No. 10

LECO MET-TIPS Ideas for Metallographic Procedures

Metallography: A Tool for Quality Control

Nowadays, more than ever before, there is a real need for some sort of quality control to be set up for checking incoming material. Without a check, the buyer must assume the seller is giving him what he ordered, but too often this is just not the case—and it is not a deliberate attempt to defraud, but the result of negligence on the part of the individual who must go out to the stockpile to fill an order. For example, a customer orders SAE 1060 cold-rolled steel but receives SAE 1030 cold-rolled steel; to any examination by eye they look alike, they feel alike, and even weigh the same, but they certainly will react differently when heat treated. In fact, if the customer wanted the SAE 1060 material to be heat treated to achieve a through-hardened condition and he inadvertently received SAE 1030, through-hardening could never be achieved; he would have a hardened surface but not a hardened core. He in turn blames the heat treater for faulty heat treating practices, and so the circle of blame starts. In another situation, a customer has been ordering a certain grade of steel over a period of time, but all at once the material does not perform as it had. A grain size measurement performed on incoming material showed the grain size to be much larger than what had been received in the past. Big differences in grain size will affect the behavior of the material. As a result, customers are now specifying grain sizes to be within a certain range and to be furnished to the customer along with mill specifications. Situations like these are not hypothetical, they can and do happen, and they can easily be avoided by having an incoming quality control inspection involving metallography.

Metallography is the preparation of specimens for microscopic examination and then the study of the microstructure in relation to the physical and mechanical properties of that particular alloy. This is a text book definition and to one schooled in metallography makes good sense, but in layman's terms what does it mean? Key words in the definition are "preparation", "microstructure", and "physical and mechanical properties". Preparation involves taking a piece of metal, cutting off a small representative piece, and imbedding it in some molding material because the piece is usually too small to handle conveniently. Next it is ground with several successively finer abrasive grits, polished until all the scratches from the last grind are removed, subjected to a corrosive environment (usually acidic), then viewed on a microscope at magnifications ranging from 50 to 1500 diameters. Microstructure is the microscopic structure of the polished and etched specimen. Physical and mechanical properties deal with hardness, hardenability, ductility, and tensile strength, and all relate to how the material will perform in its designed purpose.

There are two types of metallography: research and quality control. Of the two, research metallography is perhaps the more sophisticated in that it requires a knowledge of phase diagrams, TTT diagrams, and certainly a vocation to devote one's career to research. On the other hand, quality control metallography is the most important to industry. In quality control, a very rapid turn around—from the time a specimen is submitted for metallographic preparation until the results are known—is paramount. Many times production is halted until the met lab has performed the work for evaluation—has the heat treating yielded the correct hardness or the desired grain size; has the carburization treatment given the desired case depth; is the carbon potential too high or too low? Some or all of these questions must be answered before production can resume. These jobs are sometimes referred to as "rough-and-dirty" jobs, but what they really want is a quick and good job. Unfortunately, these "rough-and-dirty" jobs can sometimes be misleading when interpretation is performed because of poor preparation in the laboratory. Poor preparation can be avoided quite easily and it only takes a few seconds longer. The satisfaction, of course, is having a sample that is not only quick but one you know is good, and no apologies are necessary.

There are three distinct processes in the overall preparation of samples for metallographic examination: **preparation,** which consists of sectioning, mounting, and grinding; **polishing and etching**; and **microscopic examination**. Of these three processes, the one most often overlooked is the first process, and in particular, the sectioning and grinding processes. Once a specimen has been properly sectioned and ground, it constitutes approximately 95% of the effort involved in obtaining a good specimen. Mounting is important but not nearly as important as sectioning and grinding. Moreover, many samples can be processed without mounting.

Let's take these various processes and go through them one by one, and point out the pitfalls that are ever present.

Sectioning

In sectioning, one must obtain a representative section from a larger section, and the cutting or sectioning equipment used is very important. Abrasive cut-off wheels will produce a better surface than other types of sectioning equipment provided the proper abrasive wheel is used. A good rule to follow when sectioning relatively soft material (such as ferrous alloys not hardened to a great degree), is to select a hard abrasive wheel; and conversely, when sectioning hard materials (such as hardened tool steels, metal carbides, or metal borides), one should select a soft wheel. One wants the abrasive wheel to wear away fairly rapid so it will always be presenting a good cutting edge. If one makes an improper selection, such as using a hard wheel for hard material, the cutting edge of the blade becomes glazed and the cutting edge is lost. The use of any type of sectioning apparatus results in deformed or smeared metal. The depth of deformation is far less when an abrasive wheel is used for sectioning than by other sectioning means, such as a band saw or an oscillating hacksaw. A bandsaw or an oscillating hacksaw can be used to cut a large piece from an even larger piece, but hopefully, the sectioned piece would then be further sectioned using an abrasive wheel. A bandsaw cut will not deform the metal as extensively as an oscillating hacksaw because it runs at a relatively low speed and not too much heat is generated, but in the case of an oscillating hacksaw, the deformation can be so great it can permeate completely through the sectioned piece, particularly if the alloy is prone to deformation.

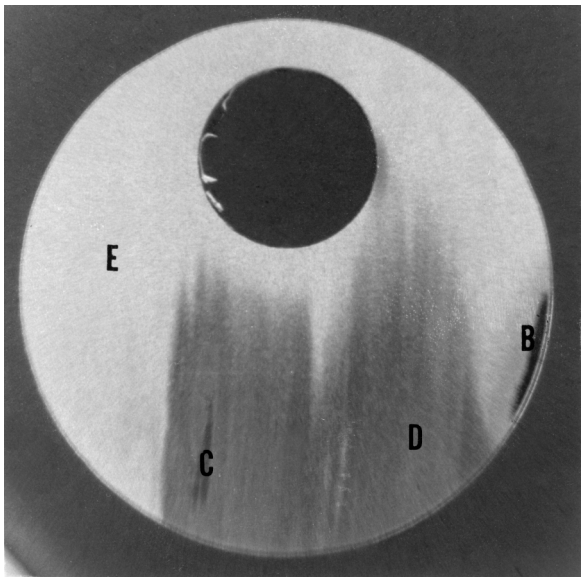
One should always section with a coolant, preferably one with a rust inhibitor to prevent rusting of moving parts. Without a coolant, the specimen can be "burned" and this results in an altered microstructure. This is a common occurance but one that can easily be overcome. For example, and for the sake of illustration, let's assume an alloy has been quenched and tempered to produce a tempered martensitic microstructure, but for some inexplicable reason, the tempering temperature was too low and the as-quenched martensite was not tempered properly. A sample of the alloy is submitted to the met lab for preparation and microscopic examination, and sectioning is performed dry. Burning will occur over most of the surface. Sometimes it is quite visible in the form of dark streaks, other times it may not be quite so visible being a light straw color. The sectioned piece is metallographically prepared, and on examination, the microstructure is found to be tempered martensite, and even microhardness tests in the microstructure confirm the hardness specifications. As a result, the part is passed through the Q.C. inspection department as having received a proper heat treatment. Based upon the metallographic examination of this one part, all the heat-treated parts are sent into service. Eventually the parts fail after a short service life and an irate customer is the result. A situation such as this happens frequently but is one that can be avoided by a few simple applications. By being aware that dry sectioning can result in a "burned" sample, the sectioned piece can be deep-etched in 2% Nital to see if burning has occurred. The burned areas will etch darker and much more quickly than unburned areas. If burning is evident, one resorts to wet grinding by belt or discs and alternately etching and regrinding to get below the burned area. Several such grinding-etching steps may be required to remove the burned layer, and only when the evidence of burning is completely removed is the sectioned piece ready to be processed in a normal manner.

An example of how burning can affect and change the microstructure is illustrated in Figure 1. The material is a quenched and tempered nodular cast iron that was sectioned dry using an abrasive cut-off wheel. The effect of dry sectioning was not evident until after the specimen was given a routine metallographic preparation and etched with 2% Nital to reveal the microstructure. Although the normal microstructure is quenched and tempered martensite, burning has altered it considerably. The microstructure shown in Fig. 1B is as-quenched martensite. At this location, the metal reached an austenitizing temperature and the surrounding metal acted as a quenching media. This structure is extremely hard but very brittle and prone to cracking. Eventually, cracking would initiate in this area and propagate through the sample. Fig. 1C is from a similar area, but here the microstructure is a very fine tempered martensite with proeutectoid ferrite and small iron-carbide particles, further evidence of high temperature and rapid quenching from the adjacent metal. The iron-carbide is probably a result of slight dissolution of the graphite nodules and gives a local area rich in carbon. Fig. 1D is from the dark etching area and is representative of the microstructure there. It is a coarse, acicular tempered martensite and illustrates different cooling rates; the light etching areas are fine tempered martensite. Fig. 1E is the normal microstructure consisting of tempered martensite.

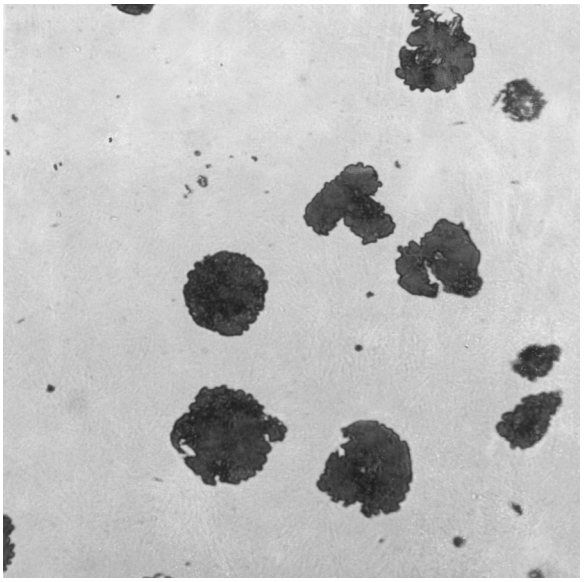
Grinding

The purpose of grinding is to remove the deformation brought about by the sectioning operation. Although a grinding step removes deformation, it also introduces more deformation (but to a lesser degree), and as one proceeds through a series of decreasing grit sizes, the depth of deformation becomes quite small.

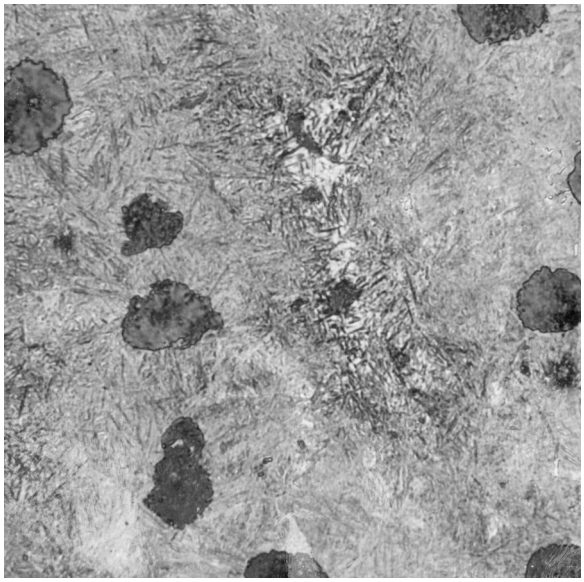
Just as in sectioning, and for the same reasons, a coolant should be used to avoid burning of the specimen. Burning can occur very quickly, particularly with the coarser grits. Again, if burning is suspected, etching in 2% Nital at any stage of the grinding sequence will reveal the extent of burning, and only repetitive etching and grinding will get below the effects.



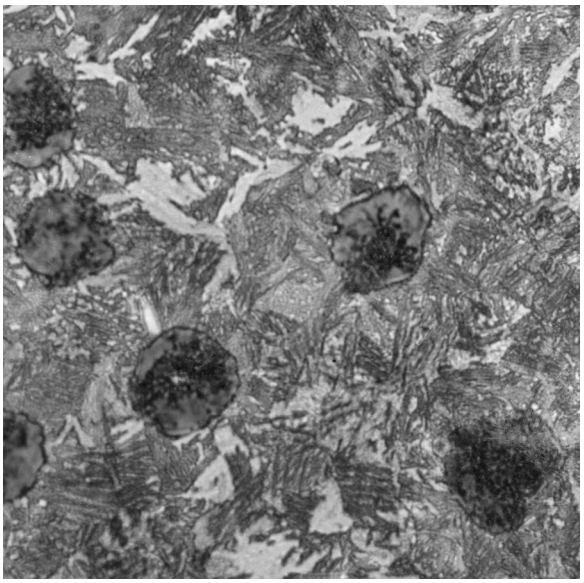
A **2% Nital** **4X**



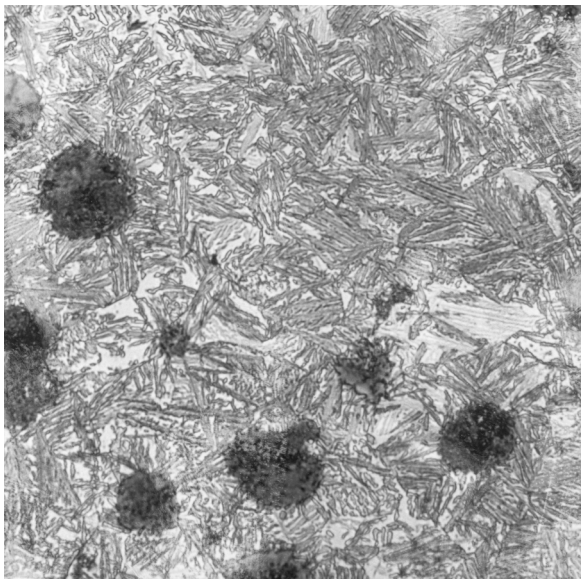
B **2% Nital** **200X**



C **2% Nital** **200X**



D **2% Nital** **200X**



E **2% Nital** **200X**

FIGURE 1.

Different microstructures in a nodular cast iron sample resulting from "burning" during the sectioning operation.

A. Burning Streaks

B. As-Quenched Martensite

C. As-Quenched Martensite, Ferrite and Iron-Cabide in Fine Martensite matrix

D. Coarse Tempered Martensite

E. Normal Microstructure

Dry grinding can also result in non-metallic inclusions being torn out, cracked carbides from thermal shock, and extensive deformation.

During grinding, care should be taken to keep the sample as flat as possible. Heavy pressure is not recommended, nor is it necessary. Heavy pressure will result in extremely deep gouges and a beveled specimen. If one grinds at an angle where the specimen is wedge-shaped and subsequently polishes the specimen, several things can happen: keeping the image in focus during microscopic examination is almost impossible; and if one is measuring a plating thickness or determining the effective case depth by means of a hardness traverse, the measurements will not be exact. There is a taper effect, and it is like measuring the hypotenuse of a right triangle when one should be measuring a leg.

When going through the various grinding steps, one should rotate the sample 45 to 90 degrees between each step. This enables the operator to visually check the effectiveness of the grind. Grinding should continue until all the scratches are uniform and running in one direction before going to another grinding step. Samples should be cleaned before going to the polishing step to eliminate carry-over of grinding debris that will contaminate the polishing wheels.

Polishing

Polishing is brought about by covering a polishing wheel with an appropriate polishing cloth and impregnating the cloth with the polishing media and using a suitable lubricant.

Most laboratories use two or three polishing stages—usually two diamond polishing steps and one final polish. For most applications in a Q.C. lab (where one is checking grain size or visual case depth measurements for instance), one diamond polish will usually suffice. A 6-micron or 1-micron diamond compound, impregnated on a nylon or red felt cloth, and using oil as a lubricant will effectively remove 600-grit SiC scratches in approximately 2 minutes. If it is necessary to obtain a finer polish, then one can go to 0.05-micron alumina oxide on a medium nap cloth using ordinary tap water as a lubricant.

There are literally hundreds of cloths that can be used for the polishing steps, but one criterion should be followed when polishing with diamond compound—a napless cloth such as nylon should be used to insure the entire surface of the specimen comes in contact with the diamond particles. Polishing is continued until all traces of the grinding process are removed.

Etching

Etching is accomplished by subjecting the specimen to a corrosive attack by acid or basic chemical—usually in an alcohol or aqueous solution. The polished sample is either immersed in or swabbed by the etchant until the as-polished luster is gone and a frosted appearance is achieved.

During the polishing steps, a layer of disturbed metal is usually present over some or all of the specimen surfaces. Disturbed metal is a slight layer of deformation or metal that is removed by the abrading action of the polishing media and redeposited at other locations. The first etch usually will serve only to etch this disturbed layer, and sometimes, depending upon the alloy, a pseudo-microstructure. Only repeated etching and polishing will get below the effects of disturbed metal.

To briefly summarize the do's in good specimen preparation and to avoid erroneous interpretation based upon poor preparation.

1. Use a coolant during the sectioning operation to avoid the possibility of "burning" the samples which can result in an altered microstructure.

2. Etch sectioned carbon steel surface in 2% Nital as a visual aid in determining if "burning" has occurred.

3. Grind as flat as possible to avoid a beveled mount or worse yet, a many-faceted one.

4. If necessary, tch-polish several times during the final polishing step to remove disturbed metal layers.

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