

## New Attempts on Preparing Tungsten FIB Sample

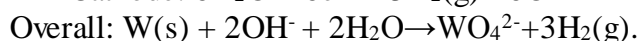
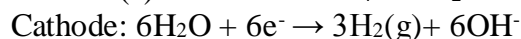
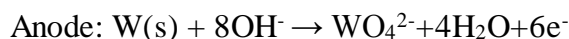
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Twin-jet electro polishing is a widely used method for preparing thin focused ion beam (FIB) and TEM samples of materials. The electro-polishing of tungsten-based foils is generally performed in an alkaline solution (predominantly containing NaOH) [1] but has several drawbacks. For example, the alkaline solution sometimes damages the glass windows in the twin-jet device and also produces poor quality foils [2], mandating the use of cyanides to mitigate the surface damage [3]. In this study, we propose an alternative method to reduce the thickness of a 70  $\mu\text{m}$  thick tungsten foil below 10  $\mu\text{m}$ , while simultaneously obtaining a flatter surface finish and better machinability under the FIB.

A schematic of the electro-polishing system is shown in Figure 1 (a). A solution of 2 M NaOH and 1 M NaNO<sub>3</sub>, heated to 60 °C, is used as the electrolyte. A constant voltage of 3V is applied between the tungsten sample and the copper ring, with tungsten as the anode and the copper ring as the cathode. On immersing the sample in the electrolyte, a sub-10  $\mu\text{m}$  thin tungsten FIB sample can be prepared in a few seconds. Figure 1 (b) are the optical micrograph and scanning electron micrographs of a sample prepared by this method. The scratches caused by mechanical milling in the pre-polished surface is eliminated and a 5  $\mu\text{m}$  thick, flat surface is obtained.

The overall electrochemical reactions are the following:

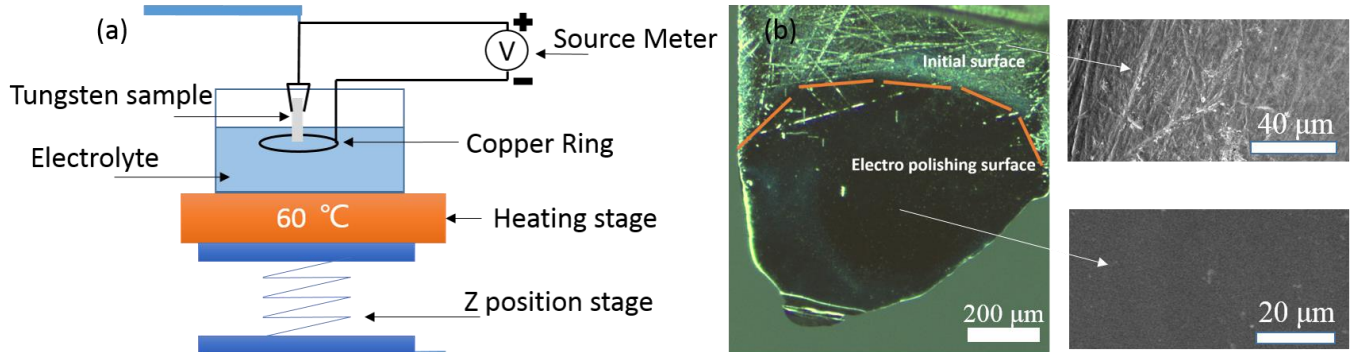


Though the chemical reactions in the traditional method and the present study are the same, an important difference is that the latter process involves heating whereas the former relies on a cooling system. Figure 2 illustrates the difference between the two processes. As seen in Fig. 2 (a) and 2 (b), when the temperature is low, the anodic dissolution of tungsten generates a vortex of OH<sup>-</sup> ions that accelerates etching at this location in the meniscus formed at the gas / liquid / solid interface. Also, the WO<sub>4</sub><sup>2-</sup> ions flow downstream over the surface and hinder the etching process below the meniscus level, thus creating a 'neck-in' formation in the resulting sample [5]. Heating the solution accelerates the diffusion of both ions such that the etching is accelerated with increasing depth as shown in Figure 2 (c) and 2 (d). This implies that the lowest part dissolves faster (see Fig. 2 (d)), and a uniformly shaped sample is obtained.

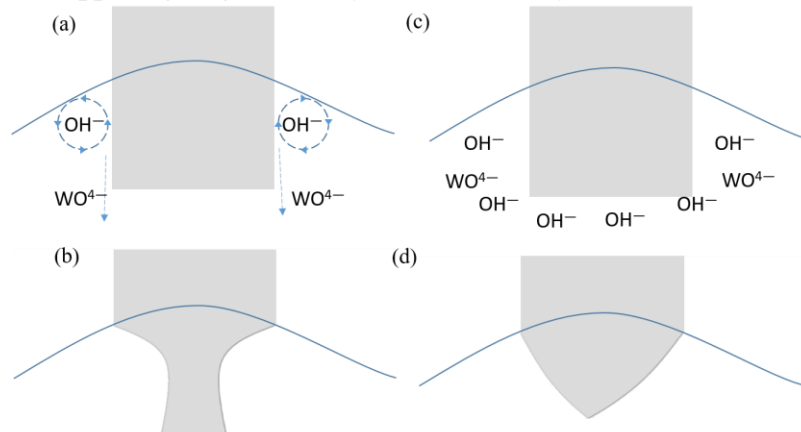
Addressing the issue of improving surface quality, Bucki M et al. recommended the use of aged NaOH or Na<sub>2</sub>CO<sub>3</sub> [2], though he did not explain the underlying phenomenon. However, we obtain an equally good surface finish with the use of a fresh NaOH and NaNO<sub>3</sub> solution. We therefore conclude that, even though the extra anion, CO<sub>3</sub><sup>2-</sup> or NO<sub>3</sub><sup>-</sup>, does not participate in the reaction, it will form a protective layer on the sample. Since this layer is thicker around the lower area compared to that around the raised area, the OH<sup>-</sup> prefer to dissolve the raised area, and leads to a smooth surface as shown in Figure 3.

[1] Mastenbr.A, Hamburg G, Journal of Physics E-Scientific Instruments, **5** (1972), p. 10

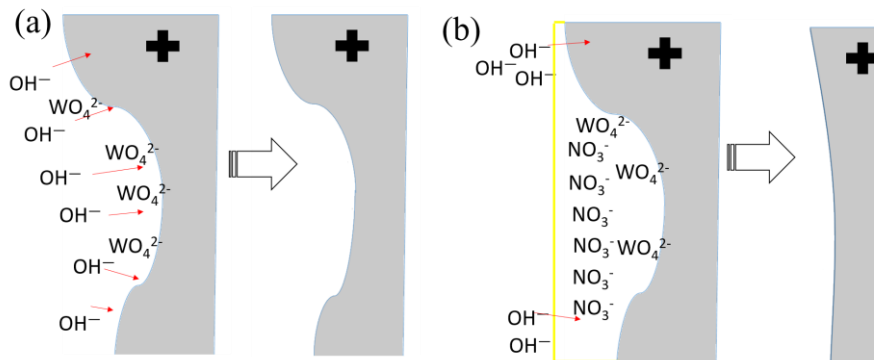
- [2] Bucki M, Gemperle A, Burkhanov GS, *Metallography*, **13** (1980), p. 89.  
 [3] Reiser J, Rieth M, Dafferner B, *Journal of Nuclear Materials*, **424** (2012), p.197.  
 [4] Ibe JP, Bey PP, Brandow SL, *Journal of Vacuum Science & Technology A*, **8** (1990), p.3570.  
 [5] Ju B-F, Chen Y-L, Ge Y, *Review of Scientific Instruments*, **82**(2011), p.13707.



**Figure 1.** (a) Schematic of the electro polishing system, including source meter, position stage, heating stage, tungsten sample, coppering ring, electrolyte, and electrolytic cell and (b) the prepared FIB sample.



**Figure 2.** Illustration of the electrochemical etching process (a) and (b) at lower temperatures showing vortexing of  $\text{OH}^-$  ions; (c) and (d) at high temperatures, where accelerated diffusion of ions eliminates the “necking” effect. The grey area is the sample and the blue line indicates the liquid level.



**Figure 3.** Illustrations of protective ionic layers (a) where only a few  $\text{WO}_4^{2-}$  are present, allowing the  $\text{OH}^-$  ions to easily etch the surface and (b) where the additional  $\text{NO}_3^-$  ions from the solution will also attach to the anode and form a thicker protective film (indicated by the yellow line), causing  $\text{OH}^-$  to only react with the raised area.