International Tables for Crystallography (2006). Vol. C, Figure 3.5.1.2, p. 172.

2 mm in diameter is used or when the specimen edges are masked with the holder. The disc specimens can be profiled on both surfaces or one surface can be left flat. A flat surface is preferable for electron-diffraction analysis as well as for secondary electron imaging.

The mechanical preparation of specimens has been greatly simplified by the development of two instruments by Gatan Inc. (6678 Owens Drive, Pleasanton, CA 94566, USA) (Alani & Swann, 1990). The first is a holder for disc samples that is used on a polishing wheel to grind and polish discs to a specific thickness. This holder has a height adjustment for the specimen, which can be ground and polished to a thickness of 50 μm by the use of various grades of abrasive. With the second instrument, called a 'dimpler™', the polished disc is profiled or dimpled on one side. This dimple is ground and polished with a sensitivity of 1 μm. The dimpled disc is then ready to be thinned in an ion-bombardment instrument.

3.5.1.3. *Final thinning by argon-ion etching*

Argon-ion bombardment or sputter etching is the simplest method for the final thinning of electron-microscope specimens. The application of the technique to ceramics and minerals was demonstrated in the early and mid-1960's with an apparatus commercialized by Paulus & Reverchon (1961; Tighe & Hyman, 1968) or similar designs (Bach, 1964; Drum, 1965). Since that time, numerous commercial instruments have been developed and are available in most electron-microscope laboratories.

The schematics in Fig. 3.5.1.1 show two types of arrangement of the instruments. There are two ion sources for etching from both sides of a specimen, a specimen holder that can be rotated, a viewing port, and a vacuum system. In the instrument in Fig. 3.5.1.1(*b*), the ion sources tilt instead of the specimen holder and an airlock system is used for sample exchange and for monitoring the sample during thinning. The new instruments are relatively trouble free and simple to use compared with the first-generation instruments. The ion sources operate at 4 to 10 kV with variable current to control desired thinning rate and the amount of specimen damage. Thinning rates of 1 $\mu m\,h^{-1}$ per ion source are average for normal specimens. The sputtering rates depend also on the angle of tilt (Fig 3.5.1.2) with respect to the ion beam. Faster rates cause more specimen heating and greater ion damage.

The Dual Ion Mill system has two chambers such as the one shown in Fig. 3.5.1.1(*b*) (Gatan Inc.). The chambers function independently, so that two specimens can be thinned simultaneously. The sample holder is raised through an airlock to the observation window at intervals in order to monitor the thinning process. A special beam detector can be used to stop the operation when the specimen perforates.

The specially designed Precision Ion Polishing System 'PIPS™' provides precise control over the specimen thinning area and is a dedicated low-angle instrument with a high thinning rate (Alani & Swann, 1992). The ion beams can be adjusted individually to specific angles, and can be switched on and off regularly during the thinning process. Additionally, the beams can be oriented with respect to specific line features of the sample to preserve edge detail, for example, in a stacked sample (Alani, Harper & Swann, 1992). Gases other than argon can be used for special etching conditions.

Ionic bombardment produces uniquely etched surfaces that are easily recognized in light and electron micrographs. With stationary specimens, closely spaced grooves and ridges are etched parallel to the direction of beam impingement. When the specimen is rotated slowly, these ridges are smoothed and an undulating orange-peel surface is produced. The severity of etching decreases when the angle of incidence to the ion beam is decreased to near grazing angles but uneven etching is never eliminated. The orange-peel texture is randomly located with

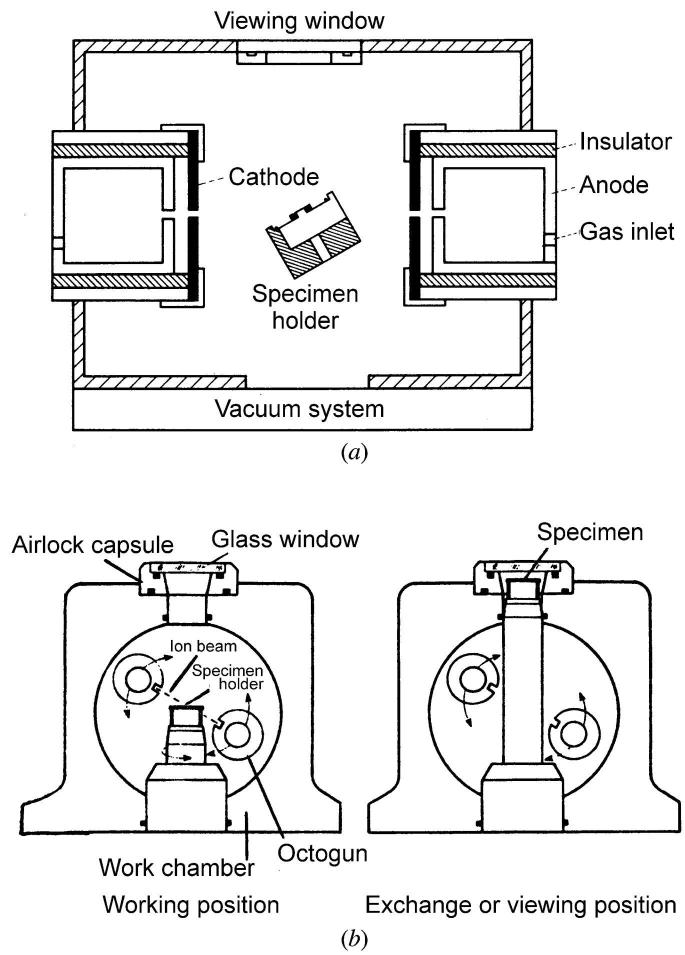

Fig. 3.5.1.1. The two types of arrangement for final thinning by argon-ion etching. (*a*) The system of Paulus & Reverchon (1961) with fixed ion sources, made by Alba. (*b*) The system of Swann with movable ion sources and an airlock for specimen viewing (drawing courtesy of Gatan, Inc.).

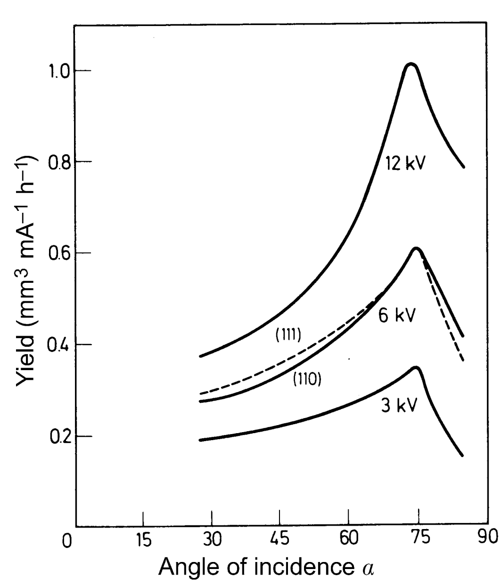

Fig. 3.5.1.2. Dependence of sputtering rate on the angle of tilt.

references