Trends in SPECIMEN PREPARATION

New approaches to the preparation of metallographic samples can reduce costs, improve efficiency, and reveal more useful information.

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Metallographic preparation procedures have changed more in the past decade than in any comparable period of time since Henry Clifton Sorby successfully prepared his first specimen in 1863. For many years, the well-established preparation wisdom has been to sequentially grind specimens with a series of progressively finer water-cooled silicon carbide abrasive papers (120-, 240-, 320, 400, and 600-grit sizes). Typically, each was used for 60 to 120 seconds, and the paper was worn out.

This was followed by polishing with finer abrasives, generally with one or two steps (6-, 3-, and 1-μm diamond abrasives were the most common sizes), followed by polishing with still finer abrasive slurries. Alumina has been the preferred final abrasive, but colloidal silica has gained dominance over the past fifteen years. Again, one or two sizes of alumina (0.3- and 0.05-μm alumina, α and γ crystal forms, respectively) were used, depending upon the material being prepared and the nature of the work.

This procedure, with minor variations, became commonplace in the 1960s and has been refined and modified by combining various surfaces, abrasive sizes, speeds, pressures, times, etc. The many possible combinations reflect the "art" side of metallography and individual preferences. In the 1970s, these procedures began to be automated, as labs began to downsize and reduce costs. Such methods generally provide adequate quality specimen surfaces for most applications and are still widely practiced today.

**Fellow of ASM International*

This article describes new abrasive materials and methods, shows how to assure sharp edge retention, and details a rigid-disk method of specimen preparation.

Abrasive materials

Alumina is making a comeback as new methods of manufacture that are based on the sol-gel process have been introduced. In the past, calcined alumina particles always had some degree of agglomeration, which reduced the effectiveness of the finest abrasives. However, new sol-gel alumina slurries, such as Masterprep suspension, provide vastly improved surface finishes, in some cases exceeding those possible with colloidal silica suspensions.

Over the past decade or so, a concerted effort has been made to streamline preparation methods using new surfaces and abrasives, fewer steps, and automation. The primary goal has always been the exposition of the true structure without preparation-induced artifacts, in minimal time, and with reduced cost. Important secondary goals include improving edge retention; enhancing inclusion, precipitate, and intermetallic retention; and minimizing relief (surface height differences) between constituents of different hardness or abrasion resistance.

Edge retention

Achieving excellent edge retention in particular has been a long-sought goal, due to the frequent need to examine the extreme surfaces of components, often at high magnification. If the outer edge of a specimen abrades faster than the bulk, the surface edge will be curved rather than flat. Unfortunately, as higher magnification objectives resolve finer details, the numerical aperture rating increases and the depth of focus is reduced to well under 1 μm with a 100X, 0.95 NA objective (for 1000X nominal magnification at the film/image plane).Consequently, if the detail at the edge of the specimen is to be observed at the same time as the rest of the structure beneath the edge, then the flatness must be nearly perfect right out to the edge. This is not a simple task.

Many procedures have been put forth over the years to enhance edge retention. Simply mounting the specimen in a polymer was a good first step;

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The most satisfactory approach for edge retention was to first plate the surface of interest with electroless nickel, or some other compatible metal.

but by itself, this was usually inadequate. Most polymeric mounting resins, whether used hot under pressure or "cold" as a castable resin, do not provide this degree of edge flatness. The best are the thermosetting epoxy resins that contain filler material, such as Epomet resin.

The most satisfactory approach for edge retention was to first plate the surface of interest with electroless nickel, or some other compatible metal (by electroless or electrolytic procedures) prior to encapsulating the specimen in a polymer. If plated correctly, the edges would become round at the outer surface of the plating, not at the interface between the plating and the specimen. However, if done incorrectly, the plating could actually be pulled away from the specimen, leaving a gap between specimen and plating, which would make the plating ineffective. Naturally, the plating composition must also not create galvanic effects that would interfere with etching. In addition, plating is a slow process, not suitable for production labs.

For the castable resins, metallographers have tried adding a variety of hard filler materials (some of these have also been added to compression mounts, but with minimal improvements) to improve edge retention. For example, very fine alumina shot was recommended at one time. However, with a hardness of ~2000 HV, its grinding and polishing characteristics were incompatible with metallic specimens. When alumina shot is mixed with epoxy and placed around a metallic specimen, the mount can be prepared as if it is a ceramic or a metal. The difficulty is that the techniques are different, and generally incompatible.

If the mount is prepared as a ceramic, the true metal structure is not revealed properly, with the difference increasing as the metallic specimen hardness decreases. If the mount is prepared as a metal, then it takes a very long time to prepare. Recently, we have introduced a soft ceramic shot, Flat-Edge Filler shot, which has a hardness of ~775 HV. When added to epoxy, it has grinding and polishing characteristics compatible with metals. Figure 1 shows an example of edge retention in which the Flat-Edge Filler is added to an epoxy mount containing an annealed 5140 alloy steel specimen (note the cracks present near the edge).

Fig. 1 — Surface of forged and annealed 5140 alloy steel exhibits surface cracks (arrows) using Flat Edge Filler shot in an epoxy mount for edge retention. (1000X, 4% picral etch).

Mounting presses

In the late 1970s, automated mounting presses were introduced that cooled the mount back to ambient temperature under pressure. This is ideal for thermoplastic resins such as Transoptic resin, which polymerizes during the cool-back to near ambient (to at least 70°C) under pressure. Although it was quickly recognized that cooling a thermosetting resin back to near ambient temperature under pressure reduced the size of the shrinkage gaps, this influence on edge retention was not immediately recognized. In the past, when a thermosetting resin such as a phenolic was made, it was typically held under pressure for five minutes at 150 to 180°C to cure the resin, and then it was ejected hot. In those days, manual ("hand") preparation was most common, so the mount was usually quickly cooled in water so that it could be prepared without burning the fingers.

However, this practice was very damaging, because the metal cooled faster than the polymer and contracted away from the polymer, leaving a gap between specimen and mount. It is almost impossible to retain a good edge in the case of an unsupported edge caused by a shrinkage gap. A free edge will be rounded by any abrasive surface that compresses under load. The gaps also cause bleeding problems before and during examination of the structure with the microscope. Any etchant or solvent in these gaps will seep out and obscure the edge detail, and may fall onto the objective lens (with an inverted microscope) causing damage, or merely blurring detail.

Improving edge retention

In more recent years, a variety of new types of surfaces have been developed for preparing specimens; these include new types of polishing cloths, laps, grinding disks, and rigid grinding disks. It became apparent that preparation could be speeded up if some or most of the silicon carbide grinding steps could be eliminated. Silicon carbide paper works very well for most metallographic specimens, but it does have a short life.

With automation came the concept of planar grinding, in which several specimens are placed in a holder with the side to be prepared facing down. However, the locations of the faces of those specimens are not identical initially. One object of planar grinding is to move all of the surfaces to a common plane, so that the subsequent steps will remove material equally from all of the specimens. Also, planar grinding must remove all of the damage produced by sectioning of each specimen.

Silicon carbide and alumina papers are suitable for planar grinding, but other abrasive surfaces have also been tried. For very hard sintered carbides and ceramics, metal-bonded diamond disks are effective, but they are not satisfactory for metals. Grinding stones are effective for planar grinding and they have a high removal rate, but they must reach speeds above 1500 rpm to cut, which requires a separate machine. They also generate considerable debris that must be contained, and they must be trued regularly with a diamond tool. Further-

more, grinding stones produce substantial damage, and the alumina abrasives may embed in certain materials.

Some grinding disks have surfaces partially covered by abrasive, unlike the metal-bonded diamond disks. This new type of disk has small surface pads that contain abrasive of a specified size, generally diamond. An example of such a disk is the Ultra-Prep disk. The diamond may be metal-bonded or resin-bonded to the surface. The surface is only partially covered, allowing a reduction in surface tension, which improves the removal rate. Resin-bonded diamond disks are suitable for the planar grinding of soft metals such as aluminum, while the metal-bonded disks can prepare a wide variety of ceramics, carbides, and metals, even copper alloys. These disks produce excellent edge retention.

Many other surfaces have been considered. Laps have been tried and they work quite well in the beginning, but they get out of flat with use. Keeping them flat is a problem. Screen-like metal mesh pads with diamond abrasive bonded to the mesh by a metallic plating have been introduced, and they too are successful with very hard materials, but less useful for softer metals. Woven stainless steel "cloths" may be combined with coarse diamond to prepare hard metals and other hard materials, but again, they are not practical for softer metals. Several rigid grinding disks have been developed, and these may be selected after the planar grinding step to replace the remaining silicon carbide grinding steps. Abrasive must be added to these disks, generally diamond as suspensions. In some cases, they are also acceptable for the planar grinding step. To date, they have been most successful with medium and high hardness materials. Development of similar disks for low hardness metals has been more challenging.

Beyond planar grinding

After the planar grinding step, modern preparation procedures aim to yield specimens adequately prepared within two to four more steps, depending on the material and the degree of perfection needed. For example, for routine production evaluations of microstructure, many materials can be adequately prepared with three or four steps. However, some materials are very difficult to prepare correctly. Also, if publication-quality images is required or if image analysis is to follow, an extra step or two may be necessary. These new procedures are reliable and reproducible, and generally generate results better than could be achieved with the "traditional" method described at the beginning of this article.

Rigid grinding disks (RGD) are being selected more often for the second step, that is, after planar grinding. In some cases, they are satisfactory for the planar grinding step as well. They generally are 15-, 9-, and 6-μm diamond suspensions, and replace all of the SiC steps after planar grinding. Up to now, 9-μm probably has been the most common diamond abrasive size for the second step with a rigid grinding disk. Today, even finer abrasive sizes are possible, which makes it easier to get publication-quality images with as few as three steps, for some materials.

Examples of contemporary preparation procedures are being published frequently. They highlight a variety of surfaces and possible approaches. Most of these surfaces are relatively new in metallography, and may be unfamiliar to many metallographers, as it is often difficult to find time to experiment with new methods and materials. However, specimens of these materials may be prepared successfully by a wide variety of materials and methods.

This is both a blessing and a curse! It is a blessing in that materials of varying cost and availability are acceptable, although some may work better than others on a given specimen. It is a curse in that new metallographers can feel overwhelmed with the number of choices available. Years of experimentation may be needed to master all of the possible combinations and permutations available (the "art" of metallography). Relatively few metallographers have experience with more than a small fraction of all the consumable products currently on the market.

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Stainless steel disks

We have recently introduced a new rigid grinding disk called the Hercules H rigid grinding disc (H-RGD). It differs from others in that it consists of a thick stainless steel disk upon which a number of approximately 12-mm diameter pads of epoxy are bonded. The epoxy pads contain filler metal and some porosity. This disk can be held in place magnetically (by an Apex M magnetic psa disk applied to a platen) or it can be glued to a platen with double sided tape (for example, with a Met-Grip liner). The pads cover only a portion of the surface, so that surface tension can be controlled and swarf can be easily removed. If the percentage covered by the pads is too low, the cutting rate will decrease. Hence, the amount of area covered has been optimized.

In this type disk, abrasive is added during grinding, rather than being embedded. Diamond has been the preferred abrasive for rigid grinding disks, and polycrystalline suspensions have higher removal rates than monocrystalline suspensions. Charging the pads with paste is impractical. The H-RGD is best suited for preparing hard metals and alloys, although nearly all types of steels have been successfully prepared with it. A version for softer nonferrous metals and alloys (S-RGD) is under development.

The following are examples of the application of this disk.

• *Sintered carbides:* The first example demonstrates both planar grinding and the second preparation step in preparing sintered-carbide cutting tools that typically exhibit hardnesses in the 1200 to 1500 HV range. The inserts were cut in half by an Isomet 4000 precision saw with a 15 HC blade at 4000 rpm and a feed rate of 0.2 inch/min, which required 4 to 5 minutes per cut. They were mounted in Epomet thermosetting epoxy resin, and were cooled under pressure. Six specimens were placed in a

Table 1 — Four-step practice for sintered carbides

Surface	Abrasive/ Size	Load, lb (N)	Speed, rpm/ Direction	Time, minutes
H-RGD	Metadi Supreme / 30-μm PC	5 (22)	250/ Contra	5
H-RGD	Metadi Supreme 9-μm PC	6 (27)	250/Contra	4
Ultra-Pol cloth	Metadi Supreme 3-μm PC	6 (27)	150/Contra	3
Ultra-Pol cloth	Mastermet 2 0.02-μm Colloidal Silica	6 (27)	150/Contra	2

Note: Contra means specimen holder and platen rotate in opposite directions.

Table 2 — Four-step practice for steels

Surface	Abrasive/ Size	Load, lb (N)	Speed, rpm/ Direction	Time, minutes
SiC paper disk	120-grit (water cooled)	6 (27)	240-300 Comp	Until plane
H-RGD	Metadi Supreme 9-μm PC	6 (27)	120-150 Comp	5
Trident cloth	Metadi Supreme 3-μm PC	6 (27)	120-150 Comp	3
Microcloth pad	Masterprep 0.05-μm alumina suspension	6 (27)	120-150 Contra	2

Note: Comp means specimen holder and platen rotate in same direction.

Table 3 — Three-step practice for steels

Surface	Abrasive/ Size	Load, lb (N)	Speed, rpm/ Direction	Time, minutes
SiC paper disks	120-grit (water cooled)	6 (27)	240-300 Comp	Until plane
H-RGD	Metadi Supreme 3-μm PC	6 (27)	120-150 Comp	5
Microcloth pad	Masterprep 0.05-μm alumina suspension	6 (27)	120-150 Contra	5

Table 4 — Four-step practice for superalloys and stainless steels

Surface	Abrasive/ Size	Load, lb (N)	Speed, rpm/ Direction	Time, minutes
SiC paper disk	120-grit (water cooled)	6 (27)	240-300 Comp	Until Plane
H-RGD	Metadi Supreme 9-μm PC	6 (27)	120-150 Comp	5
Trident cloth	Metadi Supreme 3-μm PC	6 (27)	120-150 Comp	5
Microcloth pad	Masterprep 0.05-μm alumina suspension	6 (27)	120-150 Contra	5

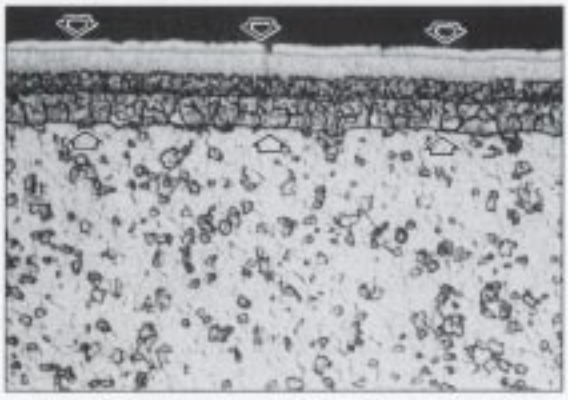

Fig. 2 — Complex coating (bracketed by arrows) on an enriched sintered carbide cutting tool mounted in Epomet resin reveals perfect edge retention. (1000X, Murakami's reagent).

holder, and they were prepared as described in Table 1. The H-RGD was chosen for both the planar grinding step (30-mm diamond) and the second step (9-mm diamond). Ultra-Pol cloth, a psa-backed silk cloth that gives excellent edge retention, was used for both step 3 (3-mm diamond) and step 4 (Mastermet 0.02-mm colloidal silica suspension). After the third step, specimens were examined and found to be adequate for photography, although better detail of the coatings was achieved after step 4, Fig. 2.

• *Steels:* Tables 2 and 3 present four- and three-step practices for preparing steels with the H disk. Colloidal silica may be substituted for the Masterprep alumina suspension if preferred. Figure 3 shows the microstructure of a quenched and tem-

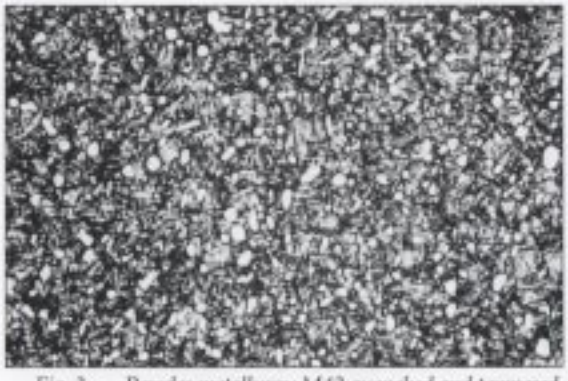

Fig. 3 — Powder metallurgy M42 quenched and tempered high-speed steel prepared in three steps, as described in Table 3. (1000X, 2% nital etch).

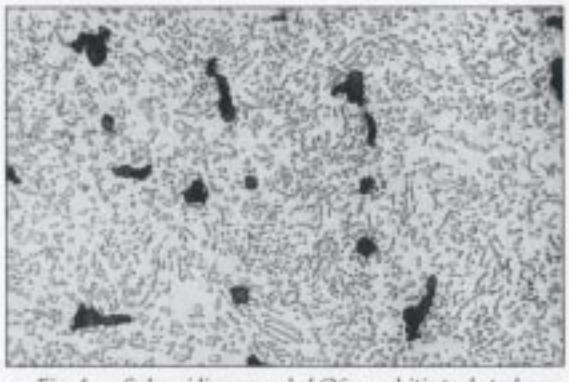

Fig. 4 — Spheroidize annealed O6 graphitic tool steel prepared with the H-RGD, see Table 2. Note the perfect graphite retention and the mixture of spheroidized and partially lamellar cementite in the ferrite matrix. (1000X, 4% picral etch).

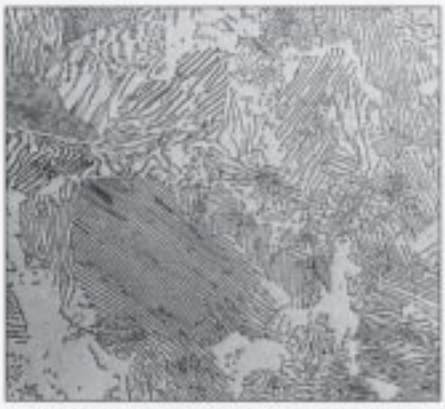

Fig. 5 — Fully resolved coarse lamellar pearlite in annealed 4140 alloy steel prepared according to Table 2. (1000X, 4% picral etch).

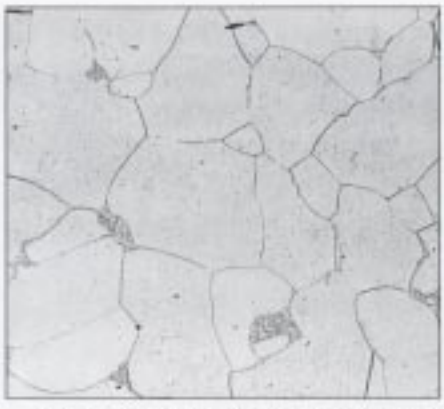

Fig. 6 — Ferritic 1005 sheet steel prepared according to Table 2. (500X, 2% nital etch).

Fig. 7 — Microstructure of solution annealed and aged 18Ni250 maraging steel prepared according to Table 4. (1000X, Fry's reagent).

Fig. 8 — Microstructure of 416 free-machining stainless steel prepared according to Table 4, but with 6-µm diamond for step two. (200X, Vilella's reagent).

Fig. 9 — Microstructure of an annealed dual phase stainless steel (ferrite darkened) prepared according to Table 4, but with 6-µm diamond for step two, viewed with differential interference illumination mode. (200X, 20% NaOH at 1.5 V dc).

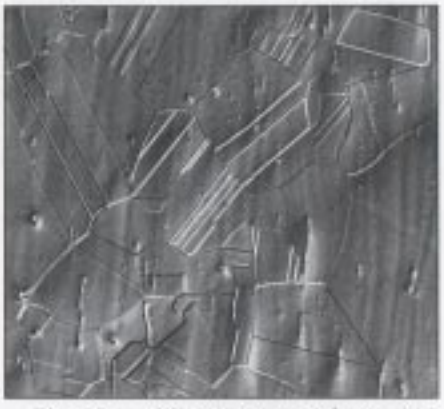

Fig. 10 — Microstructure of type 316 austenitic stainless steel after a 30% cold reduction in thickness and solution annealing prepared according to Table 4. (100X, glyceregia etch).

pered powder metallurgy M42 high-speed steel (about 68 HRC) prepared in three steps by the method of Table 3. The fine, well-dispersed alloy carbide, typical of P/M high-speed steels, is in a martensitic matrix. Figure 4 shows the microstructure of spheroidized annealed type O6 graphitic tool steel (about 180 HB) prepared with four steps. Note that the graphite is fully retained, the carbide is clearly visible, and the ferrite is deformation-free.

Figure 5 shows well-resolved lamellar pearlite in a fully annealed SAE 4140 alloy steel specimen, prepared in four steps with the H disk. Note the small manganese sulfide inclusion that is completely retained. As a final steel example, Figure 6 shows a sheet steel specimen prepared in four steps with the H disk. These examples cover a huge range in hardness from about 68 HRC for the M42 specimen to about 80 HRB for the sheet steel. Microstructures are shown from fully martensitic to fully ferritic. All show the true structure.

• *Maraging and Stainless Steels:* Maraging steels are easily prepared with a four-step method as described in Tables 2 and 4. Figure 7 illustrates the microstructure of 18Ni250 maraging steel prepared with four steps (Table 2).Stainless steels also can be prepared with any of the methods in Tables 2 and 4. Martensitic stainless steels are easily prepared using this type of technology as illustrated by Fig. 8 which shows FM type 416 stainless steel. This was done in four steps with 6-mm diamond for step 2. The microstructure is tempered martensite with stringers of delta ferrite and sulfide inclusions. Duplex stainless steels can be prepared easily, as shown in Fig. 9, which illustrates the ferritic-austenitic (white) structure in DIC.

Austenitic stainless steels are the most difficult type to prepare with the H disk. Figure 10 shows the structure of type 316 stainless after cold drawing with a 30% reduction in diameter followed by solution annealing at 1200°C. The structure is shown in DIC that will reveal any preparation-induced, or process-induced, deformation to the austenitic structure. This reveals some alloy banding (faint longitudinally oriented lines) and some fine precipitates and inclusions. This degree of perfection is not always achieved in four steps for austenitic grades. ■

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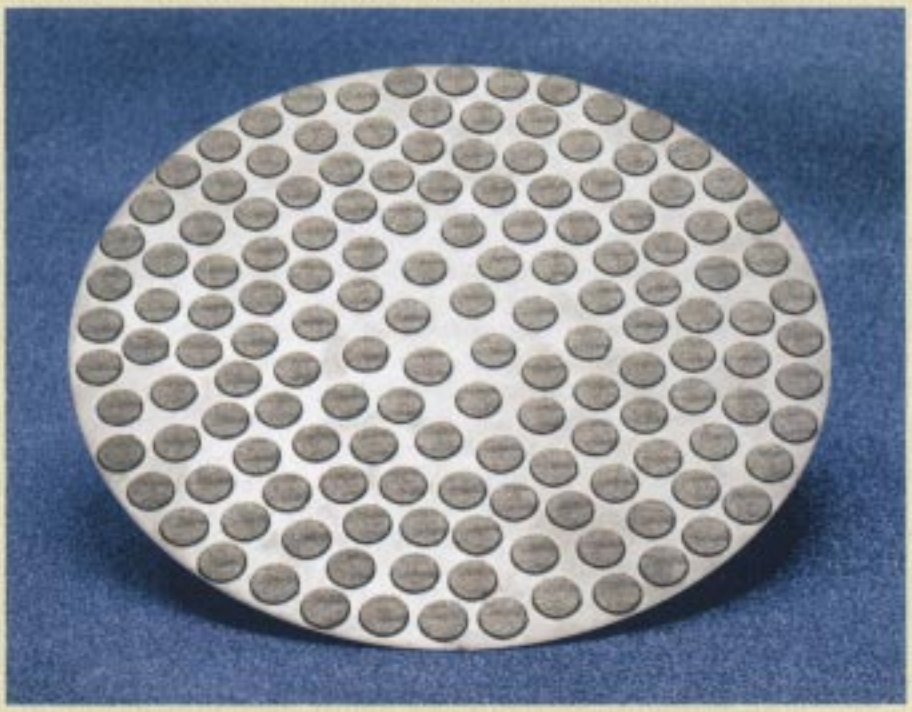

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