TECHNotes

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Efficient Sample Preparation of Commercially Pure Ti

Introduction

Titanium and its alloys are invaluable in aerospace, defense, and marine applications due to their high strength-to-density ratio and excellent corrosion resistance. Additionally, their biocompatibility makes them highly useful in biomedical applications. Titanium has the strength of steel alloys but only a fraction of the density.

However, when preparing metallographic samples, titanium proves to be more challenging than steel. This difficulty arises because titanium is ductile and readily damaged, with also a relatively low material removal or recovery rate, posing a challenge to sample preparation.

Sectioning

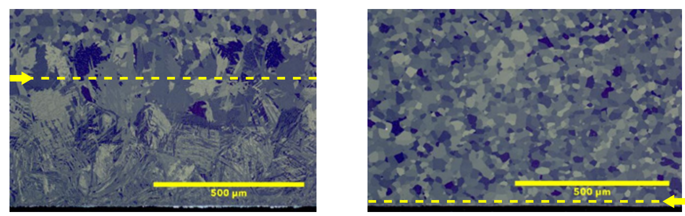
During the sample preparation process, sectioning is performed to remove a relatively small sample from a larger section to enable efficient preparation of the surface. This must be done in such a way to minimize thermal and mechanical damage to the sample surface. Sectioning-induced damage can alter the microstructure of the material, any such damage must be removed in subsequent grinding stages. Abrasion-resistant materials are particularly susceptible to burning during the cut as they readily wear the abrasive on cutting blades, reducing cutting efficiency and increasing friction at the cut surface.

The two most common sectioning techniques in metallurgy are precision and abrasive cutting. Precision cutting is most commonly performed using diamond abrasive bonded to the cutting surface of metal blades. Precision cutting is recommended for smaller components, perhaps up to ~30mm^2 cross-section, and is ideal for cutting a specific location with a high degree of precision (within microns of the area of interest, for example). Recommended precision blade type is the series 15 high-concentration diamond at 4000 to 5000 RPM and moderate feed rate in the range of 6 to 10mm/min.

Abrasive cutting is distinguished from precision cutting as it generally involves larger, higher power cutting machines using resin-bonded abrasive blades with alumina or silicon carbide abrasive. These blades wear appreciably during the cut as the abrasive is consumed and the bond holding the abrasive at the edge of the blade breaks free. Abrasive cutting is used to rapidly section large parts up to thousands of mm^2 cross-section. When using the technique to cut abrasion-resistant materials such as Titanium, correct blade choice is critical to minimizing deformation. Abrasive in blades for Titanium should be silicon carbide, blade bond should be relatively soft such that it wears readily and continuously exposes fresh abrasive for cutting. When cutting with manual abrasive cutters, pulsing the cut (applying and releasing pressure intermittently) is often employed to ensure blade bond wear and ensure free cutting. Automated cutting machines such as the ABL, ABXL and AB300 can provide pulse cutting automatically.

Whether choosing precision or abrasive cutting, coolant is critical to achieving good quality surface finish of the sample. Mix coolant in the recommended mix ratio (5% for Cool 3) and periodically check the ratio by refractometry (Brix factor 2.0 for Cool 3). Coolant provides lubricity that enhances capability of the cutting blade to cut cleanly, lessening potential for thermal damage. Coolants such as Cool 3 also contain corrosion inhibitors that enhance longevity of cutting machines.

After sectioning is complete it is likely there will be coolant residue and other contaminants on the sample surface that are best removed prior to mounting. Thorough cleaning is in order. This is best accomplished by rinsing in tap water, spraying with ethanol and drying with a lint-free towel.



(a) Deformation induced by incorrect cutting

(b) Correct cutting, showing virtually no deformation

As-polished cross section showing damage induced in titanium from improper cutting technique (cross-polarized light)

Mounting

Titanium can be mounted using cast (epoxy) media or compression (molding press) techniques. Cast mounting involves mixing two-part epoxy resin and pouring into a mold containing the sample. Curing time depends on the epoxy selected. Best options for Titanium include EpoHeat and EpoKwick, either of which provide excellent quality molds without shrink gaps in 90 minutes. Acrylic media is not recommended for Titanium due to poor qualities relative to epoxy. Acrylic media exhibits greater shrinking than epoxy and is less abrasion-resistant, leading to poor edge retention.

Compression mounting using resin with silica filler in a mounting press is typically the preferred mounting technique for Titanium. EpoMet G contains hard silica filler and an epoxy component that aids in adhesion of the resin to the sample edges. This eliminates the potential for shrink gaps in the mold. The silica filler provides abrasion resistance necessary to ensure the media and sample grind and polish at a similar rate, keeping the two materials in a common plane which is highly useful for microscopic analysis near the edge of the sample. This is what is meant by the term edge retention.

If both molding types are available compression mounting is typically preferred due to speed and high quality of the mold with respect to edge retention and shrinking. Compression mounting requires no mixing of mount media and molds can be made in as little as a few minutes. One exception to the preference for compression molding is the case where inspection of the sample for hydrides is of interest. The compression molding press operates at 150C, this is high enough temperature that hydrides in the sample may go into solution during the mounting process. When hydrides are of interest, cast mounting using low-exotherm epoxy such as EpoxiCure is in order.

Grinding and Polishing

Titanium is readily ground using silicon carbide grinding paper. One must bear in mind this paper wears quickly and must be changed every 2 minutes of grinding. This is because the sharp cutting facets of silicon carbide shear during grinding, leaving dull abrasive in resin that begins to burnish rather than grind. Fixed abrasive diamond discs are an alternative to silicon carbide in the event changing abrasive papers frequently is undesirable. Buehler's Color discs are the recommended diamond discs for Titanium.

Initial grinding is performed until cutting deformation is removed and a plane surface is achieved. After the first grinding step a flat surface with randomly oriented, similar size scratches should be visible. A single grinding step using 320-grit CarbiMet, or 35-micron Color diamond disc, is typically all that is needed prior to polishing.

Polishing is performed over three stages, the time for each stage is extended for titanium relative to processes for other materials to account for the abrasion-resistance of the sample. This ensures deformation from prior stages of preparation is removed prior to continuing to each successive step. The first two polishing steps after grinding are performed using MetaDi Supreme, a carefully formulated, finely graded polycrystalline diamond suspension that ensures the most efficient material removal and uniform resulting surface finish. We recommend 9-micron MetaDi Supreme on UltraPad, a relatively hard cloth surface that presents the diamond in an aggressive fashion to ensure grinding damage is removed while the sample is kept flat. Following this, 3-micron MetaDi Supreme is used with Trident, a woven cloth that is softer than UltraPad yet also serves to maintain flatness of the sample surface.

The third polishing step of MasterMet silica ensures an optically scratch-free surface finish. ChemoMet, a chemical-resistant, napless cloth is the best option here, particularly for automated procedures. In the case of Titanium and alloys it is common to add an oxidizer to MasterMet silica. One approach is to use 30% hydrogen peroxide in a ratio of 1 part peroxide to 5 parts silica. Care should be taken to use appropriate PPE as 30% hydrogen peroxide is a class 2 oxidizer and can cause burns on contact with skin. Another option is ammonium persulfate (class 1 oxidizer). Mix 10 grams ammonium persulfate into 100mL distilled water. Add one parts of this solution to 5 parts MasterMet silica.

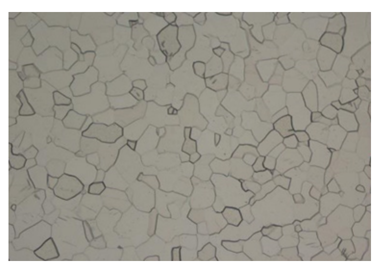
Cleaning between polishing steps is essential to ensure contamination of cloth surfaces do not occur. Specimen holders must be rinsed, and the polisher wiped clean after each step. Store polishing cloths in a clean storage cabinet. Rinse samples under the tap between steps, after the final step, rinse, spray with ethanol or IPA then dry using a dryer.

If polarized light capability with an optical microscope is available it can be used to observe grain contrast in alpha titanium in the as-polished condition. Polarized light response is a measure of preparation quality. In the event an optically scratch-free surface finish is not obtained at this point, it is likely that remaining deformation originates from the cutting or grinding stage. Further polishing on the final, silica step is not likely to remedy the circumstance, rather one should re-grind at 320-grit silicon carbide (or 35-micron diamond disc) then re-polish according to the procedure given.

Etching

Etch rate of titanium often depends on the duration between polishing and etching as titanium passivates readily in air. Extended rest time (30 to 60 minutes) after polishing prior to etching results in slower etch rates, some prefer this as it may enable greater control of the etch rate.

Perhaps the most common etch solution for Ti is Kroll's reagent, 100mL distilled water, 2mL HNO_3, 1mL HF, though there are variants of this etch using differing amounts of nitric and hydrofluoric acid. There is considerable interest in etch solutions that do not contain HF due to the associated hazards. Most etch solutions for Titanium contain HF, alternatives often contain chemicals that are similarly hazardous. Pre-mixed etch solutions are readily available, it is sound practice to use them thus avoiding the need to work with concentrated HF when mixing etch solutions. Titanium may be color-etched by immersion in a solution consisting of 5 grams ammonium bifluoride in 100mL distilled water. The interference film resulting from this etch creates color contrast based on grain orientation.



Contrasting of the titanium grade 2 sample with Kroll's etchant.

Table 1: 4-step method for polishing Titanium. Step 3 can be omitted for many alloys

	Surface	Abrasive	Lubricant/Extender	Force (Per Specimen)	Time (min:sec)	Platen Speed	Head Speed (rpm)	Relative Rotation
1	CarbiMet	320 Grit SiC	Water	5lbs [20N]***	Until Flat	250	60	>>
2	UltraPad	9 µm Metadi Supreme	Water	5lbs [20N]***	10:00	150	60	><
3	Trident	3µm Metadi Supreme	Metadi Fluid	5lbs [20N]***	5:00	150	60	>>
4	ChemoMet	0.06 µm MasterMet**	•	5lbs [20N]***	10:00	150	60	><

>> Comp, >< Contra • Rinse platen last 15-20 seconds *(optional, for difficult materials only)
** Attack polish For best results mix 5 parts Colloidal Silica to 1 part $(NH_4)_2S_2O_8$ solution.
*** Pressure recommendations are for 1.25"-diameter molded samples, adjust pressure to approximately 5psi to accommodate different sample sizes

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