Automatic Unit for Thinning Transmission Electron Microscopy Specimens of Metals

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A specimen holder and a polishing unit with an automatic shutoff were designed for thinning disks taken from preselected areas of large metallic specimens or from specimens with small cross sectional areas. Since the disk precisely fits the specimen holder of the microscope, a supporting grid is unnecessary and deformation from cutting and handling after thinning is eliminated. The automatic shutoff device greatly improves conditions for transmission by stopping the polishing current at the optimum time.

INTRODUCTION

IN 1949 Heidenreich successfully electrothinned a 3 mm aluminum disk for electron transmission.[1] Since then a number of techniques have been developed for thinning disks of a variety of metals. Basically, they are the same as that used 17 years ago. A stream of electrolyte is directed onto one side of the disk until a dish shaped depression about one-half the thickness of the sample is obtained. The disk is then inverted and the operation repeated on the opposite side. One of the prime concerns in thinning disks is the stopping of thinning in the final stage, at the optimum time. In semiconductors, because of their ability to transmit light, the thinning can be stopped just before the sample is perforated. In 1961 Riesz and Bjorling[2] thinned Ge by utilizing an electrolytic jet thinning technique, a light source, and a photomultiplier tube which automatically halted the etching process at the right time. A

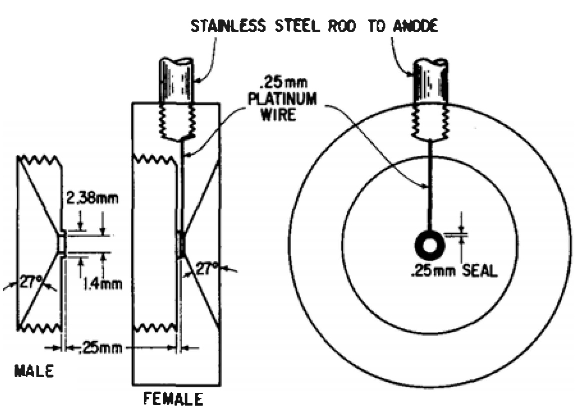

FIG. 1. Cross section of disk specimen holder.

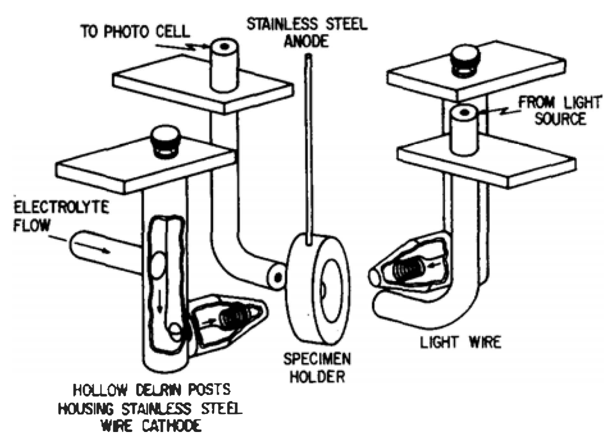

FIG. 2. Polishing arrangement.

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[2] R. P. Riesz and C. G. Bjorling, Rev. Sci. Instr. 32, 889 (1961).

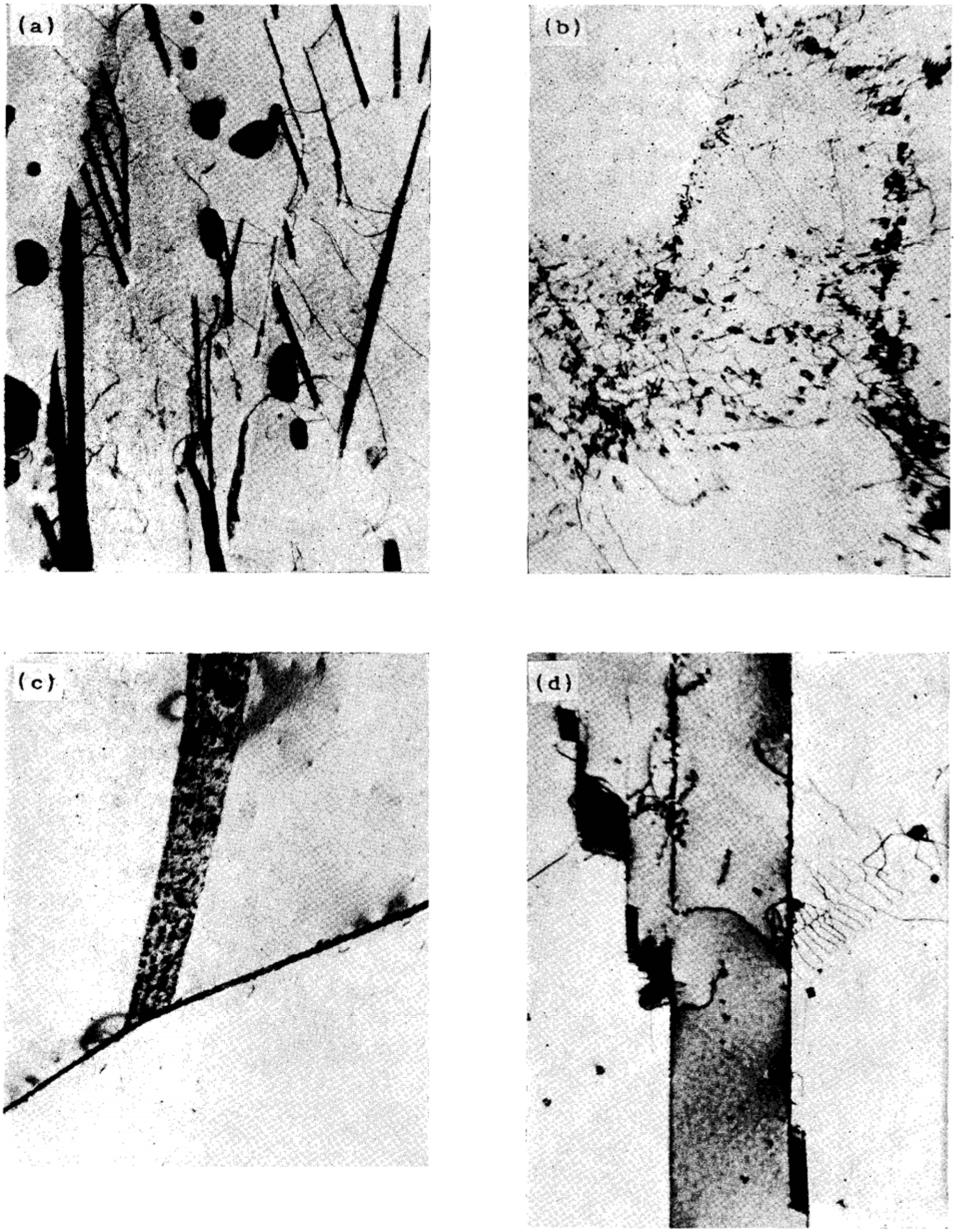

Fig. 3. Examples of results obtained from the automatic shutoff technique. (a) Type 410 stainless steel; (b) low carbon steel; (c) zone refined iron; and (d) type 303 stainless steel. All ×20 000.

variation of this technique was developed by Booker and Stickler[3] to thin Si and Ge. Since light cannot be transmitted through metals, the best time to stop polishing is the instant the metal is perforated. To detect perforation, light striking one side of the specimen can be seen by a low power microscope focused on the opposite side,[4,5] or, with the specimen in a horizontal position and the electrolyte impinging on the bottom, electrolyte can be seen on the top surface.[6]

This paper describes a simple, rapid technique for polishing metal disks simultaneously from both sides and automatically stopping the polishing current the instant perforation occurs. The procedure utilizes a submerged double jet technique,[7] a photocell, and Light Wires (the Bausch & Lomb trade name for noncoherent bundles).

CONSTRUCTION OF DISK HOLDER

Figure 1 illustrates the construction of the specimen holder. Delrin resin was chosen as the material for the specimen holder because it is resistant to most acids and is easily machined. Black Delrin resin was used to eliminate transmission of light through the holder. However, Delrin resin is attacked by perchloric acid solutions.[8] The disk holder was machined from 25.4 mm bar stock. It consisted of a 16 mm diam$\times$4 mm thick threaded male section that screwed into a 25.4 mm diam$\times$8 mm thick female section. A 2.4 mm diam$\times$0.25 mm recess on the inside of the female section accommodated the disk and a 2.38$\times$0.25 mm projection on the male section completed the seal. A 1.4 mm diam hole in each section determined the area of the specimen exposed to the electrolyte.

Electrical contact between the disk specimen and the stainless steel anode is accomplished by a 0.25 mm platinum wire recessed in the female section. This wire is formed into a ring 2.4 mm in outside diameter that is in contact with the periphery of the disk specimen. The remaining 0.25 mm Delrin resin provides a seal between the electrolyte and the anode, which prevents preferential attack at the points of contact of the platinum wire and the specimen.

CONSTRUCTION OF POLISHING UNIT AND AUTOMATIC SHUTOFF DEVICE

The polishing arrangement is shown in Fig. 2. A 1600 rpm motor drives the pump which forces the electrolyte at the rate of 600 ml/min through hollow moveable Delrin resin posts which house the cathodes in the form of a 3 mm diam coil of stainless steel wire. The cathode orifice is also 3 mm in diameter. Polishing was done with an anode-cathode separation of 1 cm.

The automatic shutoff of the polishing current is accomplished by the use of a Clairex CL603AL Cd-Se photocell and 3 mm Bausch & Lomb Light Wires. The Light Wires are enclosed in glass tubing and submerged in the polishing solution. Light from an outside source is transmitted by a Light Wire and impinges on one side of the specimen. Perforation of the specimen allows the transmitted light to impinge on a Light Wire on the opposite side of the specimen which in turn transmits the light to the outside photocell, energizing a solenoid which shuts off the polishing current and sounds an audio alarm. The simultaneous sounding of an alarm with the current shutoff helps minimize contamination that can occur when some materials are left in an electrolyte after polishing has ceased.

PROCEDURE

Specimens were ground on abrasive papers to a thickness of approximately 0.1 mm. Disks were punched out with a standard Siemens grid punch. If a particular area was desired, the specimen was given a light etch to reveal the area of interest and the disk was taken from that site.

A sodium chromate-acetic acid solution was commonly used as the electrolyte (100 g anhydrous Na_2CrO_4 to 500 cc glacial acetic acid). The thinning current varied between 12 and 14 mA at 30 to 35 V. Despite the fact that the thinning was done in a double walled glass container designed for cooling the electrolyte, no cooling was needed because of the small size of the specimen, the short polishing time and constant stirring by the jets. Polishing time for a 0.1 mm thick disk of low carbon steel was approximately 10 min. Perforation of a 100 to 200 μ hole normally occurred in the center of the disk, far removed from any deformation that may have resulted from the punching operation. At the sound of the audio signal, the specimen holder was removed from the electrolyte, rinsed in clean acetic acid and then in methyl alcohol. The disk was removed from the holder, rinsed in alcohol, and dried under a blower. Electron micrographs of typical materials so thinned are shown in Fig. 3.

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