Metallographic Preparation of HASTELLOY® and HAYNES® Alloys

High Performance Alloys
Technical Information
Metallographic Preparation Of HASTELLOY® AND HAYNES® Alloys

These preparation procedures and guidelines for etching have been examined carefully and are considered to be the best for producing satisfactory samples.

Note: Metallographic preparation involves use of abrasive saws and handling of acids. It is the responsibility of the individuals following these guidelines to develop safe practices and to use protective equipment in accordance with all appropriate local, municipal, state, and federal regulations covering safety and health.

I. SECTIONING

Cut the specimen to a convenient size using any of various types of silicon carbide cut-off blades. Deformation damage can be minimized by using thin cut-off wheels (1/32 inch [0.78 mm] thick as opposed to 1/16 inch [1.58 mm]). This is especially beneficial if the sectioning is to be done dry. Adequate water coolant is desired to reduce the amount of disturbed metal created, in part, from frictional heat during this phase of preparation. The original microstructure of a specimen may also be radically altered, (at least superficially, on the cut surface) due to metallurgical changes if an excessive amount of frictional heat is generated.

II. COARSE GRINDING

Use a 120 grit silicon carbide (SIC) wet-belt grinder and light contact pressure to obtain a plane surface free from deep grooves. In addition to producing a flat surface, this procedure removes burred edges or other mechanical abuses which may have occurred during sectioning.

III. MOUNTING

To assure flatness, and facilitate handling, it is recommended that specimens be mounted in phenolic, acrylic or cold-setting epoxy resins. Epoxy resins involve the blending of a liquid or powder resin in a suitable hardener to initiate an exothermic reaction to promote hardening and curing at room temperature. This usually requires an overnight operation. However, an advantage of epoxy is that the mount is semi-transparent and permits observation of all sides of the specimen during each phase of the preparation.

Compression molding techniques may be used with phenolic powders to produce the standard 1¼-inch (31.7 mm) diameter mounts. Phenolic mounts are convenient when time constraints do not permit an overnight cold-setting operation.

IV. FINE GRINDING

Rotating discs flushed with running water are recommended with successively finer grit papers of 220, 320, 400, and 600 grit SIC. (A light to medium amount of pressure is exerted on the specimen to minimize the depth of deformation). Best results are obtained on the 600 SIC paper by grinding the specimen twice. Specimens should be rotated 90 degrees after each step until the abrasive scratches from the preceding grit have been

removed. In each step, the grinding time should be increased to twice as long as that required to remove previous scratches. This ensures removal of disturbed metal from the previous step. Considerable care should be used in the fine grinding stage to prevent the formation of artifacts.

V. **ROUGH POLISHING**

The specimen should be hand washed and, perhaps, ultrasonically cleaned to ensure the complete removal of silicon carbide carry-over from the fine grinding stage. A pellon pan-W type cloth should be charged with 9-micron diamond paste, and water should be used as the lubricant. The specimen is moved counter to the direction of the rotating polishing wheel from the center to the outer periphery around the entire lapping surface. Extremely heavy pressure is used with diamond abrasive techniques to gain the maximum cutting rate. At the conclusion of this stage, the specimen should again be cleaned to remove any diamond polishing residue remaining in pin-holes, cracks, and cavities.

VI. **VIBRATORY POLISHING**

Semi-final and final polishing operations on a major portion of metallographic specimens can be completed on vibratory polishing units such as the Syntron units. A nylon polishing cloth using a slurry of 30 grams of Linde type "A" alumina polishing abrasive and 500 ml of distilled water are recommended for this operation. Additional weight in the form of a stainless steel cap must be placed on the specimen. The suggested weight to achieve a satisfactory polish in 30-60 minutes on a 1¼-inch (31.7 mm) diameter mount is 350 grams.

Samples should be cleaned with a cotton swab under running water to remove Type “A” alumina film, placed on a short nap micro-cloth with a slurry of 30 grams of Linde type "B" alumina abrasive and 500 ml of distilled water, and polished until a scratch-free surface is obtained. Again a 350-gram weight is used to augment polishing. Specimens usually require 25 to 30 minutes to produce a satisfactory final polish. The specimen can usually be polished an additional 10 to 15 minutes without producing harmful over polishing effects, but too much time may create relief on thin samples.

VII. **SURFACE PREPARATION**

The surface, prior to etching, should:

- be free from scratches, stains, and other imperfections which mar the surface;

- contain all non-metallic inclusions intact;

- not exhibit any appreciable relief effect between micro-constituents.

VIII. **ETCHING PROCEDURES**

The composition of the alloy to be etched determines the etchant which can be used. Structural components of an alloy are revealed during etching by a preferential attack or staining of the various constituents by the reagents. This is due to differences in the chemical composition of the phases and attending rates of solution. Immediately prior to etching, specimens should be lightly polished (Linde type "B" wheel) and swabbed with cotton under running water to remove any air formed oxide film, to reduce chances of

staining.

. Electrolytic Etching

Place the specimen immersed face up in the etching reagent. The cathode is placed approximately one inch from the specimen, and the anode is put in contact with the sample. During etching, the cathode is moved to assure a uniform action of the etching reagent on the specimen. The sample is then washed and repolished lightly, if needed, to remove any traces of disturbed metal on the surface, and then re-etched.

The following etchant is used for most HASTELLOY® and HAYNES® alloys, with the exception of HASTELLOY® B, B-2, B-3®, N, and W alloys (see Section VIII-B).

5 gm. oxalic acid mixed with

95 ml HCl (reagent grade)

Electrolytic - 6 volts DC

Carbon cathode

Stainless anode probe

1 to 5 seconds - depending on heat treated condition and size of sample

1. The sample must have a fresh polish. If the surface has been dry, even for a few seconds, give the sample 6 to 10 laps on final .05 micron alumina (Linde "B") cloth then place directly under running water and swab with a cotton pad.
   Important - Sample surface must be kept wet.
2. Put sample face up in etchant. With good overhead light to visually see sample surface: make contact at end or corner of sample with anode probe, dip carbon cathode into etchant watch to see any surface change – break contact.
3. **Important** - Before removing sample from etchant, agitate to remove any film on surface. Pull sample and put under running water. Rinse with Methanol, then place sample under hair dryer until it is thoroughly dry.
4. If etch is too light and needs to be heavier, do not take sample back to running water and then into etchant. Instead it must go back to the final cloth for 6 to 10 laps making sure that no part of surface dries - failure to do this can, and most likely will, result in staining. If the sample does stain do not try to remove stain on final cloth. Rather, go back to the papers - at least to the 400 and 600 grit; then 9 micron diamond and then to .05 alumina – and again, keeping sample surface wet, repeat as described before.

. Immersion Etching

Immersion etching techniques are usually used for the high molybdenum alloys, namely HASTELLOY® B, B-2, B-3®, N, and W alloys. The preferred etchant for this family of alloys is chrome-regia (1 part chromic acid to 3 parts reagent grade HCl). Stock chromic acid is made by mixing 300 grams chromic acid with 300 ml of hot water.

For immersion etching, it is equally important to work with a wet, freshly polished surface, (i.e., follow step VIII A1 above for electrolytic etching). The wet sample is then immersed face up into the chrome regia for 1 to 3 seconds, depending on heat treated condition and sample size. Then pull sample, put under running water, rinse with methanol, and blow dry. If etch is too light, follow procedure described in Step VIII A4 above.