TITANIUM SPECIMEN PREPARATION

A three-step polishing procedure has been developed and found to be quite successful for preparing titanium and titanium alloy specimens for metallographic examination.

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Titanium and its alloys are more difficult to prepare for metallographic examination than steels. Like all refractory metals, titanium and its alloys have much lower grinding and polishing rates than steels. For example, deformation twinning can be induced in alpha alloys by over-aggressive sectioning and grinding procedures, and elimination of smearing and scratches during polishing can be difficult.

In addition, it is safest to mount relatively pure titanium specimens in castable (“cold”) resins rather than hot compression mounting, due to the potential for altering the hydride content and morphology, especially in those taken from service in a hydrogen-containing environment. However, the heat of polymerization can become an issue if these resins are not handled carefully.

This article describes a three-step preparation procedure that effectively reveals the microstructure of titanium and its alloys while preventing these problems.

Early preparation methods

Early mechanical preparation methods tended to be rather long, involved procedures that nearly always incorporated an attack polishing solution in the last step or last two steps. The problems associated with achieving well-prepared surfaces have prompted considerable interest in electro-polishing procedures. However, the inherent hazards associated with some electrolytes have prompted interest in chemical polishing methods.

Preparation methods for titanium and its alloys continued to rely upon these older procedures into the 1970s and 1980s. Perhaps the first publication of a modern approach to preparing titanium was that of Springer and Ahmed in 1984. This was a three-step procedure, assuming that the planar grinding step can be done with 320-grit SiC paper, which may not always be possible. If the specimens are sectioned with a wafering blade or an abrasive blade of the proper bond strength, and they produce a smooth surface with minimal damage, then 320-grit SiC paper may be suitable. If a rougher surface with greater damage is produced, such as would result from a power hacksaw, then grinding must commence with a coarser grit paper to remove the damage in a reasonable time. Grinding and polishing rates of titanium are much lower than for many other metals and alloys.

Although titanium and its alloys can be readily sectioned with band saws, power hack saws, and similar machine shop tools, these devices cause a great deal of damage. Figure 1 demonstrates the substantial depth of damage caused by sectioning commercial purity (CP) titanium. This damage is difficult to remove in rough grinding, as the grinding rate is very low. Consequently, for perfect surfaces, section titanium and its alloys with only laboratory abrasive saws or precision saws with blades designed for metallography.

Strictly speaking, any mounting compound is suitable for titanium and its alloys. However, if specimens for applications where hydrogen can be picked up are to be mounted, a low-viscosity epoxy resin and a conductive mounting approach would minimize the exotherm during polymerization. If the heat involved in polymerization is substantial, titanium hydrides could be dissolved.

Specimens never placed in service are unlikely to contain hydrides, and more freedom of choice in mounting is possible. To minimize the heat of polymerization, wrap ordinary aluminum foil around a block of steel or copper (a heat sink). Then, glue a phenolic ring form (a cylinder) to the
Fig. 1 — Polished surface of alpha-Ti, ASTM F67, Grade 2, in the annealed (1038°C) condition showing (left edge) extreme surface damage due to band sawing (modified Week’s reagent, polarized light plus sensitive tint). The magnification bar is 100 μm in length.

Many more details of these procedures are available in Metallography: Principles and Practice, by George F. Vander Voort. Electrolytic and chemical polishing solutions for titanium and its alloys are also summarized in this book. It is available from ASM by calling Customer Service at 800/336-5152; customer.service@asminternational.org.

Fig. 2 — Appearance of titanium hydride at the inner diameter of a CP Ti tube that broke in service showing the greatest amount of TiH in (a) where a low-viscosity, slow curing epoxy was used with a conductive mounting approach to keep the heat of polymerization below 30°C. (b) ID of tube mounted in fast-curing EpoKwick resin with a polymeric mold. (c) ID of a tube mounted in a press at 150°C using EpoMet thermosetting resin. Magnification bars are 20 μm long in each image. The specimens were not etched.

Improved preparation method

A series of experiments was conducted to develop an improved method to prepare titanium and its alloys. Many surfaces were tried with the aim of producing a damage-free surface in CP titanium, so that good polarized light images could be made after the last step. The method developed works best when cutting damage is minimized.

Cutting is an aggressive process, and the vast majority of problems encountered in specimen preparation can be attributed to failure to remove the damage from sectioning. Therefore, introduce the least possible amount of damage in sectioning (which will also produce a good surface finish). Next, mount the specimen for ease in identification and for facilitation of edge retention.

• **Step 1** is grinding to remove the sectioning damage. Then, commence grinding with the finest possible SiC grit size: 240-grit is usually adequate and 320-grit SiC may be acceptable if you are careful in placing the specimens in the specimen holder so that the surfaces are flat and parallel to the SiC paper surface.

Coarse abrasives introduce more damage than fine abrasives. Automated grinding and polishing is highly recommended, not only because it yields superior results compared to hand polishing, but also because the final step includes an attack-polishing agent.

• **Step 2** is rough polishing. Use a psa (pressure-sensitive adhesive) backed UltraPol silk cloth and 9-μm diamond abrasive. Charge the cloth with diamond in paste form by setting the platen speed at about 100 rpm, placing the syringe tip at the center of the cloth, and slowly pulling the tip towards the cloth periphery. This deposits a concentric track of diamond on the cloth. Turn off the polisher and rub the paste into the cloth surface. Then, add some MetaDi Fluid (a petroleum-based lubricant) and commence polishing at 150 rpm, with a 6-lb (27-N) load with “contra” rotation. In this approach, the head rotates clockwise while the platen rotates counterclockwise. Every 30 seconds, squirt onto the cloth a small amount of 9-μm MetaDi Supreme diamond suspension to keep the cutting rate high. Continue polishing for ten minutes. After ten minutes, clean the specimens and the holder and change the surface.

• **Step 3** is final polishing. Use a psa-backed...
MicroCloth pad (synthetic suede) with the same load, rpm, time, and rotation direction, with MasterMet colloidal silica as the abrasive. Mix five parts colloidal silica with one part hydrogen peroxide (30% concentration – avoid skin contact) as the attack-polish agent.

Contra rotation works best when the head speed is under 100 rpm. The machines for these experiments have a 60-rpm head speed. This helps to keep the abrasives on the cloth surface. If the head and platen both rotate in the same direction (called “complementary” rotation), centrifugal force throws the abrasive and the lubricant off the surface as fast as you add them. It is important to keep the polishing surfaces uniformly covered with abrasive and lubricant to minimize smearing, pull-out, and deformation.

Because it is more difficult to clean specimens polished with colloidal silica than with alumina, and because 30% concentrated hydrogen peroxide is added to the abrasive, the cloth and specimens should be cleaned and washed before final polishing ends. Stop adding any abrasive at least 20 seconds before the ten-minute polishing cycle ends. With ten seconds remaining, direct the water jet onto the polishing cloth to clean both the cloth and the specimens. Colloidal silica is more difficult to remove from specimens than other abrasives.

CP titanium can be examined as-polished with crossed polarized light to observe the grain structure. Figure 3a shows an example of ASTM F67 Grade 2 CP Ti revealed (a) after the three-step method and (b) after 20 minutes of vibratory polishing after the three-step method. The specimens are in cross-polarized light and are not etched. The magnification bars are 100 μm long.

![Fig. 3 — Microstructure of as-hot-rolled ASTM F67 Grade 2 CP Ti revealed (a) after the three-step method and (b) after 20 minutes of vibratory polishing after the three-step method. The specimens are in cross-polarized light and are not etched. The magnification bars are 100 μm long.](image)

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