Metallographic Preparation of Titanium and Its Alloys

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ANNOTATION
Experiments were conducted using three-step preparation procedures for titanium and its alloys. For CP titanium and alpha-titanium alloys, use of an attack polishing agent in the third step was required to obtain good results. The experiments defined optimum surfaces for each step and operating conditions. Two-phase alloy specimens are significantly easier to prepare than single phase (alpha) specimens.

Key words: metallography, titanium, grinding, polishing, microstructure.

1. INTRODUCTION

Titanium and its alloys have become quite important commercially over the past fifty years due to their low density, good strength-to-weight ratio, excellent corrosion resistance and good mechanical properties. On the negative side, the alloys are expensive to produce.

Titanium, like iron, is allotropic and this produces many heat treatment similarities with steels. Moreover, the influences of alloying elements are assessed in like manner regarding their ability to stabilize either the low temperature phase, alpha, or the high temperature phase, beta. Like steels, Ti and its alloys are generally characterized by their stable room temperature phases - alpha alloys, alpha-beta alloys and beta alloys, but with two additional categories: near alpha and near beta.

Titanium and its alloys are more difficult to prepare for metallographic examination than steels. They have much lower grinding and polishing rates. Deformation twinning can be induced in alpha alloys by overly aggressive sectioning and grinding procedures. It is safest to mount relatively pure Ti specimens in castable ("cold") resins rather than using hot compression mounting due to the potential for altering the hydride content and morphology. Elimination of smearing and scratches can be quite difficult.

Early mechanical preparation procedures [1-5] tended to be rather long, involved processes nearly always incorporating an attack polishing solution in the last step or last two steps. Some of the more commonly used attack polishing solutions are summarized in [6]. The problem of obtaining well prepared surfaces has prompted considerable interest in electropolishing procedures [3-5, 7, 8]. The inherent danger of some of these electrolytes has fostered interest in chemical polishing procedures [9]. Electrolytic and chemical polishing solutions for Ti and Ti alloys are also summarized in [6].

Mechanical polishing methods for titanium and its alloys continued to relie upon these older procedures into the 1970's [10] and 1980's [11]. Perhaps the first publication of a modern approach to preparing titanium was that of Springer and Ahmed [12] in 1984. This was a three-step procedure, assuming that the planar grinding step can be performed with 320-grit SiC paper, which may not always be possible. If the specimens are sectioned using a wafering blade or an abrasive blade of the proper bond strength, which produce a smooth surface with minimal damage, then 320-grit SiC paper may be used. If a rougher surface with greater damage is produced, such as would result from use of a power hacksaw, then grinding must commence with a coarser grit paper in order to remove the damage in a reasonable time.

The procedure developed by Springer and Ahmed was:

1. Wet grind with 320-grit SiC paper for 2-3 minutes to obtain a flat surface free of cutting damage.
2. Rough polish with 9-µm Metadi® diamond paste on a perforated Texmet® cloth for 10-15 minutes with distilled water as lubricant.
3. Final polish with Mastermet® colloidal silica suspension on either a Microcloth® or a Mastertex® cloth for 10-15 minutes.

G. Müller, as reported by Leistner [13], also developed a three-step procedure for titanium. Again, it will take three steps only if sectioning produces a smooth surface with minimal damage. His procedure was:

1. Wet grind with P500 SiC paper, 300 rpm, 100N load (6 specimens), until all surfaces are co-planar.
2. Wet grind with P1200 SiC paper, 300 rpm, 100N load, for 30 seconds.
3. Polish with colloidal silica suspension containing an attach polishing agent on a synthetic napless cloth, 150 rpm, for: 10min. at 200N, 2 min. at 100N, 2 min. at 50N, and 1 min. without any load.

The polishing suspension consisted of 260mL of colloidal silica, 40mL H_2O_2 (30%), 1mL HNO_3 and 0.5mL HF.

2. PREPARATION EXPERIMENTS

2.1 METHOD OF SPRINGER AND AHMED

The initial preparation experiments centered on evaluating, expanding and updating the method of Springer and Ahmed [12], S&A. As that specific type of perforated Texmet cloth is no longer available, other surfaces had to be substituted and evaluated. Also, the published method did not specify the applied load, platen rpm or rotational directions.

The first experiment with the S&A method used:

1. Grind with 320-grit SiC paper, water cooled, 240 rpm, complementary* rotation, 27N (6 lbs.) per specimen load, until plane.
2. Rough polish with 9-µm Metadi diamond on an Ultra-Pad™ cloth, 120 rpm, 27N/specimen, Metadi Fluid, contra** rotation, 10 min.
3. Final polish with Mastermet 2 colloidal silica on a Microcloth pad, 120 rpm, contra rotation, 10 minutes.

Results for titanium alloys were generally acceptable, although some scratches were observed; but, they were inadequate for CP titanium, as shown in Figure 1.

Figure 1a Figure 1b

Figure 1. Results of preparation using the method of Springer and Ahmed for a)Ti-6Al-2Sn-4Zr-2Mo (977°C-1h, fan cool, 593°C-8h, air cool, 500x) and b)CP Ti, ASTM F67, Grade 2 (1040°C anneal, 200x). Both etched with 0.5% HF.

Addition of a 3-µm diamond step between steps 2 and 3 did not improve the alpha alloy and the CP Ti microstructures. Whether the cloth used was hard and napless, or softer and napped, the results were the same for the CP Ti specimens. The use of an Ultra Pol™ silk cloth in step 2 resulted in less scratches after this step and yielded acceptable results for the CP Ti specimens. A Texmet 1000 pad was also tried for step 2 and found to produce nearly as good results as with the Ultra Pol cloth. Figure 2 shows the results of three successive experiments where the surface of the same specimen was ground with 320-grit SiC (step 1) and then rough polished with 9-µm diamond (step 2) on Ultra-Pad, Ultra-Pol and Texmet 1000 cloths. Based on these experiments, Ultra-Pol was selected as the preferred cloth for step 2 and Texmet 1000 pads were considered to be nearly equivalent.

As another example of these experiments, Figure 3 shows the microstructure of two specimens using an Ultra-Pol cloth for step 2 and a Microcloth Supreme cloth for step 3. Etching with aqueous 0.5% HF produced colored alpha grains on CP Ti (Figure 3b). Higher magnification examination revealed some roughness in the alpha grains. Using a Microcloth pad for step 3 produced excellent results. If Kroll's reagent is used, instead of 0.5% HF, a flat grain boundary etch for the CP Ti specimens is obtained rather than a color grain contrast etch, but only when the preparation is near perfect. Figure 4 shows an α-β alloy, an alpha alloy and a CP Ti specimen. The two phase alloys were well prepared but the alpha alloy and the CP Ti specimens were inadequate.

* In complementary rotation, the head and platen rotate in the same direction.

** In contra rotation, the head and platen rotate in opposite directions.

Figure 2a

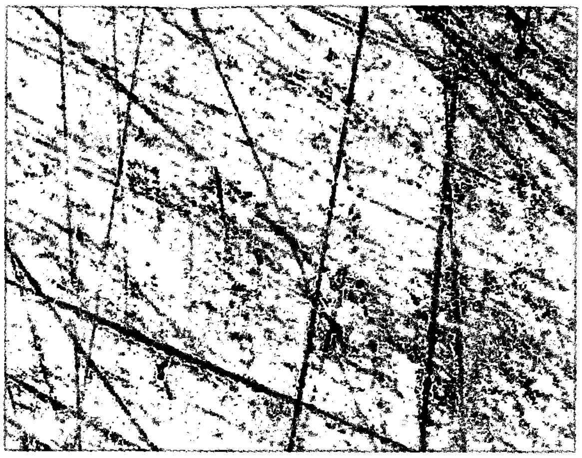

Figure 2b

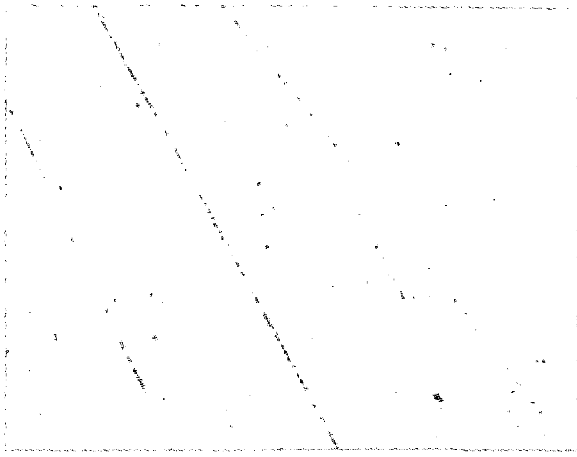

Figure 2c

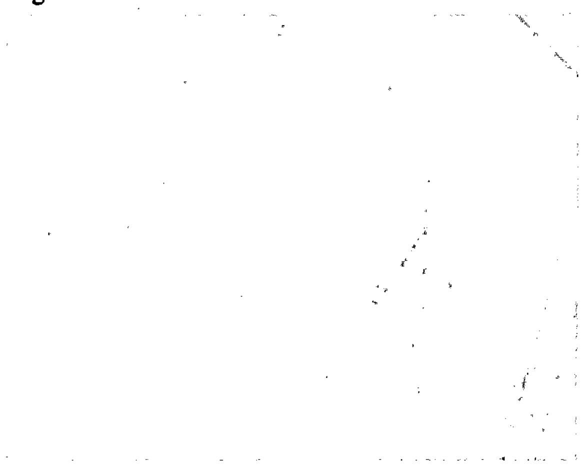

Figure 2. Scratch pattern after steps 1and 2 on as-cast Ti-6Al-2Sn-4Zr-2Mo after using for step 2: a) Ultra-Pad cloth, b) Ultra-Pol cloth, and c) Texmet 1000 pad (200x).

Figure 3a

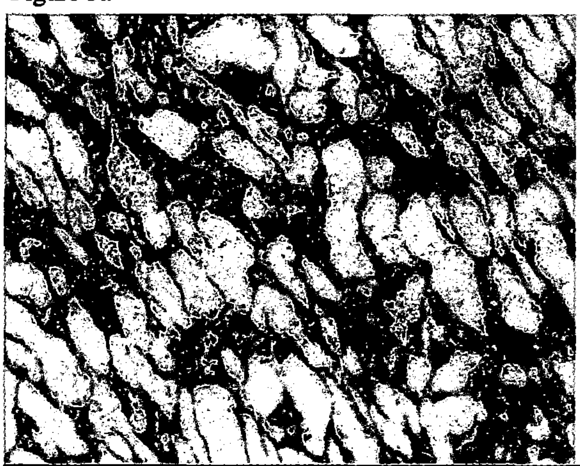

Figure 3b

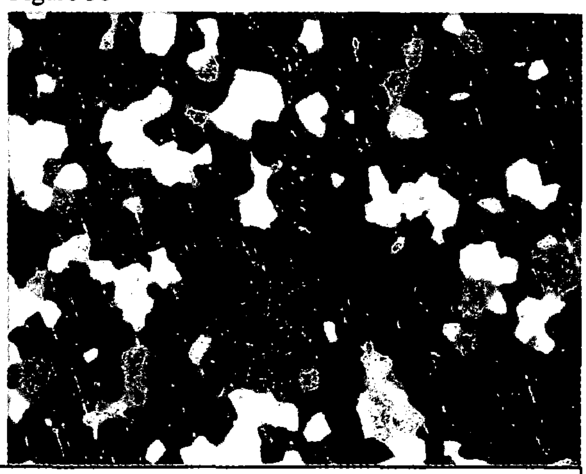

Figure 3. Results of preparation using an Ultra-Pol cloth (step 2) and Microcloth Supreme pad (step 3) for: a) as-rolled Ti-8Al-1Mo-1V (500x) and b)CP Ti, ASTM F67, Grade 2 (1040C anneal, 100x). Both etched with 0.5% HF.

Figure 4a

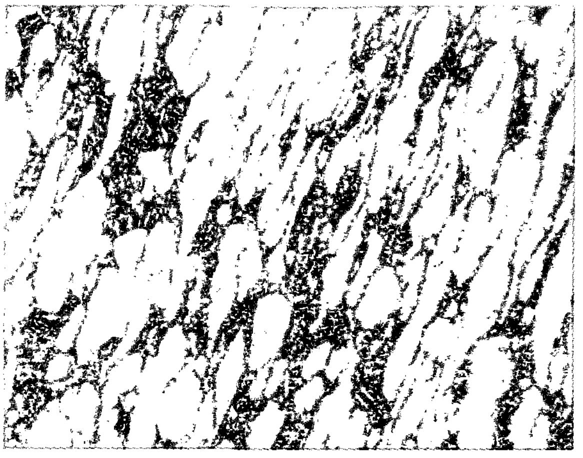

Figure 4b

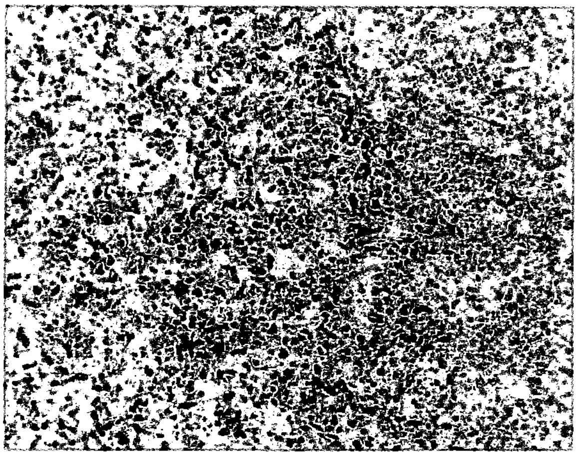

Figure 4c

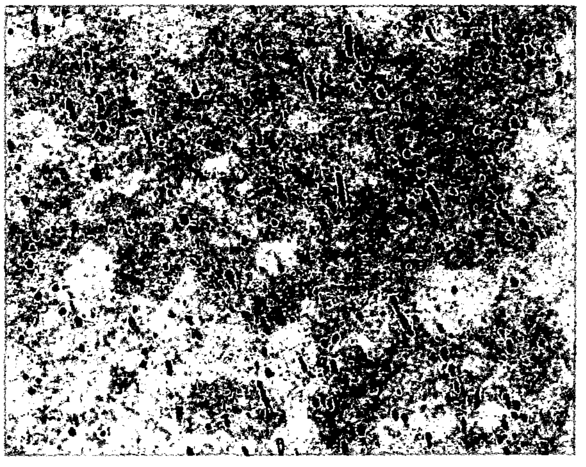

Figure 4. Same preparation as in Figure 3, but etched with Kroll's reagent: a) as-rolled Ti-8Al-1Mo-1V (500x), b) annealed Ti-5Al-2.5Sn (200x), and c)CP Ti, ASTM F67, Grade 2 (1040C anneal, 200x).

2.2 ADDITION OF ATTACK POLISHING AGENT

If an attack polishing solution is added to the colloidal silica abrasive for step 3, the structures are all revealed more clearly, as Figure 5 shows. In this experiment, step 2 used an Ultra-Pol cloth, step 3 used a Microcloth pad and the step 3 abrasive contained: 50mL Mastermet 2 colloidal silica, 10mL H_2O_2 (30%) and 5mL Kroll's reagent. Figure 5b shows a well developed alpha grain structure in an annealed CP Ti specimen with little cold work in the alpha grains.

Figure 5a

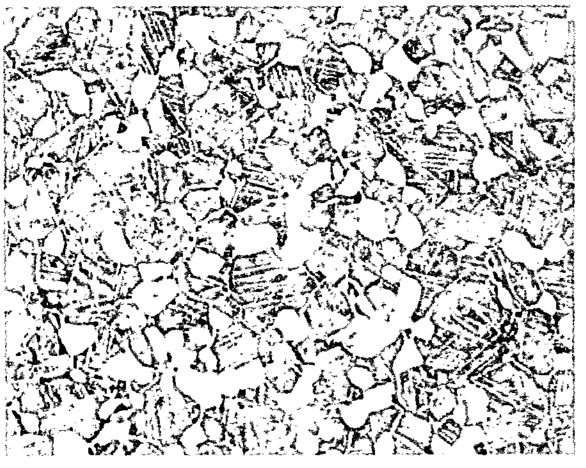

Figure 5b

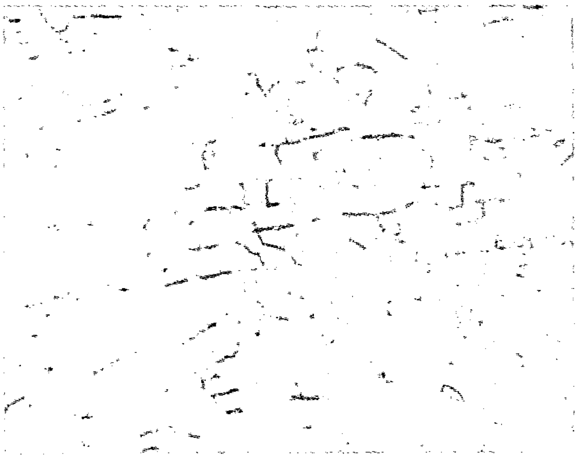

Figure 5. Results of adding an attack polishing agent to the abrasive in step 3: for a)Ti-6Al-2Sn-4Zr-2Mo (977°C-1h, fan cool, 593°C-8h, air cool, 500x) and b)CP Ti, ASTM F67, Grade 4 (704°C anneal, 200x). Both etched with Kroll's reagent

This experiment, and numerous subsequent experiments, revealed that an attack polishing agent must be used in step 3 to get satisfactory images of alpha alloys and CP Ti specimens. For step 3, napped cloths, such as Microcloth, Microcloth Supreme and Mastertex, yield better results for CP Ti than harder, napless cloths like a Chemomet® I cloth. For edge retention and cavity wall retention (cast alloys), the Chemomet cloth will yield the best results and holds up best with attack polishes. All of these cloths work well for two-phase Ti alloys, such as the popular Ti-6Al-4Valloy.

If a Texmet 1000 pad is used for step 2, a Microcloth pad is used for step 3, and attack polishing is employed, results are still excellent, as shown in Figure 6.

Figure 6a

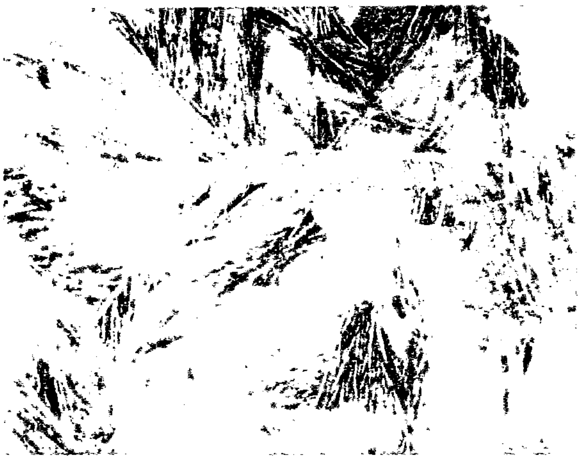

Figure 6b

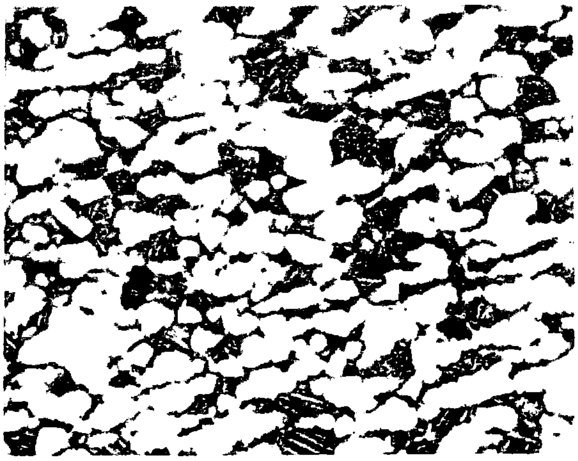

Figure 6c

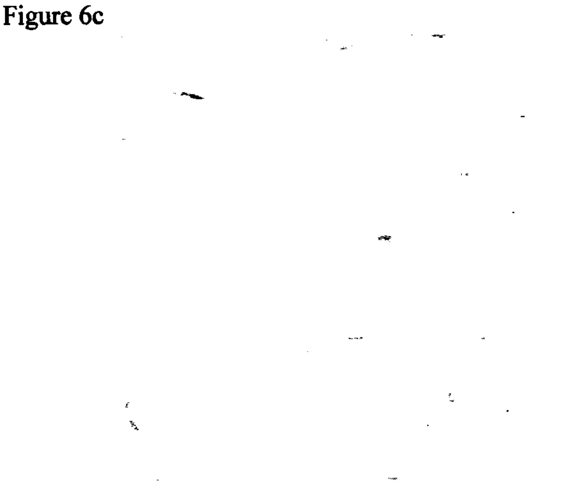

Figure 6. Results using a Texmet 1000 pad (step 2) and attack polishing on a Microcloth pad (step 3): a) as-cast Ti-6Al-2Sn-4Zr-2Mo (200x), b) Ti-6Al-2Sn-4Zr-2Mo (950°C-1h, fan cool, 593°C-8h, air cool, 500x) and c)CP Ti, ASTM F67, Grade 2 (as rolled, 200x).

Figure 7

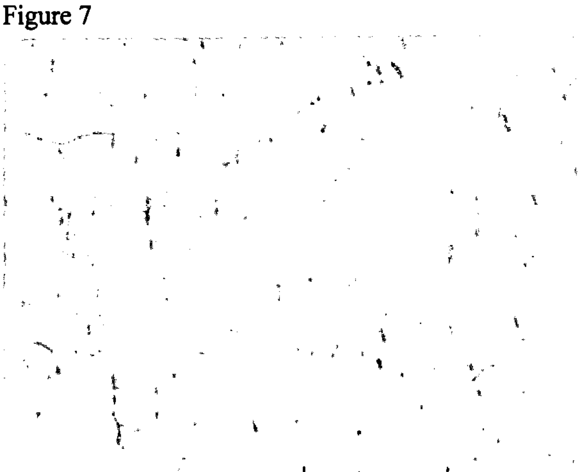

Figure 7. Best results on CP Ti (ASTM F67, Grade 2, 1040°C anneal, 200x, Kroll's reagent) using the procedure of Müller.

2.3 METHOD OF MÜLLER

The three-step procedure of G. Müller [13] was also tried. The results were quite comparable although there was a bit more cold work visible in the alpha grains of the CP Ti specimens. Figure 7 shows an example of one of the best CP Ti structures obtained with this procedure.

2.3 OTHER VARIATIONS

Two final three-step preparation procedures were tried. First, 240-grit SiC paper was used for step 1 rather than 320-grit paper. Most of the alpha-beta alloy specimens were prepared adequately, although some exhibited excessive cold work in the alpha phase, as in Ti-6Al-4V containing equiaxed alpha. The alpha alloy and alpha CP Ti specimen microstructures exhibited substantial cold work. The other experiment involved substitutions of 0.05-µm gamma alumina for colloidal silica in step 3 (using the same attack polishing additive). Results were generally acceptable, especially for the two-phase alloys, but the alpha phase in CP Ti specimens contained substantial cold work, Figure 8.

Figure 8

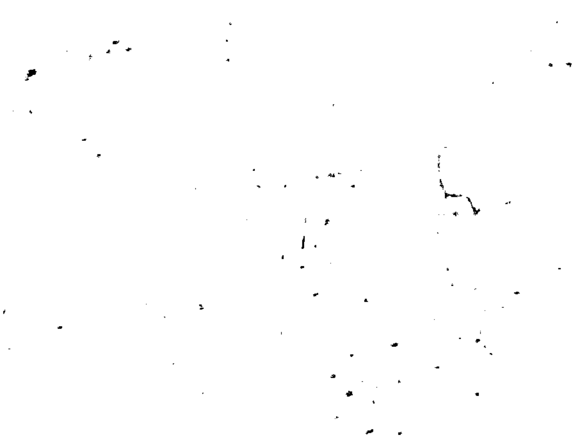

Figure 8. Results using gamma alumina, with an attack polishing agent in step 3 for CP Ti (ASTM F67, Grade 2, 1040°C anneal, 200x, Kroll's reagent).

A few variants of the attack polishing solution were tried. Leonhardt [14] uses a mixture of: 150mL colloidal silica, 150mL water, 30mL H_2O_2 (30%), 1-5mL HF and 1-5mL HNO_3. Results with this attack polishing additive to the abrasive were equivalent to the one used. Buchheit [6] added 5mL of a 20% aqueous CrO_3 solution to 30mL of an alumina slurry. To try this, but using collidol silica instead, 10mL of the 20% CrO_3 solution was added to 75mL of colloidal silica. This also produced excellent results. In using these attack polishing solutions, care must be taken in handling, mixing and using these additives as they contain very strong oxidizers and acids. Avoid physical contact with the ingredients and the prepared attack polishing abrasives.

3. CONCLUSIONS

A three-step procedure can be used quite successfully to prepare titanium and titanium alloys. Use of an attack polish additive in step 3 is required to obtain good results with CP titanium and alpha Ti alloys. Most two-phase Ti alloys can be satisfactorily prepared without using an attack polishing additive, although results were better when it was used. Optimal and alternative surfaces and operating conditions were defined, see Table 1.

Table 1. Three-Step Preparation Procedure for Titanium and Its Alloys.

1. Wet grind with 320-grit SiC paper, 27N (6 lb.) load per specimen, complementary rotation, 240 rpm, 1 minute, or until surfaces are co-planar.
2. Rough polish with 9-µm Metadi paste on an Ultra-Pol silk cloth, 27N per specimen, 120 rpm, contra rotation, Metadi Fluid for lubricant, 10 minutes.
3. Final polish with Mastermet 2 colloidal silica on a Microcloth pad, 27N per specimen, 120 rpm, contra rotation, 10 minutes.

Note:
Complementary - head rotates in the same direction as the platen.
Contra – head rotates in the direction opposite that of the platen.
For alpha titanium alloys and for pure titanium, add an attack polishing agent to the colloidal silica.

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