

## In-Situ Metallography

Many times a metallographer needs to examine the microstructure of a material without physically destroying the piece. At first glance this seems to be an impossible feat; however, with the techniques involving in-situ, or in-place metallography, this task can become a reality. In fact, as one becomes highly proficient in this technique it is very difficult to differentiate a microstructure that is the result of careful preparation in the laboratory from a microstructure resulting from in-situ metallography.

In-situ metallography is the metallographic preparation of an area located on large pieces that cannot be destroyed by sectioning or taken to a laboratory. Large rolls, shafts, tubes, pipes, inside surfaces, outside surfaces, curved surfaces, vertical, or horizontal surfaces can be successfully examined by replicating the prepared surface with softened cellulose acetate and then inspecting the replica under normal microscopic conditions back in the laboratory.

There are no special tools required to perform in-situ metallography. All the necessary items are found in the laboratory: abrasive papers for grinding, polishing cloths and compounds, etchants to reveal the microstructure, cellulose acetate and acetone, containers to squirt water or alcohol on the prepared surface to flush away grinding debris or an etchant, containers to hold the removed replicas, scissors, tweezers, and blocks of wood to wrap the grinding papers or polishing cloths around. As one becomes more involved with this technique, the necessity for a portable kit containing all the above items soon becomes evident.

### Procedure

When in-situ metallography is required, the first thing to determine is how uncomfortable it will be for the person doing the preparation. If the part to be examined is in a cramped area like the inside of a roll or on a vertical surface high in the air, then a rapid removal of the initial surface is desired. An electric sander (orbital or otherwise), or even a portable drill that has sanding attachments can be used. If these are not available, or if the piece is in an area where electricity cannot be furnished, then small pieces of abrasive paper wrapped around a block of wood can be used to manually grind the surface.

The grinding procedure should always start with the finest silicon carbide grit that will remove the original surface. A finely machined surface can usually start with a 120 grit SiC paper, but if the surface is heavily oxidized or extremely rough, then one should start with a coarser grade of silicon carbide paper or equivalent grinding disc. Grinding steps should follow the same procedures as used in the laboratory, (i.e. 600 grit grind). Water should be used as a lubricant, or if water is harmful to the part(s), other liquid media can be used. Each grinding step should be 90 degrees to the previous grind to enable one to tell when all the previous grinding scratches are effectively removed. Also, at the end of each grinding step, the area should be flushed with water (or other liquid media) to flush away loose abrasive particles.

Once the grinding steps are completed, the area is ready for polishing. Polishing will usually consist of three steps—polishing with 6-micron diamond compound, followed by 1-micron diamond compound polish, and the final polish with 0.05-micron gamma alumina.

The polishing cloth used for the diamond polishing should be red felt, billiard, or canvas in which the diamond compound is impregnated. Although other polishing cloths can be used, these will have greater durability and longer life. A water soluble oil is recommended as a lubricant. The final polishing step will consist of a medium napped cloth using 0.05-micron gamma alumina as the polishing abrasive. Water is usually preferred as a lubricant. As in the case of the grinding steps, a small piece of wood wrapped with the polishing cloths is used. Although one's fingers can be used, more pressure can be applied using the cloth-covered blocks of wood.

Once the grinding and polishing procedures are completed, the area is ready for etching. Etching is the most difficult part of in-situ metallography. Where the effects of grinding and the replication technique can be visibly seen, the correct degree of etching is difficult to ascertain with the eye. When in doubt as to how long to etch, the best method is to etch in degrees. Etch until a frosty appearance is observed, replicate this result, etch heavier and replicate again. The metallographer may want to do this several times, particularly if he or she has no previous experience. After each etching interval the area must be flushed with water or alcohol and dried before replicating.

### **Replication**

As the etching step is the most difficult procedure, the replication technique is the easiest. A small piece of cellulose acetate, 3 to 5 mils thick and approximately three times the size of the area to be replicated, is held by tweezers and softened on one side only. Apply acetone with an eyedropper, allow to set for approximately 30 seconds, then shake to remove excess acetone. The softened side is then pressed firmly against the area with the eraser end of a pencil or the tip of the fingers and held several seconds. Care must be taken that the acetate is not allowed to slip or a smudged replica will be obtained. Let the replica remain in this position until it hardens, usually 10 to 15 minutes, depending upon the size of the replica. Once it has hardened it can be stripped with tweezers. At this point, the

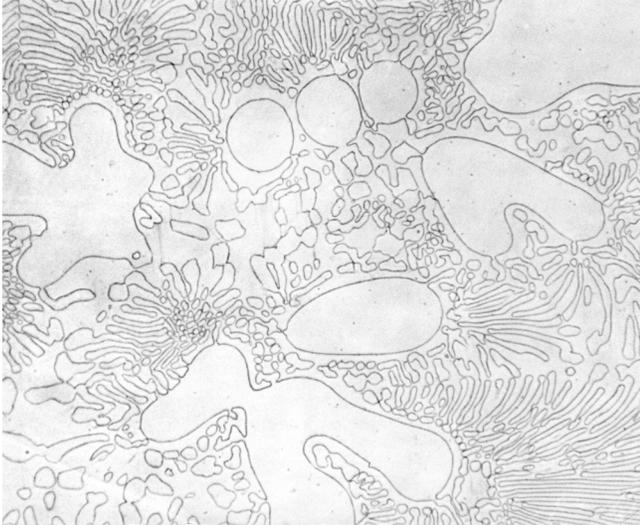
stripped replica should be put into a separate container (usually a small plastic box), or taped to a glass slide with double-sided tape. Several replicas should be made of each area for a better chance of obtaining a good replica. While one replica is hardening, other replicas can be made at different locations. Needless to say, all replicas should be labeled appropriately, particularly if several replicas are taken with different degrees of etching.

### **Examination of Replica**

Examination of a replica is carried out with normal microscopic procedures. However, there are several things that can be done to enhance the viewing of the replica. If possible, the replica should be shadowed at 45 to 60 degrees with carbon in an evaporator. While this is the ideal situation, some laboratories do not have an evaporator and must view the replicas as they are. Placing the replica (using double-sided tape) on a glass slide that has been spray painted black will greatly enhance the contrast. Insuring that all points of the replicated surface are flat will increase the viewing field. Excess acetate can be trimmed off before shadowing with carbon or affixing to a glass slide. Another way to increase contrast is to very lightly spray the replica with flat black paint, however, extreme care must be exercised so there will be no build-up of paint that will obliterate features.

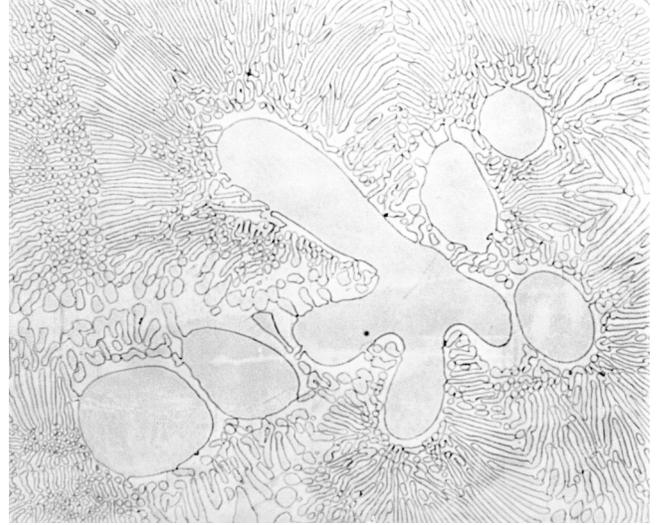
The replica is examined in the same manner as a specimen prepared in the laboratory. Photographs can be taken and semi-quantitative evaluations can be made. One thing to remember is that the replica is just that, and features that are up or down on the surface will be reversed on the replica. For example, porosity will show up as elevations on the replica and hard intermetallics that have a tendency to polish in relief will be recessed on the replica. Also, scratches will show up slightly raised. Keeping these things in mind, one will have no trouble in interpreting the micro-structure.

The following photomicrographs have been taken (1) on laboratory prepared samples and (2) by the in-situ technique. Can you tell the difference between the two?



(a)

400X



(b)

400X

**As-Cast Brazing Alloy**



(c)

400X



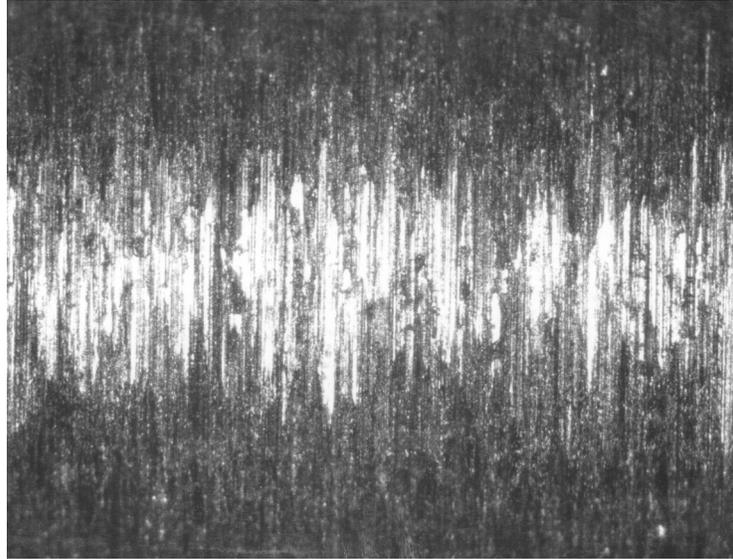
(d)

400X

**Quenched and Tempered SAE 8620 Steel**

**Photomicrographs (a) and (d) are from replicated surfaces.**

**In-situ metallography is ideal for round material where the curved surface can be replicated and the replica placed flat onto a glass slide to give a large, flat surface to be examined.**

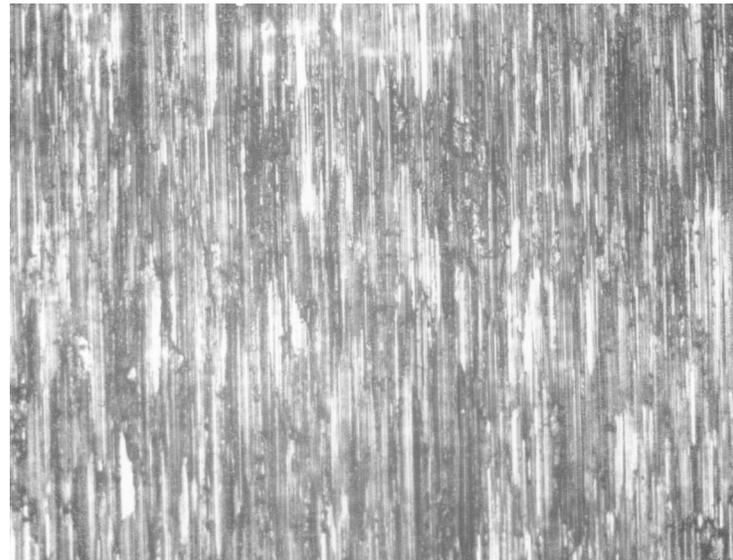


**As-Received**

**50X**

**Machining Grooves on 0.25 inch rod**

***Note limited viewing area***



**As-Received**

**50X**

**Replica of Machining Grooves**

***Note large viewing area***

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