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Title: NEW METALLOGRAPHIC PREPARATION TECHNIQUES FOR TANTALUM AND TANTALUM ALLOYS

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New Metallographic Preparation Techniques for Tantalum and Tantalum Alloys

Ann M. Kelly, Sherri R. Bingert, Robert D. Reiswig

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Abstract

Two new metallographic techniques have been developed for tantalum and its alloys. The first is a chemical polishing method that can even be used on specimens immediately after grinding on silicon carbide papers. The second is an etching technique that even delineates low-angle grain boundaries, making it particularly suitable for quantitative grain size measurements. It has also been determined that these are suitable for the preparation of a surprisingly large variety of other metals and alloys, including, titanium, tungsten, Ti-6Al-4V, molybdenum, a Zr-Ti-Cu-Ni alloy, a Ti-Ta-Sc alloy, Fansteel 85, and a Hf-Zr alloy to name a few.

In certain applications, it is becoming increasingly important to control processing parameters for tantalum and tantalum alloys. Grain size is one measure of process consistency. Previously available metallographic techniques did not always delineate grain boundaries reliably enough for grain size determination, particularly in the case of low-angle grain boundaries. Other drawbacks associated with the earlier preparation procedures included lengthy processing times, and the development of pits during polishing and etching. These problems were more apparent in, but not limited to, porous powder metallurgy products. A study was therefore undertaken to develop preparation methods that could be more readily used for measurements of grain size, as well as improved surfaces for imaging in bright field and differential interference contrast. Combinations of abrasives, cloths, chemical polishes, and etchants were evaluated, resulting in a technique that produced superior results in a much shorter time.

Initial Sample Preparation

The samples were mounted in a low-viscosity epoxy resin by first evacuating the samples and resin separately, using a mechanical vacuum pump, then pouring the resin, while under vacuum into mold cups containing the samples. After venting the system to atmospheric pressure, the mold cups were placed in an autoclave to cure at room temperature in a dry nitrogen atmosphere of 600 to 1000 psi for a minimum of 5 hours. The solidified mounts were then given a post-cure at 60° C to further harden the resin. This procedure ensures excellent penetration of the resin into all surface-accessible porosity, making it easy to distinguish between porosity and pullout in relatively porous specimens. The samples were then wet ground through 1200 grit SiC grinding papers.

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Original Mechanical Polishing Method

The initial preparation was followed by our original polishing method, which consisted of polishing on Perforated Texmet(1), using a 3μm aqueous diamond suspension with a 50/50 H_2O/propylene glycol lubricant for approximately 6 - 8 hours. Final polishing was done on Microcloth(1) with a 1μm diamond paste abrasive and a 50/50 H_2O/propylene glycol lubricant for approximately 2 hours. Figure 1 shows examples of various tantalum materials when prepared in this manner. As shown, the use of interference contrast reveals only slight polish relief at the grain boundaries in the mechanically polished condition.

Original Etching Technique

Following the mechanical polishing, these samples were swab etched with a solution consisting of :

15 ml lactic acid

5 ml HNO_3

5 ml HF

for approximately 20 - 30 seconds. Figure 2 illustrates the results obtained by using this etchant, ASTM etchant #178 (2), which is recommended for tantalum. Although this method produced satisfactory results for general evaluation of microstructure, it was a very time consuming process, low angle grain boundaries were not clearly defined, and severe etch pits developed in some wrought tantalum alloys and powder metallurgy specimens. This was especially apparent for the CIP'ed (cold isostatically pressed) material. To correct these deficiencies it was necessary to develop a preparation sequence which would produce satisfactory results in a minimal time frame.

Improved Preparation Technique

Efforts to minimize polishing time included the following steps. Rough polishing was performed using Texmet(1) with a 15μm diamond abrasive suspension for approximately 10-15 minutes. This was followed by an intermediate polish using Texmet with a 3μm diamond paste and a 50/50 water/propylene glycol lubricant. Polishing progress was checked at 20 minute intervals during the 3 hours of polishing time. The final polish was done on Microcloth, using 1μm diamond abrasive with a 50/50 H_2O/propylene glycol lubricant for approximately 20 minutes.

Optical examination of the specimens during the polishing sequence revealed severe pitting which developed during the intermediate polishing step. This pitting was correctable only with prolonged intermediate polishing, but reappeared to a lesser degree during the final polish. After several unsuccessful attempts to eliminate the pitting problem using various combinations of polishing cloths and lubricants, it appeared that the source of the problem was the diamond abrasive, the only constant in this polishing process. It was later determined that rough polishing with a 15μm diamond abrasive suspension decreased polishing time and had no adverse affect on specimen preparation. However, subsequent polishing with finer diamond abrasives caused pitting. The final solution to this pitting problem was to use alumina slurries in place of the diamond paste for the intermediate and final polishing steps.

A chemical polish was developed, which not only eliminated the scratches, but also produced sharper, better defined grain boundaries. The composition of this polish is:

25 ml lactic acid

15 ml HNO_3

5 ml HF.

It is applied by swabbing vigorously with heavy pressure for 90 to 120 seconds. Porous samples, such as powder metallurgy products, require less chemical polishing time, typically 30-45 seconds. Examples shown in Figure 3 display the effect of the new mechanical/chemical polishing technique on various tantalum base materials.

To further enhance the grain boundaries for grain size determination, an etchant was needed that would define structure without causing additional pitting or an increased pore size. This was accomplished by experimenting with

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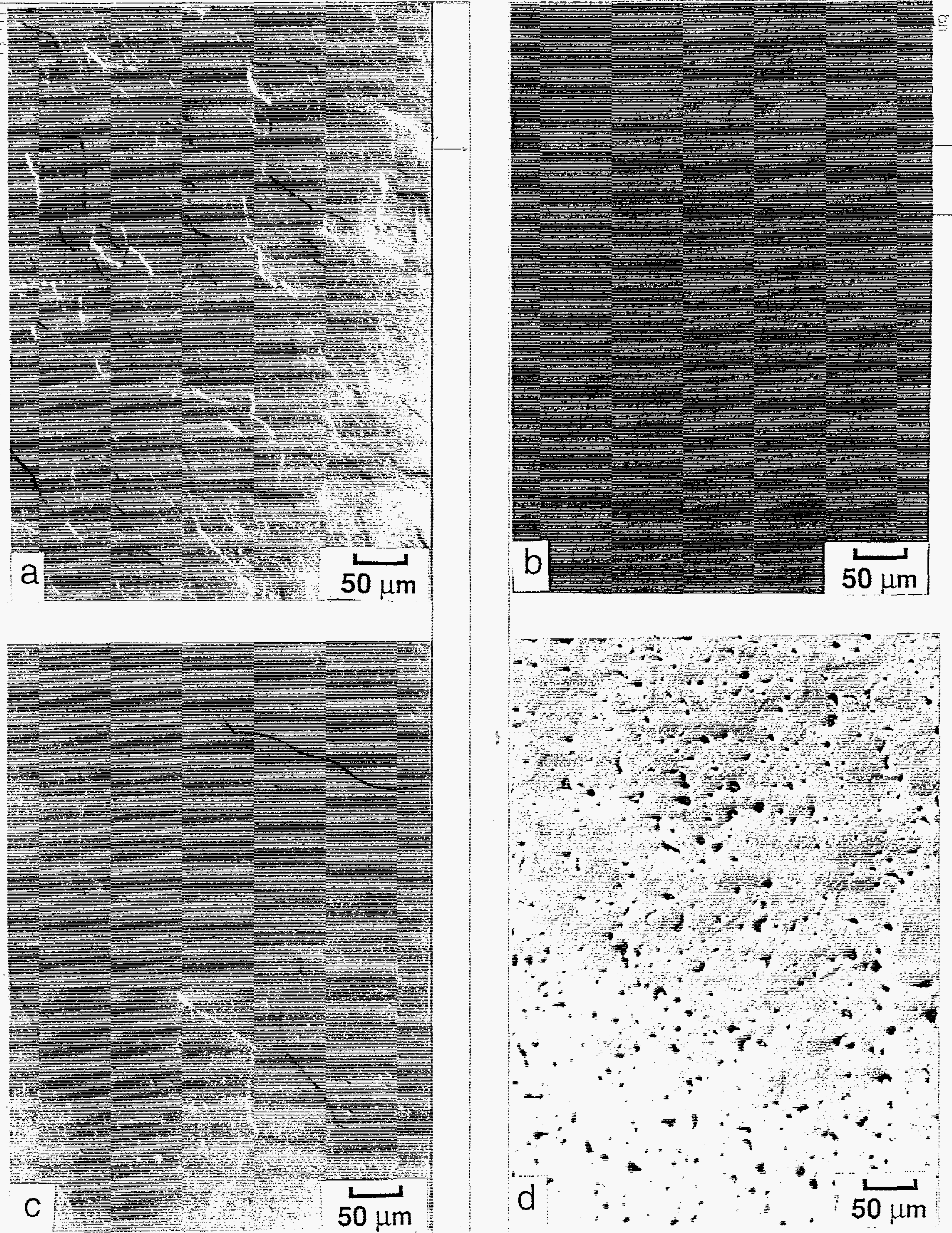

Fig. 1. Samples of (a) wrought Ta, (b) Ta-10W alloy, (c) low oxygen content P/M Ta HIP'ed at 1600°C, and (d) high oxygen content P/M Ta CIP'ed and sintered at 2400°C in the old mechanical polished condition. Light micrographs, interference contrast.

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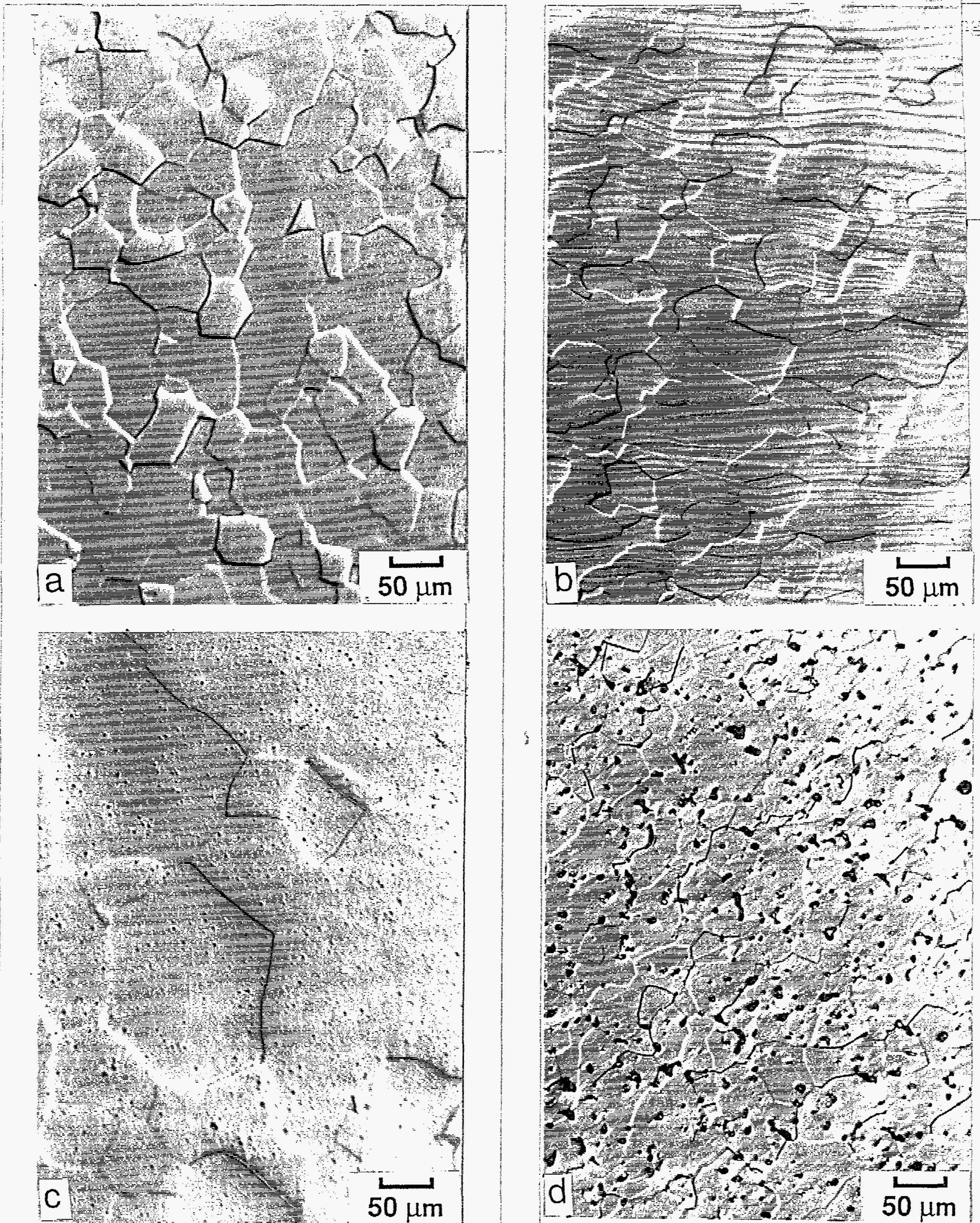

Fig. 2. Examples of (a) wrought Ta, (b) Ta-10W alloy, (c) low oxygen content P/M Ta HIP'ed at 1600°C, and (d) high oxygen content P/M Ta CIP'ed and sintered at 2400°C using a standard tantalum etchant (ASTM E407-93 #178). Light micrographs, interference contrast.

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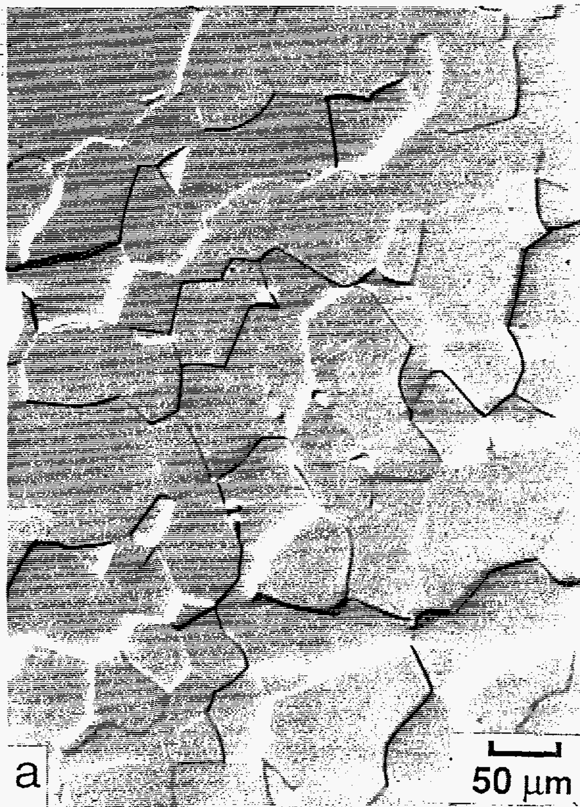

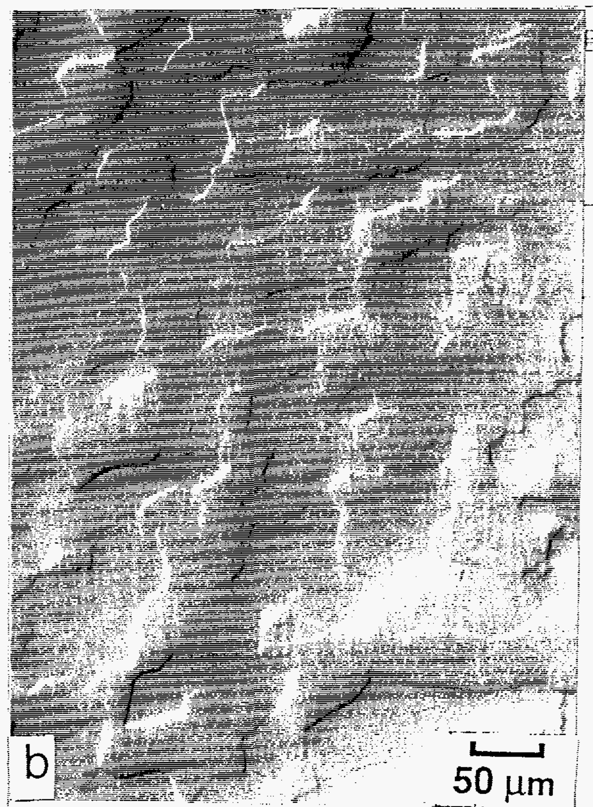

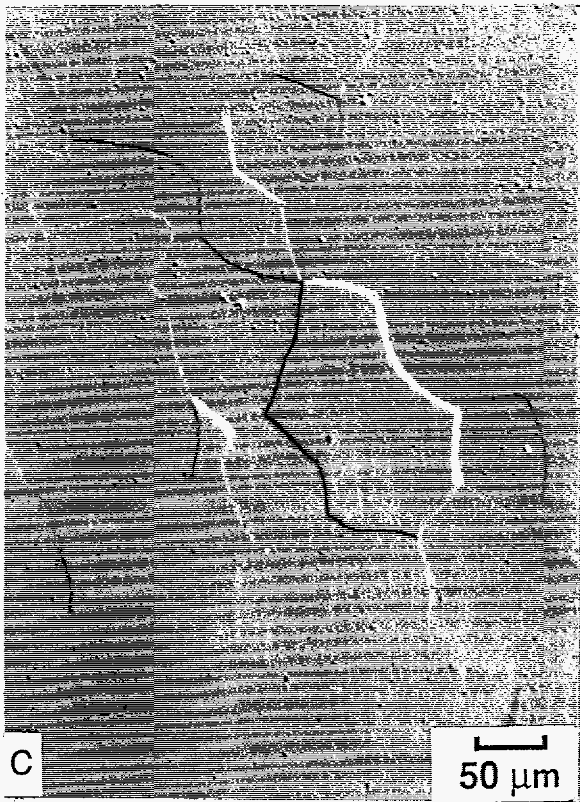

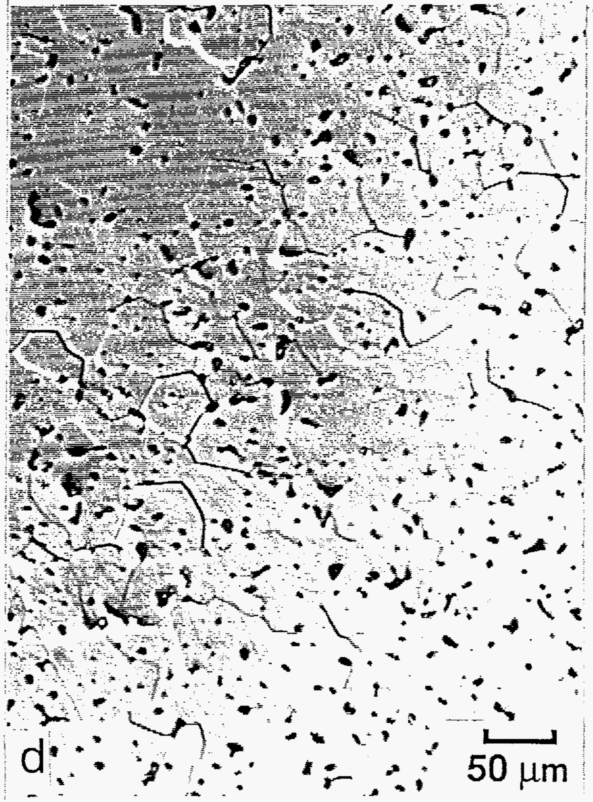

Fig. 3. Mechanically/chemically polished samples of (a) wrought Ta, (b) Ta-10W alloy, (c) low oxygen content P/M Ta HIP'ed at 1600°C, and (d) high oxygen content P/M Ta CIP'ed and sintered at 2400°C. Light micrographs, interference contrast.

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etchants not specifically designed for tantalum. The best results were obtained using the following etchant:

20 ml HNO_3

20 ml HF

60 ml H_2SO_4.

Specimens should be swabbed for 30 seconds for dense samples or 5-10 seconds for porous samples. This etchant produces somewhat sharper grain boundaries, with only a slight degree of additional pitting.

Note - This etchant should be mixed in a fume hood. It is recommended that the mixing beaker containing the HNO_3 and HF be placed in an ice bath while slowly adding the H_2SO_4 with constant stirring to prevent overheating. It should not be stored for more than one month.

Typically the sample appears dull gray during the beginning of the process but will brighten as the etching cycle is completed. Advantages of this etchant include better defined low angle grain boundaries and substructure, little or no apparent enhancement of porosity, and the possibility of photomicrography not only in interference contrast, but also in bright field. Figures 4 and 5 illustrate the effectiveness of this new etch used in combination with the new chemical polish. Shown in Figure 4 are examples of etched tantalum samples photographed in interference contrast. Figure 5 shows examples of etched tantalum photographed in bright field.

This combination of chemical polishing and etching was also used successfully on samples having only a 30 minute polish on Texmet (1) with a 0.3μm alumina slurry. To further test the capabilities of the chemical polish, it was used on a sample directly after grinding on 1200 grit SiC grinding paper; after nine minutes of chemical polishing the grain boundaries were well defined. This sequence was followed by swabbing for thirty seconds with the new etchant. The result following the chemical polish, as shown in Figure 6, was a specimen well suited for a quick grain size determination, but which would not necessarily be of report quality.

Results

In general, the following preparation technique has proven to be effective for tantalum and tantalum alloys. Samples should be ground through 1200 grit SiC grinding papers, followed by rough polishing on Texmet(1) using a 15 μm diamond abrasive suspension for approximately 10-15 minutes. This should be followed by an intermediate polish on Texmet with a 0.3μm alumina slurry for approximately 30 minutes, and then a final polish on Microcloth(1) with 0.3μm alumina slurry for approximately 20 minutes.

After this mechanical polishing sequence, the samples should be chemically polished using the previously described solution, swabbing for 90-120 seconds using heavy pressure. Following the chemical polish the sample should be swab etched with the new etchant for approximately 30 seconds.

This technique has also been successfully used on other refractory materials by varying the chemical polishing and etching times. One example that is particularly noteworthy is a comparison etchant study made on a titanium-60wt% tantalum specimen. This sample was mechanically polished and plater's tape was used to give distinct boundaries between etchant test areas. Etchants tested on this material included Kroll's etchant - ASTM # 192 (3), ASTM etchant #178 (2), and the new chemical polish followed by the new etchant. As the comparison photographs in Figure 7 indicate, the new chemical polish and etchant produce sharper grain boundaries with little or no indications of etch pitting, whereas the other etchants tested show less well-defined structure and regions of etch pitting.

Summary and Conclusions

Although previously used metallographic techniques produced satisfactory results with Ta and Ta alloys for most purposes, they proved to be unsatisfactory for powder metallurgy Ta. Problems associated with traditional techniques include lengthy mechanical polishing time, etch

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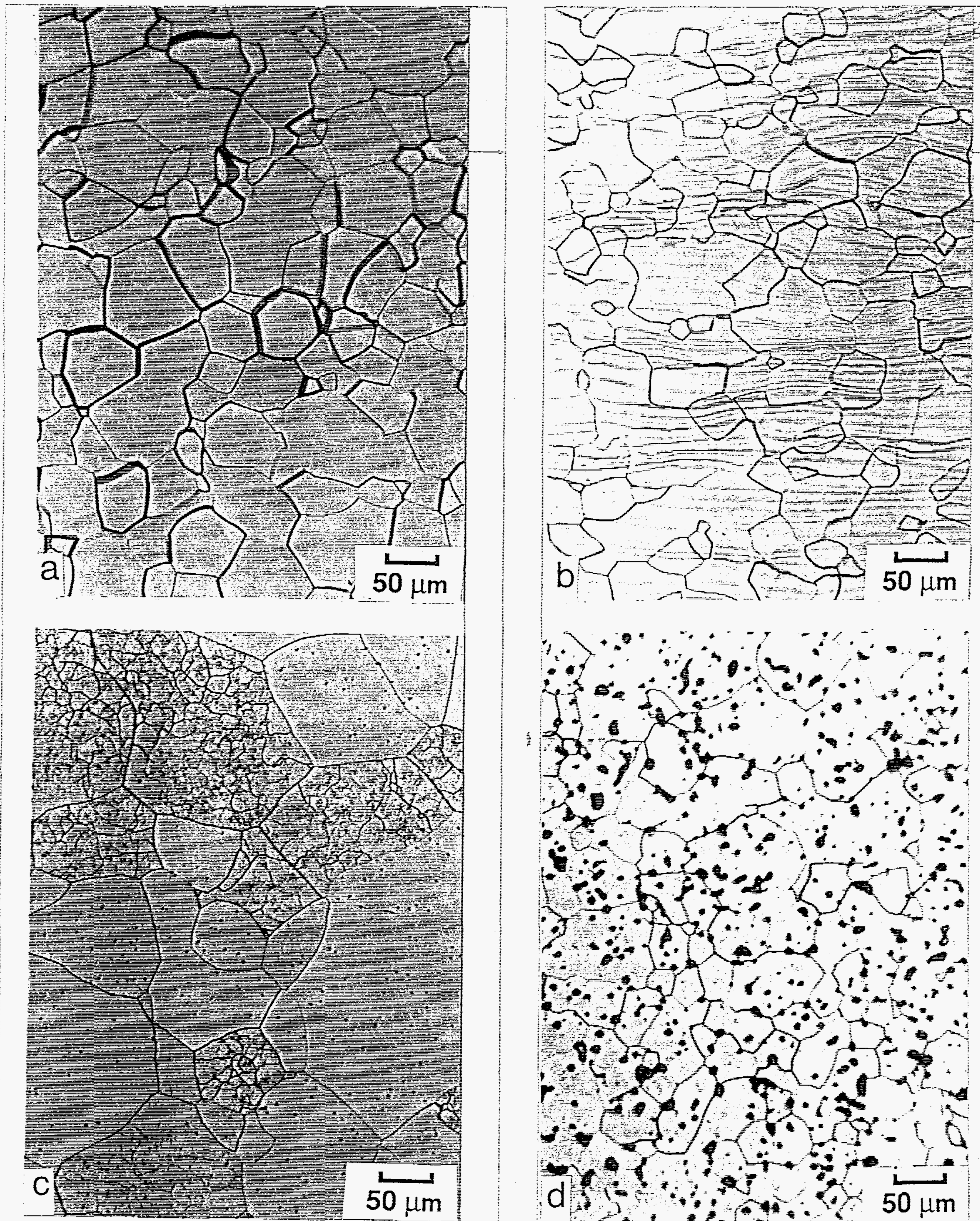

Fig. 4. Samples of (a) wrought Ta, (b) Ta-10W alloy, (c) low oxygen content P/M Ta HIP'ed at 1600°C, and (d) high oxygen content P/M Ta CIP'ed and sintered at 2400°C prepared using the new chemical polish and etch. Light micrographs, interference contrast.

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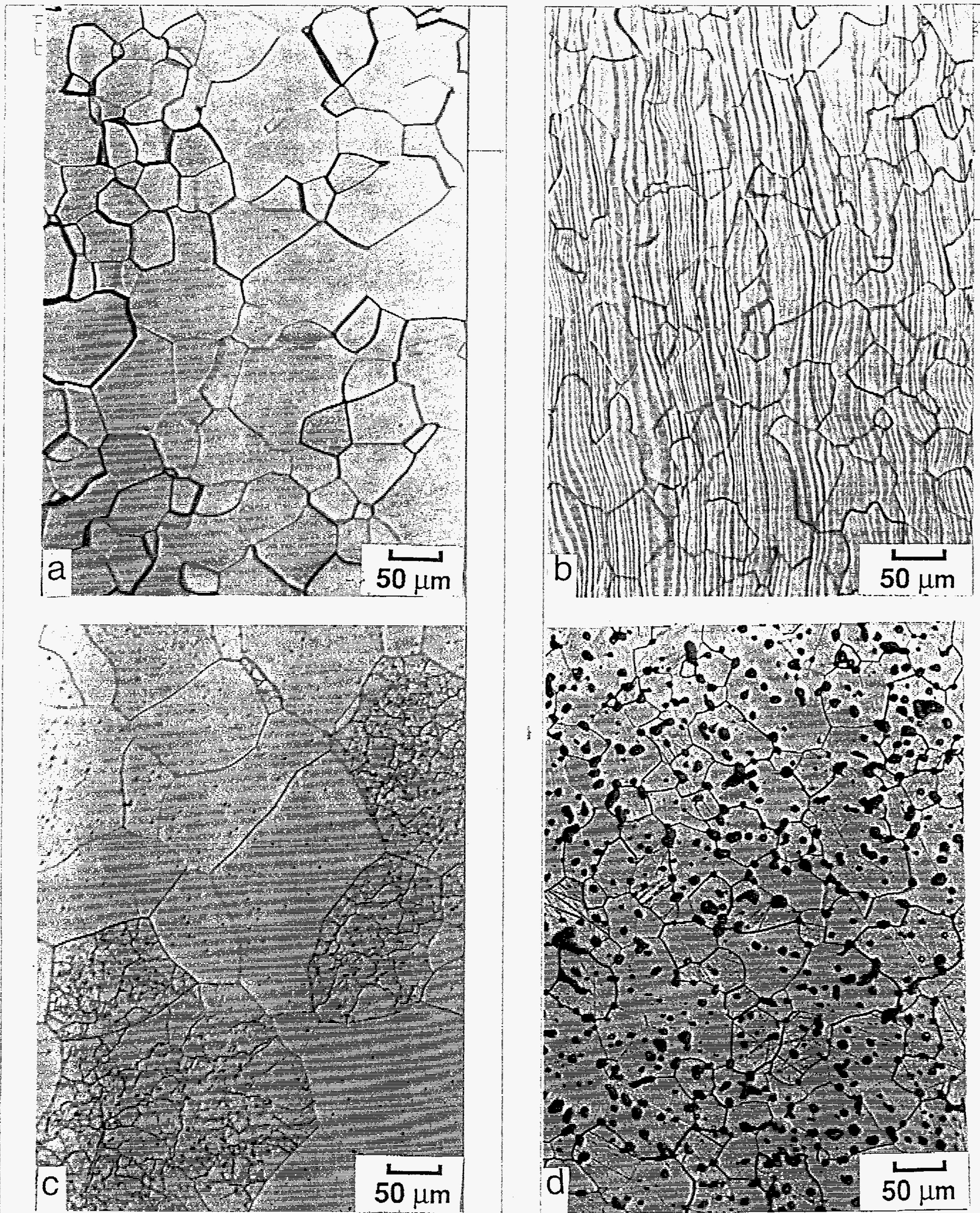

Fig. 5. Brightfield micrographs of (a) wrought Ta, (b) Ta-10W alloy, (c) low oxygen content P/M Ta HIP'ed at 1600°C, and (d) high oxygen content P/M Ta CIP'ed and sintered at 2400°C, prepared using the new chemical polish and new etch.

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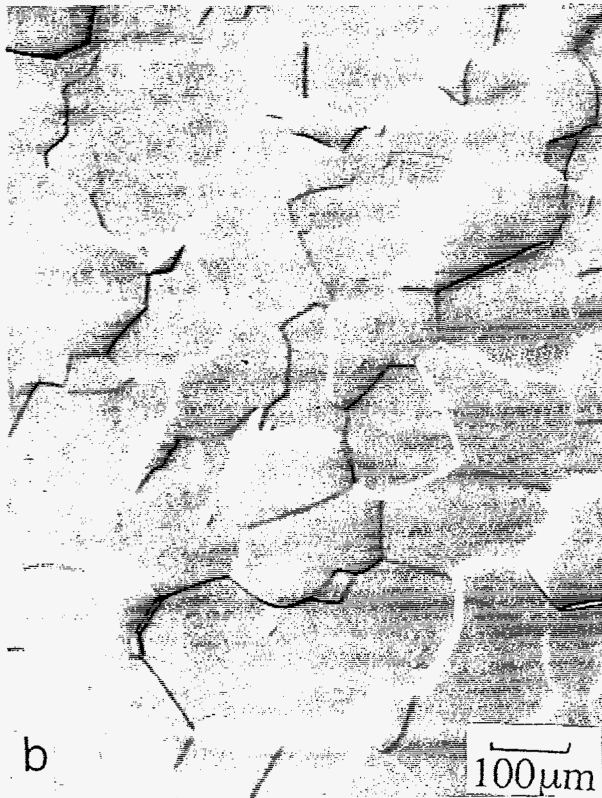

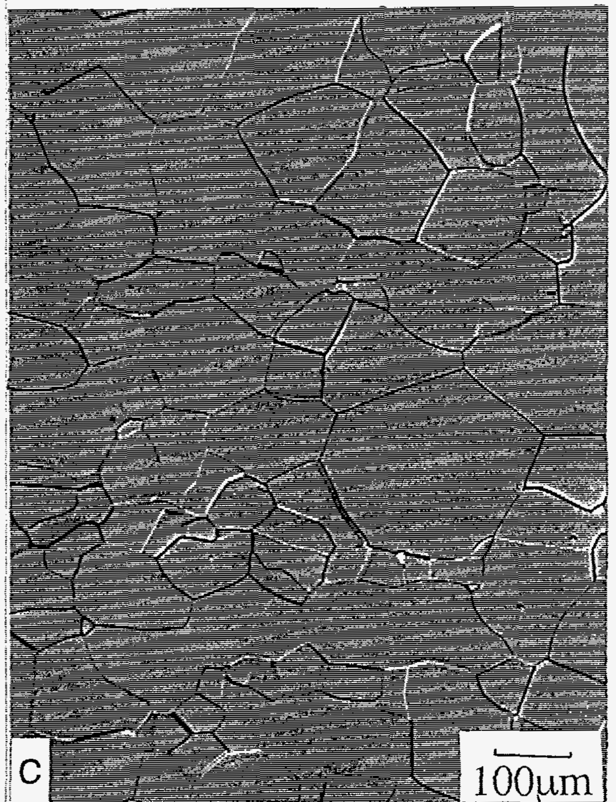

Fig. 6. Results of the preparation sequence of (a) tantalum ground through 1200 grit SiC grinding paper, followed immediately by (b) a nine minute chemical polish, and (c) the same specimen etched for 30 seconds with the new etchant. Light micrographs, interference contrast.

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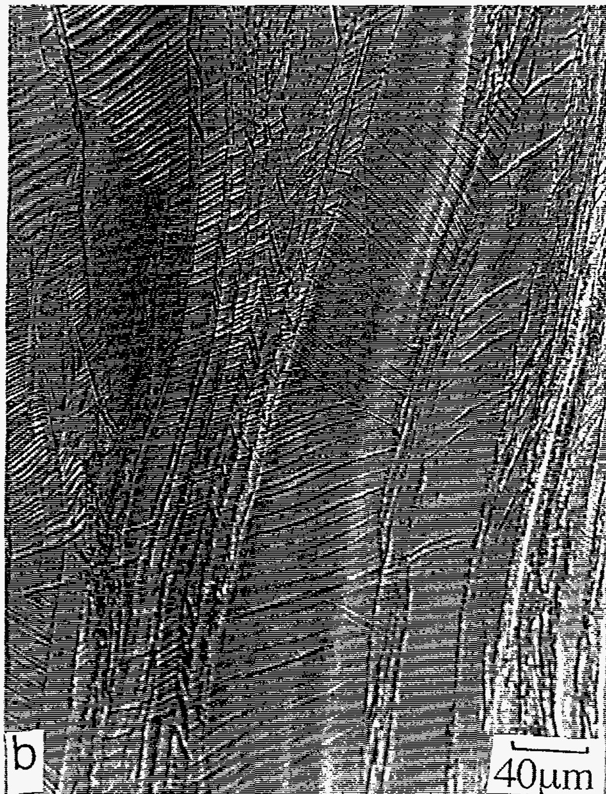

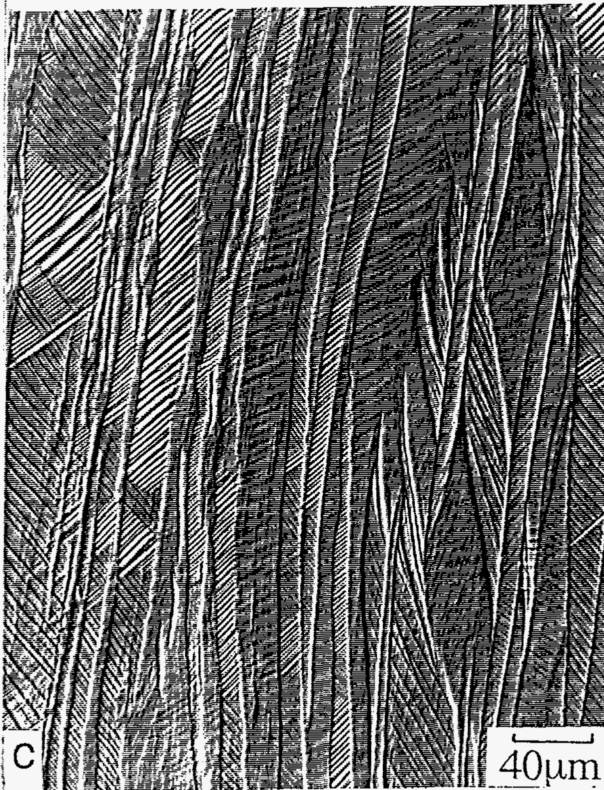

Fig. 7. Comparison etches on a Ti-60 Ta specimen using (a) ASTM etchant #192, (b) ASTM etchant # 178, and (c) the new chemical polish and etch procedure. Light micrographs, interference contrast.

pitting, and a strong tendency toward indistinct low-angle grain boundaries, which made it difficult to make accurate, quantitative grain size and porosity measurements.

The new preparation sequence has several advantages. Considerable preparation time is saved, because there is sufficient relief between grains in the chemically-polished condition to show most grain boundaries clearly, using interference contrast. When the new etching technique is applied after the chemical polish, even low-angle grain boundaries and substructure are well defined, and the accuracy of grain size measurements using intercept methods (4) is greatly improved. In addition, less etch pitting is encountered compared with most other etchants. Samples in the chemically polished condition are satisfactory for measurement of the area fraction and size distribution of porosity, while the etched condition is better for intercept counting measurements of grain size. The new etching technique also makes it possible to photograph specimens in either bright field or interference contrast . It should be noted that, if interference contrast is not available, similar results can usually be obtained with oblique illumination. This can be attained either by decentering the aperture diaphragm or by partially blocking its opening.

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