used, can be of great value. Table 2 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-step preparation method with annealed O6 graphitic tool steel as an example. Note that the graphite has been fully retained regardless of the surface used, and that there is no residual deformation or scratches in the ferrite and the cementite is clearly revealed using picral. 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.

Experiments with a number of tool steel grades in either the annealed or quenched and tempered condition, or both, were conducted to assess the selectivity of the etchants listed in Table 2 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 etchant was used the specimens were completely re-prepared. Results are given in Table 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, use 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.

Table 2. Etchants for Tool Steels

Composition	Comments
1-10 mL HNO3 99-90 mL Ethanol	Nital. Most commonly used reagent. Reveals ferrite grain boundaries and ferrite-carbide interfaces. Excellent for martensite. Do not store solutions with >3% HNO3 in a tightly stopped bottle.
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. Aqueous 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 NH4F•HF. Immerse until the surface is colored. Ferrite and martensite are colored; carbides are not affected. Good for high chromium tool steels.
2 g Picric acid 25 g NaOH 100 mL water	Alkaline sodium picrate. Colors cementite and M6C carbides. Immerse specimen in solution at 80-100 °C for 1 minute or more.
10 g K3Fe(CN)6 10 g NaOH or KOH 100 mL water	Murakami's reagent. Use at 20 °C to outline and darken M7C3 and M6C, and to outline M2C. M23C6 is faintly colored.
4 g KMnO4 4 g NaOH 100 mL water	Groesbeck's reagent. Use at 20 °C to outline M2C and to outline and darken M6C. M7C3 is faintly colored.
1 g CrO3 100 mL water	Blickwede and Cohen's etch. Use at 2-3 V dc, 20 °C, for 30 seconds with a stainless steel cathode. Outlines M23C6, outlines and colors M7C3, colors MC and attacks M2C.

Table 3. 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	Outlined/ Colored	Outlined/ Colored	NA	Outlined
Groesbeck	NA	NA	Faint	Outlined/ Colored	NA	Outlined
1% CrO3	NA	Outlined	Outlined/ Colored	NA	Colors	Attacks

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