Metallographic Techniques for Cu-Au Alloys

by Robert Bakish and William D. Robertson

IN the course of a study of stress corrosion cracking of AuCu₃, it was necessary to develop new electropolishing and metallographic etching methods. The techniques are generally useful for Cu-Au alloys, including the AuCu composition in which transformation markings are well delineated.

For electropolishing, the only satisfactory electrolyte and procedure that has been found¹ has the composition given in Table I. The solution must be vigorously stirred while polishing and the temperature maintained in the range of 55° to 62°C. The electrolyte is very unstable and can be used only for 10 to 15 min after which its color changes and a dark-brown precipitate forms. The polishing time is between 1 and 4 min depending on the initial surface preparation. The use of a hood is recommended to avoid the danger of cyanide gas which may form at one of the electrodes even though the solution is alkaline. The optimum current and voltage conditions for vertical anode and platinum cathode are on the plateau shown in Fig. 1, in which voltage is measured across the cell terminals. At voltages below the plateau, etching of grain boundaries occurs and under some conditions, which have not been clearly defined, octahedral etch figures are observed.

For general purpose grain-size determination in polycrystalline alloys, a 3 pct solution of iodine in methyl alcohol is used with excellent results. However, when it is necessary to reveal small orientation differences or concentration gradients in an alloy, the iodine etch is not satisfactory. For this purpose, a highly satisfactory etching procedure was developed during this investigation. It involves the use of the vapor given off by freshly prepared aqua regia. The solution employed is 3HCl to 1HNO₃, which decomposes in accordance with the reaction $\text{HNO}_3 + 3\text{HCl} = \text{NOCl} + 2\text{H}_2\text{O} + \text{Cl}_2$.

The polished specimen surface is held about 1 to

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Fig. 1—Current voltage characteristics are shown for electropolishing of AuCu₃. Voltage was measured across the cell terminals.

Table I. Composition of Electrolyte

Compound	Quantity*
Potassium cyanide	67.5 g
Potassium-sodium-tartrate	15.0 g
Potassium-ferro-cyanide	15.0 g
Phosphoric acid, 85 pct	18.5 ml
Ammonium hydroxide (Sp.G. 0.9)	2.5 ml

* Based on one liter of solution.

2 cm above an aqua regia solution for 2 to 10 sec depending on the intensity of the contrast required and the surface is not subsequently washed. The results obtained with a coarse grained cast structure are shown in Fig. 2.

Subgrain boundaries in a single crystal and concentration gradients within the boundaries are developed by the same treatment with the results shown in Fig. 3. Obviously, any conclusions regarding structure-dependent properties will be modified by the pronounced heterogeneity, which is not clearly evident when more conventional methods are used.

Interesting structural details are also revealed by treatment for 4 hr at 850°C in unpurified tank argon. Fig. 4 shows a pattern characteristic of orientation-dependent oxidation.

¹ *Metal Finishing* (August 1950) 50, p. 84.

Fig. 2—Specimen etched with aqua regia vapor shows dendrites in a coarse grained cast structure. X50. Area reduced approximately 15 pct for reproduction.

Fig. 3—Cell structure of a single crystal etched with aqua regia vapor is shown after heating 24 hr at 850°C. X200. Area reduced approximately 15 pct for reproduction.

Fig. 4—After 4 hr at 850°C in unpurified argon, specimen shows oxidation figures. X800. Area reduced approximately 15 pct for reproduction.