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A Jet Polishing Technique for Thinning Two Phase Materials

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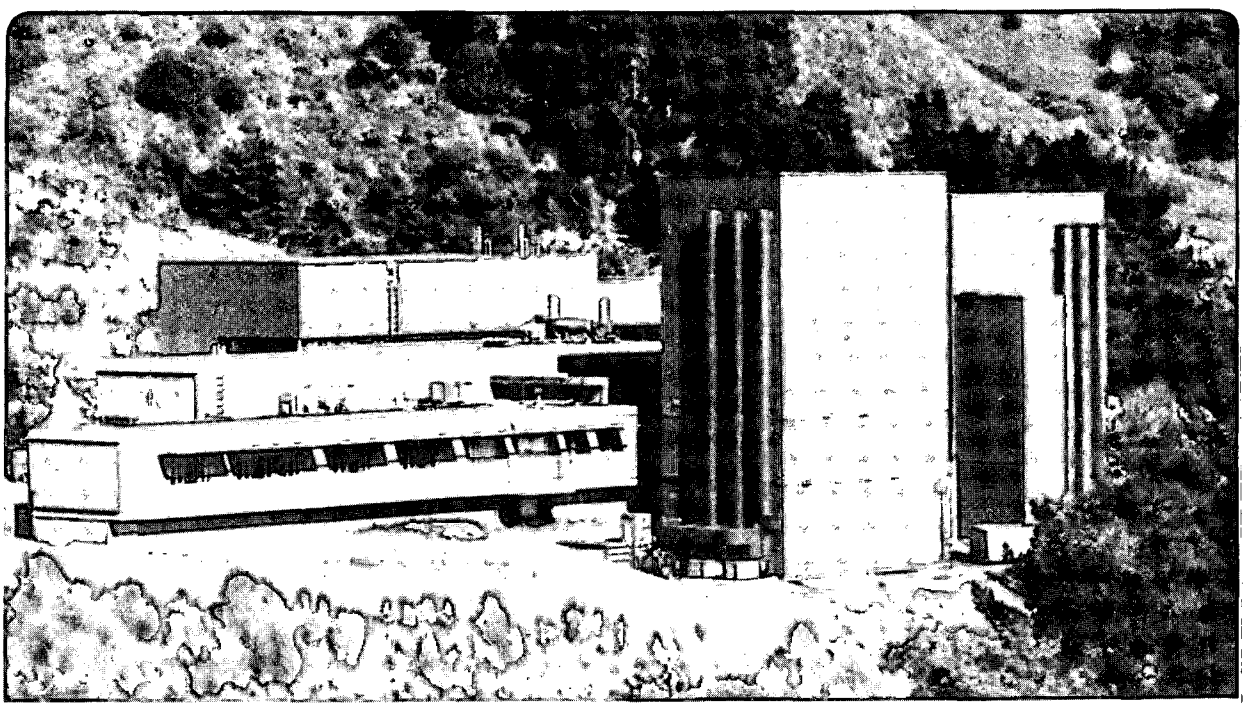

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A Jet Polishing Technique for Thinning Two Phase Materials

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A JET POLISHING TECHNIQUE FOR THINNING TWO-PHASE MATERIALS

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A common problem in the preparation of thin foils for transmission electron microscopy is the different thinning rate in two-phase materials. Often this leads to foils in which the majority, or matrix, phase is evenly polished while the minority, or precipitate, phase is either etched out or stands proud of the surrounding material. In the present report we describe a two-stage jet polishing technique that has been used successfully on different relatively coarse two-phase structures.

The apparatus used in this work was a Fischione twin jet polisher, but similar behavior was found using a Struers design. The technique relies on the standard polishing characteristics of alloys in jet electropolishers. As illustrated in fig. 1 the polishing behavior usually is a sensitive function of the applied voltage or the current drawn. If the voltage is too low the sample develops a smooth double-concave profile but the dull appearance of the surface indicates etching conditions (a). At increased voltage a well-polished region begins to appear around the perimeter of the disc while the center still shows the dull surface typical for etching (b). This behavior shows that the perimeter region must draw higher current density than the center region, probably an effect of the jet flow profile. A further increase in the voltage leads to a well-polished foil (c). Finally, when the voltage is raised further, shiny dimples appear in the high-current density regions at the perimeter of the film (d). Typically, this condition leads to premature perforation of the foil at the edge without producing a usable hole in the center even though the center region may still be in good polishing condition. Under these pitting conditions, inclusions or second phase particles are often attacked preferentially. Each particle becomes the center of a pit usually with very steeply wedged sides. By combining this behavior with the known polishing behavior of the matrix it is possible to set up a two-stage process in which the sample is polished under conditions appropriate for the matrix until just before perforation followed by a few seconds at greatly increased voltage to thin preferentially the second phase particles.

Fig. 2 illustrates the results obtained on an overaged Cu-Cr alloy. In (a) the foil was thinned to perforation at a single voltage. From its thickness it is apparent that the particle has been attacked more slowly than the matrix. If the two-stage process is used, the particles are thinned preferentially (b), often with a small hole in the center of each precipitate. Two-stage polishing of Fe-Cr and Mo-C alloys is shown in fig. 3.

Although the specific current/voltage characteristics of each solution and alloy system will vary, it is believed that this technique is of general applicability for those cases where a satisfactory electrolyte with behavior as shown in fig. 1 has been found.

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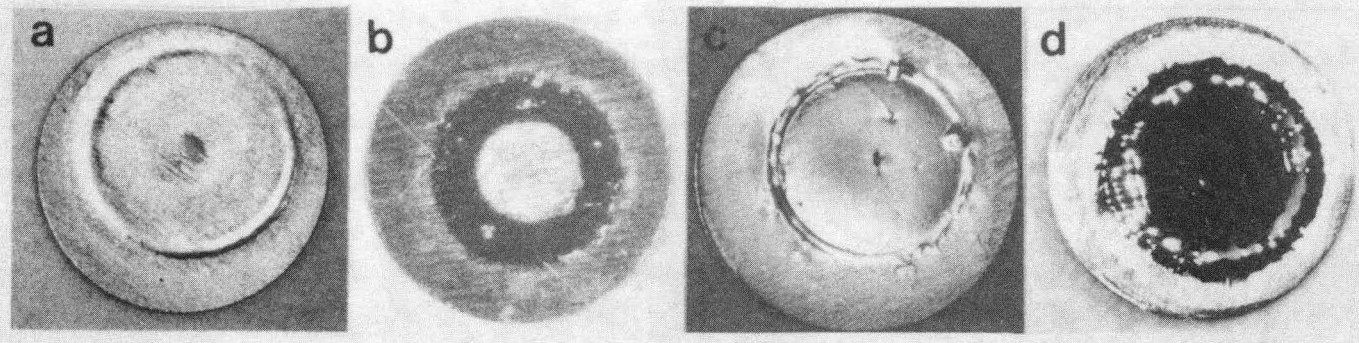

Fig. 1 Characteristic appearance of jet polished discs as a function of voltage.

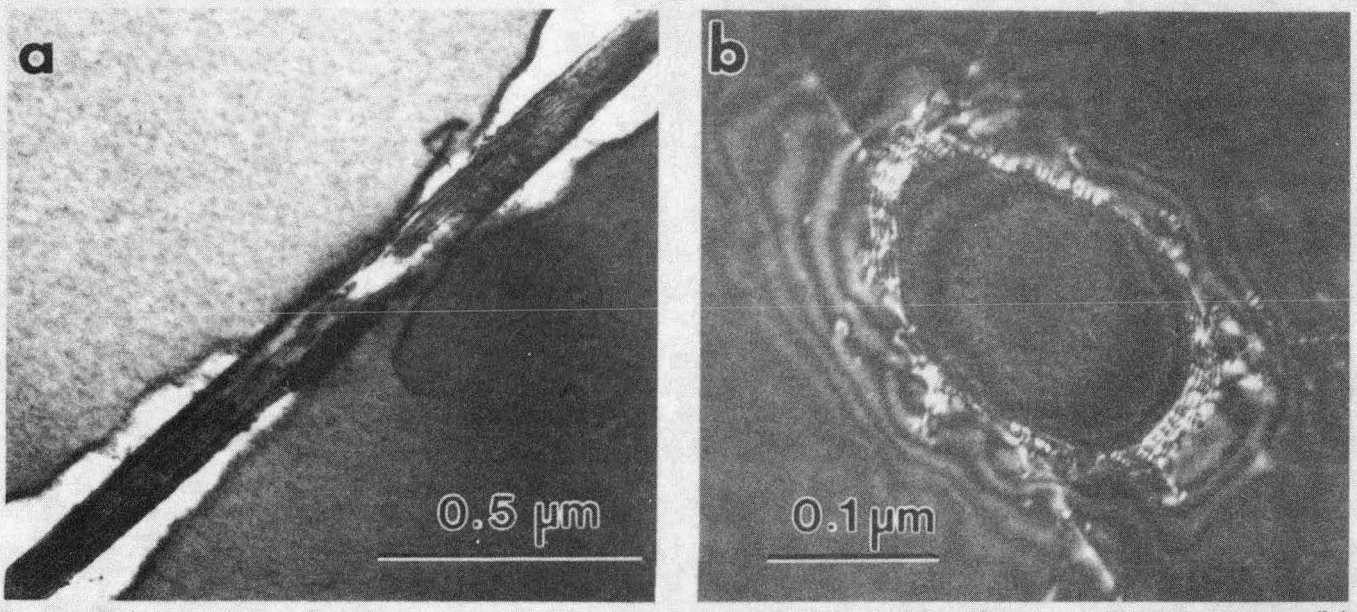

Fig. 2 Overaged Cu-Cr alloy thinned in a single stage (a) and by the two-stage process (b).

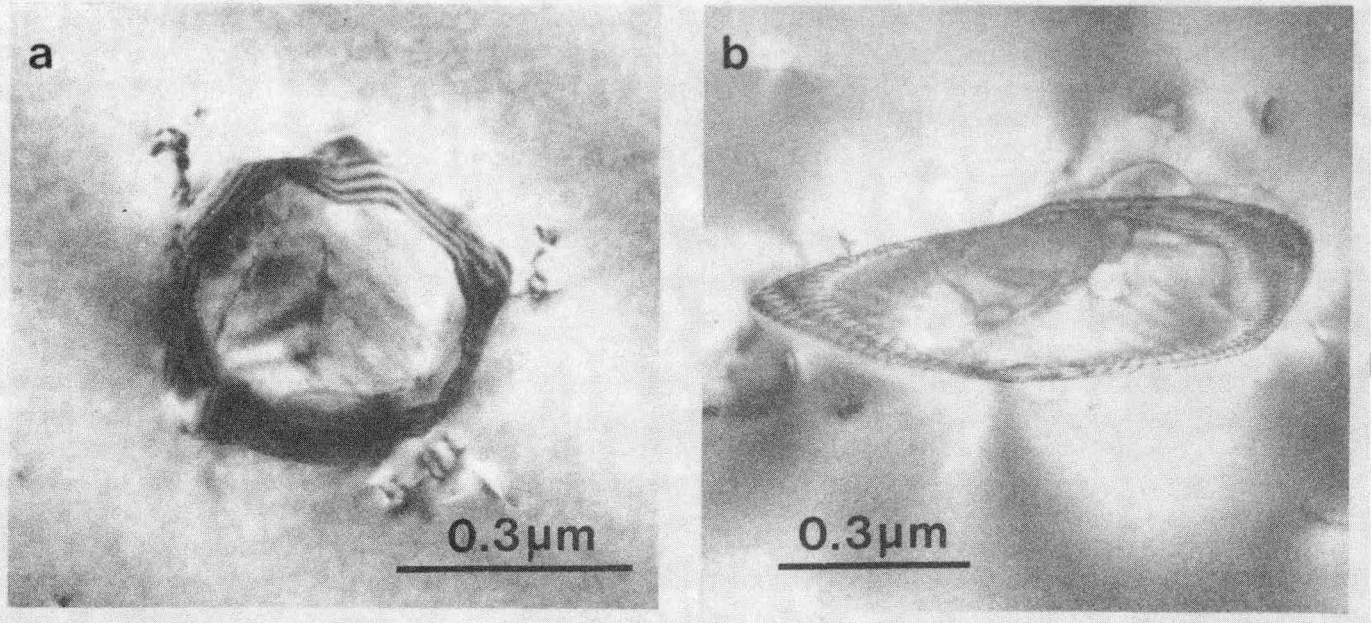

Fig. 3 Samples thinned by the two-stage technique. Fe-Cr alloy (a), Mo-C alloy (b).

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