TECHNotes

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Efficient Sample Preparation of Titanium Grade 2

Introduction

Titanium and its alloys' high strength to density ratio and good corrosion resistance make them invaluable in aerospace, defense, and marine applications. Good biocompatibility also makes it quite useful in biomedical applications. It is as strong as some steels but a fraction of steels density.

When preparing metallographic samples, one quickly learns, titanium is more difficult to prepare than steel as it ductile and readily damaged, but also has a relatively slow material removal or recovery rate, which poses a challenge to sample preparation.

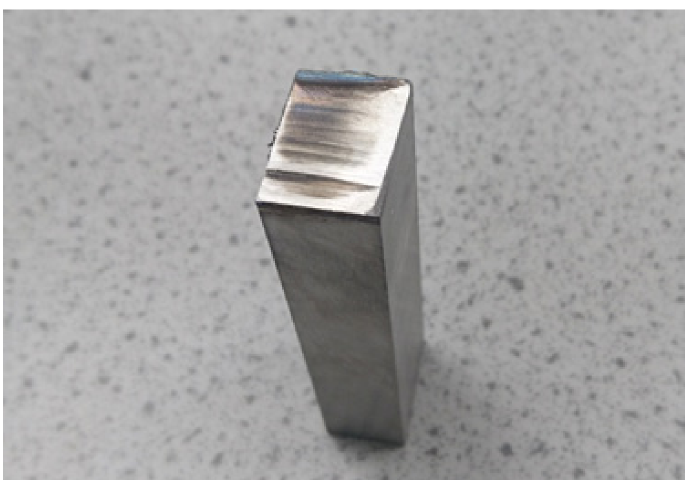
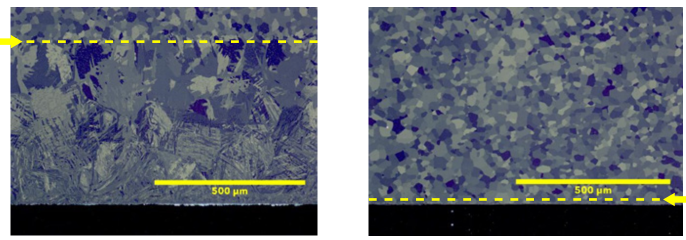


Figure 1. *Tarnishing of a titanium sample due to incorrect selection of the cutting disc and insufficient cooling during the cutting process.*

Sectioning

Sectioning titanium can be difficult as it is quite tough. Grade 2 can be especially difficult as it is more easily deformed. Commercially pure titanium is softer and ductile. Sectioning can very easily cause twinning and thermal damage if one is not careful. Therefore, great care must be taken during the cutting process. Proper blade selection and cooling is important. Abrasive blades with bonding strengths optimized for titanium and other refractory metal should be used. The binder of these blades is designed to break down at the right moment to release dull, used abrasives and expose sharp, fresh abrasive particles. This helps abrasive blades maintain optimum cutting efficiency and prevents blades from plowing into the sample with dull abrasive. Using a cutter with a robust cooling system helps prevent thermal damage. As an extra precaution, conservative loads and feed rates should be used. As a result, the microstructure is protected from coarse destruction (see Figure 2).



(a) Deformation induced by incorrect cutting

(b) Correct cutting, showing virtually no deformation

Figure 2. *As-polished cross section showing damage induced in titanium from improper cutting technique (cross-polarized light)*

Mounting

Selecting a chemically resistive mounting media with little or no shrinkage generally makes the preparation of titanium and titanium alloys easier. Whether using compression or castable mounting method epoxies are best. When using compression mount consider using a hard, mineral filled, epoxy like EpoMet. They tend to have very little to no shrinkage can resist chemical attack from etchants.

If the hydride content needed to be measured it is best to mount specimens in a low exotherm castable epoxy like EpoxiCure 2. Castable epoxies like EpoxiCure 2 are chemically resistive, adhere well to specimen edges and have low shrinkage providing great edge retention properties.

Before mounting it is important to make sure specimens are clean and dry; not doing so could cause shrinkage gaps between the specimen and the mounting media. Shrinkage gaps are sites that can collect and disperse contaminates during grinding and polishing. Removing contamination scratches from titanium is time consuming.

Grinding and Polishing

Grinding

Titanium does not abrade very easily. Material removal rates are low across all stages of preparation. Titanium, especially grade 2 can be very easily deformed during grinding. Using aggressive grinding to shorten preparation time can cause smear, gross deformation and twinning. Initial grinding steps often use silicon carbide (SiC) discs with moderate pressure. SiC is fairly hard and contains sharp abrasive particles that cleave while grinding creating new cutting edges as the disc is used. This makes the initial material removal rate quite high.

Use each disc for only 1-2 minutes. Grinding with degraded SiC discs may create excessive damage. If properly sectioned one can use 320-Grit SiC for the initial grinding step. It is also important to ensure adequate water cooling during grinding, since titanium is prone to local overheating due to its thermal conductivity (local structural change).

Polishing

After the initial grinding step use 9-micron polycrystalline diamond suspension to recover grinding damage on a hard woven cloth. Hard woven cloths like UltraPad tend to have higher material removal rates and help maintain flatness. The polycrystalline diamonds like the ones used in MetaDi Supreme have an increased number of cutting edges. This allow the diamonds to remove damage more quickly with minimal subsurface deformation and leaves a better surface finish.

For many titanium alloys such as Ti-6Al-4V, one can move directly to a final polish after the 9-micron stage. This 3-step method is very effective for most titanium alloys. More sensitive alloys such as Grade 2 and commercial purity titanium may benefit from an added 3-micron stage. The 4-step method can increase clarity of grain orientation of more sensitive titanium alloys.

The final polishing stage, like other steps in titanium preparation, takes more time than with most other materials because of the low material removal rate. We highly recommend polishing using MasterMet colloidal silica mixed with a 10% $(NH_4)_2S_2O_8$ solution on a chemically resistant, no-nap polishing pad such as ChemoMet. For best results mix 5 parts Colloidal Silica to 1 part $(NH_4)_2S_2O_8$ solution.

As with many non-cubic materials, it is possible to see microstructural details in titanium using cross polarized light on a light optical microscope, with no etching. This is a very quick and easy assessment of the quality of polish. A good response to cross-polarized light (more contrast, and sharper detail) indicates low surface deformation. A bad response to cross-polarized light (poor contrast and lack of sharp detail) indicates higher surface deformation and poor sample preparation.

Etching

To illustrate the grain structure, titanium can be contrasted well with standard etchants such as Kroll's etchant (see Figure 4). If relatively long polishing times occur in the final polishing, it is recommended to let the sample rest for at least half an hour in air. Due to the semi-reconstituted passivation layer, the material reacts less intensively with the etchant. Without the passivation layer, the samples are usually over-etched quite quickly ($t \leq 1$ sec).

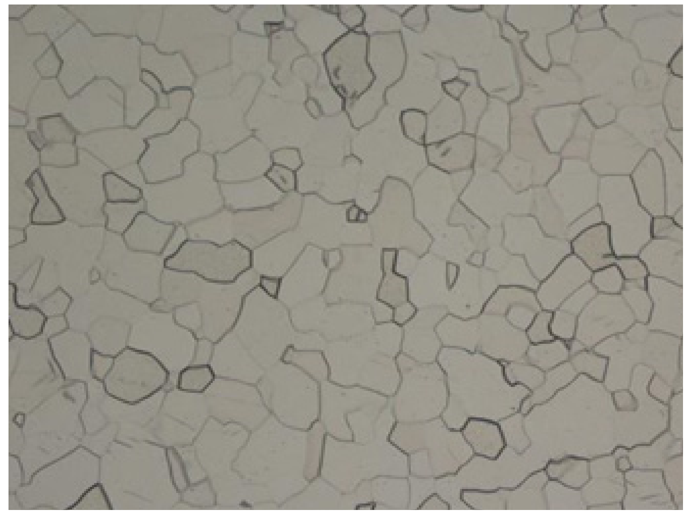


***Figure 4**. Contrasting of the titanium grade 2 sample with Kroll's etchant.*

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	4lbs	Until Flat	250	60	>>
2	UltraPad	9 µm Metadi Supreme	Water	4lbs	10:00	150	60	><
3	Trident	3µm Metadi Supreme	Metadi Fluid	4lbs	5:00	150	60	>>
4	ChemoMet	0.06 µm MasterMet**	•	4lbs	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 (NH4)2S2O8 solution.

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