Observation of Dislocations in a Conventional Scanning Electron Microscope

P. Guyot

LTPCM, laboratoire associé au CNRS, n° 29, Enseeg, Domaine Universitaire BP 44 — 38401 Saint-Martin-d'Heres

J. M. Gjurasevic

Aluminium Pechiney, Centre de Recherches — 38340 Voreppe

(Z. Naturforsch. **29 a**, 1381—1382 [1974]; received June 15, 1974)

A special transmission device has been adapted on a scanning electron microscope, in which the image is formed by secondary electrons generated by the transmitted ones in an underlying foil of metal. It is shown how this stage allows diffraction patterns and dislocations to be imaged in a conventional microscope (30 kV voltage, hairpin tungsten filament).

During the recent past, dislocations have been observed in scanning electron microscopy, either by transmission or reflexion on thin foils, and with sufficiently bright beams to get a convenient signal to noise ratio. This is easily demonstrated by considering that the beam diameter d and divergence α must be respectively lower than the linear and angular image width of the dislocation, and that the beam intensity is proportional to $Bd^2\alpha^2$, B being the brightness of the gun, Clarke et Howie [1], Guyot [2]. Consequently, dislocations have been observed only with high voltages (80 or 100 kV), LaB_6 or pointed filaments. In conventional apparatus, i. e. 30 kV acceleration voltage and classical hairpin tungsten filament, individual dislocations have not been resolved. We show here that their observation can nevertheless be made possible in such a microscope providing to introduce some beam current magnification in the transmission mode (STEM).

For that purpose a simple transmission stub has been realised. The scanning transmission micrograph is generated according to the Nemanic and Everhart's device [3]: as shown on Fig. 1, the 30 kV transmitted electrons generate secondary electrons in a block of Al or Pt underlying the transmission stage, and these secondaries are collected on the metal-coated scintillator-photomultiplier standard detector. The stub is about 1 mm from the bottom of the objective lens: this short working distance allows to screen the secondary electrons generated at the top surface of the specimen from the detector and to increase the beam current at the specimen. An objective aperture and a collection aperture determine the divergence of the incident, 2α, and the transmitted, 2β, beam. In our simplified stage, the collection aperture is attached to the specimen stage and does not allow for dark field images to be performed. The secondary emission foil being inclined of 60° with respect to the horizontal, current measurements [3] have shown a high conversion efficiency of this foil, characterised by a secondary emission coefficient between 1.25 and 1.3. We show below that diffraction patterns and dislocation images can be obtained with a conventional scanning microscope equiped with such a transmission-detection stage.

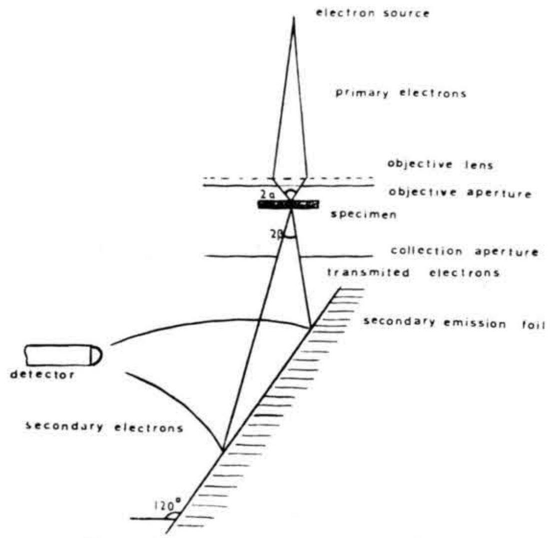

Fig. 1. Transmission — detection device.

When taking transmitted images of cristalline specimens, the following features based on the reciprocity theorem, Cowley [4], have to be considered:

- the illumination angle 2α is equivalent to the objective aperture angle in conventional electron microscopy (CTEM),
- the collection aperture 2β is equivalent to the illumination angle in CTEM.

Consequently, diffraction patterns with good angular resolution necessitate small β angle, while the image resolution is in principle not affected by it due to the fact that the energy loss in the specimen is unimportant (no focussing performed on the transmitted beam).

Examples of aluminium diffraction patterns taken in rocking mode (no objective aperture) in a Cambridge S4 stereoscan are shown on Figs. 2 and 3 *; on Fig. 2, the collection diaphragm is circular, determing an angle $2\beta = 0.030$ rad; on Fig. 3 the diaphragm is a slot with $2\beta = 0.050 \text{ rad} \times 0.30$ rad:

Reprint requests to Dr. P. Guyot, LTPCM, laboratoire associéau CNRS, n° 29, Enseeg, Domaine Universitaire, *BP 44 — 38401 Saint-Martin-d'Heres*, Frankreich.

* Figures 2, 3, 4 and 5 on page 1382 a, b.

the pattern is of poorer quality, but the large collection angle allows bands of the channeling pattern to be distinguished.

In the image mode, small objective apertures must be used to produce minimum probe size. In our working conditions, with a 50 μm aperture the beam diameter at the specimen is of $\sim$100 nm.

STEM dislocations images in aluminium are shown on Figs. 4 a and b. It has first been checked by 100 kV CTEM that they are not topographic contrast effects at the excit surface of the specimen: as shown on Figs. 4 a and 5, the same dislocation details, indicated by letters, are found on both micrographs.

Figure 4 a has been obtained with an objective diaphragm of 200 μm, and 4 b with 50 μm; it is impossible to decrease it below 50 μm, in order to get a smaller beam diameter, due to the increase in noise. Similarly we are obliged to use a large collection aperture (above slot diaphragm) to receive a sufficient signal to noise ratio. With such a collecting aperture the images are of multibeam origin.

The dislocation images show the expected black-white contrast, with an image width of about 30 nm.

Attempts to form pure bright field images have been, for the above reason, unsuccessful. In the future, use of pointed filaments and modification of the transmission stage should allow bright and dark fields to be performed, making accessible the cristallographic identification of dislocations.

[1] D. R. Clarke and A. Howie, Phil. Mag. **24**, 959 [1971].
[2] P. Guyot, Rev. Phys. Appl. **9**, 393 [1974].
[3] M. K. Nemanic and T. E. Everhart, Scanning Electron Microscopy **26**, Chicago 1973.
[4] J. M. Cowley, Appl. Phys. Lett. **15**, 58 [1969].

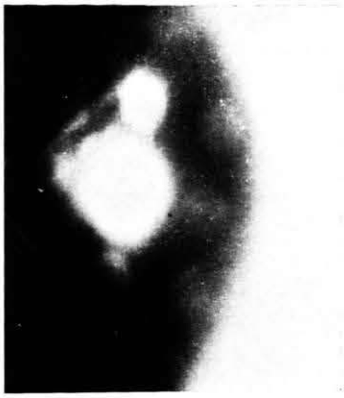

Fig. 2. [100] diffraction pattern in Al. $2\beta = 3 \cdot 10^{-2}$ rad.

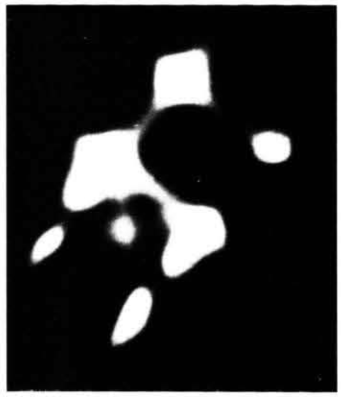

Fig. 3. [100] diffraction pattern and channeling pattern $2\beta = 50 \cdot 10^{-3} \times 30 \cdot 10^{-2}$ rad.

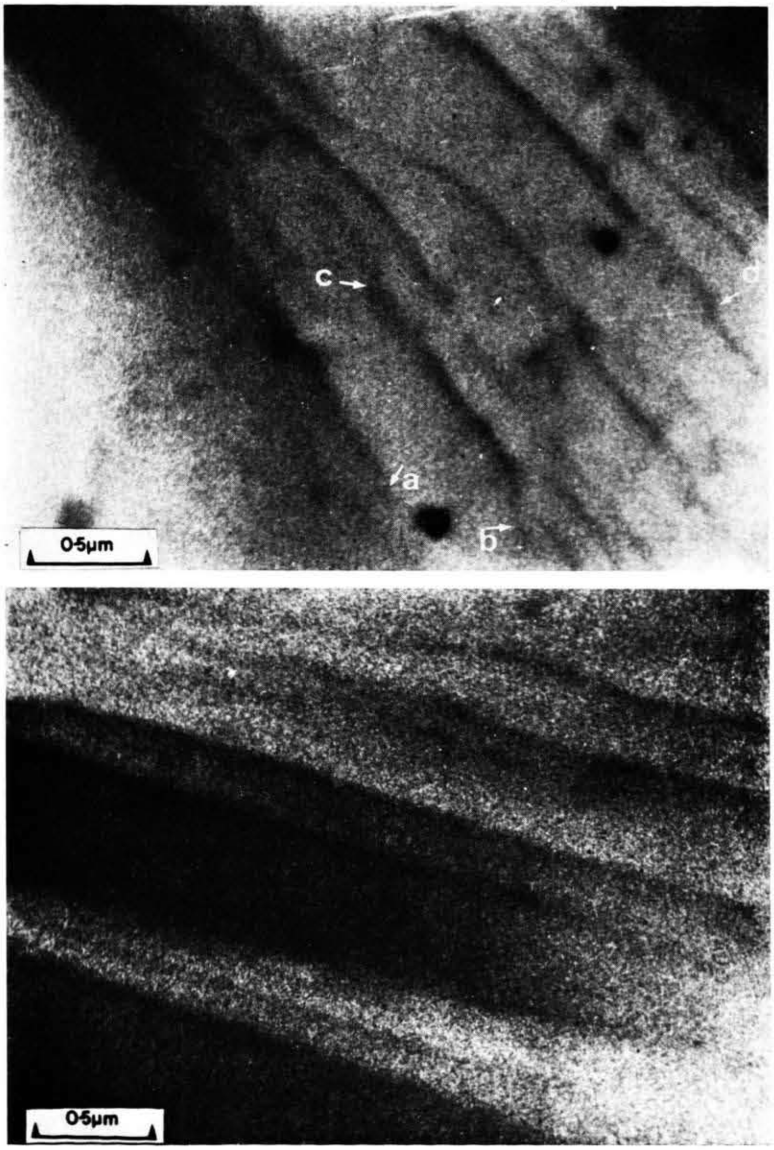

Fig. 4. STEM images of dislocations in Al. 30 kV hairpin filament a) $2\alpha = 10^{-1}$ rad; b) $2\alpha = 25 \cdot 10^{-3}$ rad.

Fig.5. CTEM micrograph. 100 kV. Same specimen as for micrograph 4 a.