

SHORT COMMUNICATIONS

A Simple Thinning Technique for Transmission Electron Microscopy Observation of an Al-Cu Alloy

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During the course of a study of the age-hardening characteristics of Al-4 wt.% Cu alloy under stress the need for thinning this alloy for transmission electron microscopy became apparent. Although some microscopic investigation of Al-Cu alloys has been reported in the literature [1, 2], no standard procedure for preparing thin foils was to be found. In the following we describe in detail a quick and effective method of preparing thin foils of this alloy.

First, a specimen (approximately $\frac{1}{8}$ in. square and 0.01 in. thick) is cut from the age-hardened single crystal using an Al_2O_3 cut-off wheel (0.015 in. thick). While mounted on a small metal block with wax the specimen is very slowly wet-ground (using only 400 and 600 grade silicon carbide paper on polishing wheels) to a thickness of approximately 0.005 in. Extreme caution is necessary in order to prevent introducing any deformation in the specimen.

The specimen is then mounted in a holder devised by Goodell [3], as shown in Fig. 1. The specimen holder is simply a long (approx. 9 in.) stainless steel rod ($\frac{1}{8}$ in. diam) with a housing at one end to hold two round stainless steel inserts. The specimen is held between these two inserts.

The above assembly is lacquered with a paint (trade name Microstop) to prevent it from being attacked by the electrolyte (see Fig. 1). Important to note here is that the central portion of the inserts is left unlacquered. This ensures a free flow zone around that area of the specimen which is to be thinned [4]. As a result, the electrolyte action usually produces a fairly large thinned region around the central hole, thus providing a large overall area 'transparent' to the electron beam. The limitation, however, is that it also enlarges the hole in the inserts a little every time a thinning operation is performed. This problem is moderated by using stainless steel washers, with the desired hole, inside the inserts. A permanent solution to

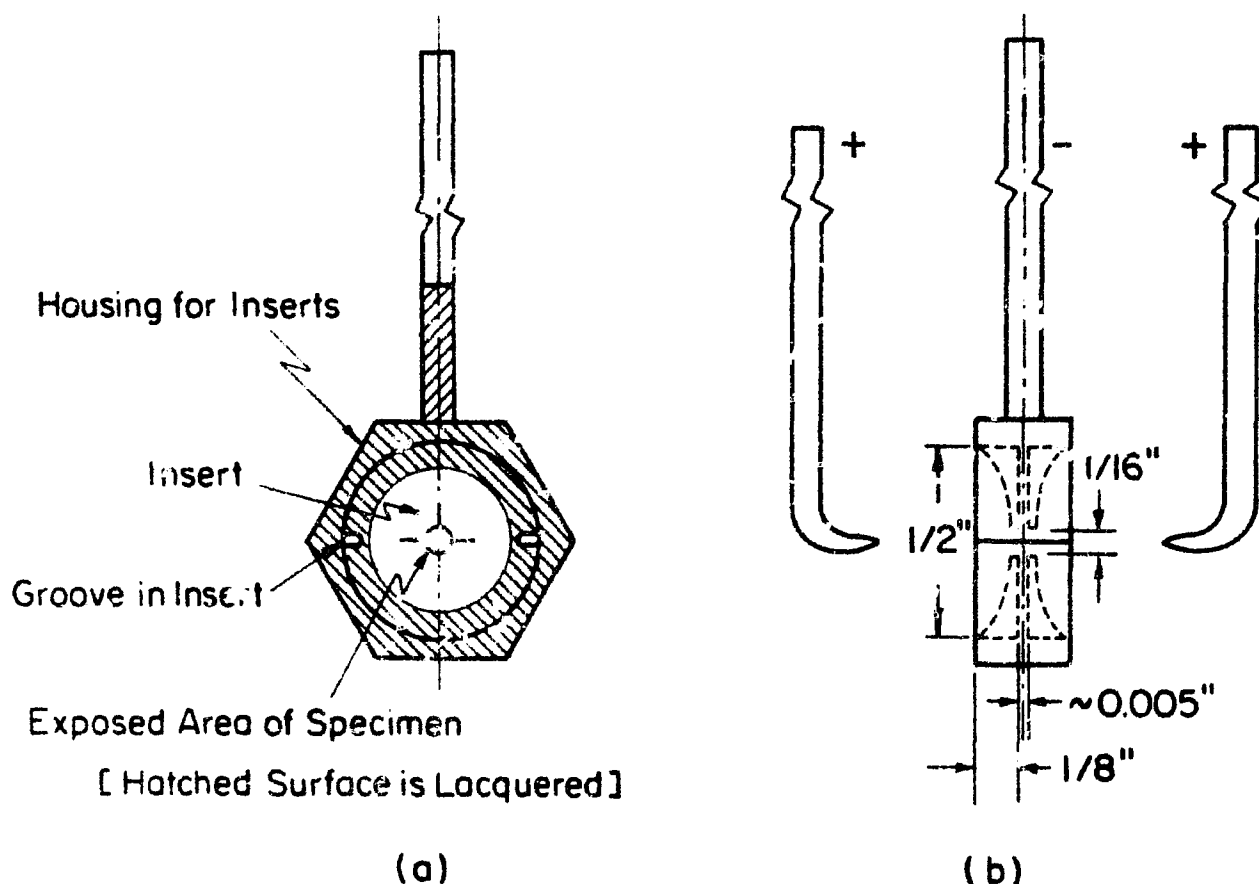


FIG. 1. Illustration of the specimen holder assembly.

this problem, however, might be to use either platinum inserts or platinum washers, since platinum is expected not to be readily attacked by this electrolyte.

The specimen holder assembly is held between two L-shaped aluminum rods ($\sim \frac{1}{8}$ in. diam, see Fig. 1(b)), which are used as cathodes to produce a 'twin-jet action' [5-7] on the exposed area of the specimen. These rods are also lacquered, so that only the ends which form the jet action are exposed to the electrolyte.

The overall experimental setup is somewhat similar to that used by Fisher and Szirmae [8]. The electrolyte (stock content: 820 cc orthophosphoric acid + 140 cc sulphuric acid + 156 g chromic oxide + 40 cc water, also known as Lenoir's solution, see [9]) is heated in a 250-ml beaker on a hot plate and stirred sufficiently to maintain an optimum temperature of $75^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

Because the electrolyte is opaque, the perforation in the specimen cannot be seen through the electrolyte with an illumination lamp. We have, however, found that it takes approximately 10 min at 7.5 V and 25 mA for the first perforation to appear. After about 9 min, therefore, the specimen is rinsed, approximately every 15 sec, with distilled water and viewed against a flash light. As soon as the first speck of light is observed, the specimen is again rinsed with distilled water and then with methanol. The

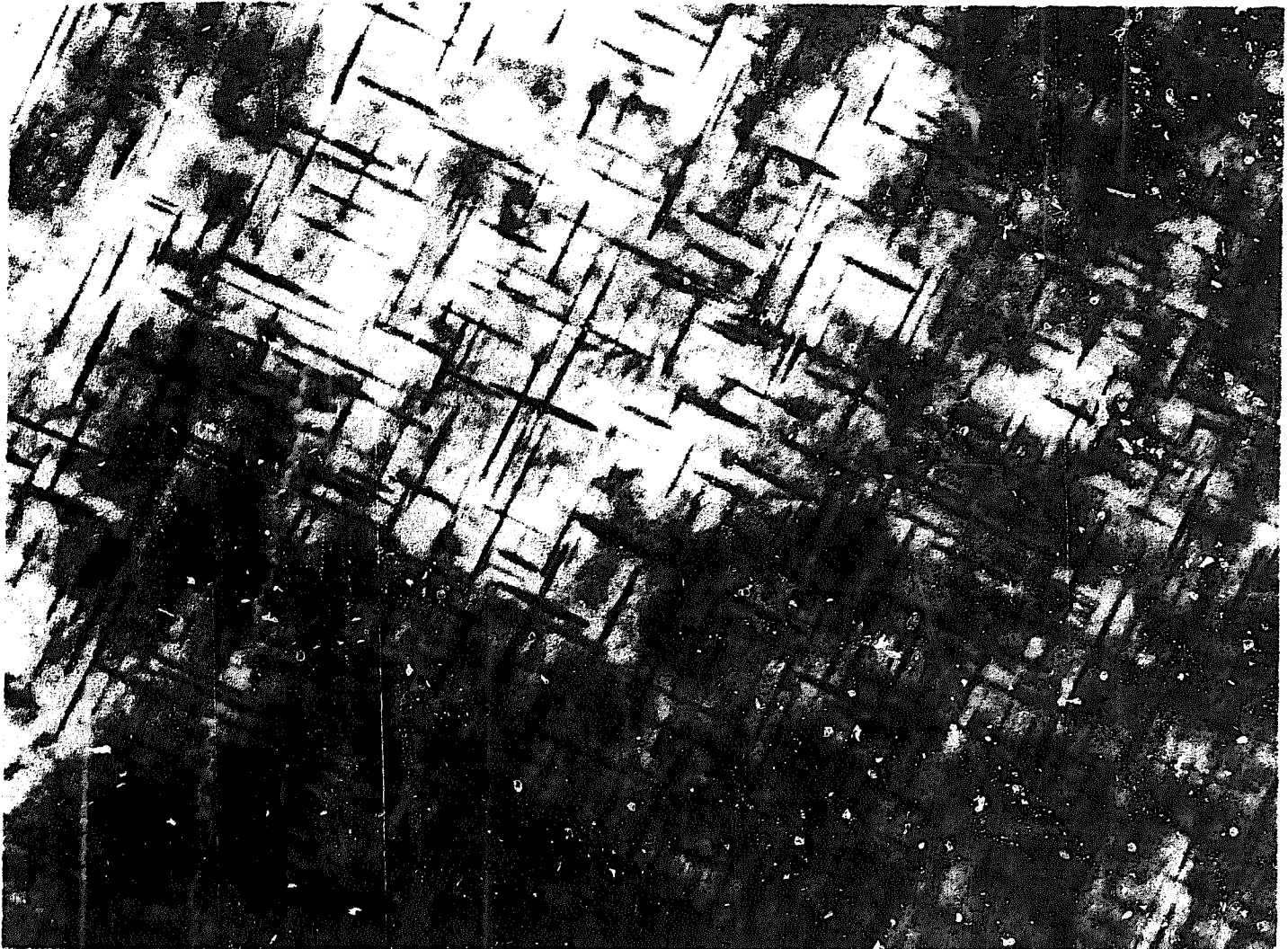


FIG. 2. Transmission electron micrograph of an Al-4Cu single crystal aged under compressive stress. Shows precipitate (θ') platelets on cube planes. Foil orientation is $\{001\}$ perpendicular to the stress axis.

specimen is removed and the final thinning is performed (at approx. 4 V for 3–5 sec) by holding the specimen in fine forceps which are connected to the positive lead from the dc source. The specimen is again carefully rinsed with distilled water and methanol and dried with a stream of hot air.

Figure 2 is a transmission electron micrograph of a thin foil, prepared in the above manner, of an age-hardened Al-4 wt. % Cu single crystal.¹ A JEOL electron microscope operating at 100 kV was employed for this examination.

The authors would like to acknowledge the help of the following in the initial experimental setup: Professors W. F. Hosford, W. C. Bigelow, and W. C. Leslie, and Mr. G. Brooks of The University of Michigan. Financial support for this investigation was provided by the National Science Foundation GH-35912.

¹ For alloy description and the preparation of single crystals see [10]. It should be noted, however, that this technique is equally applicable to polycrystalline specimens.

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Accepted January 1974