Diffraction Patterns Obtained by Scanning Electron Microscope

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Dedicated to Prof. Dr. K. MOLIÈRE *on his 60-th birthday*

Kossel-Möllenstedt and Kikuchi patterns are obtained by transmission scanning electron microscopy and compared with those obtained by the convergent beam technique from the same portion of the specimen. The identity of corresponding patterns obtained by both techniques shows the validity of the reciprocal theorem in electron diffraction for both elastic and inelastic scattering. The variations of Kossel-Möllenstedt patterns with the conditions of the incident beam and the position of the detector are also shown.

1. Introduction

COATES [1] has obtained "pseudo-Kikuchi patterns" from single crystals of galium arsenide, germanium and silicon by scanning electron microscopy (SEM). These patterns have subsequently been interpreted by BOOKER, SHAW, WHELAN and HIRSCH [2] as due to the "inverse" electron channeling effect, although they did not mention the direct relationship between these patterns and the ordinary Kikuchi patterns.

An extention of the theory of one of the present authors (S. T.) [3] has shown that the same pattern as the ordinary Kikuchi pattern could be obtained by SEM, if proper experimental conditions would be chosen, as has been pointed out elsewhere [4, 5]. The reciprocity theorem, which was extended by POGANY and TURNER [6] to include inelastic scattering, assures quite generally the equivalence between the patterns obtained by conventional electron microscopy (CEM) and those by SEM, as has also been pointed out by TAKAGI [5] and FUJIMOTO et al. [7].

In a previous short communication [4] the authors have presented diffraction patterns obtained by transmission SEM which are equivalent to the Kossel-Möllenstedt patterns (K-M patterns) or to the Kikuchi patterns obtained by CEM. The purpose of the present paper is to describe the experimental conditions in more detail, to present diffraction patterns obtained using experimental conditions which will give equivalent patterns in both arrangements and to show the variation of these patterns with changes in the diffraction conditions and in the detector position.

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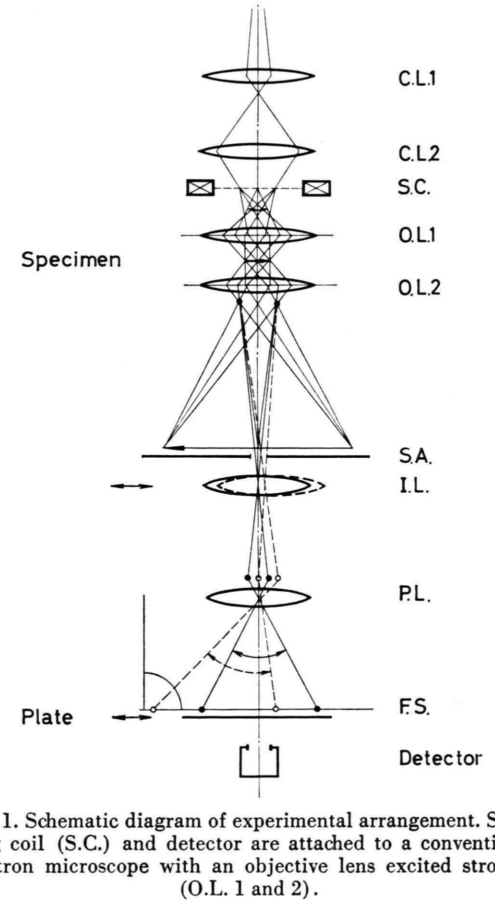

Fig. 1. Schematic diagram of experimental arrangement. Scanning coil (S.C.) and detector are attached to a conventional electron microscope with an objective lens excited strongly (O.L. 1 and 2).

2. Experimental Arrangement

The schematic diagram of experimental arrangement is shown in Figure 1. A JEM 100B, a conventional electron microscope having a scanning system (EM-ASID) between specimen and the second condenser lens (C.L. 2), was used. In the present experiment the scanning coil was displaced from the normal position along the optical axis towards the condenser lens.

For obtaining diffraction patterns by the scanning technique, the incident beam, whose angular divergence at the position of the specimen is kept smaller than 3×10^{-4} rad. by adjusting the current of the condenser lens, is deflected by the scanning coil (S. C.) and then, in the opposite direction, by the prefield of the objective lens which is excited strongly (O.L. 1), so that the same portion of the specimen is irradiated with angular scanning.

For SEM a detector with a 3 mm diameter window was placed under the usual fluorescent screen (F.S.) used in observing conventional diffraction patterns. This screen was removed when the SEM detector was in use. The detector was a photomultiplier with a fluorescent screen and was connected to a cathode-ray tube, on which the SEM images were displayed.

The electron lens system between the specimen and the detector shown in Fig. 1 was operated during scanning. The objective lens produced the image of the specimen on the selected area aperture (S.A.) which limited the effective area of specimen within a range variabe from 0.2 to 4 μm in diameter. The intermediate (I.L.) and the projector lens (P.L.) formed the image of the diffraction pattern at the position of the fluorescent screen and the detector.

The effective camera length could be varied from 20 cm to 120 cm by changing the intermediate-lens current. By this procedure the effective angular width of the detector window viewed from the position of the specimen was varied from 0.015 to 0.0025 rad. Furthermore the effective position of the detector relative to the optical axis could be varied by shifting the intermediate lens perpendicular to the optical axis, as is shown by the broken lines in Figure 1.

For the convergent beam technique (CBT), the scanning coil was switched off, the incident beam was converged on the specimen by utilizing the prefield of the objective lens (O.L. 1) and adjusting the current of the condenser lens. The K-M and Kikuchi patterns were observed on the fluorescent screen or taken as photographs as in conventional electron microscopy. The energy of electrons was 80 keV throughout the present experiments.

3. Reciprocity

According to the reciprocity theorem of the scattering problem, the diffraction process of electrons scattered elastically in SEM is reciprocal to that in usual electron diffraction and this theorem can be extended to the diffraction process including inelastic scattering [6], when the variation of the wave vector of the electrons due to the inelastic scattering is very small. Therefore, the diffraction pattern observed by transmission SEM is identical with that observed by CBT, that is, the diffraction patterns obtained by the experimental arrangement indicated in the upper side of (a) in Fig. 2 is equivalent to that of lower side, where α is the opening angle of the detector in SEM and the divergence of the incident beam in CBT. Here we must notice that, in

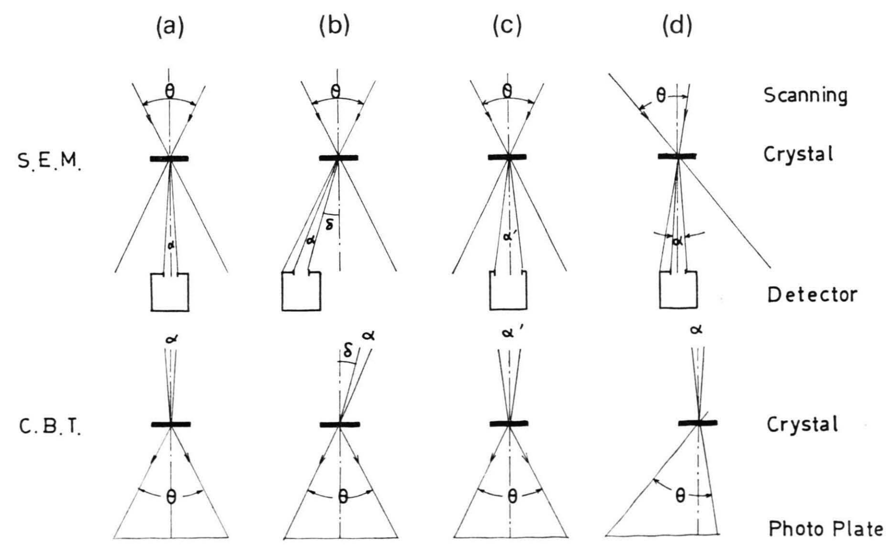

Fig. 2. Various experimental conditions in SEM and corresponding reciprocal ones in CBT (see text).

order to obtain exactly equivalent conditions in SEM and in CEM, the direction of the beam must be reversed with respect to the specimen or the specimen must be put upside down when switching over from one arrangement to the other. However, it is not necessary for centrosymmetric crystals used in the present experiment.

In Fig. 3(a) *, the K-M patterns obtained from the same portion of thin silicon film parallel to the 111 plane by SEM and CBT are shown, where the incident beam excites simultaneously the two 220 reflections. Both patterns are identical, showing that the reciprocity theorem holds in the case of elastic scattering.

Figure 3(b) shows the SEM and CBT patterns obtained from a thicker crystal of silicon and Fig. 3(c) the patterns from a much thicker part than in the case (b). In (b) the K-M patterns due to the incident and Laue spots are excluded from the figure by shifting the detector to avoid the disturbing effect of these stronger patterns. It must be mentioned in this connection that, when the diffraction patterns were observed on the fluorescent screen during scanning, the Bragg spots could not be seen due to the smearing effect of scanning, while the Kikuchi pattern was clearly observed, since the position of the latter pattern depends not on the incident beam direction but on the crystal orientation.

Comparing the diffraction pattern observed by SEM and that by CBT from the same part of the crystal, we see that both of the Kikuchi bands in the SEM and in the CBT patterns from a thin crystal shown in Fig. 3(b) are of excess type, on the other hand, those in the patterns from a thicker crystal shown in Fig. 3(c) are of defect type. These results indicate that the effects of inelastic scattering and absorption processes are identical in SEM and CBT.

4. Kossel-Möllenstedt Patterns Observed by SEM

In this section, the variation of the K-M patterns with a change in the direction of incident beam and in the detector position is studied. The experimental arrangements are shown schematically in the upper side of Figure 2. In order to understand easily the variation of the diffraction pattern observed in each case, the corresponding reciprocal arrangements are shown in the lower part of Figure 2.

Case (a) is the standard case and case (b) is that where the detector is displaced. In the present experiment the displacement was carried out by a shift of the intermediate lens perpendicular to the optical axis as is mentioned before. The variation of the K-M patterns due to the displacement of the detector is shown in Figure 4. The specimen is a thin silicon film with the surface parallel to the 110 plane. Comparing these three pictures with each other, we see that the circles corresponding to the incident and reflected spots shift with displacement of the detector, while the pattern inside each circle does not move. This situation can be easily understood by considering the reciprocal cases shown in Figure 2.

Figure 5 shows the intensity distribution inside each K-M circle observed by successive displacement of the detecor, where Roman numerals indicate the serial numbers of the original pictures, of which II, III and V are shown in Figure 4. In Fig. 5, corresponding K-M circles in the original pictures are cut out and connected to show an extended angular display of the intensity. The whole pattern in Fig. 5 has a two fold axis of rotation and is mirror symmetric concerning the 001 and $\bar{1}10$ planes.

Figure 6 shows the change of the K-M patterns for various opening angles of the detector window. The camera lengths in the cases (a), (b), (c) and (d) are 20, 40, 80 and 120 cm, respectively, and the window is 3 mm in diameter. In these pictures, circles due to the Bragg reflection decrease with increase in camera length. In the case of extremely small opening angle, we observe a net of Bragg spots.

When the scanning is asymmetric as shown in Fig. 2(d), the K-M pattern observed on the cathode-ray tube displaces from the center, as is shown in Fig. 7, where (a) and (b) are the patterns observed in the cases of symmetric and asymmetric scanning, respectively. This result can also be understood easily by considering the reciprocal case shown in Figure 2.

5. Discussion

The results given in the preceeding sections show that the diffraction pattern obtained by angular scanning of a parallel beam is identical to that obtained by the convergent beam technique, and the reciprocity in electron diffraction including inelastic

* Figures 3—7 see p. 444 a—c.

scattering is manifested in the case of transmission experiment.

On the other hand, the so called "pseudo Kikuchi pattern" was mostly obtained by the reflection method, and moreover secondary emissions from the crystal were simultaneously measured by the detector in some cases. However, in view of the reciprocity theorem, the pattern, if formed by back-scattered primaries only, must be identical to a "high angle Kikuchi pattern" obtained by the reflection method [8] with a convergent incident beam instead of a usual parallel beam.

GOODMAN and LEHMPFUHL [9] have studied various diffraction phenomena by using CBT. The same kind of experiment can be done by the present SEM method. One of the advantages of the present method is that one can observe a map of intensity variation of each reflection with crystal orientation as shown in Fig. 5, not by changing the condition of incident beam, but merely by displacing the detector. The use of a selected area aperture is very convenient also in SEM for obtaining diffraction patterns from a small flat part of thin single-crystal films, which are liable to be bent or buckled.

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CBT SEM

a)

b)

c)

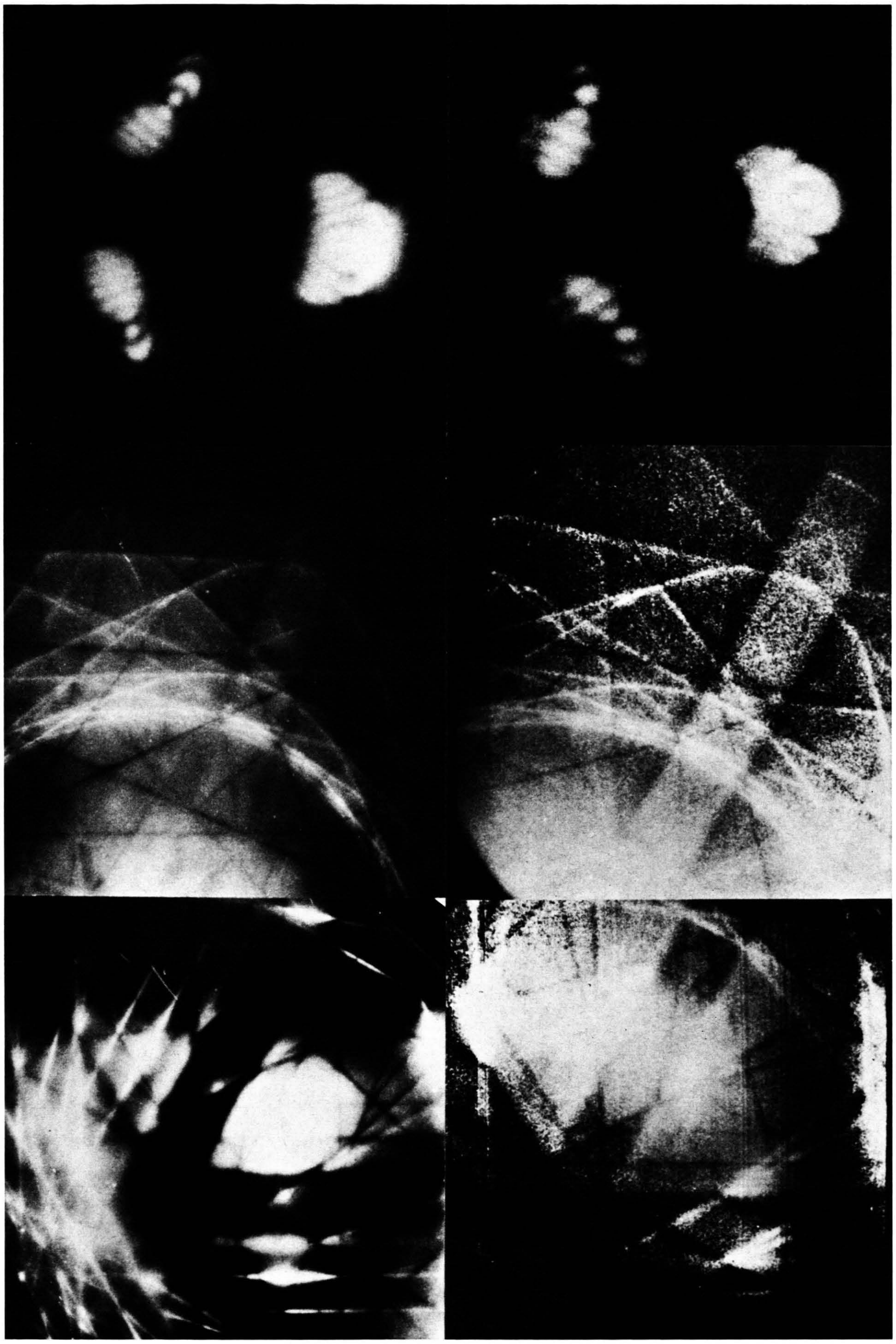


Fig. 3. SEM and CBT diffraction patterns obtained from a thin (a), a thick (b) and the thickest (c) film of silicon crystal.

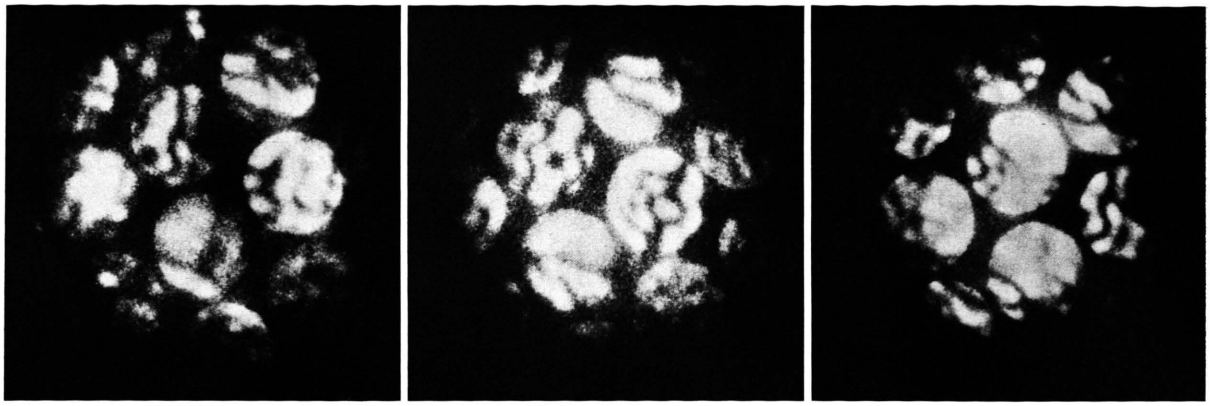


Fig. 4. Variation of K-M patterns with the shift of the detector. Roman numerals indicate the plate number which is used in Figure 5.

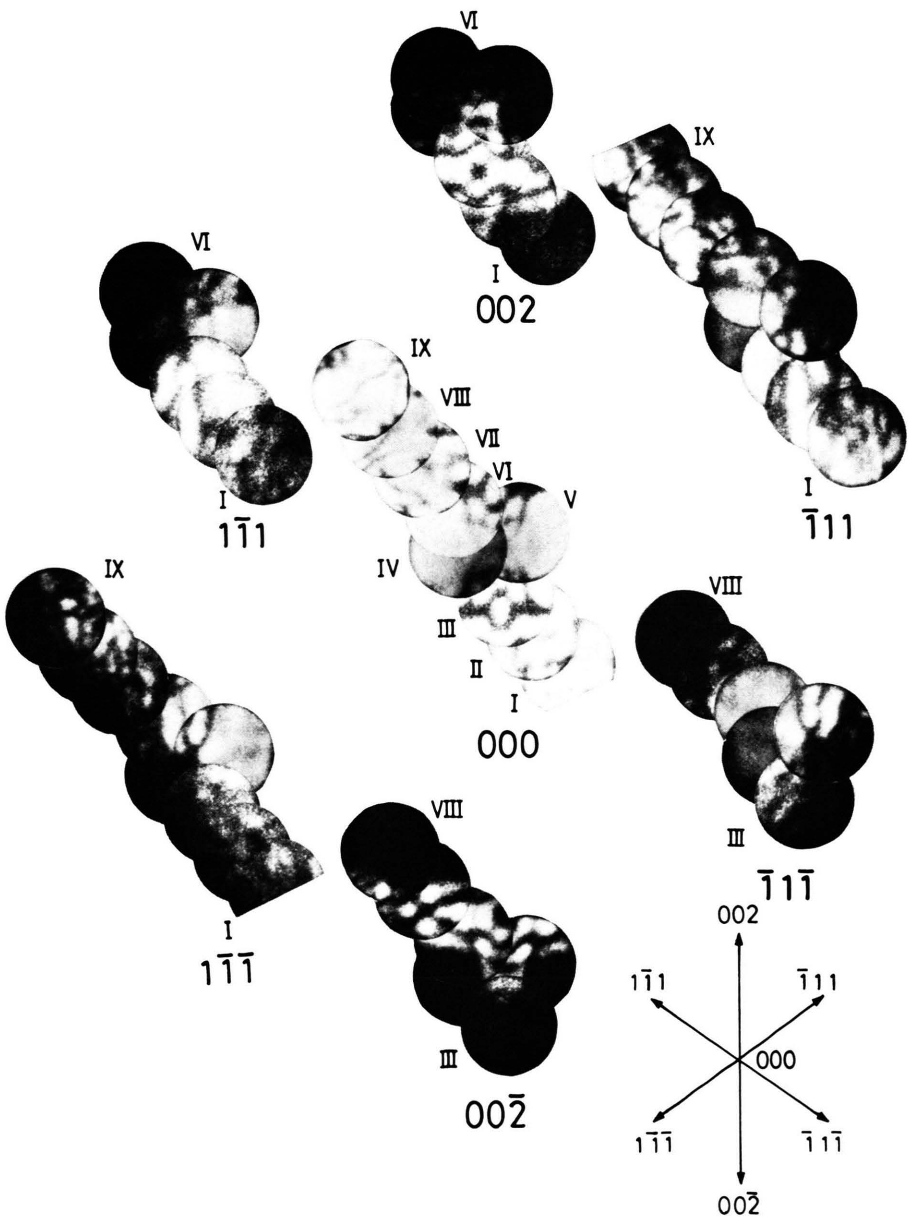
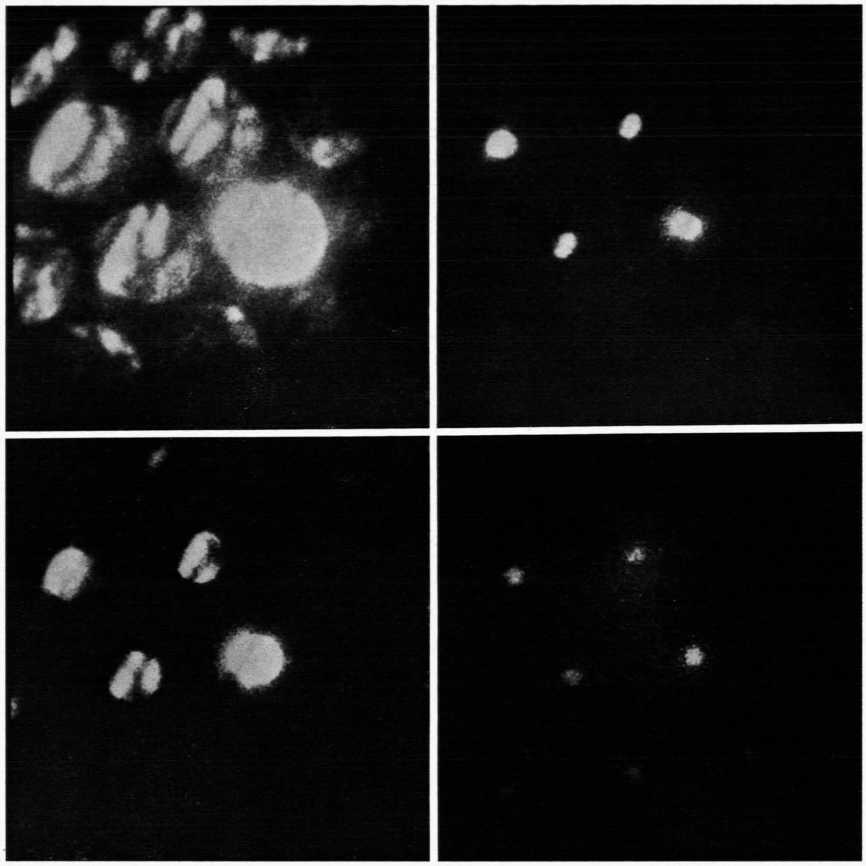


Fig. 5. Intensity distributions of each K-M circle observed by successive displacement of the detector. Roman numerals indicate the plate numbers. The pictures of II, III and V are shown in Figure 4.

a) c)

b) d)



↑ Fig. 6. Variation of K-M patterns with the opening angle of the detector window. The camera lengths in the cases (a), (b), (c) and (d) are 20, 40, 80 and 120 cm, respectively, and the window is 3 mm in diameter.

a)

b)

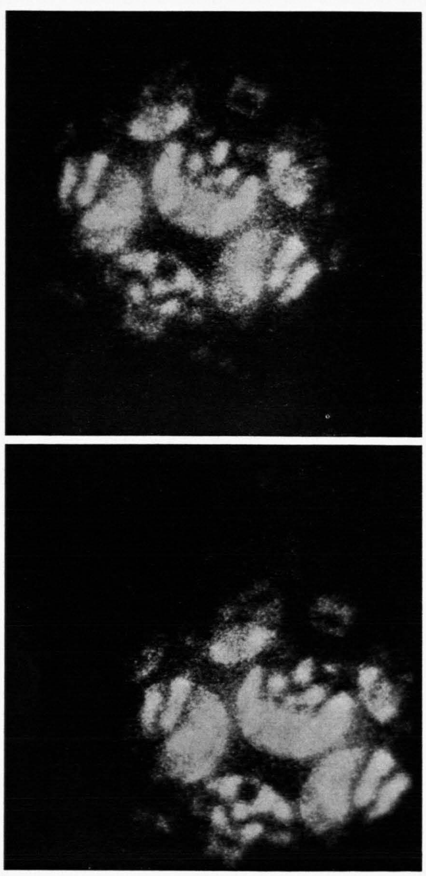


Fig. 7. K-M patterns observed in the case of symmetric (a) and asymmetric (b) scannings.