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Alternating Current Polishing and Electroetching
of Metals*

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Alternating Current Polishing and Electroetching of Metals

Alternating current electropolishing and electroetching as a generally applicable technique in metallography was suggested by Reiswig's use of an AC method on Os-Ir alloys⁽¹⁾. The etching of plutonium has been a continuing problem, the previous techniques consisting mainly of DC electrolytic polishing and etching. The AC technique was investigated because it seemed to offer an alternative to both DC electrolytic and chemical etching. After the success of the AC method on plutonium allotropes was achieved, a number of other metals were tried and, using only four electrolytes, a considerable degree of success was achieved.

Figure 1 is a schematic drawing of the equipment set-up.

The four electrolytes used are listed in Table I. The conditions for the AC etching of plutonium, but not the other metals, were extensively investigated with respect to variations in solutions, voltages, and current densities. The examples of other metals (figures 2-21) are given only to suggest the broad applicability of the AC technique and to indicate the results that may be obtained.

The conditions for specific metals are listed under the photomicrographs. The general results on metals are listed in Table II. Although a number of metals were etched poorly or not at all by the AC technique (see Table II), it is very likely that many of them and others could be successfully etched in solutions other than those listed in Table I.

Table I

Solutions for Alternating Current Electroetching

All parts of solutions are by volume unless noted otherwise.

Solution A: 10 Nitric Acid

90 Methanol

Solution B: 90 Lactic Acid

15 Water

6 Sulfuric Acid

Solution C: 8 Phosphoric Acid

5 Ethanol

5 Glycerine

Solution D: 133 Acetic Acid

25 (grams) Chromic Acid

7 Water

Table II

Figure	<u>Material Successfully Etched</u>	<u>Marginally Etched</u>	<u>No Success</u>
2	Cadium	Manganese	Zirconium
3	Indium	Vanadium	Titanium
4	Cobalt	Uranium	Niobium
5	Tin	Nickel	Gold
6	Copper		Tungsten
7	Bismuth		Molybdenum
8	Germanium		Tantalum
9	Magnesium		
10	Chromium		
11	Zinc		
12	Silver		
13	Thorium		
14	Plutonium		
15	Pearlytic Steel		
16	Austenitic Steel		
17	Most Rare Earths		
18	Scandium		
	Vitrium		
19	54 Ni - 46 Ta		
20	95 V - 5 U		
21	99 Al - 1 U		
	Beryllium*		

* Polished very well, but no etch.

Figure 1 Schematic of AC etching equipment set-up. (The specimen probe and electrode are made of stainless steel)

Figure 2

Cadium 500X

Solution B

40 volts - 10 sec - 1 am/sq cm

Etch, polish alternatively to remove cold work

Figure 3

Indium 100X

Solution B

30 volts - 10 sec

.6 amp/sq cm

Figure 4

Cobalt 100X

Solution C

1. 40 volts - 30 secs, 0.9 amp/sq cm
2. 20 volts - 10 secs, 0.7 amp/sq cm
(No. 2 cleans surface)

Figure 5

Tin 500X

Solution B

- 30 volts - 10 secs, 0.5 amp/sq cm
+ 10% Nital dip. to clean up

Figure 6

Copper ~~100X~~ (+500X)

Solution B

40 volts - 35 sec

2.1 amp/sq cm

Shorter time for smaller grain sizes

Figure 7

Bismuth 100X

Solution A

30 volts - 3 sec

10 amp/sq cm

Figure 8 Germanium 100X

Solution B
 45 volts - 3 min
 100 ma/sq cm

Figure 9 Magnesium 500X (+100X)

Solution A
 20 volts - 10 sec

Figure 10 Chromium 100X
 Solution 30% Nital (Cr doped)
 10 volts - 5 sec
 4.0 amps/sq cm

Figure 11 Zinc 500X
 Solution C
 20 volts - 5 sec
 2.5 amp/sq cm

Figure 12 Silver 500X
 Solution C
 20 volts - 10 sec
 2.8 amp/sq cm

Figure 13 Thorium 100X
 Solution D
 50 volts - 35 sec
 0.4 amp/sq cm

Figure 14

Alpha plutonium 100X

Solution A
5 volts - 20 sec
.8 amp/sq cm

Figure 15

M1020 Steel - Pearlite + ferrite 500X

Solution B
20 volts - 10 sec
1 amp/sq cm

Figure 16 Austenite + martensite needles 100X (+1000X)
 Solution B
 20 volts - 10 sec
 0.6 amp/sq cm

Figure 17 Samarium 100X
 Solution B
 10 volts - 10 sec
 70 ma/sq cm

Figure 18

Scandium $\times 500$

Solution B
10 volts - 10 sec

Figure 19 54 Ni - 46 Ta Three phases 500X
 Solution C
 40 volts - 10 sec
 2 amp/sq cm

Figure 20 95 V - 5 U 100X
 Solution B
 20 volts - 10 sec
 1 amp/sq cm

Figure 21

99 Al - 1 U 100X

Solution C
40 volts - 10 sec
1 amp/sq cm

FIGURE CAPTIONS

1. Schematic of AC etching equipment set-up. (The specimen probe and electrode are made of stainless steel.)
2. Cadmium 500X, Solution B, 40 volts - 10 sec - 1 amp/sq cm, Etch, polish alternatively to remove cold work.
3. Indium 100X, Solution B, 30 volts - 10 sec, .6 amp/sq cm.
4. Cobalt (100X, Solution C, 1.) 40 volts - 30 secs - 0.9 amp/sq cm, 2.) 20 volts - 10 secs - 0.7 amp/sq cm, (No. 2 cleans surface.
5. Tin 500X, Solution B, 30 volts - 10 secs, 0.5 amp/sq cm, + 10 Nital dip. to clean up.
6. Copper 100X (+500X), Solution B, 40 volts - 35 sec, 2.1 amp/sq cm, Shorter time for smaller grain sizes.
7. Bismuth 100X, Solution A, 30 volts - 3 sec, 10 amp/sq cm.
8. Germanium 100X, Solution B, 45 volts - 3 min, 100 ma/sq cm.
9. Magnesium 500X (+100X), Solution A, 20 volts - 10 sec.
10. Chromium 100X, Solution 30% Nital (Cr doped), 10 volts - 5 sec, 4.0 amps/sq cm.
11. Zinc 500X, Solution C, 20 volts - 5 sec, 2.5 amp/sq cm.
12. Silver 500X, Solution C, 20 volts - 10 sec, 2.8 amp/sq cm.
13. Thorium 100X, Solution D, 50 volts - 35 sec, 0.4 amp/sq cm.
14. Alpha plutonium 100X, Solution A, 5 volts - 20 sec, .8 amp/sq cm.
15. M1020 Steel - Pearlite + ferrite 500X, Solution B, 20 volts - 10 sec, 1 amp/sq cm.
16. Austenite + martensite needles 100X (+1000X), Solution B, 20 volts - 10 sec, 0.6 amp/sq cm.
17. Samarium 100X, Solution B, 10 volts - 10 sec, 70 ma/sq cm.
18. Scandium 100X, Solution B, 10 volts - 10 sec,
19. 54 Ni - 46 Ta Three phases 500X, Solution C, 40 volts - 10 sec, 2 amp/sq cm.
20. 95 V - 5 U 100X, Solution B, 20 volts - 10 sec, 1 amp/sq cm.
21. 99 Al - 1 U 100X, Solution C, 40 volts - 10 sec, 1 amp/sq cm.

REFERENCE

1. R. D. Reiswig and J. M. Dickinson, "The Osmium-Iridium Equilibrium Diagram", Trans. Metallurgical Society AIME, 230, pp. 469-472, 1964.