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ELECTRON MICROSCOPE STUDY OF
INTERLAMELLAR COMPOUNDS
OF GRAPHITE WITH BROMINE, IODINE
MONOCHLORIDE AND FERRIC CHLORIDE

by

S. AMELYNCKX, P. DELAVIGNETTE (CEN)
and M. HEERSCHAP (EURATOM)

1964



General Directorate Research and Training

Paper presented at the Sixth Biennial Conference on Carbon
Pittsburg (Pa.) USA, 17-21 June 1963

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Would you please note that the micrographs
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Investigated and discussed are interactions of a boundary dislocation and a normal dislocation ribbon as well as the widening of dislocation ribbons presumably by preferential diffusion of reactant along the ribbon.

The situation in the graphite-ferric chloride compound proved to be more complicated and the results not consistent. Evidence is given of isolated dislocations, which show no stacking fault contrast at one side. They separate however regions, resembling normal graphite, where normal dislocation ribbons are still present from regions where the contrast of the partials has become more diffuse, the partial dislocations assuming equidistant positions. It is therefore thought that these dislocations bound the intercalated ferric chloride layers. The widening of the contrast of the partials and their behaviour upon intercalation of FeCl_3 is interpreted as a result of the expansion of the interlayer spacing.

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ELECTRON MICROSCOPE STUDY OF INTERLAMELLAR COMPOUNDS OF GRAPHITE WITH BROMINE, IODINE MONOCHLORIDE AND FERRIC CHLORIDE (*)

1 — INTRODUCTION

Several substances such as potassium, bromine, iodine monochloride, ferric chloride and others (1) (2) (3) form lamellar compounds with graphite. In these compounds, which retain the aromatic planar graphite structure, the reactant is present in planar monolayers. The intercalated layers expand the spacing between the adjacent hexagon networks by a characteristic magnitude. Saunders, Ubbelohde and Young (4) concluded from the linear relationship between the fractional thickness increase and the fractional bromine uptake in their study of graphite-bromide that once a layer in the crystal compound begins to lose bromine it loses all of it and shrinks to the smaller spacing characteristic of graphite. This may mean that the process of intercalation may be considered as a shift of complete layers of foreign atoms or molecules between the hexagon networks into the crystal and out upon decomposition. The boundary of such a layer will be formed by a dislocation brought about by the presence of an excess plane of foreign material in the graphite lattice. The observation of these boundary dislocations by transmission electron microscopy, the study of their behaviour and their interactions may produce new information about the lamellar compounds of graphite, especially about the way in which residual reactants are retained in residue compounds.

Hennig (5) and Ubbelohde (6) suppose that the residual reactant is trapped at crystal imperfections. Maire and Mering (7) on the other hand suggest that in the case of bromine single layers of bromine are retained at the interfaces between imperfect and perfect layer planes. These authors observed X-ray lines of bromine in bromide residue compounds which showed the asymmetry of two dimensional layers.

Reported in this paper are the results of the electron microscope study of graphite-bromide, graphite-iodine monochloride and graphite-ferric chloride.

2 — EXPERIMENTAL PROCEDURE

Specimens were obtained by cleavage of natural single crystals of Madagascar and of purified graphite. These specimens were exposed to the vapours of the reactants only for a short time whereas in case of bromine and iodine monochloride most of the reactant was driven off by standing at room temperature in order to avoid severe damage of the crystals by intumescence, exfoliation and embrittlement.

Since the c-plane is also the cleavage plane the foil plane will be parallel to the glide plane. Dislocation arrangements are therefore in the plane of observation.

The observations are made with a Philips EM-200 electron microscope operated at 100 kV.

(*) Work performed at the Solid State Physics Department of the CEN, Mol (Belgium).

3 — STRUCTURE OF GRAPHITE

Graphite crystallizes in a layer structure. Alternate layers are displaced relative to each other and therefore not superimposable. The most common structure of graphite consists of a hexagonal stacking of sheets of hexagonally linked carbon atoms. In figs. 1a and 1c are presented respectively the projection of this structure on a plane perpendicular to the c-plane and the projection on the c-plane. The stacking sequence can be designated by a b a b ... The two positions a and b are indicated in fig. 1c. A third position c is however possible. The sequences a c a c ... and b c b c ... represent also hexagonal graphite structures. Some graphites have however the rhombohedral stacking designated by a b c a b c ... A cross section through this structure is given in fig. 1b.

4 — PARTIAL DISLOCATIONS IN GRAPHITE

The Burgers vectors AB, AD and AC in the c-plane as well as their negatives, which a basal dislocation may have, can be decomposed into two partial vectors according to the reaction $AB \rightarrow A\sigma + \sigma B$, as is demonstrated in figs. 1c and 1d. The dislocation is split into two partials of the Shockley type.

The presence of two kinds of c-planes i.e. a and b planes within the unit cell give rise to two classes of partials. For those dislocations located between "a and b" (a underneath, b on top) the dissociation reactions are of the type $AB \rightarrow A\sigma + \sigma B$. For those located between "b and a" the possible dissociation reactions are of the type $AB \rightarrow \sigma B + A\sigma$. The stacking fault associated with the first dissociation is described by the sequence

a b a b a c b c b

and for the second type of dissociation by

a b a b c a c a

The movement of a partial $A\sigma$, $B\sigma$ or $C\sigma$ between "a and b" (a underneath, b on top) changes the stacking according to the prescription $a \rightarrow b \rightarrow c \rightarrow a$, whilst the partials σA , σB and σC have the opposite effect, i.e. they change the stacking according to the scheme $a \rightarrow c \rightarrow b \rightarrow a$. On the other hand the movement of partials like σA , σB and σC between "b and a" (b underneath, a on top) changes the stacking according to $a \rightarrow b \rightarrow c \rightarrow a$; the partials $A\sigma$, $B\sigma$ and $C\sigma$ do the reverse.

The scalar product of the Burgers vectors $A\sigma$ and σB or in general of two Burgers vectors, which are indicated by symbols in which σ is in different position, is positive. The corresponding dislocations repel each other with exception however of the case that the Burgers vectors of the two partial dislocations are parallel and in different sense (e.g. $A\sigma$ and σA) under which circumstances they attract each other and annihilate when they are in the same plane.

The scalar product of Burgers vectors, indicated by expressions in which σ is in same position, is negative, as a consequence of which the corresponding partials attract each other and interact, giving rise to a new partial dislocation. Exception to this rule is the case that the two Burgers vectors are parallel under which circumstances the corresponding partial dislocations repel each other.

5 — DISLOCATIONS BOUNDING INTERCALATED REACTANT LAYERS

Of most of the graphite compounds no reliable information is available about the location of the additives in any one layer and of the relative position of the adjacent carbon layers.

When the hexagon networks on either side of the intercalated layers remain in staggered position as in the parent graphite, the Burgers vector of the boundary dislocation will only have a component along the c-axis. The magnitude of the Burgers vector is not a rational fraction of the c-parameter. Such a dislocation is not visible or will only give a residual contrast upon normal conditions of observation (c-plane perpendicular to the beam).

When however the two adjacent hexagon networks are shifted in identical positions as Rüdorff and Schulze (8) found for potassium atoms, the boundary dislocation will have a Burgers vector with a component in the basal plane in addition to the component along the c-axis. The basal component is equivalent with one of the Burgers vectors $\frac{a}{3} [10\bar{1}0]$ of the partials of normal dislocation ribbons. Such a dislocation of which the total Burgers vector is inclined to the c-plane, as is shown in fig. 2, is visible.

As opposed to normal partial dislocations, which are glissile, the boundary dislocations are sessile. They can only move in the glide plane upon diffusion of reactant.

The intercalated layers are separated by a characteristic number of carbon layers in hexagonal stacking order.

The stacking sequences of hexagonal graphite, graphite with one and with two intercalated layers present in successive planes are respectively :

a b a b a b a b a
a b γ_1 b c b c b c
a b γ_1 b γ_2 b a b a

wherein a, b, and c represent the three possible positions of the hexagons in the graphite structure and γ_1 and γ_2 the different situations of the intercalated substance. Stacking fault contrast may be observed with an uneven number of successive intercalated layers, whereas no stacking fault contrast will be observed with an even number of successive intercalated layers present upon suitable conditions of observation. The stacking sequences of hexagonal graphite, graphite with one, two and three intercalated layers present every two carbon layers are respectively :

a b a b a b a b a b a
a b γ_1 b c b c b c b c b c
a b γ_1 b c γ_2 c a c a c a c
a b γ_1 b c γ_2 c a γ_3 a b a b

Stacking fault contrast may be observed with one and two intercalated layers whereas no stacking fault contrast will be observed upon addition of the third layer etc.

6 — GRAPHITE-BROMIDE

Rüdorff (9) found that graphite forms readily a lamellar compound C_8Br by direct addition from the vapour, the interlayer spacing of the expanded layers becoming 7.05 \AA instead of 3.35 \AA of the parent graphite. The structure of the bromine layers and the relative position of the adjacent carbon layers is not known (1). The specimens were made by exposing graphite single crystals to bromine vapour at room temperature for some minutes. They were then left standing for some hours before observation.

The peripheral parts of the crystals show a mosaic of regions exhibiting contrast as is seen in fig. 3, caused by numerous islands of occluded bromine as will be discussed below. The more inner parts, resembling normal graphite, lend themselves excellently for the study of dislocations, bounding intercalated bromine layers.

The specimens reveal isolated dislocations of which some are seen in fig. 3, showing stacking fault contrast at one side. The direction of the Burgers vector \vec{b} of these dislocations is determined by means of extinction of the contrast applying the criterion $\vec{g} \cdot \vec{b} = 0$, wherein \vec{g} is the reciprocal lattice vector. The basal component proved to be always along one of the three projections of the $\{11\bar{2}0\}$ planes on the basal plane, compatible with the Burgers vectors of the partials of normal dislocation ribbons. An example is given in fig. 4, where the direction of the basal component of the Burgers vector is determined by extinction of an elongated loop formed by a moving boundary dislocation.

We suppose therefore that these isolated dislocations are bounding the intercalated bromine layers. The adjacent carbon layers are presumably shifted in identical positions upon intercalation of the bromine as was discussed in the preceding paragraph.

The boundary dislocations are highly mobile by diffusion and have a low line tension as may be concluded from their irregular shape. They leave numerous loops of irregular shape or islands of occluded bromine behind on their way through the crystal by a mechanism as is clearly presented in fig. 5. The loops are formed upon pinning of the moving boundary dislocations by other crystal imperfections. They are stable upon heating in the electron microscope by increasing the beam-intensity. In fig. 6 is presented an example of one of the typical configurations of numerous concentric bromine loops one above the other in different planes formed by moving boundary dislocations by the mechanism mentioned above as is demonstrated in fig. 3. The presence of islands of occluded bromine may be in accordance with Maire and Mering's findings about the characteristic X-ray lines of two dimensional structures. The bromine loops may account for at least part of the residual bromine retained upon decomposition. The number of loops depends on the speed of the boundary dislocations with other words on the speed of debromination.

Even after several weeks of standing in air however the crystals did still contain complete bromine layers and holes and pores filled with bromine, whereas the typical superstructure of the intercalated bromine layers could still be obtained by means of diffraction, as is shown in fig. 7.

7 — GRAPHITE-IODINE MONOCHLORIDE

Iodine-monochloride readily forms a fully intercalated compound C_5ICl (10) upon addition from the vapour. The structure of the intercalated layers, its location relative to the graphite lattice and the relative positions of the adjacent carbon hexagon networks are not known. The interlayer spacing of the expanded layers is 7.24 \AA .

1) Recently W.T. Eeles and J.A. Turnbull presented the first result of their electron and X-ray diffraction study of brominated graphite at the joint Conf. on inorganic and intermetallic crystals, Birmingham, 1963. Some of our results may well be in agreement with the arrangements of the bromine atoms, which they propose.

The specimens are made by exposing the graphite single crystals to the ICl vapour at a temperature of 30° C for some minutes and left standing for some hours before observation in the electron-microscope.

This crystal compound behaves in many respects in the same way as the graphite-bromide compound. The peripheral parts of the crystals show again a mosaic of regions exhibiting contrast, caused by numerous islands of occluded ICl one above the other, whereas the inner parts are resembling normal graphite.

The specimens reveal isolated dislocations, showing stacking fault contrast at one side upon suitable conditions of observation. The direction of the basal component of the Burgers vector of these dislocations as determined in the same way as for the boundary dislocations of the intercalated bromine layers, is along one of the three projections of the $\{11\bar{2}0\}$ planes on the basal plane as is shown in fig. 8. These dislocations are apparently bounding the intercalated ICl layers, whereas the adjacent carbon hexagon networks are presumably shifted in identical positions in the same way as was supposed upon intercalation of bromine.

The iodine monochloride boundary dislocations are much less mobile than those of bromine. Nevertheless loops of irregular shape, showing stacking fault contrast as well as the typical configurations of numerous concentric loops one above the other of which an example is given in the inset of fig. 6, are observed.

8 — INTERACTION OF A BOUNDARY DISLOCATION AND A NORMAL DISLOCATION RIBBON IN THE SAME PLANE

Fig. 9 represents schematically a cross section of a graphite crystal containing a normal dislocation ribbon and a dislocation bounding an intercalated bromine or iodine monochloride layer. The open circles represent the two partials of a basal dislocation as observed in non treated graphite. The component along the c-axis of the Burgers vector of the boundary dislocation, which is designated by $S\sigma$ or σS , depending on its direction, is perpendicular to the Burgers vector of the partials of the dislocation ribbon as a consequence of which it will not contribute to the interaction. The basal component however is equivalent to one of the Burgers vectors of normal partial dislocations, so that the discussion can be held in terms of symbols used for discussion of partial dislocations in par. 4. It follows from fig. 9 that the σ of the basal component of the Burgers vector of the boundary dislocation and of the Burgers vector of the partial to the right of the ribbon are in same position. This means that the boundary dislocation and the partial repel each other if the vectors mentioned above are parallel as is shown in fig. 10a. An example of such an interaction is given in fig. 11, where the ribbon is driven up by the bromine layer.

The two dislocations attract each other and interact however if the two vectors are not parallel. A new boundary dislocation is formed. The σ of the basal component of the Burgers vector of this new boundary dislocation and of the Burgers vector of the partial to the left of the ribbon are again in same position. These two dislocations repel each other if these two vectors are parallel as is shown in fig. 10b. An example of this interaction is given in fig. 12, where a part of the boundary dislocation has interacted with a dislocation ribbon. Of special interest is the narrowing of the $A\sigma/A\sigma (+S\sigma)$ ribbon in comparison with the width of a normal dislocation ribbon. The narrowing of ribbons under condition where bromine is introduced into the crystal can be understood as follows. Since the region containing the bromine wants to expand its stacking

fault energy γ_2 is effectively negative. A model for the ribbon is shown in fig. 13, where γ_1 is the stacking fault energy in the ribbon. The equilibrium distance between the two partials is now obtained by expressing that the net force on dislocation (2) is zero; one obtains :

$$d = \frac{a \cos^2 \Phi + \beta \sin^2 \Phi}{\gamma_1 + \gamma_2}$$

where $a = \mu b^2 / 2\pi$, $\beta = \mu b^2 / 2\pi (1-\gamma)$

μ is the "effective" shear modulus and γ the "effective" Poisson ratio, Φ is the angle between the Burgers vector b of the partials and the direction of the ribbon. The "effective" stacking fault energy can become larger than in the normal crystal and this causes the ribbon to become narrower.

If the two vectors mentioned in the second interaction are not parallel the two dislocations interact as is shown in fig. 10c. A new dislocation, which is bounding the intercalated bromine layer, is formed. This dislocation is equivalent with the boundary, dislocations of which we have given evidence in the preceding paragraphs.

The presence of the intercalated reactant is indicated in the figures by hatching.

9 — WIDENING OF NORMAL DISLOCATION RIBBONS UPON INTERCALATION OF REACTANT

A remarkable phenomenon namely the widening of dislocation ribbons upon intercalation of bromine or iodine monochloride has been observed. Examples of the two different ways in which the ribbons unfold are given in figs. 14 and 15 for bromine. One example is given only for ICl in fig. 16. We believe that this phenomenon can be explained on the basis that bromine enters the crystal preferentially along dislocations, diffusing along them and precipitating in the stacking fault ribbons. This process would be energetically favorable since the energy associated with the stacking fault would be liberated in this way.

Let us consider in detail the geometry of a ribbon along which a bromine layer is being deposited hereby shifting the layers on both sides of it in "a over a" stacking. Two essentially different possibilities occur, depending on the Burgers vector of the boundary dislocation.

We can choose arbitrarily the Burgers vector of the partials of the ribbon; we call them $A\sigma + \sigma B$. The boundary dislocation can now have a Burgers vector with a basal component $A\sigma$, $B\sigma$ or $C\sigma$ (we will ignore the vertical component which is irrelevant for the discussion that follows). Let us first consider the case that the basal component is $B\sigma$. This configuration is shown schematically in fig. 17c. The boundary dislocation reacts with the left partial hereby forming a $C\sigma (+ \sigma S)$ dislocation. The right partial forms a "narrow" ribbon of the type discussed in the preceding paragraph with the boundary dislocation. The resulting situation is shown schematically in fig. 17d. A cross section X-Y through the normal ribbon as well as a cross section U-V through the brominated part is shown in figs. 17e and 17g. The bromine layer can now extend by sweeping the boundary dislocation between the graphite layers. The configuration in fig. 14 may well have resulted in this way.

This interaction gives rise to the same kind of result as is discussed in the second case of the preceding paragraph and which is shown in fig. 12. It is difficult to decide which of the two phenomena is observed.

The situation is not essentially different if the boundary dislocation is of the type $A\sigma$ ($+\sigma S$). The only difference is that the reaction now takes place with the right partial and that the "narrow" ribbon is formed at the left.

If the boundary dislocation has a Burgers vector with a basal component $C\sigma$ however the situation is different. The boundary dislocation now reacts with both partials and forces the ribbon apart as a result of the region of negative stacking fault energy created between the partials. The repulsion of the partials aids in this process. A small segment of a boundary dislocation closes the ribbon as can be seen in fig. 15. A cross section R-S through the widened ribbon is shown in fig. 17f. It is suggested that the configuration shown in figs. 15 and 16 resulted in this way.

Further verification of the process by study of the contrast of the partials is in progress.

10 — GRAPHITE-FERRIC CHLORIDE

Since the investigation of this crystal compound is still in progress and since the results are not consistent so far, only a short summary will be given here. The graphite-ferric chloride compound is formed by intercalation of $FeCl_3$ molecules in graphite when mixtures of these reactants are heated at temperatures generally above 250° (11). The ferric chloride molecules are oriented in such a way that the iron atoms are situated in planes halfway between the carbon layers and the Cl atoms in two planes parallel to and 3.36 \AA distant from the carbon layers. The interlayer spacing of the expanded layers is 9.37 \AA . Cowley and Ibers (12) found that the intercalated molecules are distorted from their structure in the pure solid as a result of attempting to assume preferred positions relative to the adjacent carbon layers. No information is given however about the relative position of the adjacent carbon hexagon networks.

The specimens were made by heating graphite single crystals for about one hour with an excess of anhydrous ferric chloride at a temperature of 320° C . These specimens reveal isolated dislocations, which do not show contrast at one side as opposed to the bromine and the iodine monochloride boundary dislocations. So far we have not been able to determine the exact nature of these dislocations. The basal component of the Burgers vector, if there is any, is not along one of the projections of the $\{11\bar{2}0\}$ planes on the basal plane, as we concluded from extinction measurements. They separate however regions, resembling normal graphite, where normal dislocation ribbons are still present from regions where the contrast of the partials has spread considerably, the partial dislocations assuming equidistant positions as is strikingly shown in fig. 18. We therefore suppose that these dislocations are bounding the intercalated $FeCl_3$ layers. The widening of the contrast may be interpreted as the result of the expansion of the interlayer spacing brought about by intercalation of $FeCl_3$ as is demonstrated in fig. 19, where the extension of the elastically distorted region around a dislocation upon expansion of the interlayer spacing is shown schematically. The occupation of equidistant positions of the partials may be brought about by a decrease in stacking fault energy as a consequence of the same expansion of the interlayer spacing. The phenomenon results essentially from a relaxation of the strain around the dislocation over a wider region than in the normal crystal.

In a sense the pattern may be interpreted as a "moiré" pattern resulting from misfit in one direction only, e.g. the direction perpendicular to the dislocation in the case of pure edge. In such a moiré the direction of the fringes is not determined by the operating g -vector, but by the direction of the dislocations and their Burgers vector. The moiré spacing is further equal to or related in a simple manner to the dislocation spacing. If the dislocations are not pure edge the fringes may have a slightly different orientation than the dislocations, which is presumably the case in fig. 18.

The relaxation around the dislocation also decreases the energy per unit length associated with them. Since the dislocations have now different energies in the region containing ferric chloride and in the "pure" region, they will be "refracted" at the boundary. This is a consequence of the tendency of the dislocation to adopt a shape of minimum energy, which is no longer a straight line.

To a first approximation Snell's law is valid, the "refractive index" being the ratio of the energies in both regions. If it is assumed that the widened images still have the direction of the dislocations fig. 18 is consistent with the explanation given here. The "relaxed" dislocations, which have the smaller energy form a sharper angle with the boundary than the "clean" dislocations.

A typical example of a loop formed upon intercalation of FeCl_3 is presented in fig. 20. The loop does not show a clear stacking fault contrast. This contrast may be due to some bending effect. The nature of the line contrast of the dislocation loop is the same as that of a boundary dislocation. Occasionally loops are observed, which have normal dislocation ribbons inside and completely distorted ribbons outside as was discussed above for boundary dislocations so that the loops are apparently bounding regions, which are still free from FeCl_3 .

In all our specimens regions were found however where dislocations of a very raggy nature and loops with pronounced stacking fault contrast were observed as is shown in fig. 21.

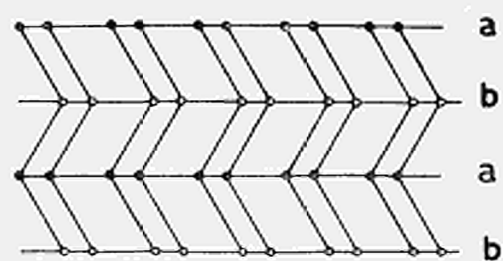
11 — ACKNOWLEDGEMENTS

We wish to thank Mr. Nicasy for technical assistance and Mr. Beyens for careful photographic work.

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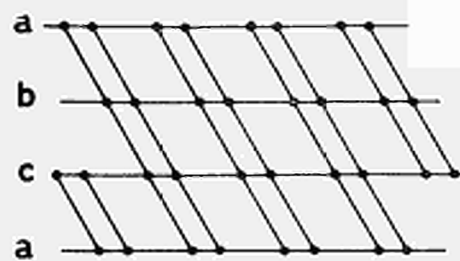
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Hexagonal.

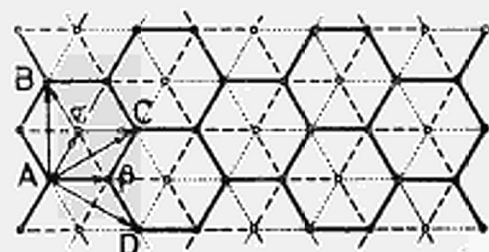


(a)

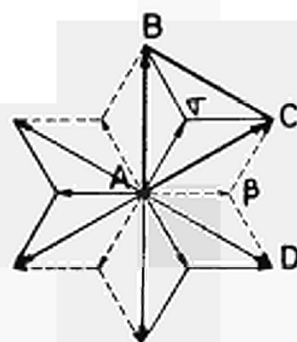
Rhombohedral.



(b)



(c)



(d)

Fig. 1 — Structure of graphite : *a*) cross section of hexagonal graphite; *b*) cross section of rhombohedral graphite; *c*) projection on the *c*-plane; *d*) directions of the Burgers vector.

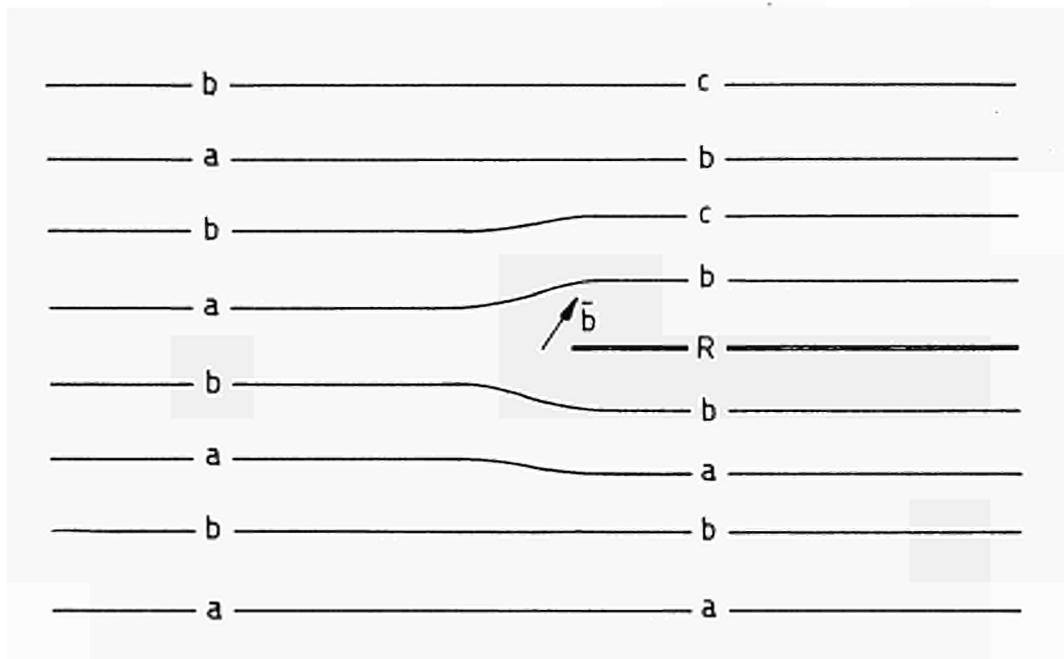


Fig. 2 — Cross section of a graphite crystal containing a dislocation bounding an intercalated layer of a reactant, which brings about a shift of the adjacent carbon layers in identical positions.



Fig. 3 — Peripheral part of a brominated graphite crystal showing regions exhibiting a mosaic of stacking fault contrast, boundary dislocations and the mechanism of the formation of typical configurations of concentric bromine loops one above the other in different planes.

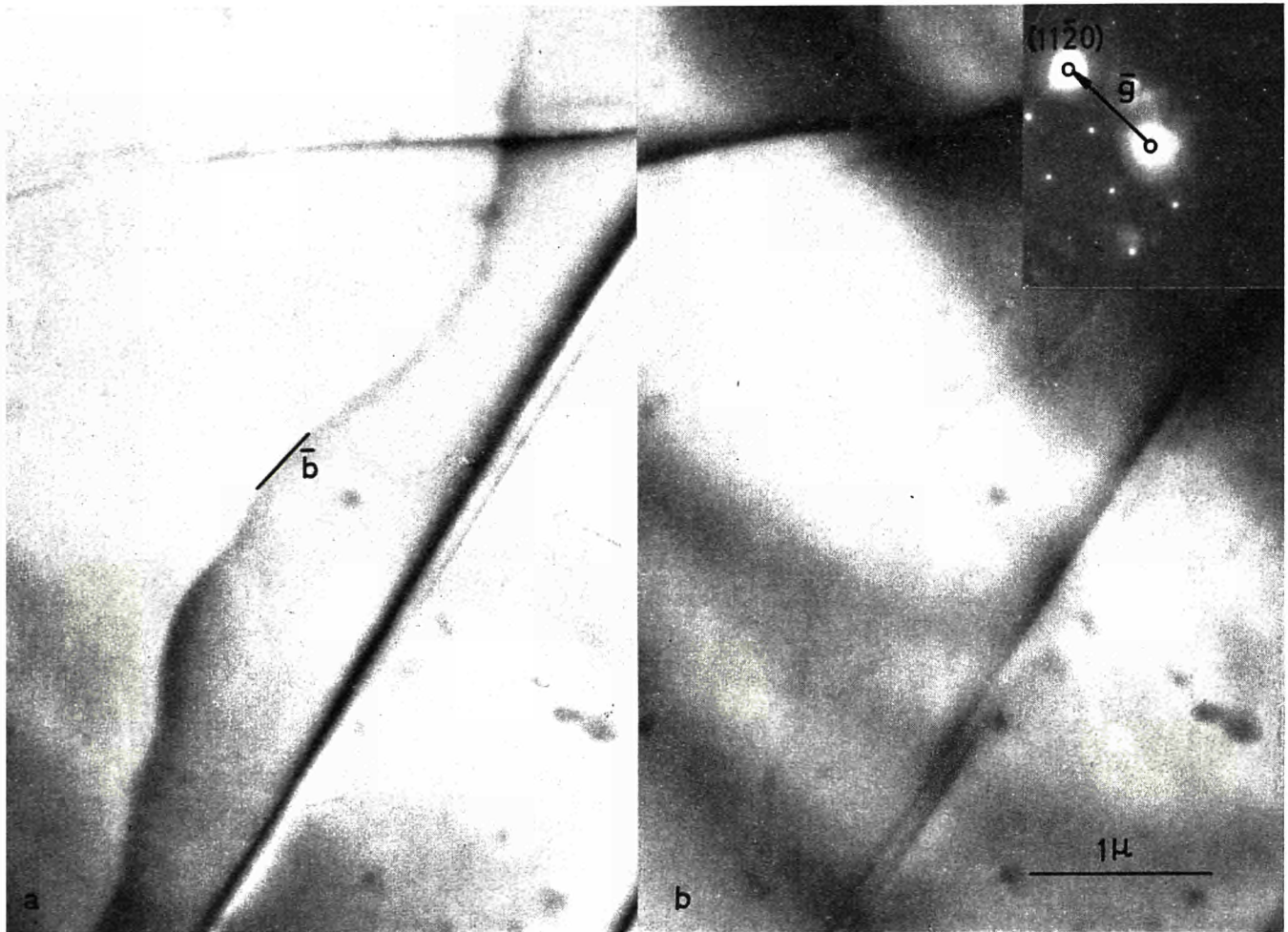


Fig. 4 — Determination of the direction of the basal component of the Burgers vector of a bromine boundary dislocation by means of extinction of a loop formed by this dislocation : *a*) elongated loop in stacking fault contrast; *b*) loop out of contrast; in inset the corresponding diffraction pattern showing the diffracting planes.

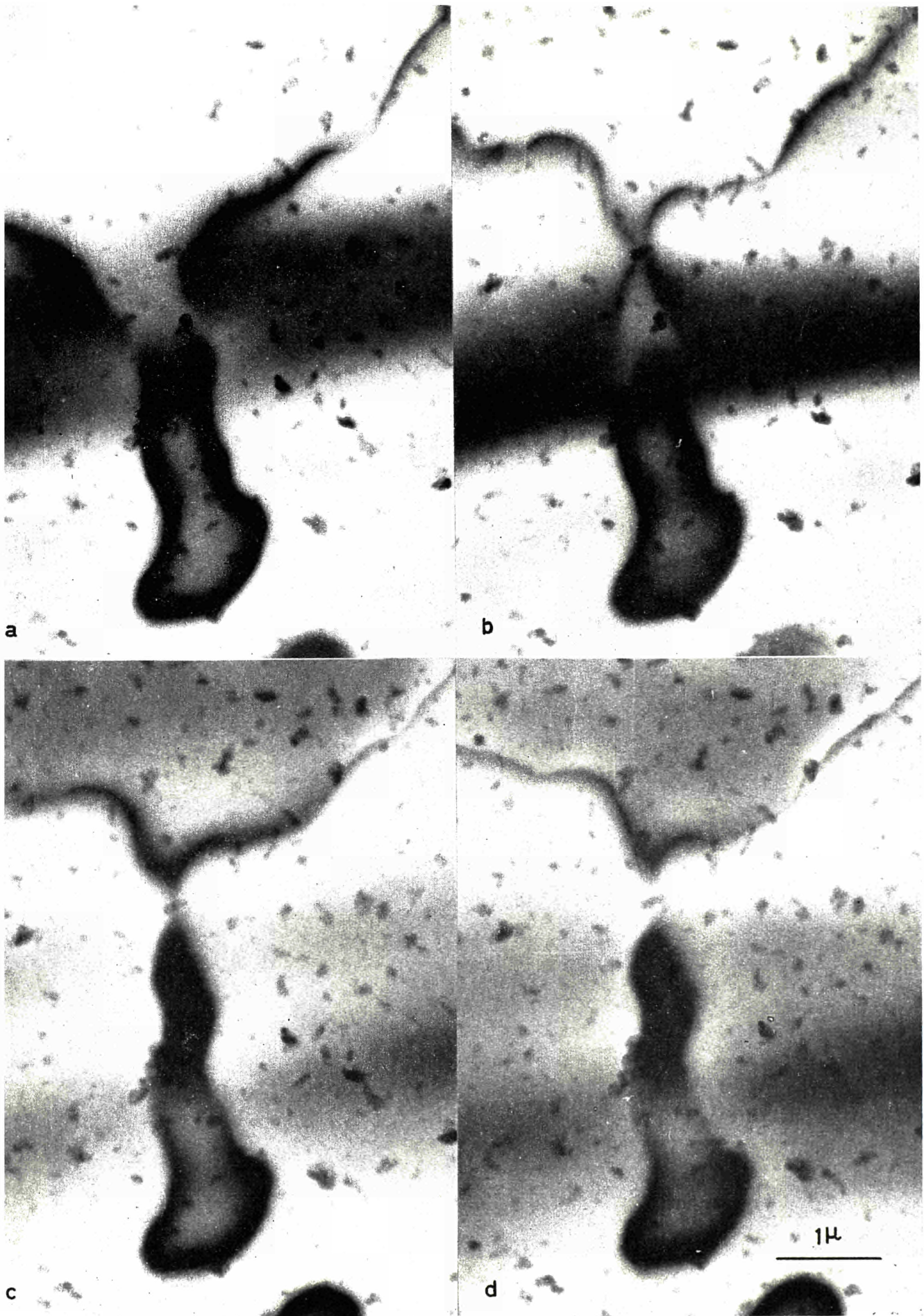


Fig. 5 — Mechanism of the formation of bromine loops by a moving boundary dislocation. The four photographs have been taken in succession at time intervals of about 30 seconds.

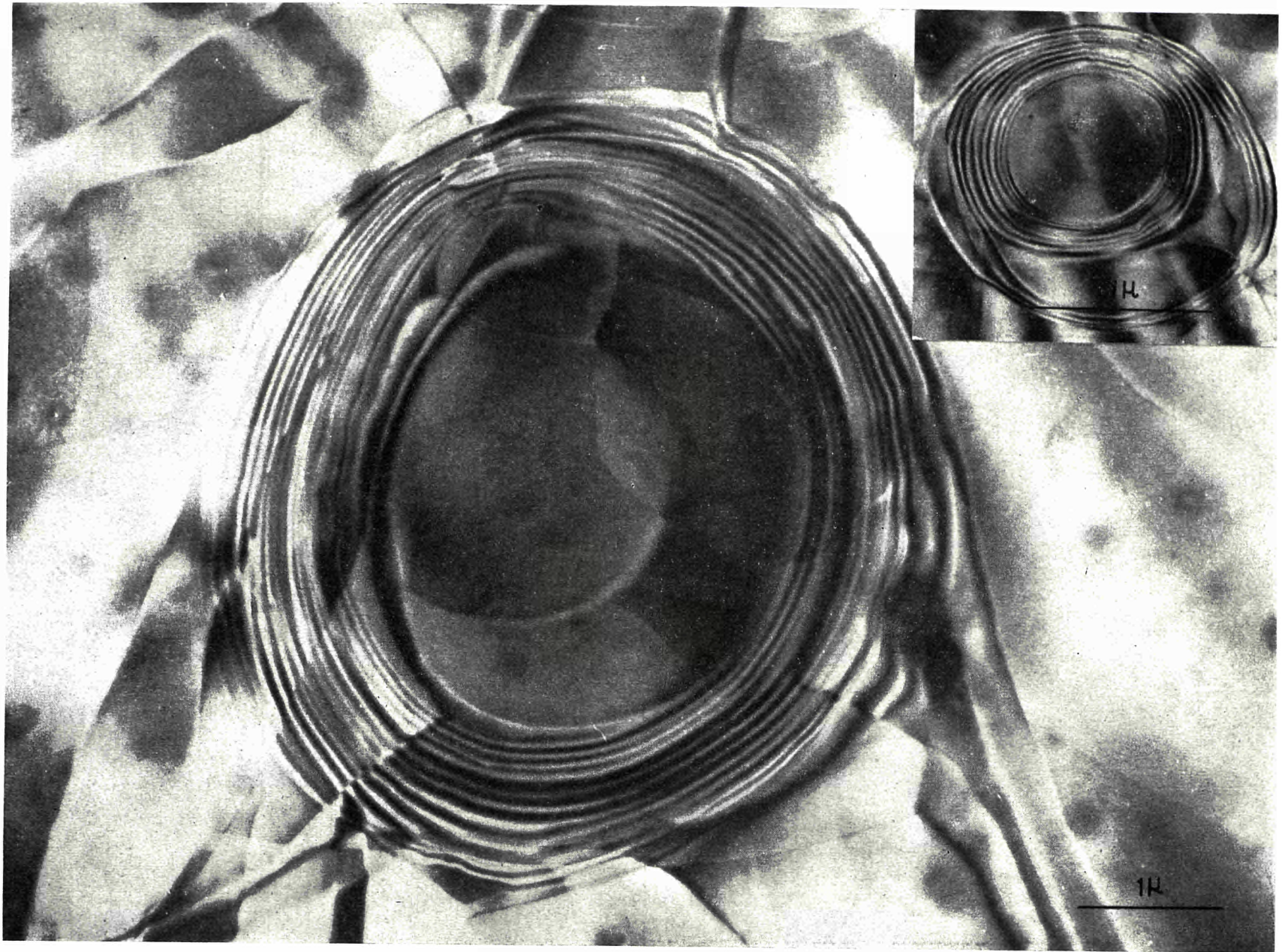


Fig. 6 — Configuration of numerous concentric bromine loops one above the other in different planes (in inset a configuration of ICl loops).

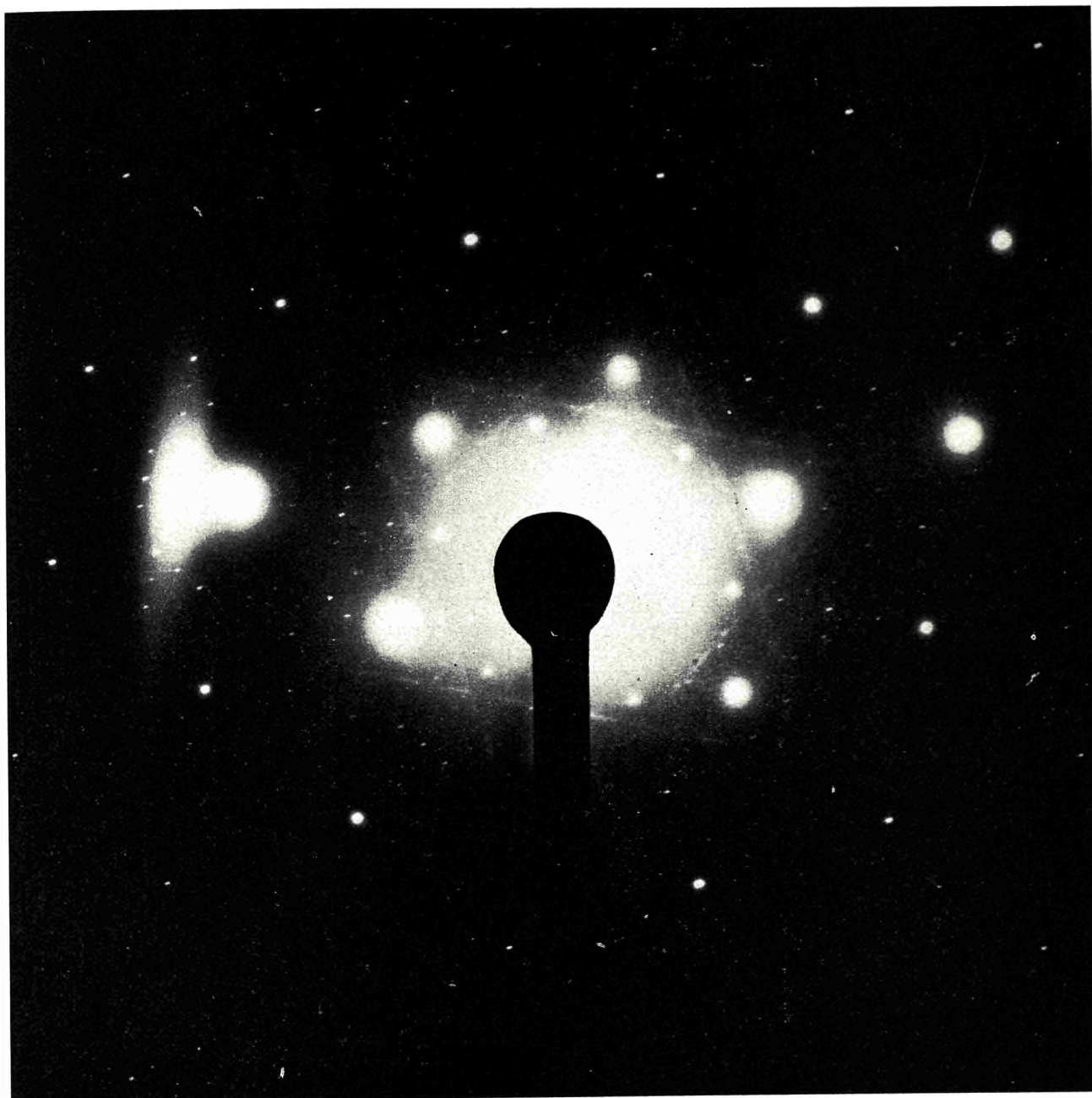


Fig. 7 — Diffraction pattern of graphite showing a superstructure brought about by bromine layers.

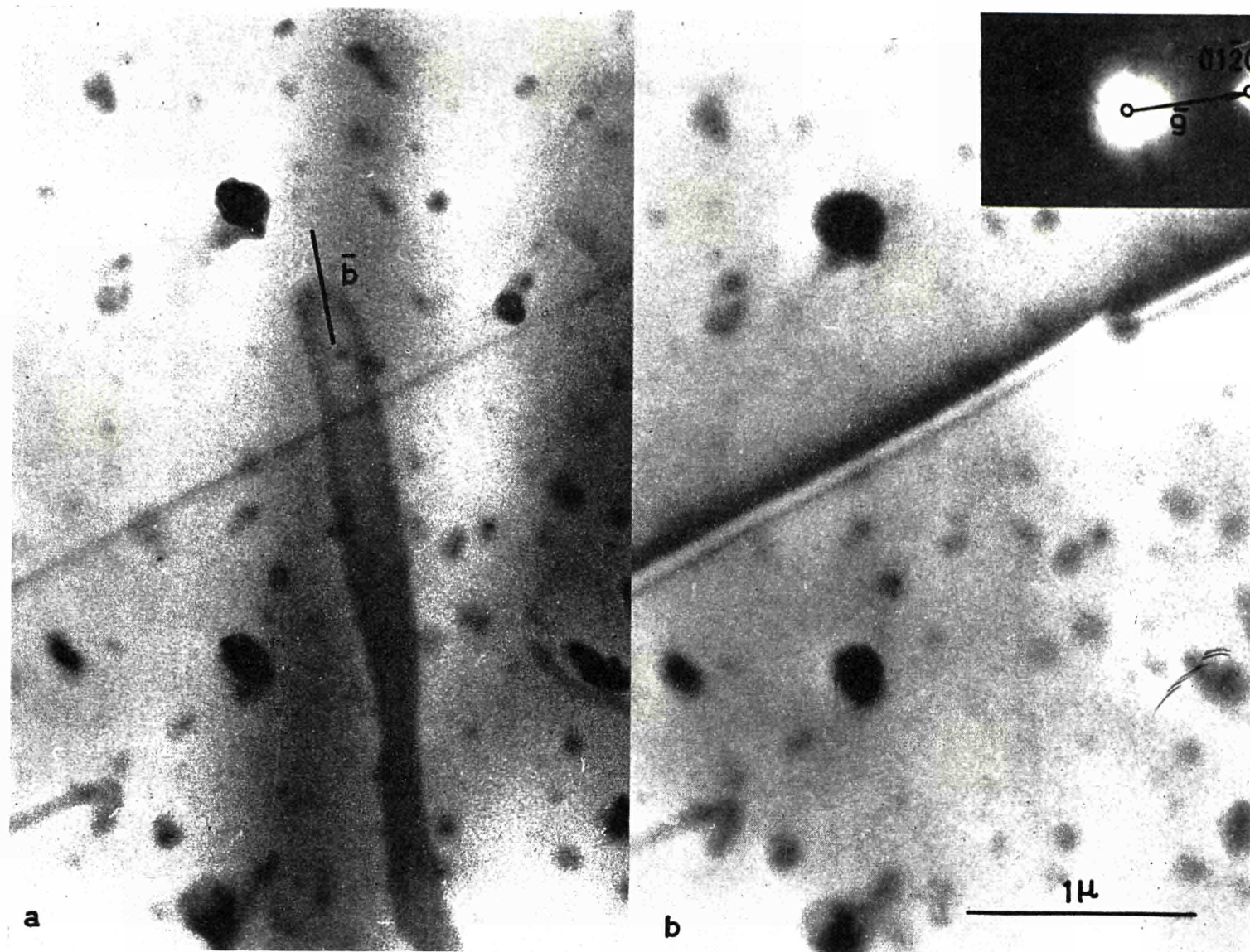


Fig. 8 — Determination of the direction of the basal component of the Burgers vector of an iodine monochloride boundary dislocation by means of extinction of this dislocation : *a*) a boundary dislocation in line contrast; *b*) a boundary dislocation out of contrast; in inset corresponding diffraction pattern showing the diffracting planes.

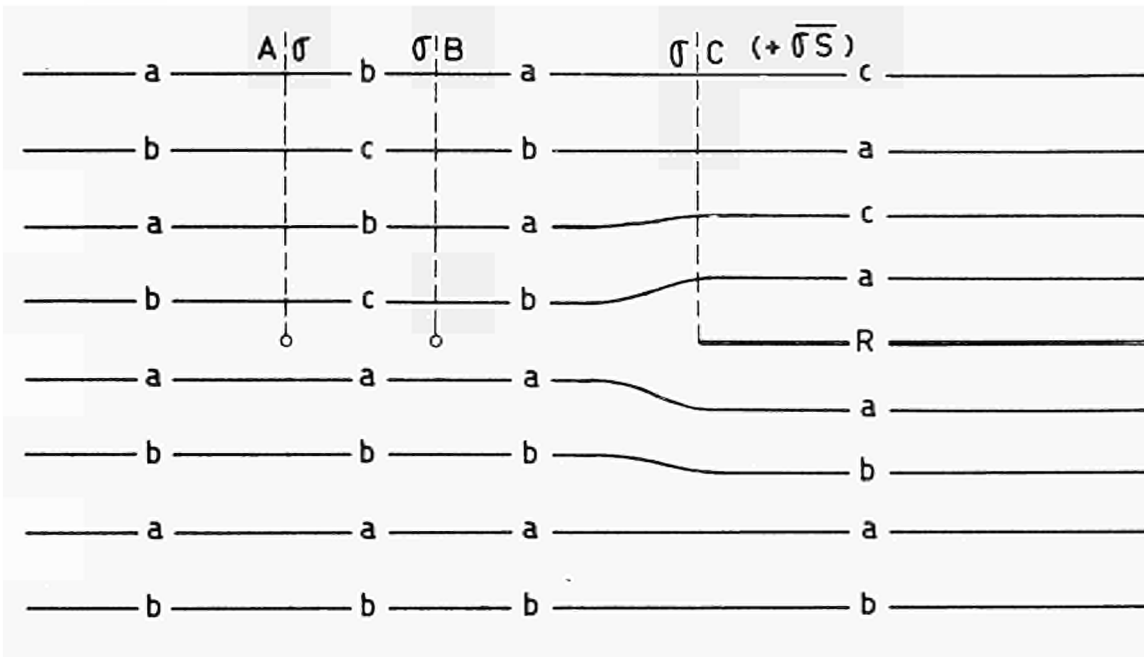


Fig. 9 — Cross section of a graphite crystal containing a dislocation ribbon and a bromine or iodine monochloride boundary dislocation in the same plane. The open circles represent the two partials of a basal dislocation as observed in non treated graphite.

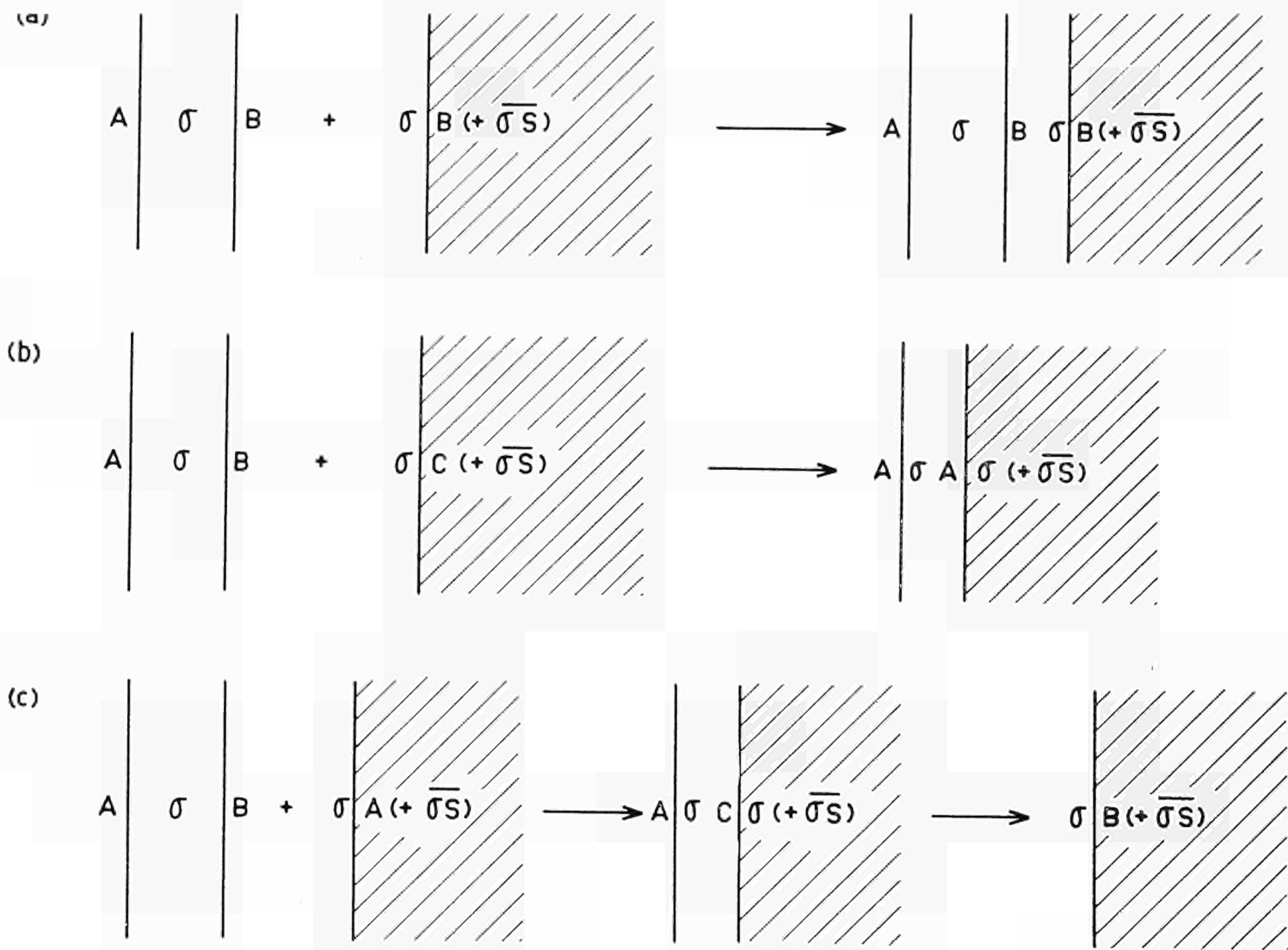


Fig. 10 — Schematic presentation of the interactions of a Br or ICl boundary dislocation and a ribbon in the same plane : *a*) the boundary dislocation and the neighbouring partial repel each other; *b*) the boundary dislocations and the neighbouring partial attract each other and interact giving rise to a new boundary dislocation; the remaining partial and the new boundary dislocation repel each other, forming a new ribbon; *c*) the remaining partial and the new boundary dislocation attract each other and interact giving rise to one boundary dislocation.

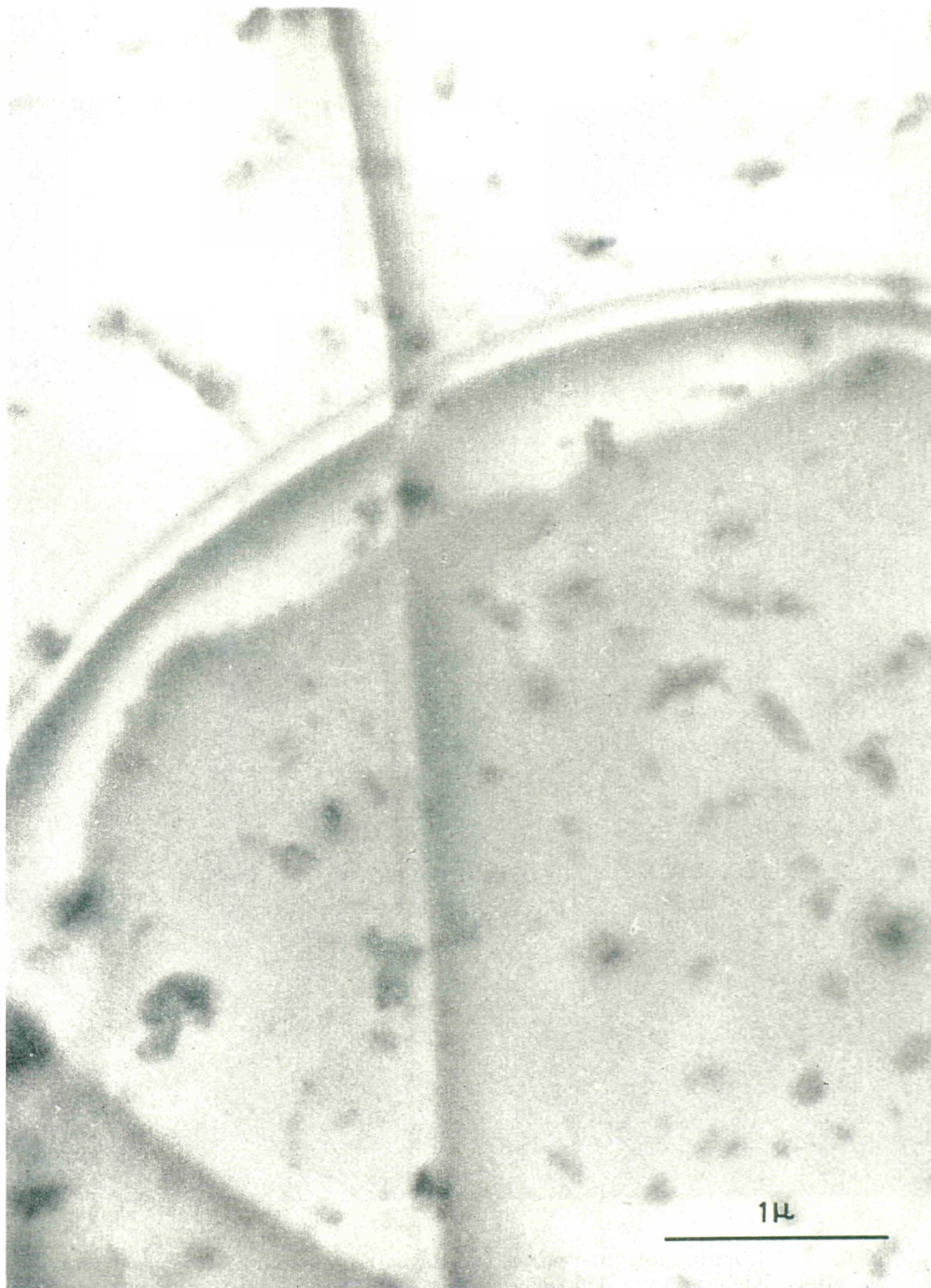


Fig. 11 — Repulsion of a bromine boundary dislocation by a dislocation ribbon as is shown schematically in fig. 10a.

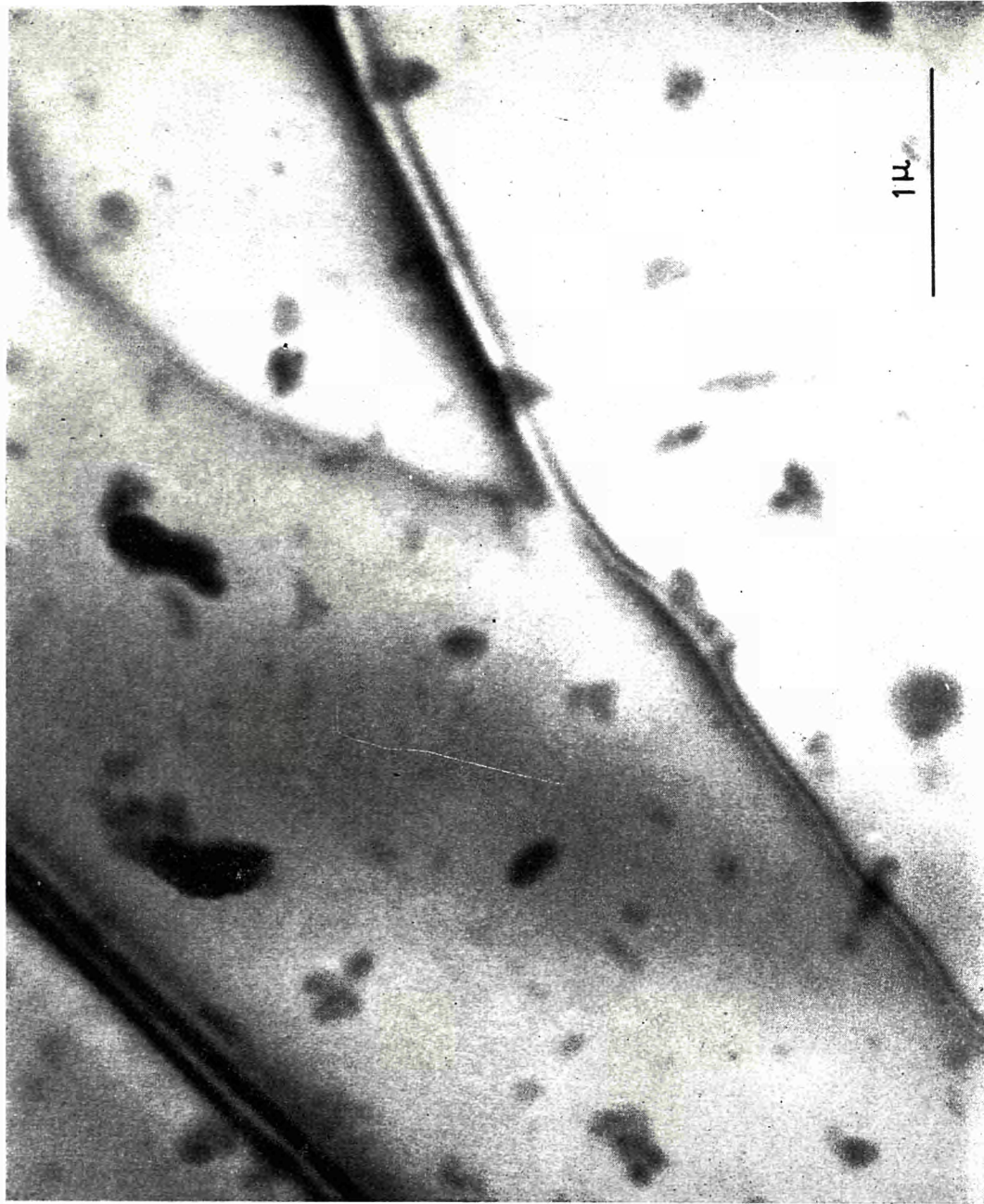


Fig. 12 — Attraction and interaction of a bromine boundary dislocation and a dislocation ribbon as shown in fig. 10b.

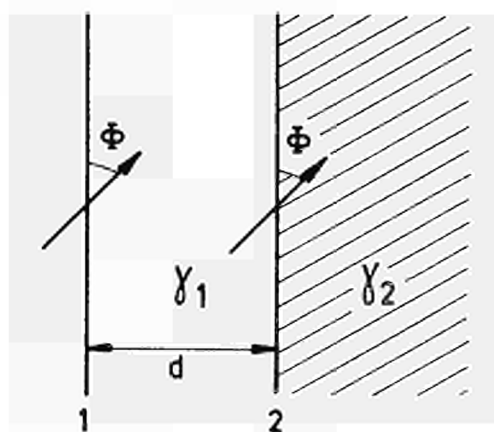


Fig. 13 — Model of the narrowing of a ribbon brought about by the presence of a reactant.



Fig. 14 — Example of the widening of a dislocation ribbon presumably by preferential diffusion of bromine along the ribbon as is shown schematically in fig. 17d.

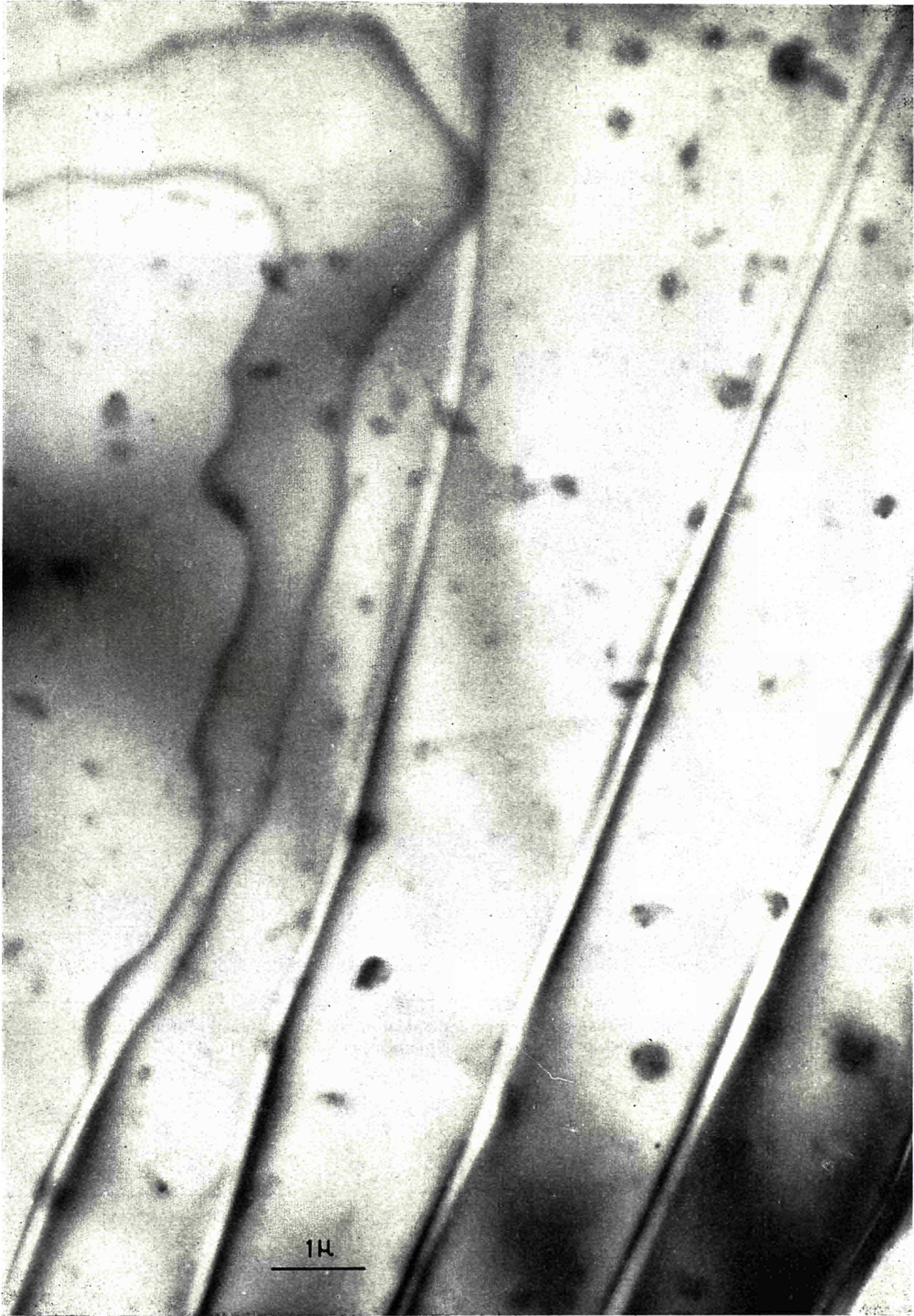


Fig. 15 — Example of another way of widening of a dislocation ribbon by preferential diffusion of bromine along the ribbon as is shown schematically in fig. 17b.

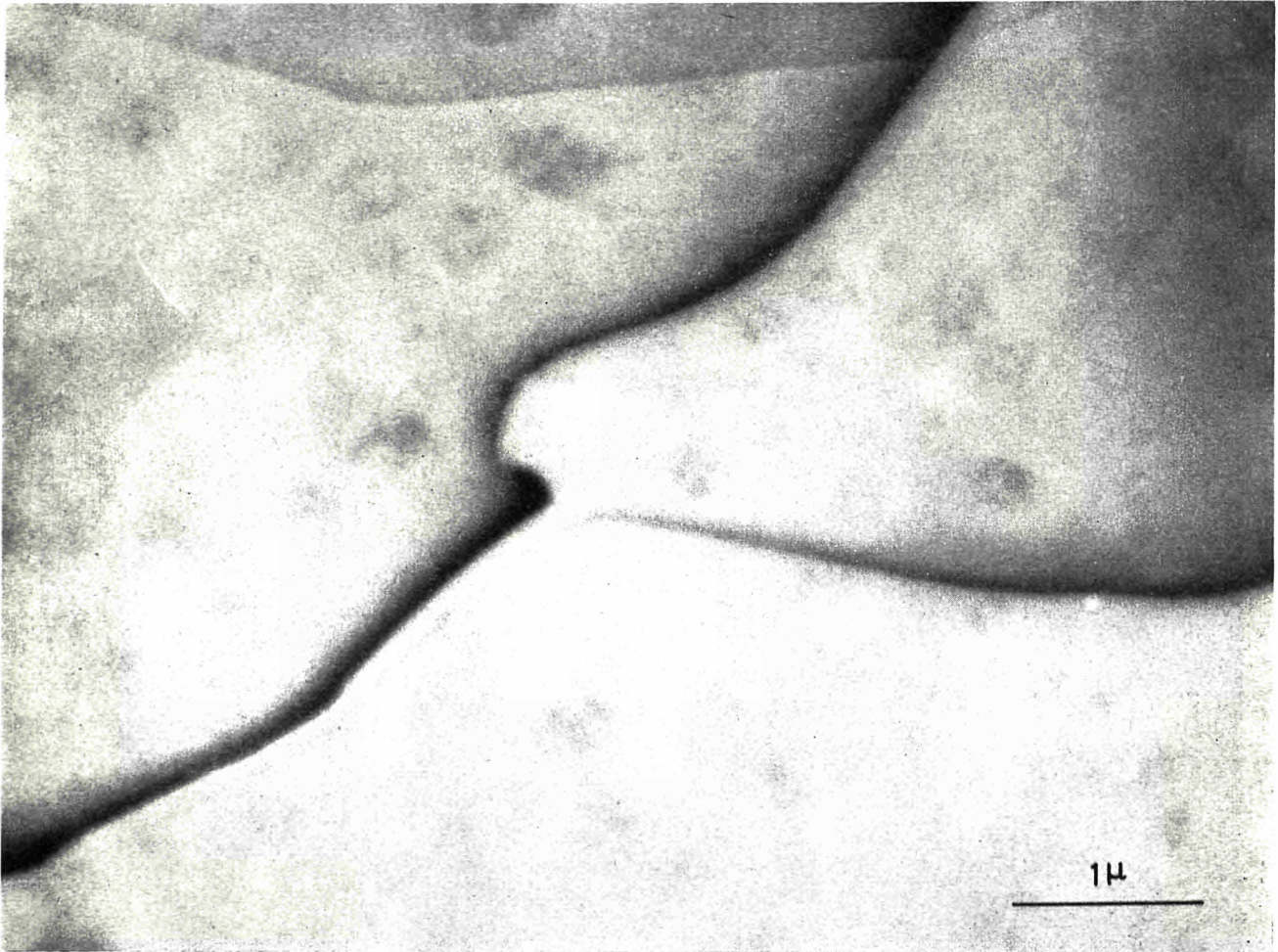


Fig. 16 — Example of the widening of a dislocation ribbon by preferential diffusion of iodine monochloride along the ribbon as is shown in fig. 17b.

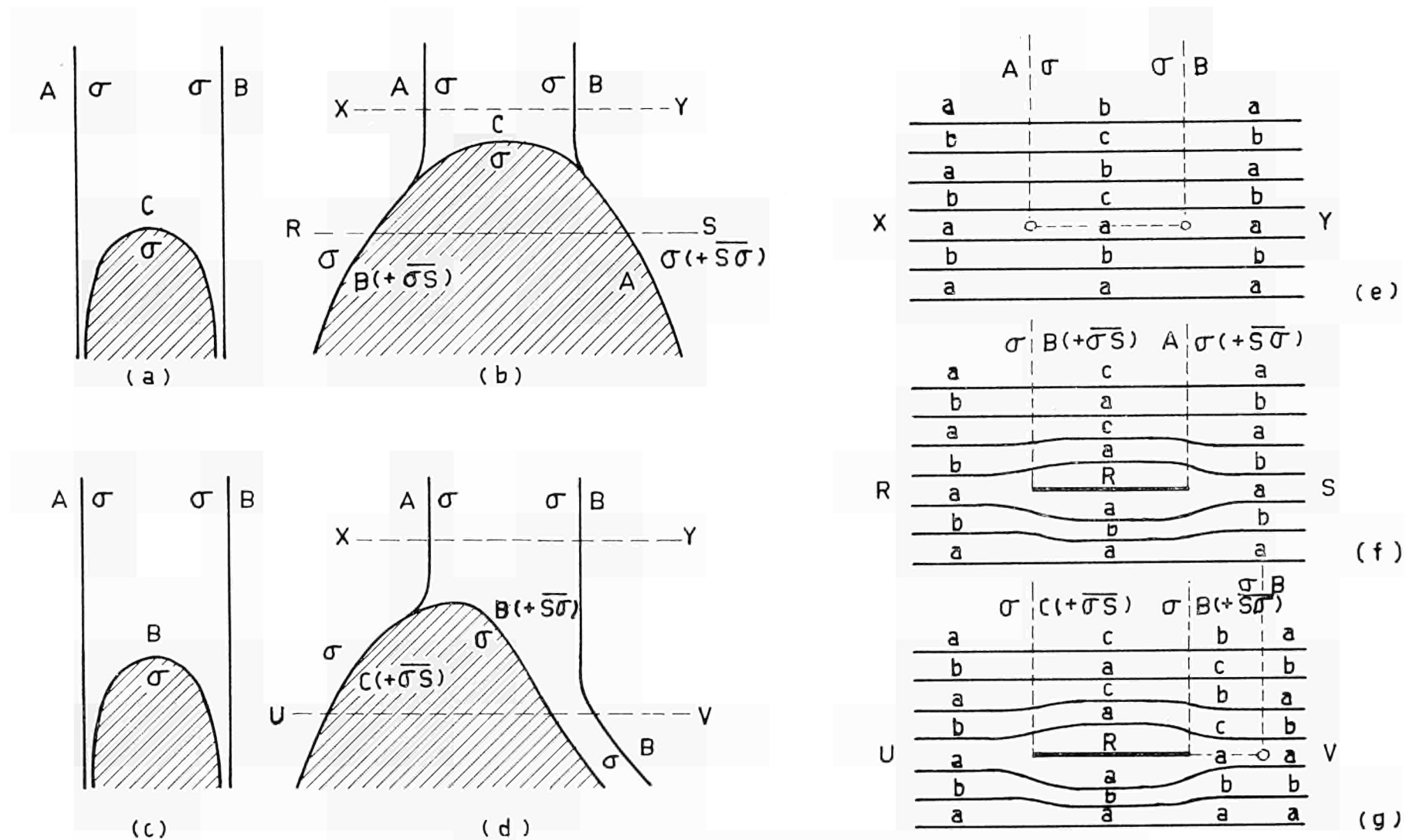


Fig. 17 — Schematic model of the widening of dislocation ribbons by preferential diffusion of bromine or iodine monochloride along the ribbon :

a) the reactant enters the ribbon; basal component of the Burgers vector of the boundary dislocation is $C\sigma$
b) the boundary dislocation has reacted with both partials of the ribbon; the newly formed boundary dislocations are pushed apart by the reactant
c) reactant enters the ribbon; basal component of the Burgers vector of the boundary dislocation is $B\sigma$

d) the boundary dislocation reacts with the left ribbon only; the newly formed boundary dislocation and ribbon are pushed apart
e) cross section X-Y through normal part of the ribbon
f) cross section R-S through widened ribbon (b)
g) cross section U-V through widened ribbon (d)

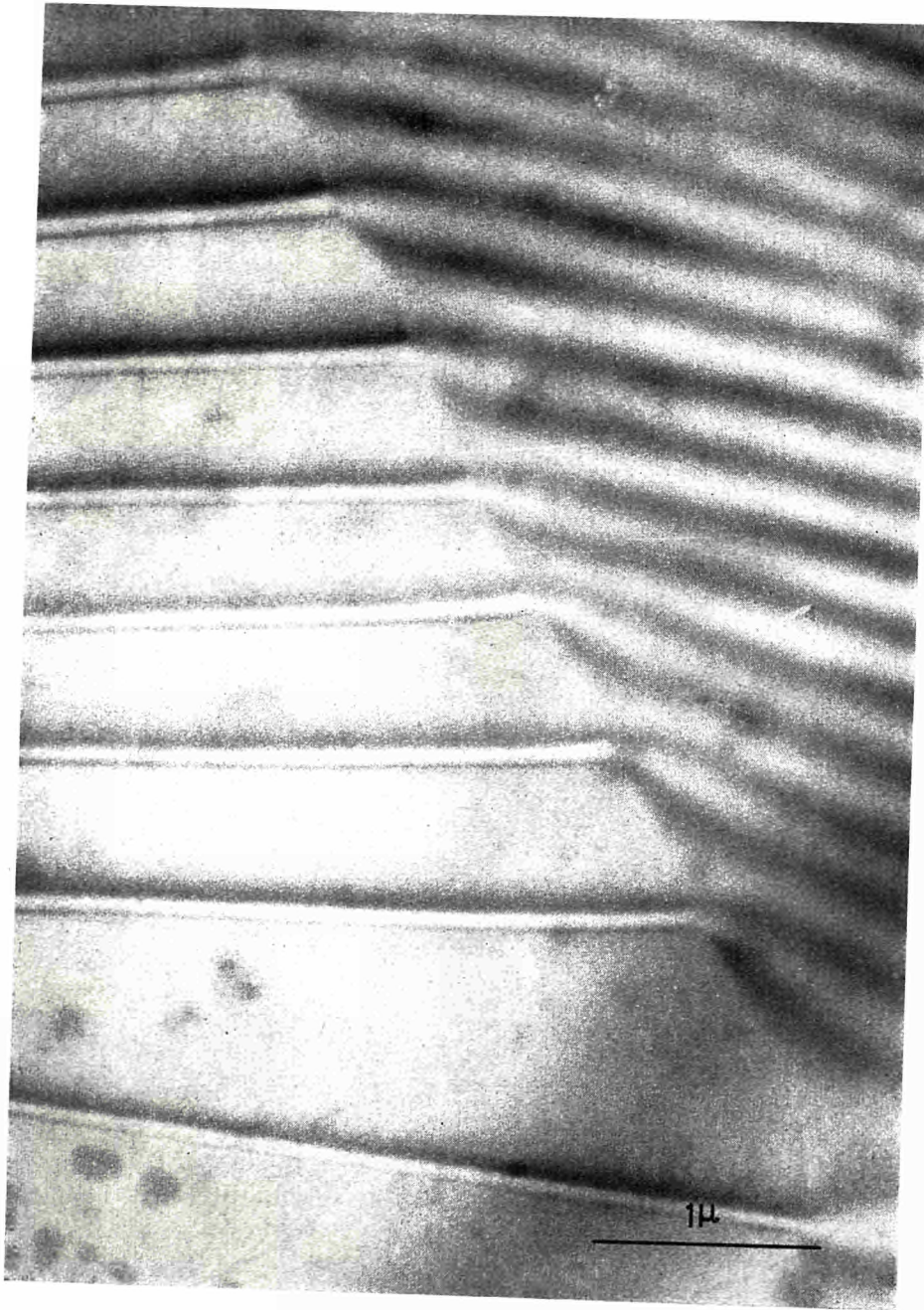
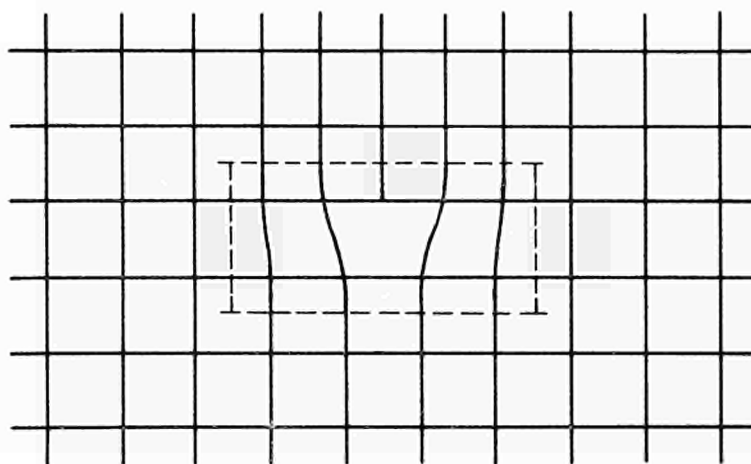
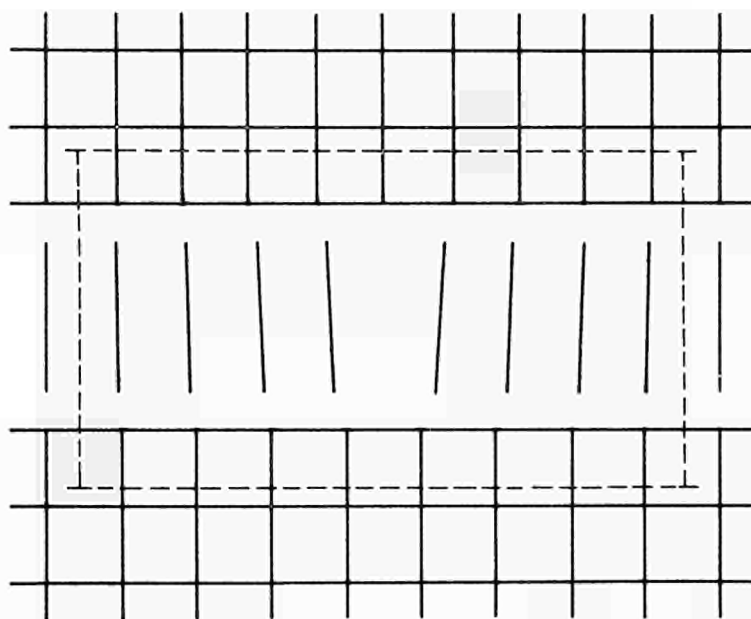


Fig. 18 — Widening of contrast of the partials of dislocation ribbons and the occupation of equidistant positions of the partials upon expansion of the inter-layer spacing caused by the presence of ferric chloride.



(a)



(b)

Fig. 19 — Extension of the elastically distorted region around a dislocation upon expansion of the interlayer spacing : *a*) normal structure; *b*) expansion of an interlayer spacing brought about by intercalation of a reactant.

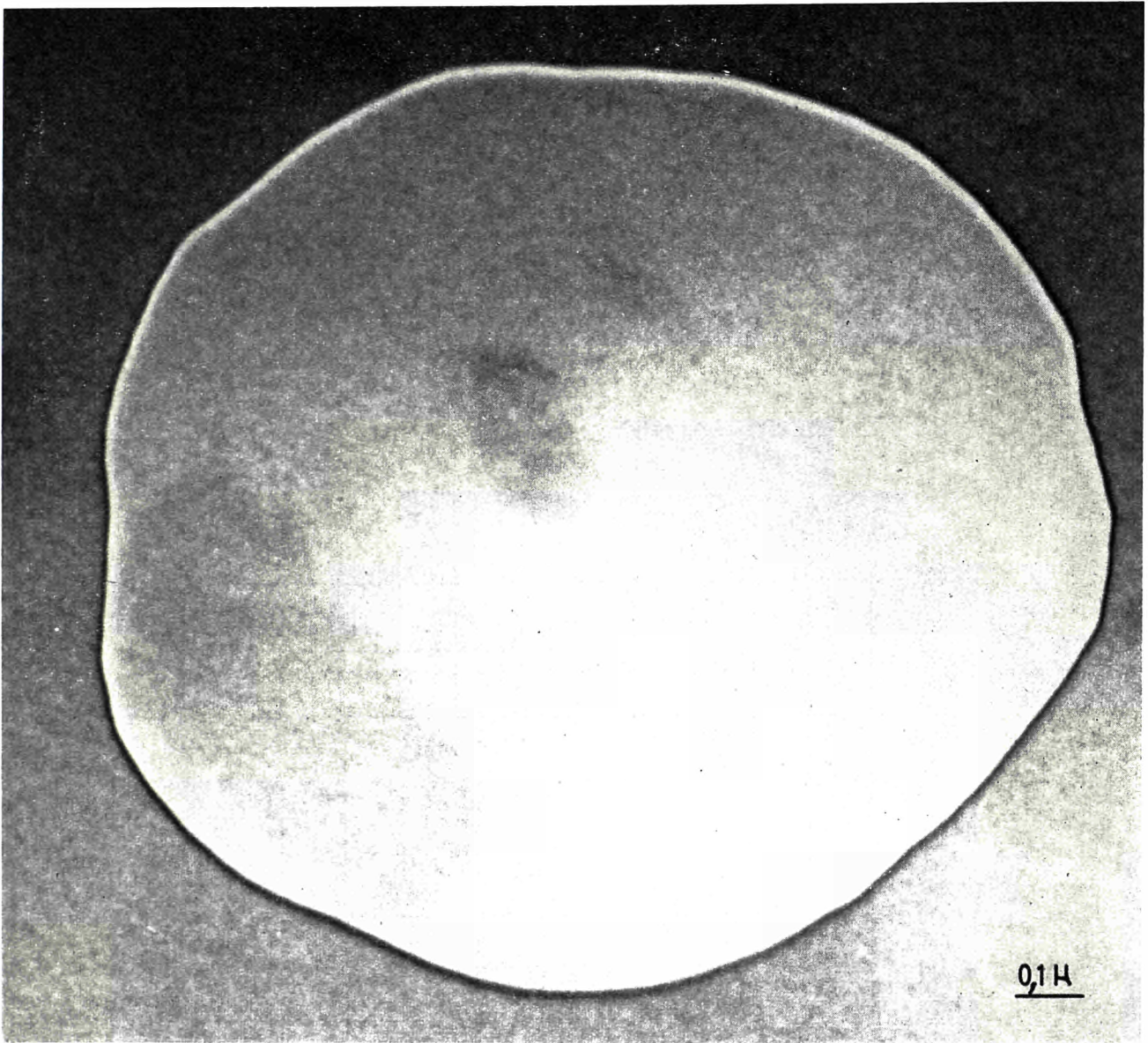


Fig. 20 — Typical example of a loop formed upon intercalation of FeCl_3 . The nature of the line contrast of the loop is the same as that of dislocations bounding the intercalated ferric chloride layers. The contrast on the upper side of the photograph is probably due to bending of the foil.

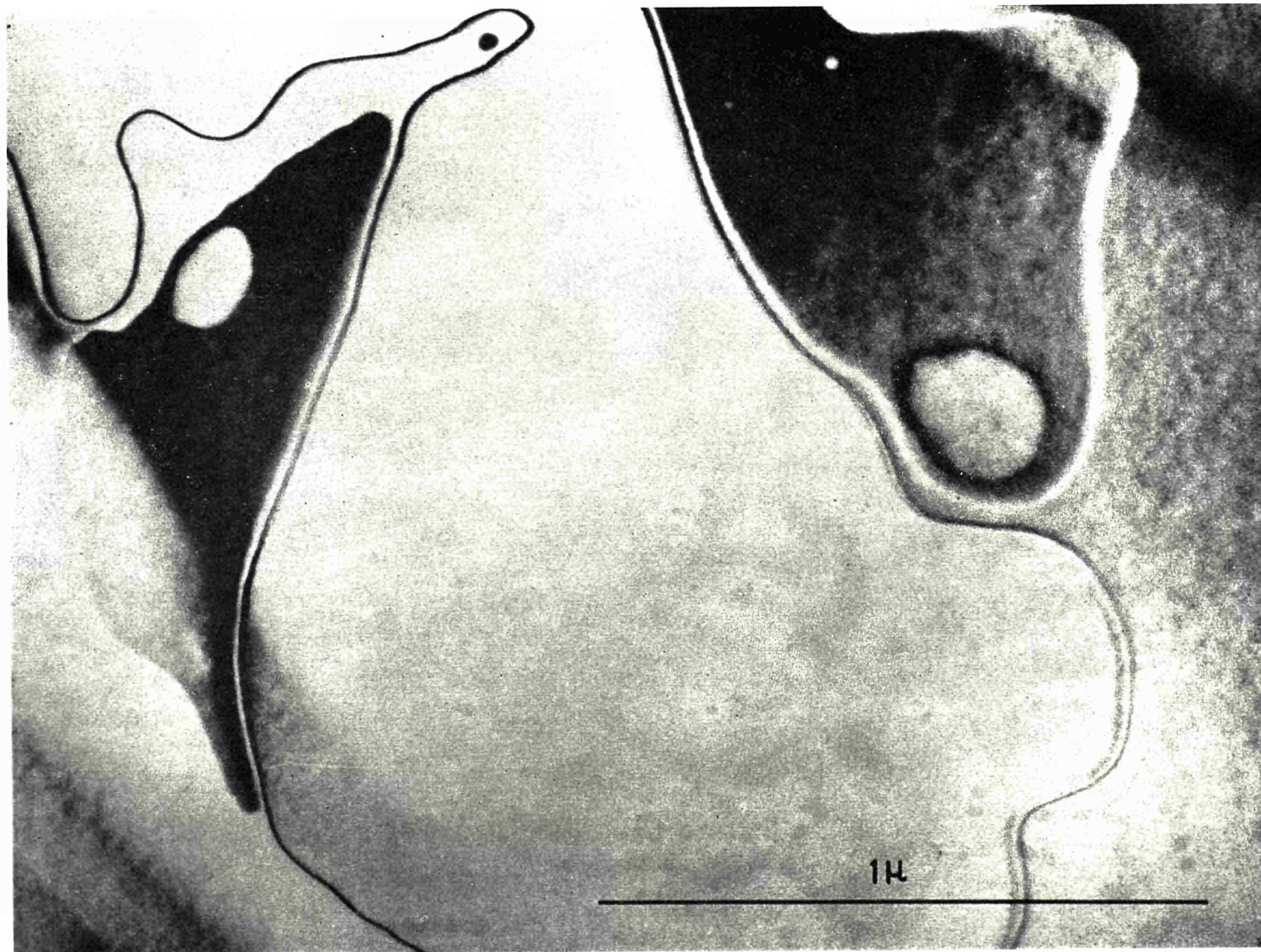


Fig. 21 — Specific region in graphite-ferric chloride specimens showing raggy dislocations and loops with a hard stacking fault contrast inside.



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