

9. MICROSCÓPIO ELETRÔNICO DE TRANSMISSÃO (MET)

9.1. APRESENTAÇÃO DOS COMPONENTES E PRINCÍPIOS DE FUNCIONAMENTO DO MET

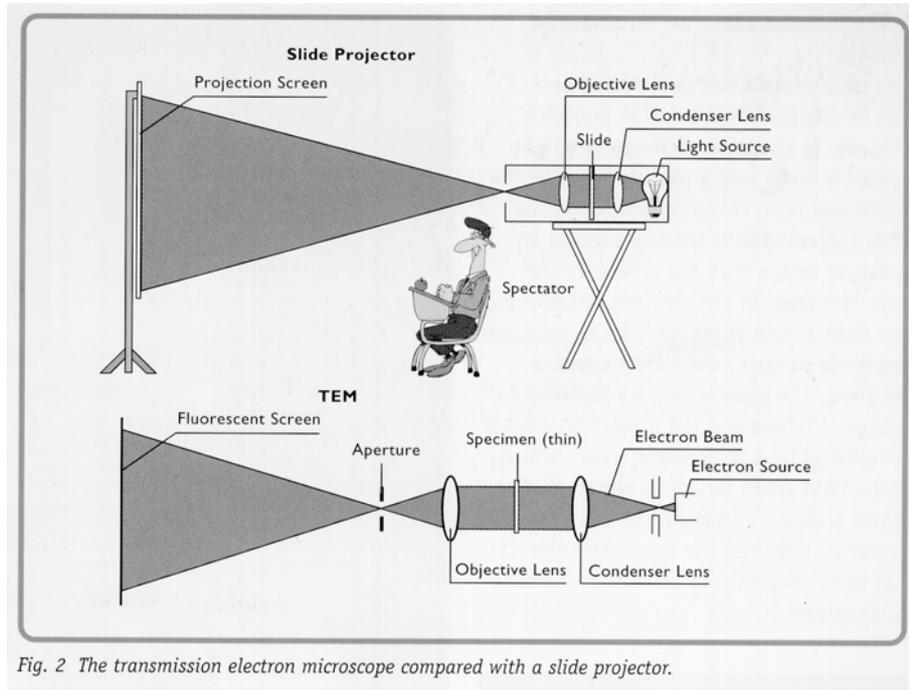


Fig. 2 The transmission electron microscope compared with a slide projector.

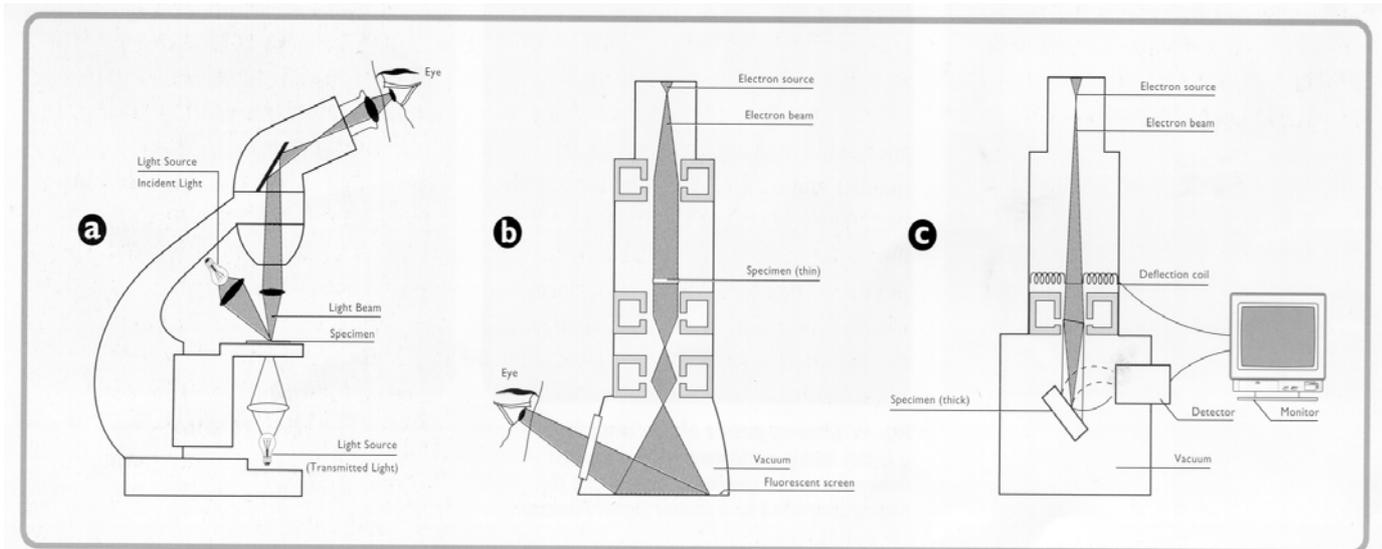


Fig. 3 Comparison of the light microscope (a) with transmission (b) and scanning (c) electron microscopes.

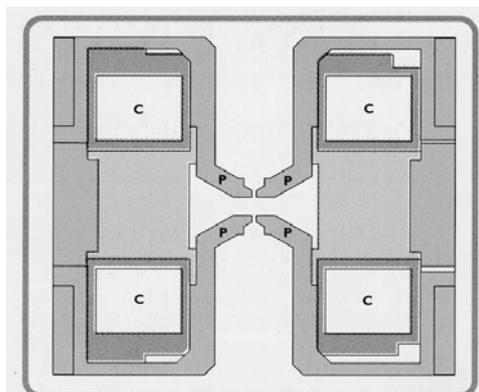


Fig. 7 Cross-section of an electromagnetic lens. C is an electrical coil and P is the soft iron pole piece.

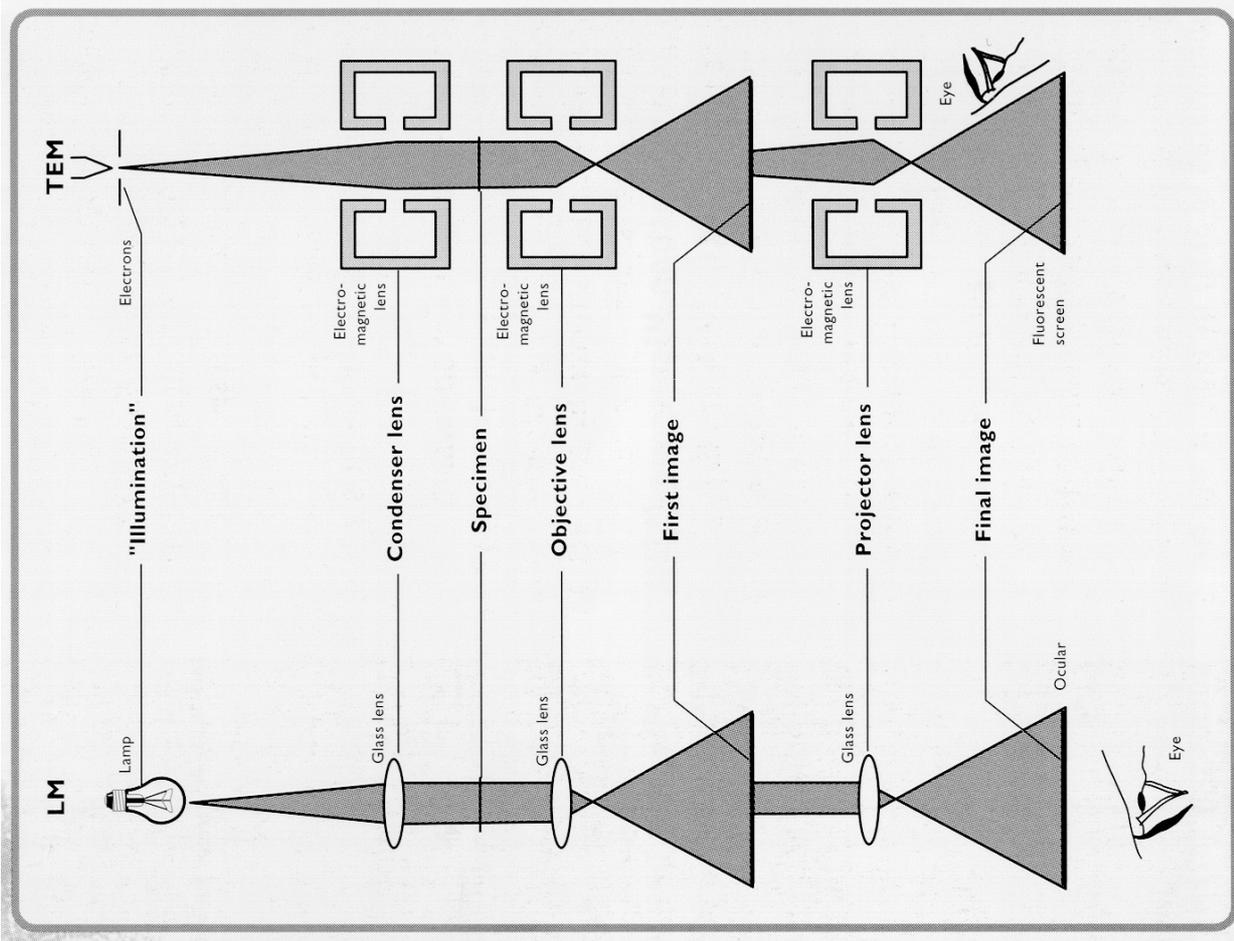


Fig. 5 Ray paths of light in a light microscope (LM) compared with those of electrons in a transmission electron microscope (TEM).

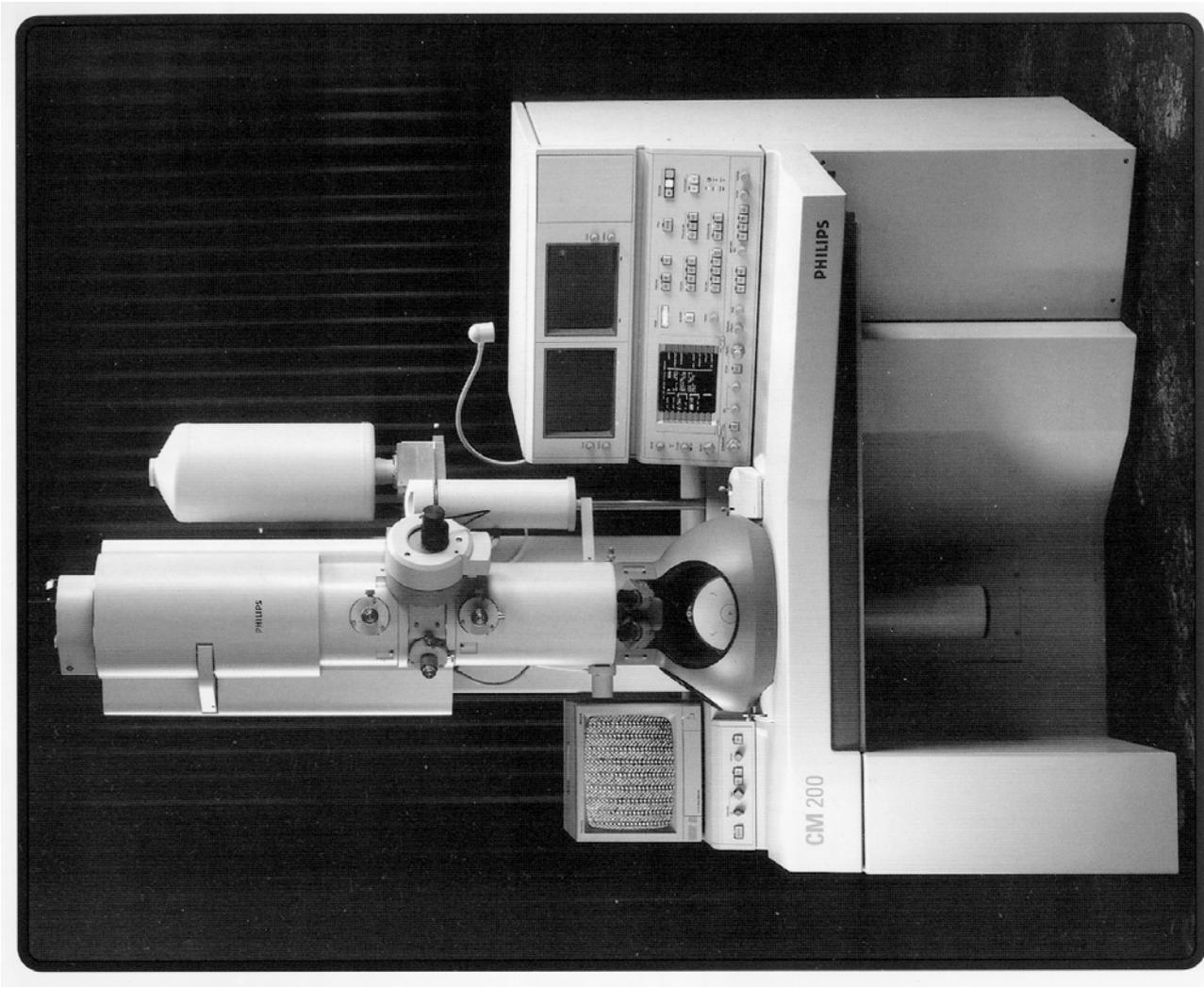


Fig. 4a. A transmission electron microscope dedicated to investigations in the material sciences – the Philips CM200.

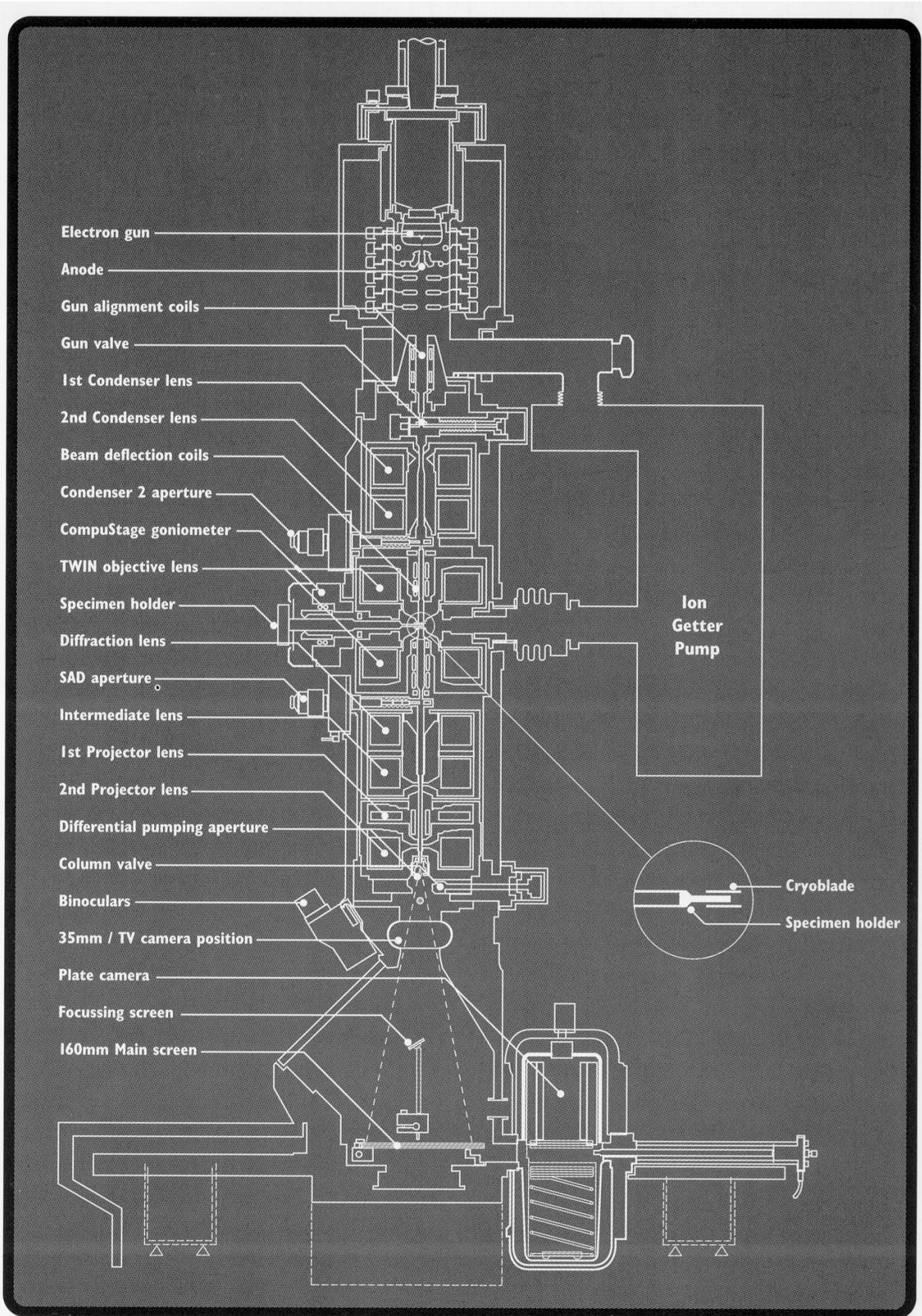
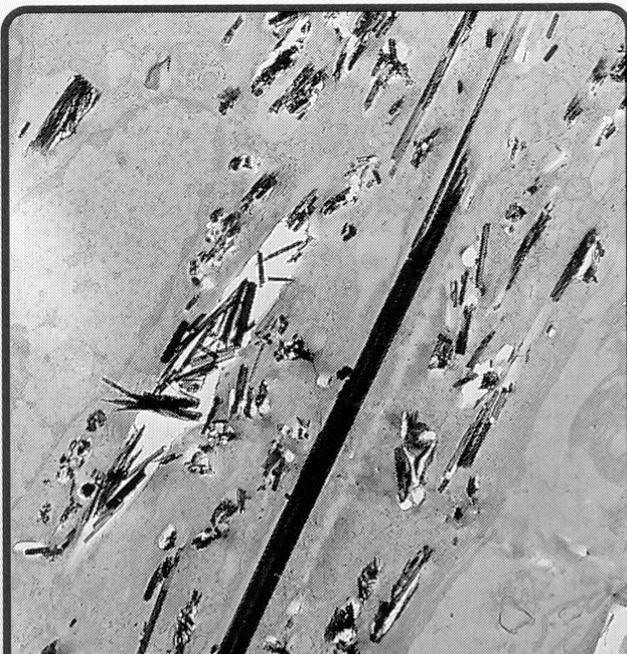
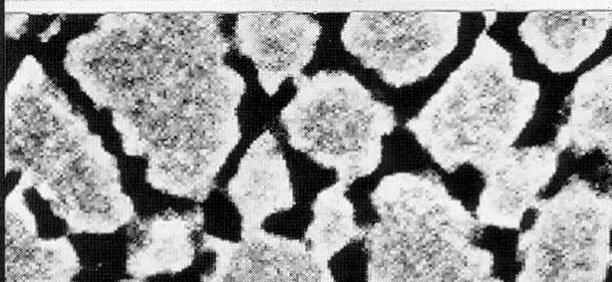
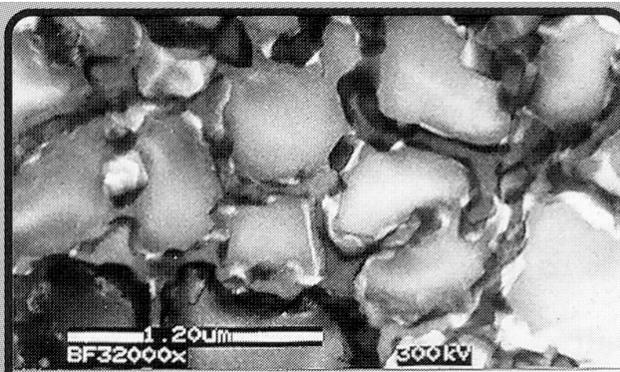


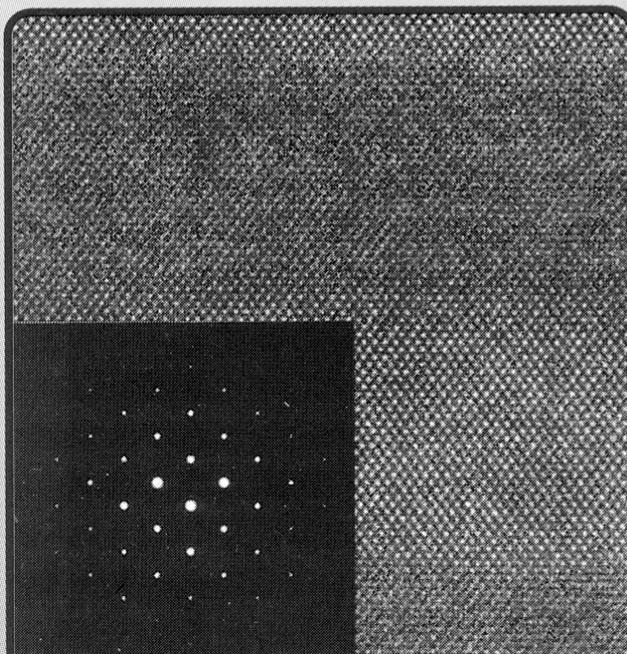
Fig. 8 Cross-section of the column of a modern transmission electron microscope.



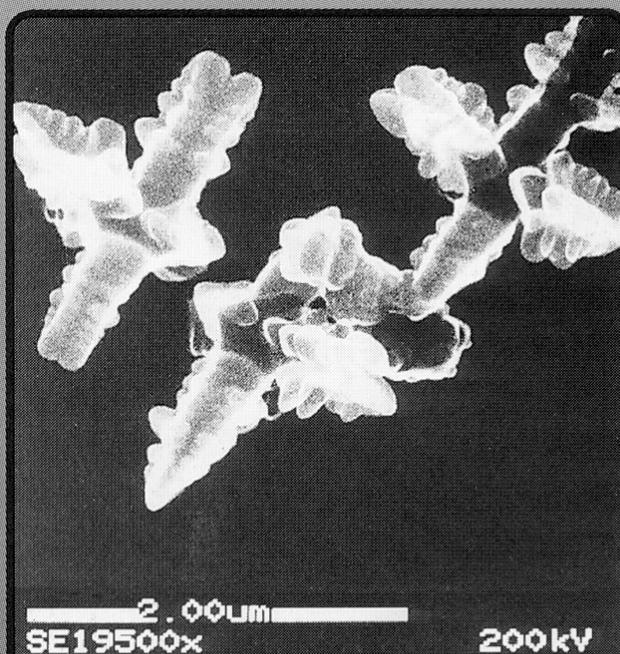
Asbestos fibres in lung tissue from lung cancer patient.



Bright-field STEM and digital Mo-X-ray image from NiMo.



High resolution TEM and diffraction pattern from Si-Si₂Ge multi-layer.



Secondary electron image of TiN particles in steel.

9.2. ARTEFATOS NAS IMAGENS DE MET

As imagens no MET são alteradas devido à artefatos similares àqueles vistos para o MEV.

A amostra pode sofrer CARREGAMENTO se não for convenientemente preparada e aterrada ao suporte de amostras. Caso necessário, pode-se fazer um recobrimento com material condutor após o afinamento da amostra. Pode-se usar carbono, ouro, etc.

A amostra pode sofrer CONTAMINAÇÃO devido à impurezas presentes no sistema devido a problemas no sistema de vácuo ou à limpeza inadequada dos componentes do MET.

A amostra pode sofrer AQUECIMENTO e aumentar o depósito de contaminantes. Para evitar este problema, pode-se fazer o recobrimento da amostra com material condutor ou utilizar um suporte de amostras refrigerado com nitrogênio líquido.

De qualquer forma, a correta preparação das amostras pode significar mais de 2/3 do trabalho em MET e ser a diferença em obter ou não bons resultados.

9.3. MODOS DE OPERAÇÃO DO MET

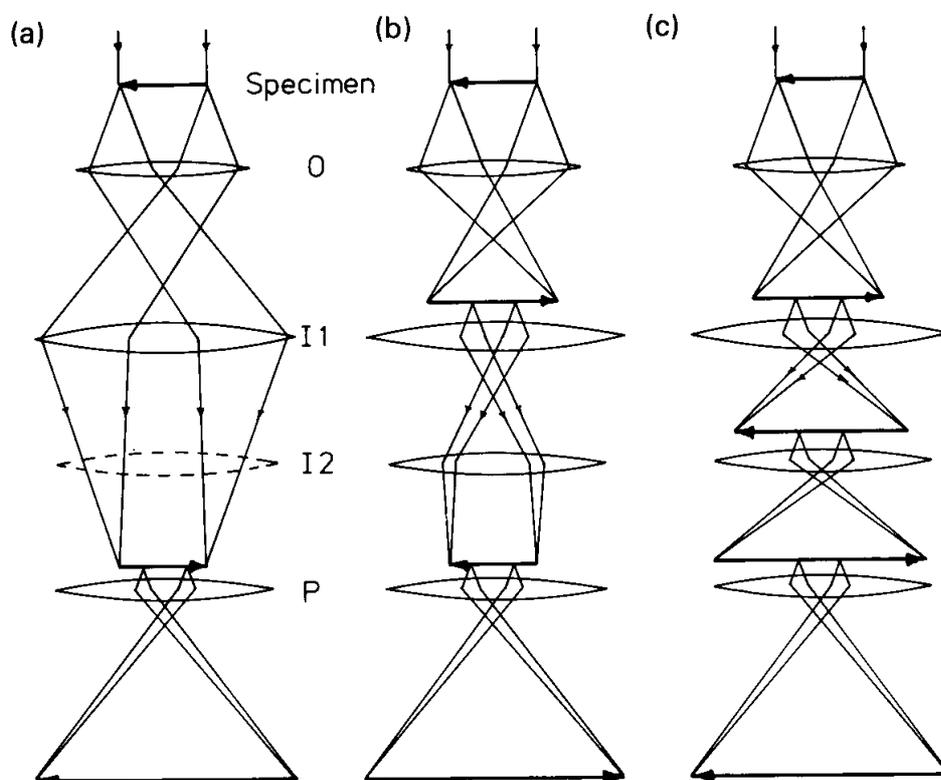


Fig. 23. Three of the commonly used optical configurations of the JEOL JEM200CX microscope, illustrating the many image inversions which may occur. (a) At low magnification the second intermediate lens (I2) is switched off. (b) At intermediate magnifications all lenses are on and there are three image inversions. (c) At high magnification all lenses are strongly excited and there are four image inversions.

9.4. FIGURAS DE DIFRAÇÃO EM MET

Todos os microscópios de transmissão oferecem a possibilidade de visualizar-se o padrão de difração (*diffraction pattern*) que inevitavelmente se forma no plano focal atrás da lente objetiva (*back focal plane*), como mostrado na figura 22(a) abaixo.

Mesmo se o usuário não tiver interesse por difração de elétrons, esta facilidade é útil no alinhamento da abertura da objetiva (*objective aperture*).

Em operação normal o padrão de difração não é normalmente visto porque a Primeira Lente Intermediária (I1) está focalizada na primeira imagem intermediária, e isto é o que finalmente é projetado na tela.

A mudança da excitação das Lentes Intermediárias pode levar à projeção dos padrões de difração magnificados na tela. Isto é tão frequente que é feito por um simples botão no controle do MET e o usuário pode ver o padrão de difração em qualquer momento. O diagrama de raios na difração pode ser visto na figura 24(b) abaixo.

A excitação da lente objetiva não é alterada na mudança entre condição de imagem e condição de difração.

A *objective aperture* deve ser removida para visualização do padrão de difração inteiro. Uma *Selected Area Diffraction Aperture* (SAD ou SAA) no plano da primeira imagem intermediária pode ser usada para selecionar a região do objeto da qual o padrão de difração é formado.

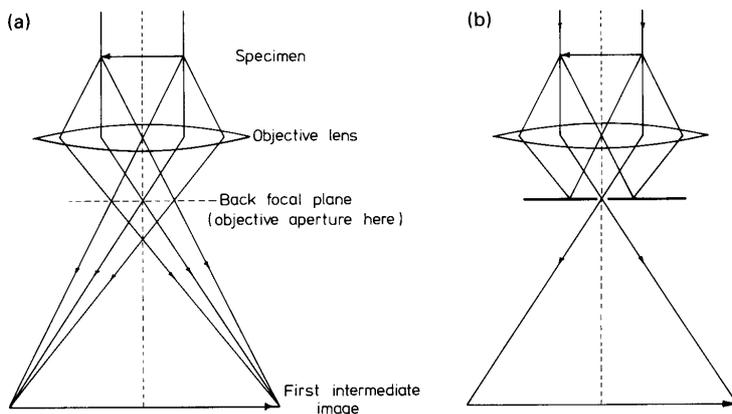


Fig. 22. The objective lens. The main function of this lens is to form a magnified first intermediate image. In the back focal plane of the lens a diffraction pattern is formed. (a) All rays leaving the specimen parallel go through a single point in the diffraction pattern. (b) Insertion of an objective aperture in the back focal plane eliminates diffracted electrons from the image.

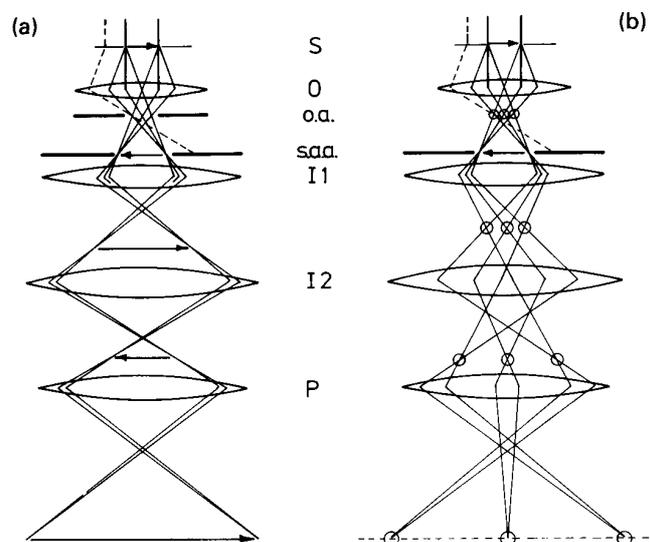


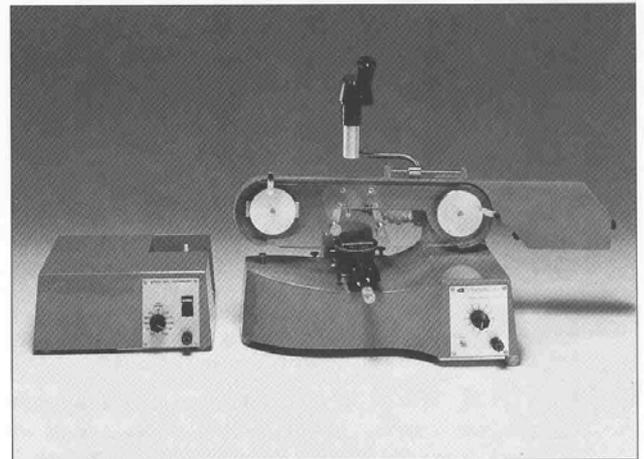
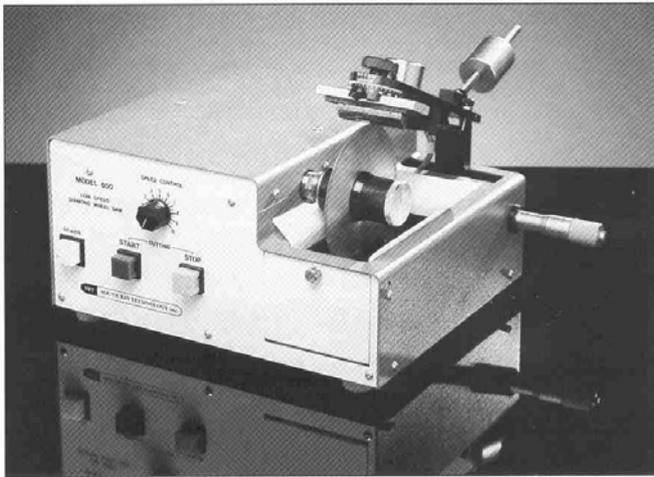
Fig. 24. A comparison of optical configurations in (a) high magnification imaging and (b) selected area diffraction. It should be noted that (a) is essentially the same as Fig. 23(c). The ray indicated by broken lines in both (a) and (b) is stopped by the selected area aperture (s.a.a.), illustrating that the diffraction pattern in (b) can arise only from the selected area.

9.5. PREPARAÇÃO DE AMOSTRAS PARA MET

As amostras precisam ser seccionadas, polidas e afinadas até a transparência para elétrons. Este procedimento de preparação das amostras corresponde, sem dúvida, à etapa para trabalhosa e consumidora de tempo nas análises em MET.

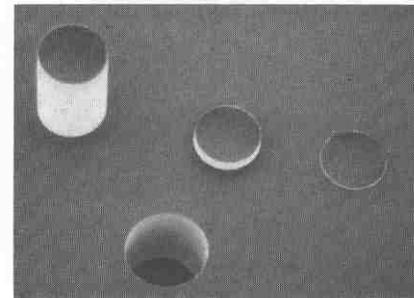
Os procedimentos adequados para preparação de uma certa amostra devem ser desenvolvidos especificamente para aquelas amostras, apesar de apresentarem características muitas vezes comuns.

9.5.1. Corte e seccionamento das amostras

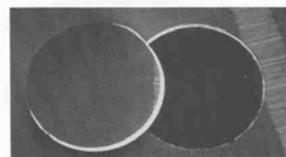
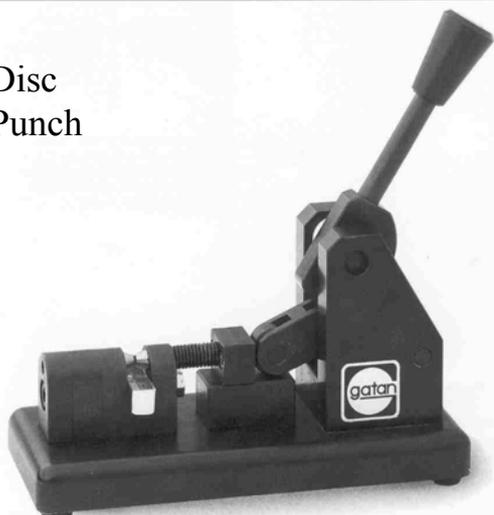


Model 850 with Model 85030 Abrasive Recirculating System and Model 85040 Microscope

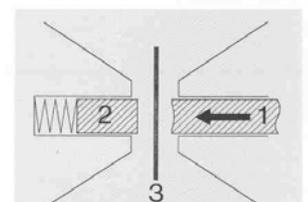
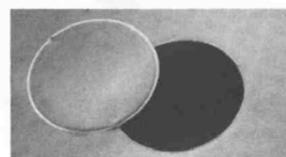
Ultrasonic
Disc Cutter



Disc
Punch



SEM micrograph of a 3 mm disc punched from near the cutting edge of a razor blade (top) and a similar disc punched subsequently from a 20µm thick aluminum foil (bottom)



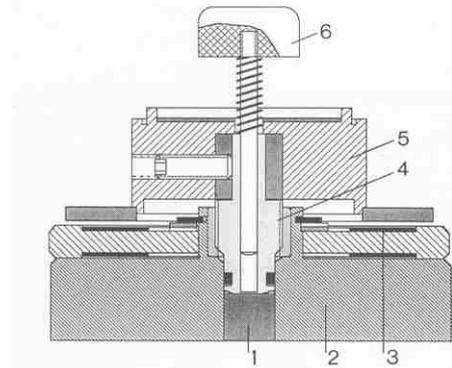
Essential Parts of Model 659 Disc Punch shown schematically:
1. Cutting piston with dimple in face
2. Spring-loaded support piston
3. Specimen (foil, sheet)

9.5.2. Lixamento, polimento e afinamento

Disc Grinder

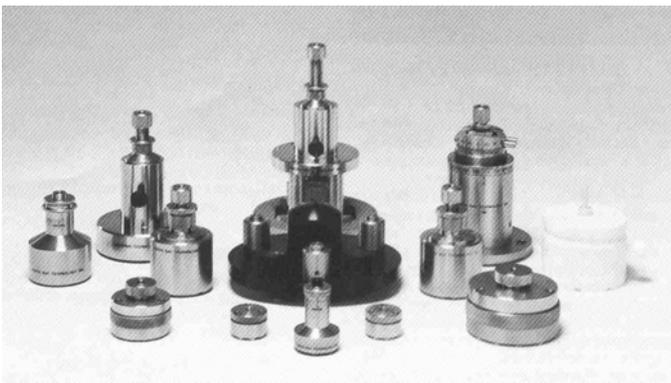


Disc Grinder



Cross section of Disc Grinder: 1 specimen mount, 2 base, 3 scale, 4 precision drive screw, 5 control knob, 6 specimen mount ejector.

Disc Grinder



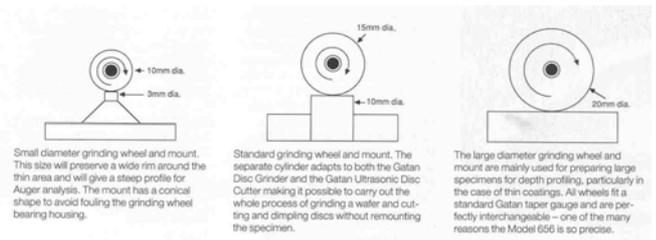
Tripod (Disc Grinder)



Dimple Grinder (Dimpler)

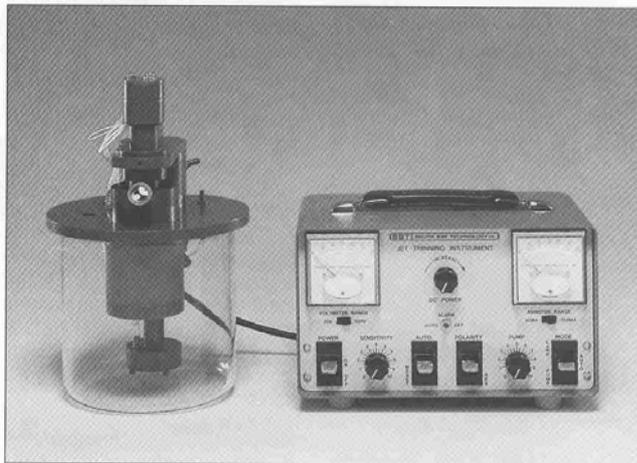


Dimple Grinder (Dimpler)



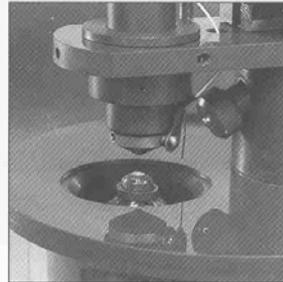
9.5.3. Afinamento final por Jet Polisher e Ion Milling

Jet Polisher (jato eletroquímico)

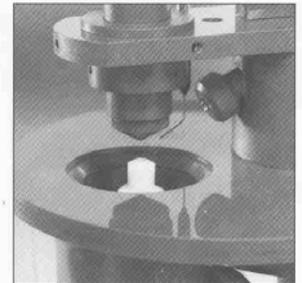


Model 550D

Jet Polisher (jato eletroquímico)

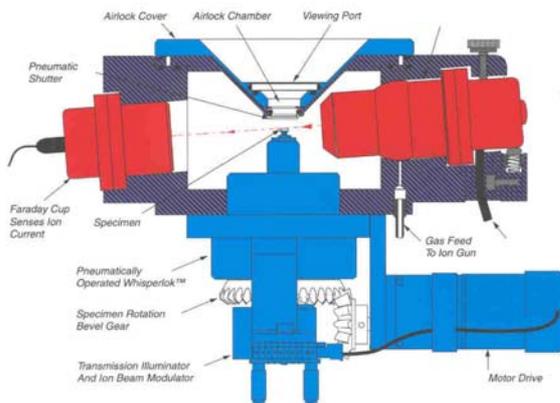


Close-up of electrolytic pedestal/jet nozzle



Close-up of chemical pedestal/jet nozzle

Ion Milling (afinamento iônico)



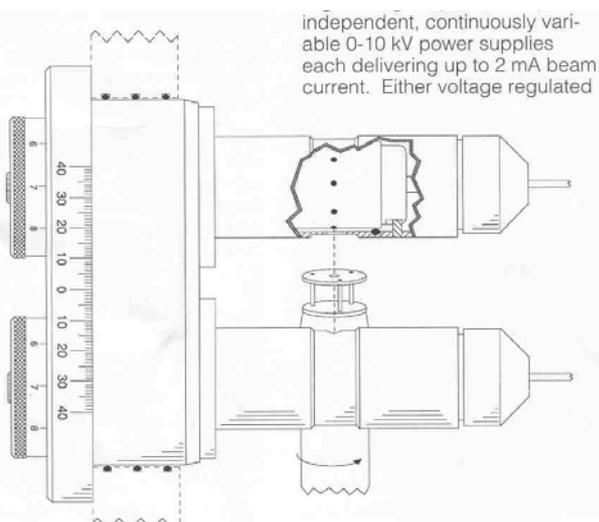
Schematic illustration of the PIPS™ work chamber showing the Panning ion gun, the specimen exchange mechanism, the Faraday cup, the airlock and the specimen post.

Dual Ion Milling (afinamento iônico)



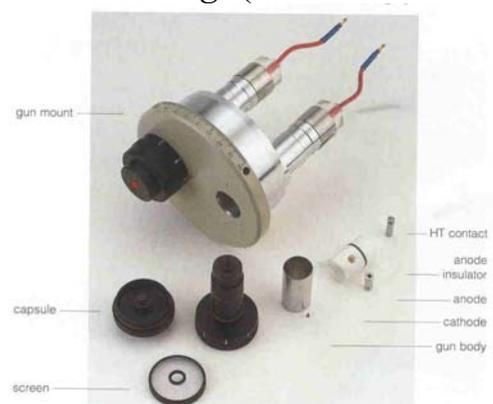
The Laser Autoterminator Control Unit operates with one or two Laser Head assemblies (which seat over the airlock capsule) to provide automatic termination of milling.

Dual Ion Milling (afinamento iônico)



As each cathode aperture wears out, a fresh aperture is simply moved into position by rotating an external knob. This is achieved without breaking the vacuum.

Dual Ion Milling (afinamento iônico)



The simple, effective design of the RIBE Octogun™ makes cleaning and maintenance easy.

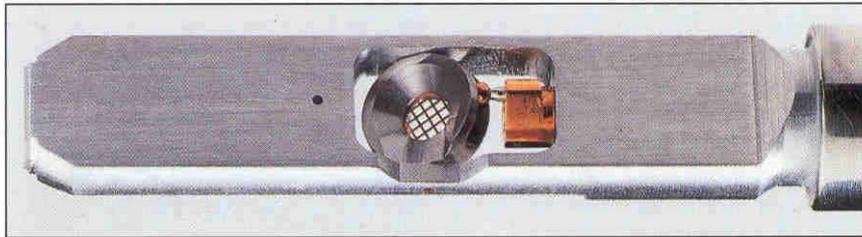


Close-up views of a cut away DuoMill™ illustrate the operation of the Whisperlok™. In the lower, working position (left) the sputter shield prevents material from the specimen coating the airlock capsule window. Simply by pressing the "Raise" switch the specimen holder is gently moved up into the airlock (center). Once in position, the "Air" button is pressed to admit air to the airlock capsule which can then be removed (right).

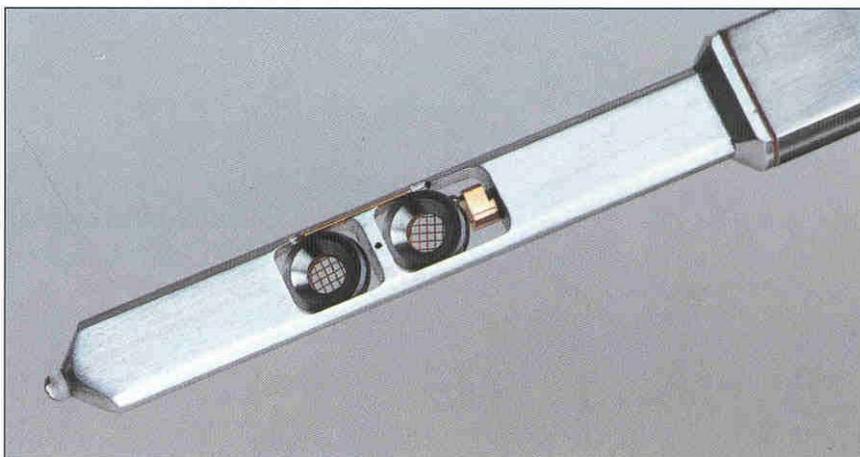
9.5.4. Exemplos de suportes de amostras para MET



Double Tilt Analytical Holder with optional motor drive for second tilt movement.

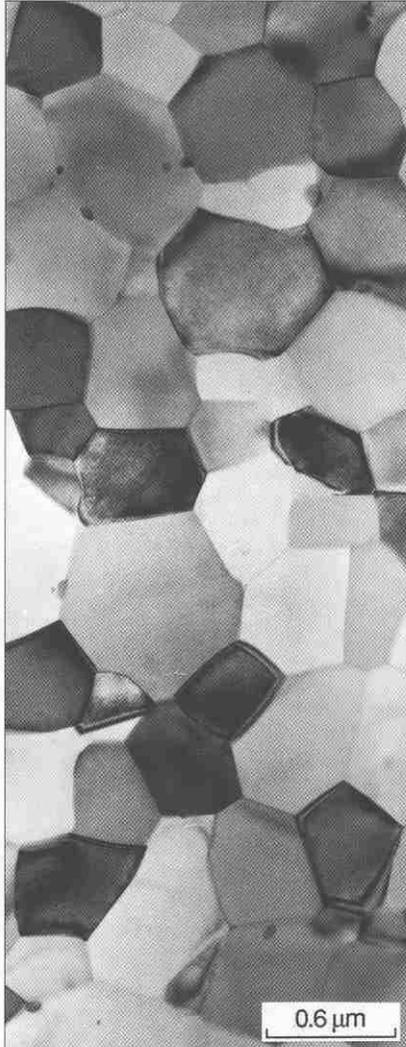


Tip of the Model 646 Double Tilt Analytical Holder, showing the ToggleTilt™ drive for the second tilt movement. Note the miniature Faraday cup and the cut-outs in the specimen cup and surrounding tip which minimize shadowing of the X-ray signal.

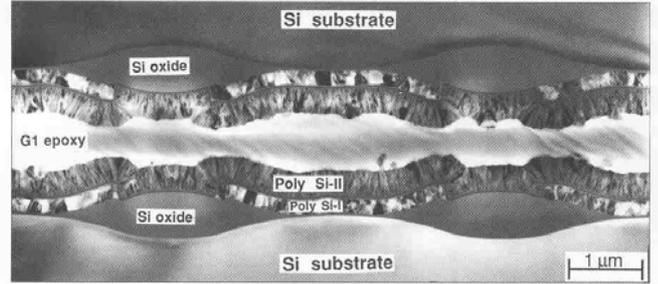


Tip of the Twin Double Tilt Holder, showing the ToggleTilt™ drive for the second tilt movement and the miniature Faraday cup between the two specimen cups.

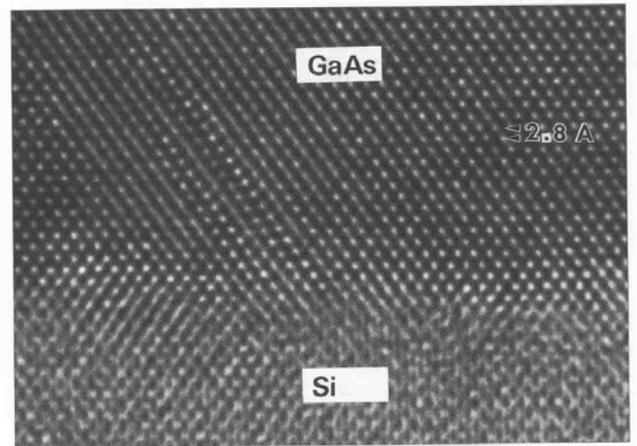
EXEMPLOS DE ANÁLISE EM MET



Al 0.5%Cu thinned on a silicon substrate at 3keV and 4°. Thinning time from a 5 m thick dimpled specimen was 20min.



Cross section through an IC device. Thinning time at 3kV 25mins.



GaAs/Si interface prepared with the Model 600 DuoMill™. Micrograph taken by Robert Hull on the Atomic Resolution Microscope, National EM Center, Berkeley. Sample grown by S. M. Kuch, Stanford University.



SiC whisker reinforced Al₂O₃ composite prepared with Model 600 DuoMill™. Material courtesy John Porter, Rockwell International Science Center. Magnification ~x60,000.