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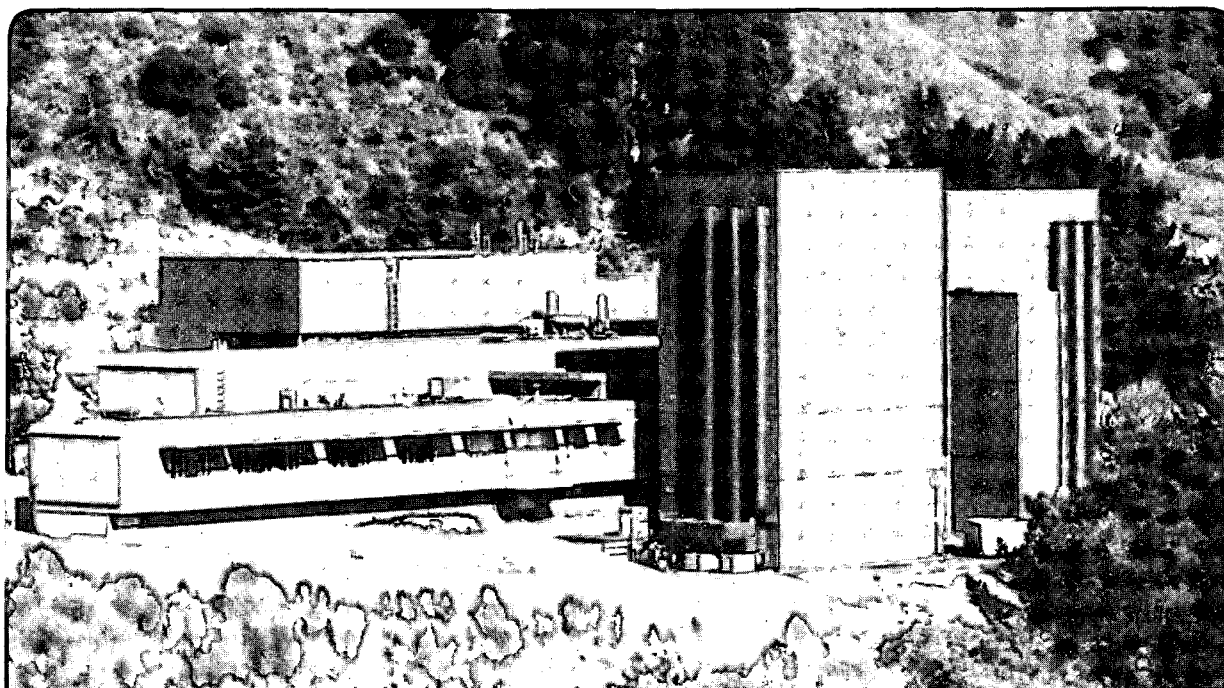
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Problems of Enclosed Crystal Interfaces

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PROBLEMS OF ENCLOSED CRYSTAL INTERFACES

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INTRODUCTION

Most studies of the structure of interfaces in materials assume that the interface is planar. This is a good approximation for thin films on substrates, for plate-shaped precipitates, or for other geometries in which the dominant part of the interface is a flat facet. However, many common microstructures contain enclosed crystals whose interface with the enclosing crystal, or matrix, is a closed, often polygonal, surface. For example, needle- or lath-shaped precipitates are often found to be faceted in cross-section so that several non-equivalent planar interfaces are joined together. Similarly, the junction of the habit plane with the perimeter of a flat plate-shaped precipitate, or the junction of planar grain boundary facets in enclosed grains, are subject to special constraints. These constraints are related to the shape of the enclosed crystal.

Whereas the shape of a small crystallite within an isotropic medium, i.e. vacuum, a vapor or a liquid, has long been a subject of fundamental interest in materials science (e.g. 1), the equivalent problem of a crystal enclosed inside another crystal has received relatively little attention. The optimum shape of a solid inclusion in a solid matrix has been treated as a problem of elasticity, and solutions have been given for different shapes, orientations and elastic constants (2,3,4,5). This approach treats a solid as an anisotropic elastic continuum without direct reference to its crystal structure. The complementary point of view takes into account the effect of crystal structure but neglects elastic or plastic distortions. The latter approach, developed only recently, has been successful in understanding and categorizing the types of crystal defects that can occur in interfaces between two crystals (6,7).

The two approaches, the continuum elastic and the crystallographic theory, address different aspects of the same problem, and in order to understand microstructures and interfaces of enclosed crystals, it is necessary to apply both or at least know which aspect dominates in a given situation.

In the present paper the problem will be examined from both viewpoints in an attempt to highlight the physical principles that underlie the shape and orientation relationship of an enclosed crystal, and these will be related to interfaces and their

defects. Although sections of this paper have been published before in a different context (8) the purpose here is to present an overview of some factors that determine solid/solid interface structure.

RESULTS

Symmetry

According to Curie's symmetry principle (e.g. 9) the shape of a single crystal inside an isotropic medium must have at least the same point symmetry as that of the crystal structure. For a cubic crystal this would be a cube, octahedron, tetrahedron or their combinations such as a cubo-octahedron etc. For a crystal inside another crystal the equilibrium shape must follow at least the point symmetry of the bicrystal, i.e. the set of point symmetry elements the two crystals have in common (6). This is directly related to their orientation relationship. Fig. 1a illustrates this for a tetragonal precipitate inside a cubic matrix. In this orientation relationship the two crystals have in common a twofold axis and two mirror planes normal to the plane of the drawing and a fourfold axis in the plane of the drawing. The equilibrium shape must have at least this common symmetry, i.e. the shape must remain unchanged during a 180° rotation or reflection across one of the vertical or horizontal mirrors. Thus the symmetry of the equilibrium shape of an enclosed crystal is the set of symmetry operations shared by the two crystals.

The remaining symmetry elements of the matrix crystal, those that are not shared by the precipitate, or enclosed crystal, generate different variants (6,10). This can be seen by reference to Fig. 1 as follows: around the precipitate shown in a) make a spherical cut (b), remove the region from the matrix (c) rotate (or reflect) it through a symmetry operation of the matrix (d) re-insert it into the matrix (e) and the spherical cut will disappear (f) because the orientation of the removed matrix material is equivalent to its starting orientation. However, the orientation of the precipitate in (f) is different from (a), and hence a new, crystallographically equivalent, variant has been generated. If the operation between (c) and (d) were also a symmetry element of the precipitate, then both the matrix and the precipitate would remain unchanged in this sequence. Therefore only those symmetry elements of the matrix that are not shared by the precipitate generate new variants.

An example showing the shape of a lath- or plate-shaped precipitate of germanium in a matrix of aluminum is given in Fig. 2. In this high resolution image the orientation relationship, shape, interface structure and internal defect structure of the precipitate are all visible at once. The common symmetry is the same as that illustrated in Fig. 1. Note how the particle shape does indeed conform to this symmetry. Note also that both crystals are cubic, yet their orientation relationship is such that only some of the symmetry elements are aligned. This lowering of the

composite symmetry is often found and, as shown below, has its origin in the lattice matching between the two crystals.

Lattice matching

Fig. 3 is an example of a composite diffraction pattern showing an asymmetric orientation relationship between an fcc matrix and a bcc precipitate. The traces of mirror planes in both phases are outlined: vertical and horizontal for the $\langle 110 \rangle$ zone axis of the fcc matrix, and in a threefold star, at 120° to one another for the $\langle 111 \rangle$ zone axis of the bcc precipitate whose $\{112\}$ spots are marked by black arrows. It is clear that the relative rotation between the lattices destroys any common mirror or rotation symmetries, leading to low composite symmetry. However, from the separation between corresponding diffraction spots (spot splitting, indicated by white arrows) it can also be seen that the same rotation produces an invariant line or invariant plane strain, characterized by a single direction of spot splitting.

This is illustrated schematically in Fig. 4. Fig. 4a shows two lattices in an orientation of high common symmetry. It is clear that the transformation from the square matrix lattice to the rectangular precipitate lattice is an expansion in the vertical and a contraction in the horizontal direction. In Fig. 4b, a small rotation has been added to the precipitate lattice, destroying the common mirror planes of the orientation relationship, but producing a direction of no strain, or invariant line.

Composite diffraction patterns for the same two situations are shown schematically in Fig. 4c and d. In the orientation of high symmetry, the direction of spot splitting, i.e. the separation between matrix and precipitate spots, varies from spot to spot but is aligned in Fig. 4d. for the invariant line strain. The direction of spot splitting is normal to the invariant line direction (11). Notice the similarity with the composite pattern shown in Fig. 3. Thus, a diffraction pattern with a single direction of spot splitting is a sign of an invariant line (or possibly an invariant plane) transformation strain.

It can be shown that the condition for the existence of an invariant line is that the principal strains be of mixed sign (12,13). The angle θ of the invariant line with the plane that is normal to η_3 is given by $\tan \theta = \sqrt{(\eta_1^2 - 1)/(1 - \eta_3^2)}$, where $1 - \eta_1$ and $1 - \eta_3$ are the two principal strains of opposite sign. The lattice rotation ϕ necessary to produce an invariant line is given by $\cos \phi = (1 + \eta_1 \eta_3)/(\eta_1 + \eta_3)$. The orientation relationship is determined by the angle ϕ while the orientation of the habit is given by the angle θ .

In general, precipitate dimensions tend to be inverse to their transformation strain, i.e. largest in the direction of smallest strain and vice versa(14). Thus precipitates with transformation strains of mixed sign tend to be elongated along invariant line directions, i.e. they will adopt needle- or plate-shape. The lattice

rotation associated with the formation of an invariant line results in low-symmetry orientation relationships and hence many crystallographically equivalent orientation variants. The lattice rotation for chromium precipitates in a copper matrix is illustrated in Fig. 3 and the corresponding complex distribution of needle shaped precipitate variants is shown in Fig. 5.

Interfaces of enclosed crystals

From the foregoing discussion it is apparent that interfaces of enclosed crystals are intimately related to their shapes, which in turn depend on the orientation relationship, which itself is given by the the minimization of the strain energy and the energy of the entire closed interface. The interface between bcc Cr precipitates in an fcc Cu matrix is dominated by the invariant line condition and is thus irrational in orientation and complex in structure. Although thoroughly investigated by different groups (15,16,17,18,19,20,21), its precise structure remains an open question. The dislocation structure in the major interface facet is clearly aligned with the invariant line direction of the needle axis. However, the Burgers vector and character of the dislocations is still uncertain.

It is worth emphasizing here that the question of the character of interface dislocations is not fully answered until it has been ascertained how the dislocation closes on itself: for an enclosed crystal each dislocation must be a closed loop. The angle of the Burgers vector with the loop plane, not just with the dislocation line in the major facet, or habit plane, determines its character. This is illustrated schematically in Fig. 6 where two different loop planes are shown with the same Burgers vector, line direction and edge character in the major interface facet, or habit plane. The set of dislocations on the left part of the precipitate is made up of shear loops and can move conservatively while that on the right is made up of edge loops and hence must climb as the interface advances. This is an important point in interface analysis that is not widely recognized.

If the transformation that relates the enclosed to the matrix crystal has unmixed principal strains, the orientation relationship, and hence the shape, is generally of higher symmetry. This leads to simpler interface structures more accessible to characterization and analysis. Nevertheless, the fact that one crystal is embedded in the other, rather than simply joined across a planar interface, leads to some important constraints. Some of these are illustrated next with examples of different plate-shaped precipitates.

θ' plates in Al-Cu

It is well-known that θ' forms as flat plates on $\{100\}$ planes, e.g. (22). This can be understood as a consequence of the good atomic match and the resulting low strain

energy associated with this habit plane. The atomic arrangement of atoms in planes parallel to the habit plane is identical in both the matrix and the precipitate, the principal difference being in their stacking sequence and chemical composition. For this reason the fourfold [001] axes of the bct θ' precipitate and the fcc matrix are always accurately aligned. The symmetry of the orientation relationship is the set of symmetry elements common to matrix and precipitate (6) and in the present case this is the 4/mmm tetragonal group. According to Curie's principle the equilibrium shape of θ' precipitates must have at least this symmetry (23). In fact, under most conditions of growth, the particles grow in the shape of a flat circular disc with cylindrical symmetry, (∞ /mm), a supergroup of the tetragonal point group. The Wulff plot of such θ' particles formed at high aging temperatures is dominated by a single deep cusp on the common (001) plane but its [001] projection is circular.

However, at low aging temperatures the decreased effect of entropy allows secondary cusps to emerge. Figure 7 shows θ' precipitates developed by aging for 176 h at 144°C. At this temperature the circular cross sectional shape gives way to a clear tendency for faceting on (100) or (110) planes. Three θ' variants are seen in Figure 7a in an $\langle 001 \rangle$ zone axis. The secondary facets are most clearly seen on the {001} variant that is face-on. The longest secondary facets follow {110} planes with smaller facets on {100} planes truncating the corners. The interfacial misfit dislocations in this particle are also aligned along low index directions, and small ledges are faintly visible in the secondary facets (see arrows).

The vertical plate that is seen edge-on clearly shows its even thickness and plate shape due to the major (001) facet. The secondary (100) facet at its top edge is parallel to the electron beam while the (110) facet at its bottom edge is inclined and consequently shows thickness fringe contrast.

Because the facets that form during low-temperature aging lie on low-index planes the direct observation of the atomic structure by high resolution microscopy becomes possible. Figure 8 shows a high resolution image of a secondary {100} end facet on one such particle. Since this facet is parallel to the beam, no overlap along the projected direction can confuse the image. A number of interesting features are seen in this micrograph. The primary (001) facet is atomically flat on the top face while a ledge with the thickness of one θ' unit cell is added at some distance from the end of the lower face. The secondary (100) facet that constitutes the end of this plate is remarkably flat, but the junctions between the (100) and (001) facets are not atomically sharp. Close inspection of the secondary facet reveals a small periodic relaxation every three (001)_{Al} planes where they meet their counterpart in the θ' lattice. The particle thickness at the end is 4.85 nm, increases to 5.45 nm further to the right and remains at this thickness over its entire length (not visible here). Elastic distortions accommodating the enclosed precipitate in the matrix are readily apparent by following the aluminum lattice along the right angle edges of the picture. An extra half plane ends at the top left edge of the particle where the major

and minor interface facets are joined (see arrow), leading to a twist of the lattice near the left edge of the picture.

A slight contraction of the Al lattice by about 0.2 nm (one (002) plane of the Al lattice) is visible near the right hand bottom edge of the image. This is consistent with the observation that in a conservative lattice correspondence 9 unit cells of θ' (9×0.58 nm) meet 13.5 unit cells of Al (27×0.202 nm). The difference of 0.23 nm must be accommodated elastically or by external dislocations.

HfN plates in Mo

The crystallography of HfN precipitates in Mo is very similar to that of θ' precipitates in Al and leads to a similar distribution of HfN plates on {001} planes of the Mo matrix (see Figure 9a) (24). However, due to the interstitial nature of nitrogen in the Mo lattice, the formation of HfN precipitates involves a large volume expansion (rather than a small volume contraction) and vacancies play an important part in the precipitation process (25). Again, the atomic match in the (001) habit plane is excellent and, as seen at high resolution in Figure 9b, this interface is perfectly coherent. This image shows that the particle thickness is such that the diagonal {110} planes of the Mo matrix are in registry, i.e. there is no net shear displacement.

As seen in Figure 9a these plates have mostly a circular or irregularly curved shape with a very pronounced facet on the habit plane. As was the case for θ' , the composite symmetry of cubic HfN in cubic Mo is tetragonal, but at this heat treatment temperature the average particle shape is that of a circular plate where only the major (001) facet is observed. However, it is likely, that under conditions of low-temperature aging, secondary facets would develop.

Carbide precipitates in Pt

In spite of its low solubility for carbon, carbide precipitates have been found to form readily in Pt during aging after a rapid quench (26). As in the two previous examples the carbides form as thin circular plates on {001} planes and thickening occurs by a ledge mechanism that can also be analyzed as a dislocation reaction. Again the interstitial nature of C in Pt necessitates vacancies for precipitation to occur, in fact C and vacancies are found to co-precipitate (27). While faceted square plates have been observed under some aging conditions, the plates investigated here were circular as shown in Figure 10a. From an analysis of stacking fault and dislocation contrast in conventional microscopy it was concluded that these particles were single layer carbides with a displacement vector of $\sim 1/3 \langle 001 \rangle$ and a vacancy strain field. The high resolution images shown in Figure 10b and c confirm this thickness and displacement directly. The image at Scherzer defocus is similar to that

of a pure {001} stacking fault while at -800 Å defocus small white dots appear at the positions of the carbon atoms. Whether or not carbon atoms are directly visible in such images remains to be determined by comparison with image simulations.

Precipitation of Ge in Al

Polygonal shapes are observed in the precipitation of Ge from Al-Ge solid solutions. Similar to the case of Mo-HfN and Pt-C, vacancies are essential for the precipitation process to occur because of the large volume increase.

Interestingly, even though the crystallography is simple, a large variation in orientation relationships and morphologies is found. This is illustrated with two dark field images in Fig 11. The micrograph in Fig. 11a was taken near the $\langle 111 \rangle$ zone axis and shows precipitates that are triangular, lath-like and trapezoidal in this projection. It is apparent from the thickness contours that some of these are plates while others are actually tetrahedral in shape (see arrows) (28). This observation is confirmed by the view along the $\langle 100 \rangle$ zone axis, Fig. 11b, where the tetrahedra project as squares. It has been pointed out by Pond (29) and by Hugo and Muddle (28) that this tetrahedral morphology ($\bar{4}3m$) was due to the intersection of the space group symmetries rather than merely the point groups of the two crystals. Laths along $\langle 100 \rangle$ directions as well as laths or plates along $\langle 110 \rangle$ directions are also visible. These images illustrate the great variety of observed morphologies and at the same time show the usefulness of observations along high-symmetry zone axes in the analysis of morphologies.

High resolution observations allow accurate determination of interface structures and faceting. Many such observations were made on $\langle 100 \rangle$ needles such as that shown in Fig. 12. For this particular orientation relationship faceting was found on {111} planes of the Ge precipitates. However, for orientation relationships of higher symmetry facets were found not on {111} planes but on the more symmetrical {100} or {110} planes (see e.g. Fig. 2). Clearly, the orientation relationship is important in the formation of particular facets and hence the development of precipitate/matrix interfaces. Particles with multiple facets such as tetrahedra or multi-faceted plates are likely to exhibit growth mechanisms that reflect the preference for flat, faceted interfaces.

Note that in the example in Fig. 12 internal twins lead to ledges at the {111} interface facets. It would be expected that these play a role in the growth of such faceted precipitates. Interestingly, the interfaces seen here show very little evidence of elastic relaxations of the type seen in the previous examples. No regular dislocations or long range distortions could be identified and it appears as though these interfaces, although faceted, could be incoherent or "reconstructed". Further investigation of these latter points is presently underway.

Continuous bicrystal structure in Al

It has recently been shown that when Al deposits in two crystallographically equivalent orientations on a {100} Si surface it forms a continuous bicrystal structure with the two crystals separated by 90° $\langle 110 \rangle$ tilt boundaries (30,31). The bicrystal diffraction pattern shown in Fig. 13 has been marked to highlight its symmetry which it has inherited, in part, from the single crystal Si substrate. The figure shows two $\langle 110 \rangle$ patterns of Al rotated 90° relative to each other. To distinguish the two patterns one has been marked by black dots. Mirror planes of the substrate have been indicated by their traces. The vertical and horizontal mirror exchange the black and white pattern while the diagonal mirrors reflect each pattern into itself. The vertical and horizontal mirrors are symmetry operations of the substrate that are not shared by the overlayer and are therefore variant-generating, or color symmetry, mirrors. By comparison, the diagonal mirrors are common to the substrate and the overlayer and hence common to the two variants. Similarly, a fourfold (90°) rotation is a color symmetry operation whereas a twofold (180°) rotation is shared by the substrate and the two orientation variants.

An intermediate temperature anneal of this bicrystal film leads to the preferential reorientation of the boundary planes (at fixed misorientation of $\sim 90^\circ$) into low-energy facets. It is interesting to note that the facets are preferentially aligned with the mirror planes of the substrate. This is illustrated in Fig. 14 with a pair of bright field micrographs taken under mirror-related two-beam conditions. The planar facets in the Al preferentially adopt the symmetrical $\{557\}_1/\{55\bar{7}\}_2$ and the asymmetrical $\{100\}_1/\{011\}_2$ boundary orientation, which they inherit from the mirror planes of the Si substrate crystal.

Due to the fact that a continuous bicrystal structure is a geometry in which the two crystal orientations are enclosed in each other, constraints at junctions of planar facets become important. A typical facet junction is found where a symmetrical meets an asymmetrical boundary. In addition to accommodating a small orientation change of 0.6° it is also necessary to accommodate the rigid body shift that has been shown to be associated with the symmetrical boundary. It has been pointed out by Pond and Vitek (32) and by Balluffi et al. (33) that this will lead to dislocations at steps and facet junctions. This effect of the enclosure of grains can have important consequences for the structure, morphology and mobility of grain and interphase boundaries.

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FIGURE CAPTIONS

Fig. 1 Schematic illustration of orientation relationship between tetragonal precipitate enclosed in a cubic matrix and related variant-generating symmetry operations.

Fig. 2 High resolution micrograph of lath- or plate-shaped Ge precipitate seen end-on in an Al matrix. The orientation relationship is as in Fig. 1. (Micrograph by J. Douin)

Fig. 3 Composite diffraction pattern of bcc precipitate in fcc matrix related by an invariant plane strain, showing misalignment of mirror planes.

Fig. 4 Schematic illustration of lattice rotation from orientation of high symmetry (a) to produce an invariant line transformation strain (b). The corresponding diffraction patterns (c) and (d) illustrate the effect on the spot splitting.

Fig. 5 Field of needle-or lath-shaped precipitates of Cr in a Cu matrix viewed along a $\langle 110 \rangle_{\text{Cu}}$ zone axis.

Fig. 6 Schematic illustration of the importance of loop closure for enclosed crystal interface dislocations. For the same Burgers vector and line direction, the shear set on the left can glide as the interface advances while the edge set on the right must climb.

Fig. 7 Faceted θ' precipitates in Al-4% Cu after aging 176h at 144°C. The three different $\{100\}$ variants all exhibit well-developed facets on $\{100\}$ and $\{110\}$ planes (a); dislocations encircling the face-on precipitate and small steps in the facets are seen in (b) (from ref. 8).

Fig. 8 High resolution image of the faceted end of a θ' precipitate shows the ledge structure, interfacial dislocations and matrix strain at the atomic level (from ref. 8).

Fig. 9 Conventional (a) and high resolution (b) micrographs of $\{001\}$ plate precipitates of HfN in Mo (from ref. 8).

Fig. 10 Conventional and high resolution images of $\{001\}$ carbide plate precipitates in Pt (from ref. 8).

Fig. 11 Triangular $\{111\}$ plates, $\langle 100 \rangle$ and $\langle 110 \rangle$ laths and needles, and tetrahedral-shaped Ge precipitates in an Al-1%Ge alloy are imaged in dark field in a $\langle 111 \rangle$ zone in (a) and a $\langle 100 \rangle$ zone in (b) (from ref. 8).

Fig. 12 High resolution image of Ge needle with rhombus-shaped cross-section, a single set of twins and $\{111\}$ interface facets. Note the disturbance in the facets where the twins emerge from the particle (micrograph by J. Douin, from ref. 8).

Fig. 13 Composite diffraction pattern from bicrystal of two 90° rotated variants of Al epitaxially grown on (100) Si. To emphasize color symmetry properties, one pattern is marked with black spots, and traces of mirror planes are outlined.

Fig. 14 Continuous bicrystal structure corresponding to the diffraction pattern in Fig. 13 showing faceting on substrate mirror planes after annealing. Complementary images in (a) and (b) were recorded in bright field under mirror-related two-beam diffraction conditions. (from ref. 34)

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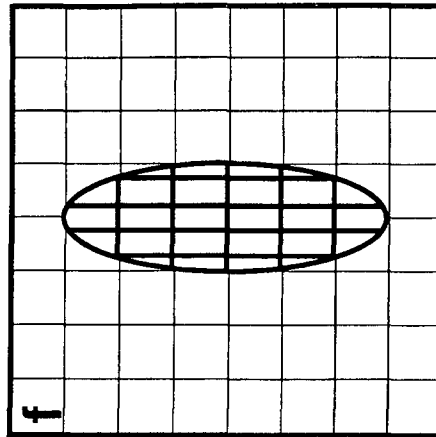
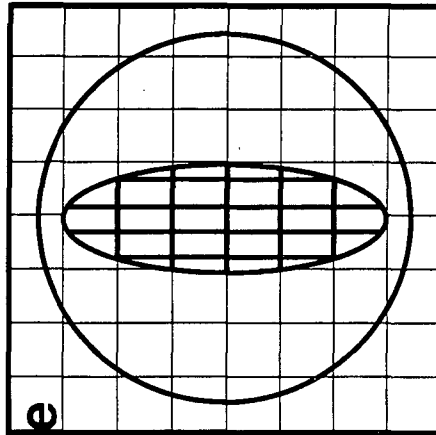
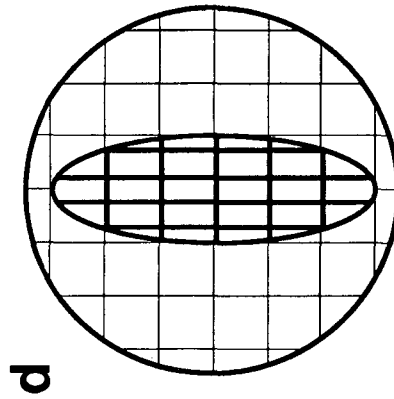
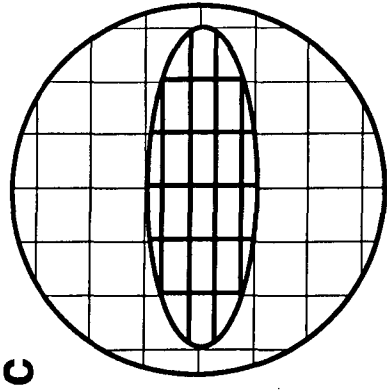
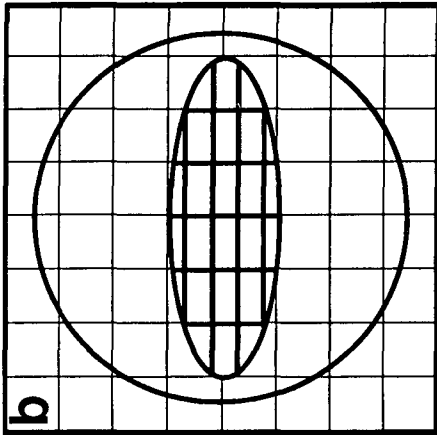
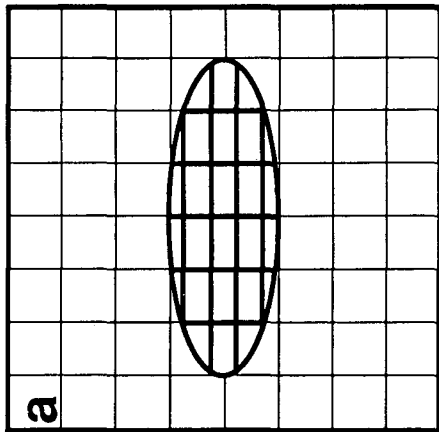


FIGURE 1

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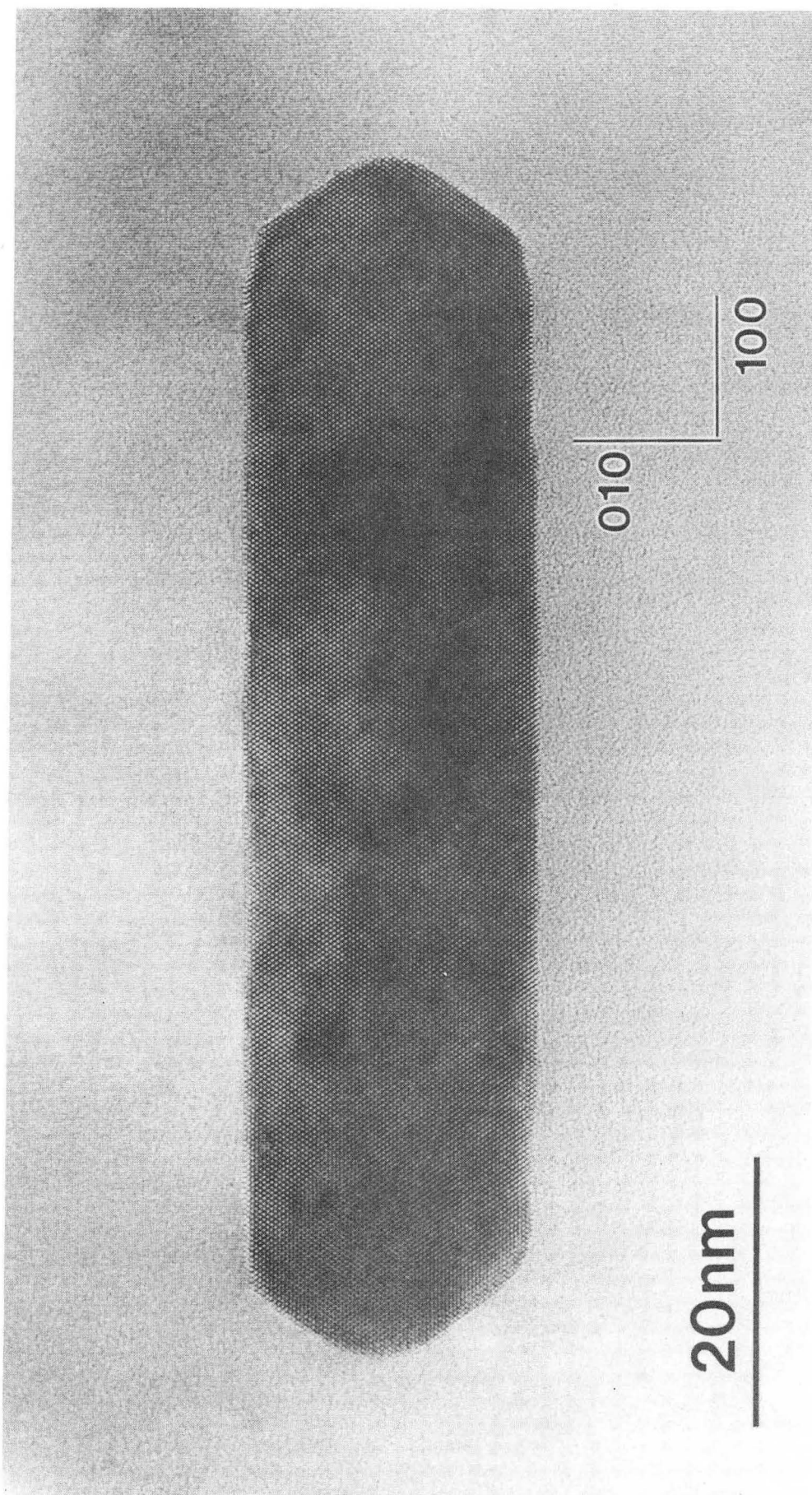
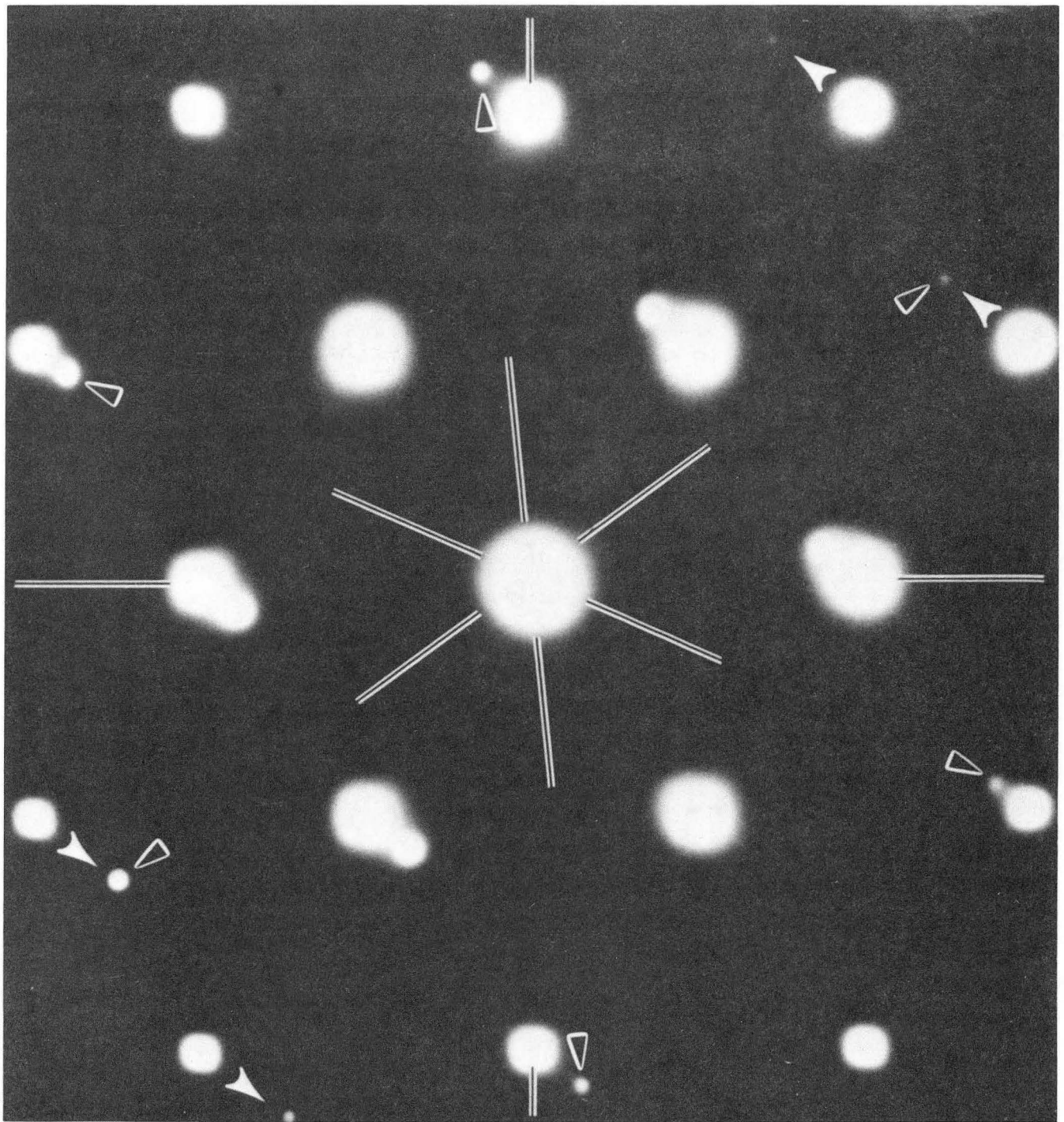


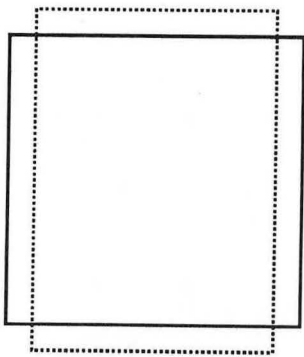
FIGURE 2



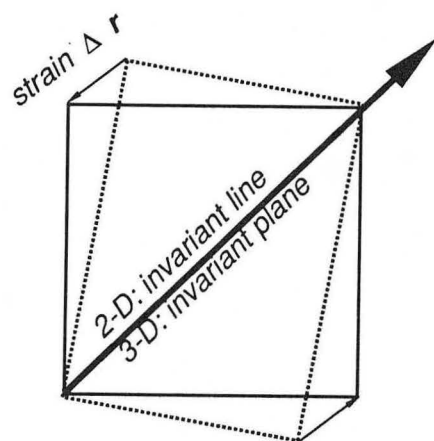
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FIGURE 3

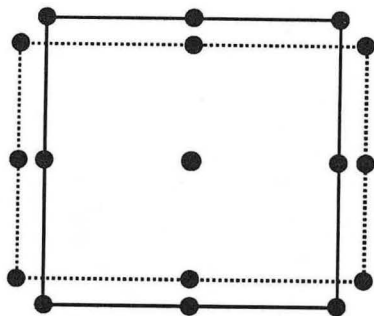
a



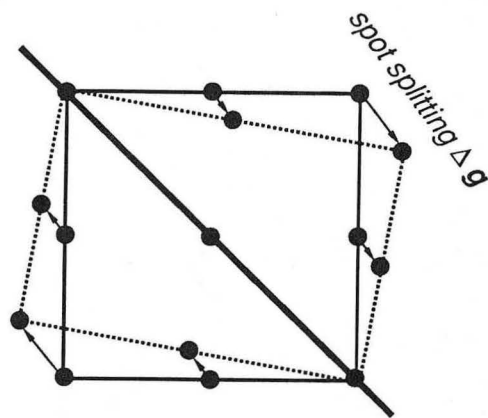
b



c

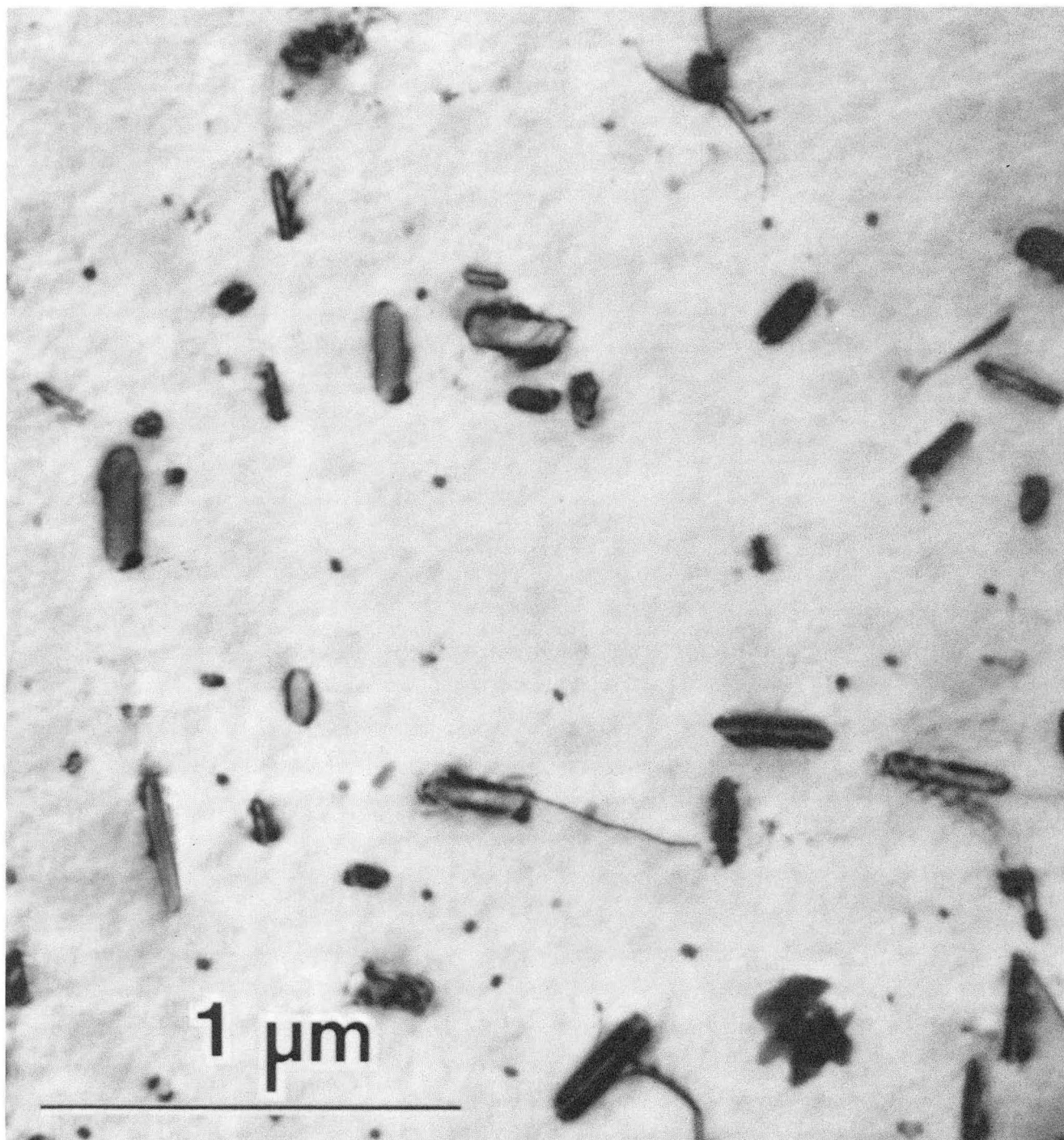


d



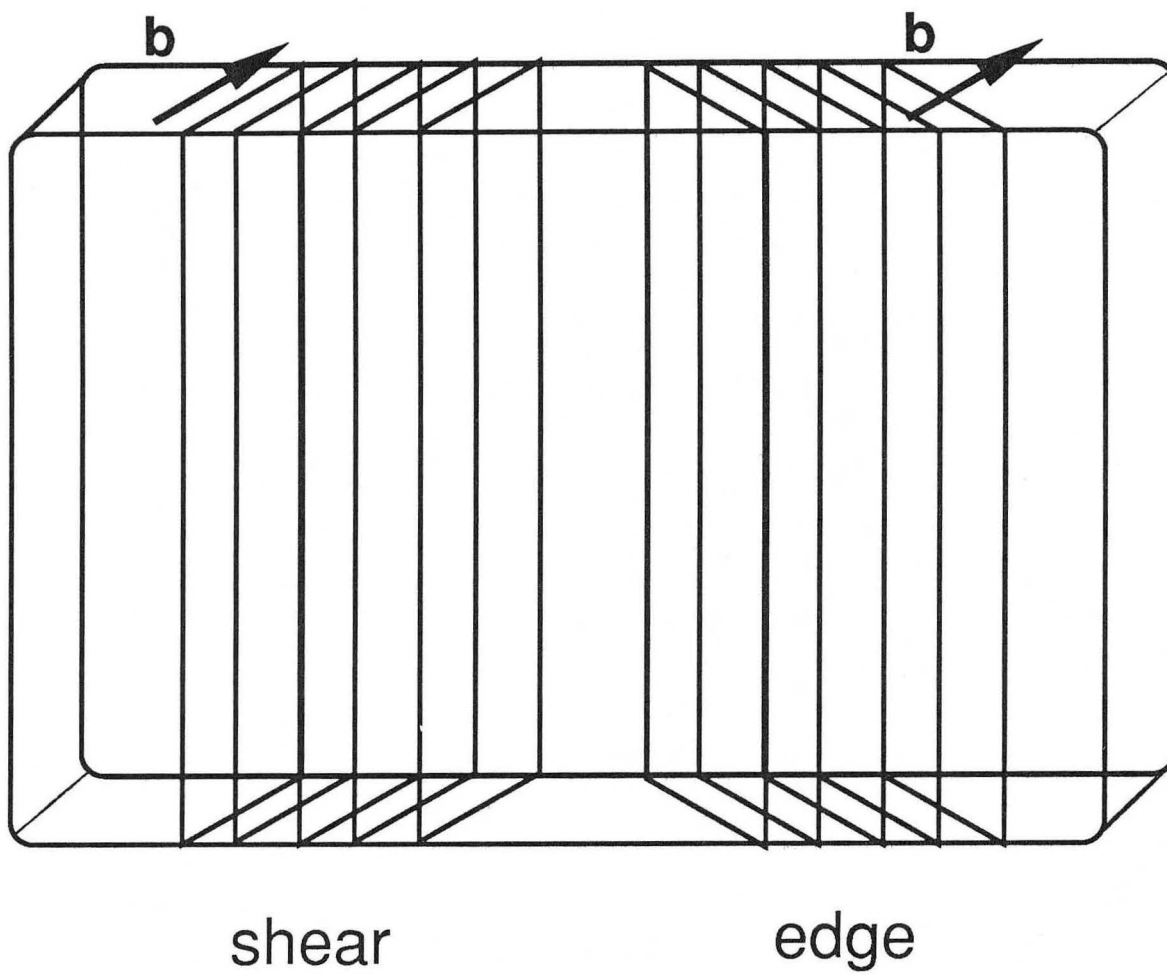
XBL 901-147

FIGURE 4



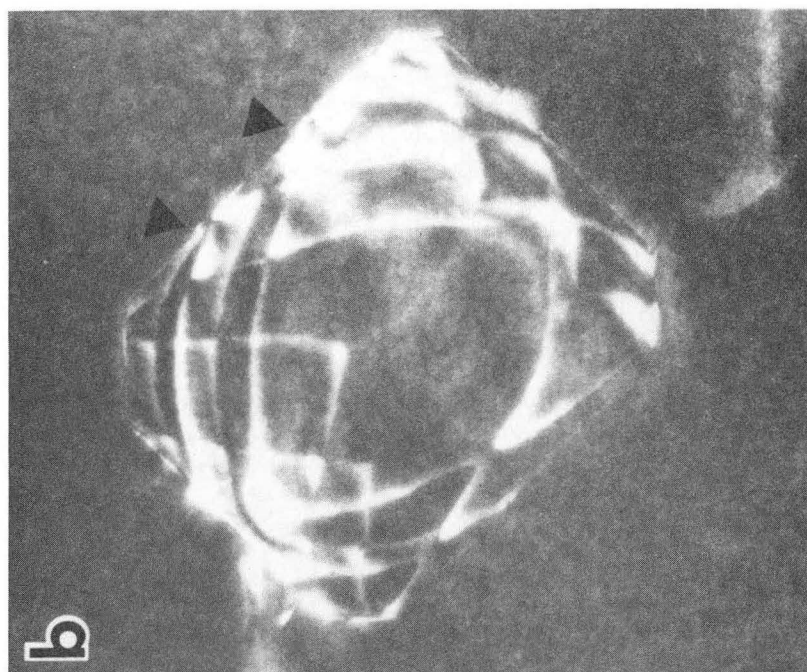
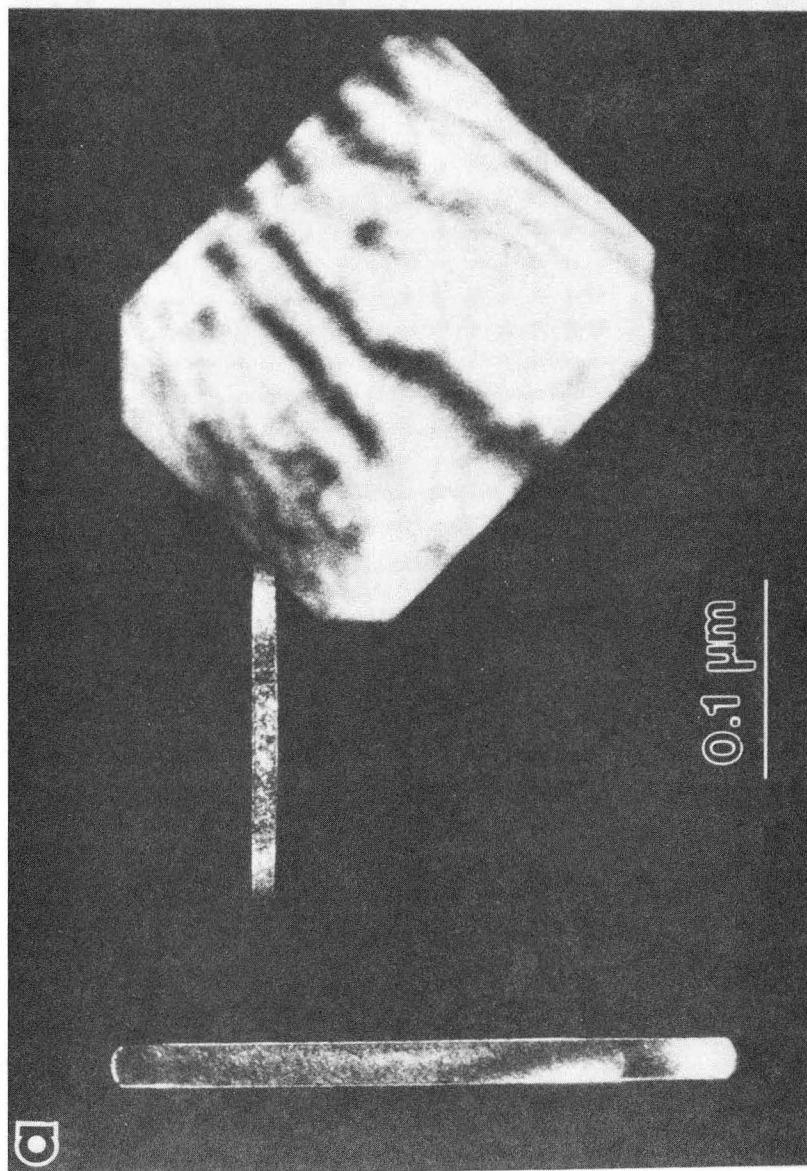
XBB 880-10428

FIGURE 5



XBL 901-146

FIGURE 6



XBB 899-8132

FIGURE 7

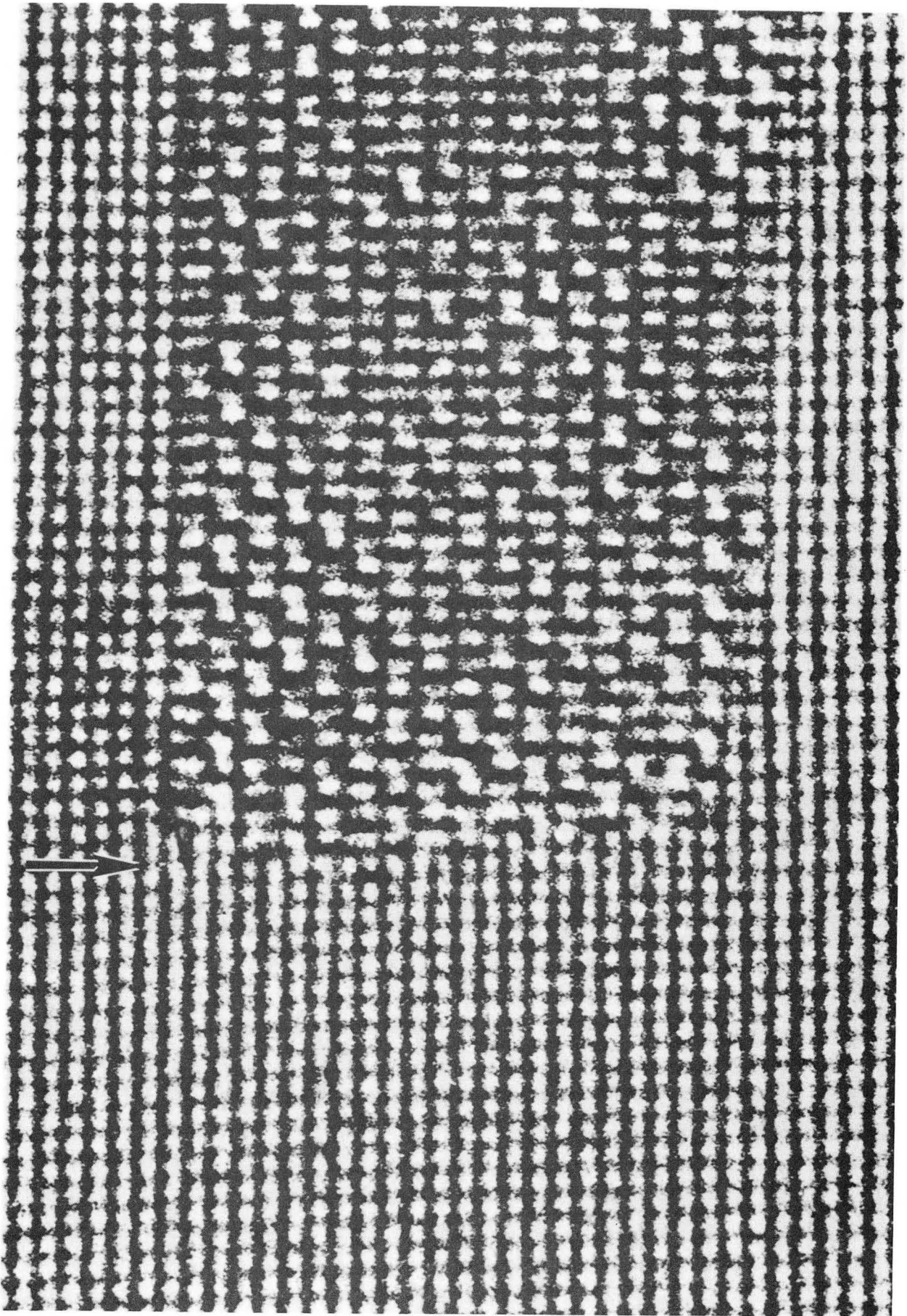
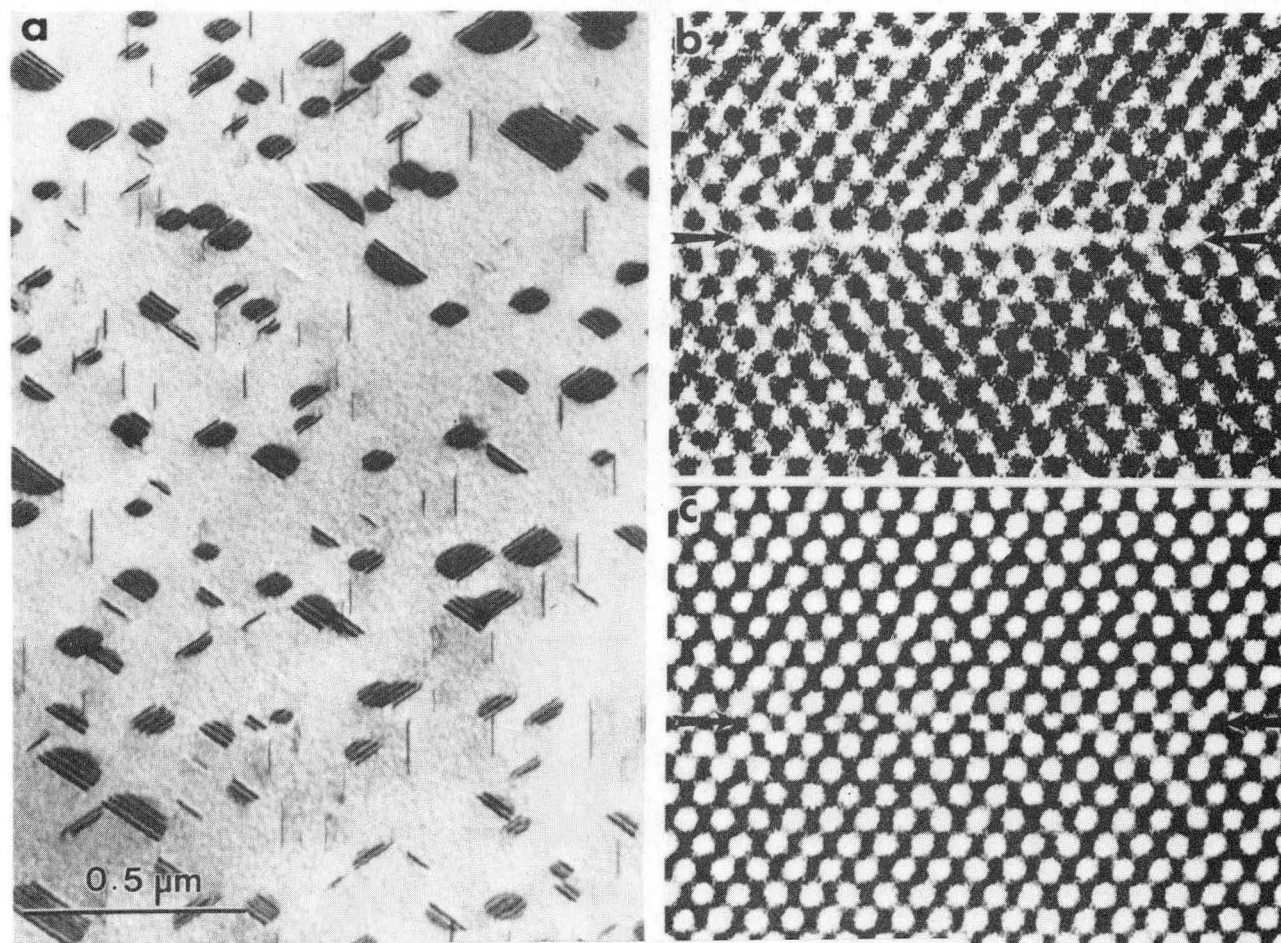
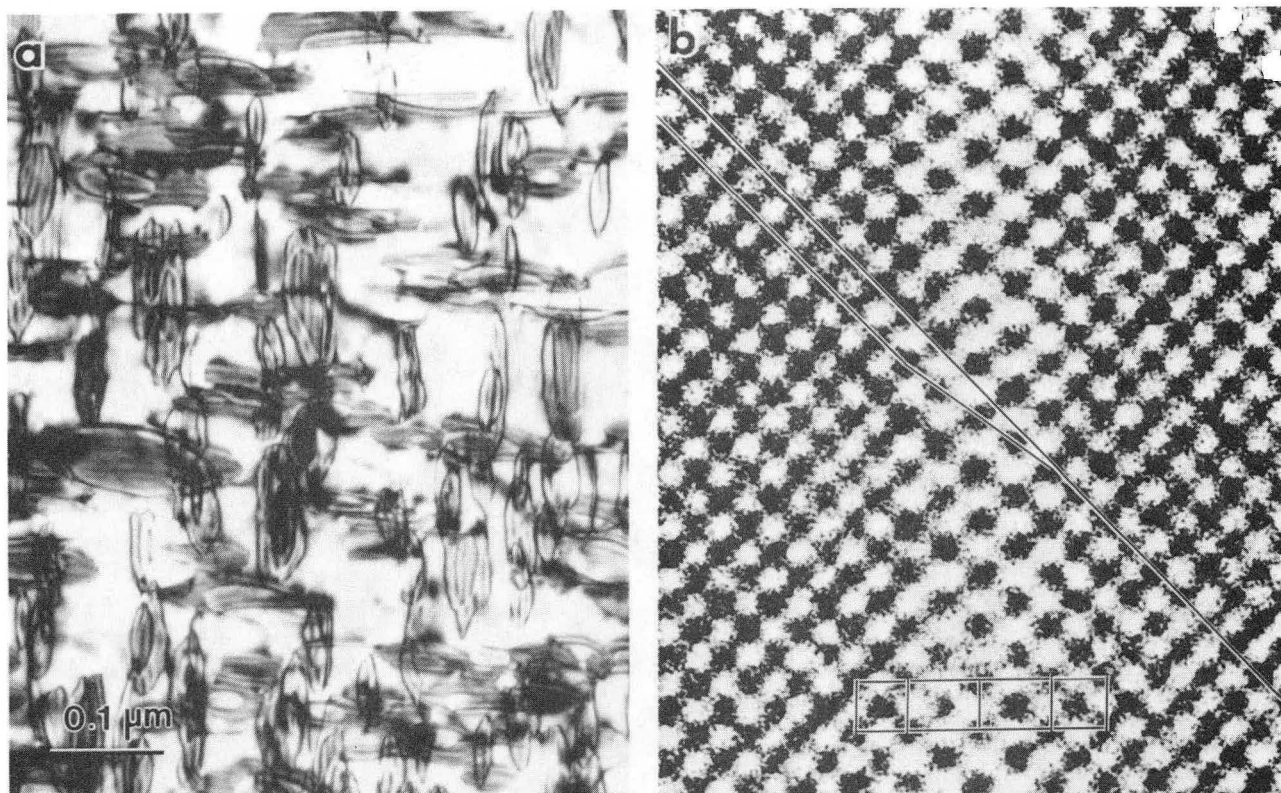


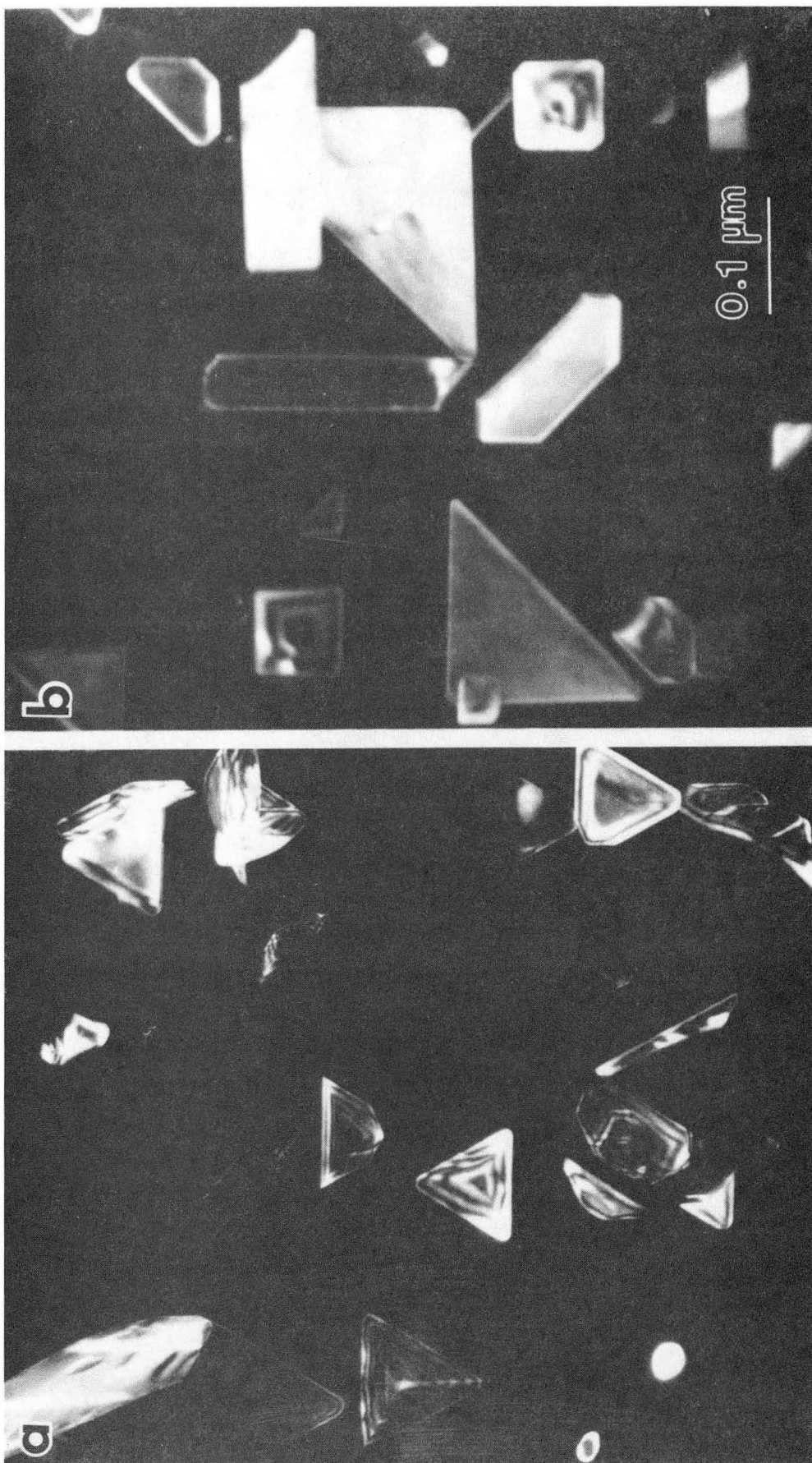
FIGURE 8

FIGURE 9



XBB 899-8136

FIGURE 10



XBB 899-8138

FIGURE 11

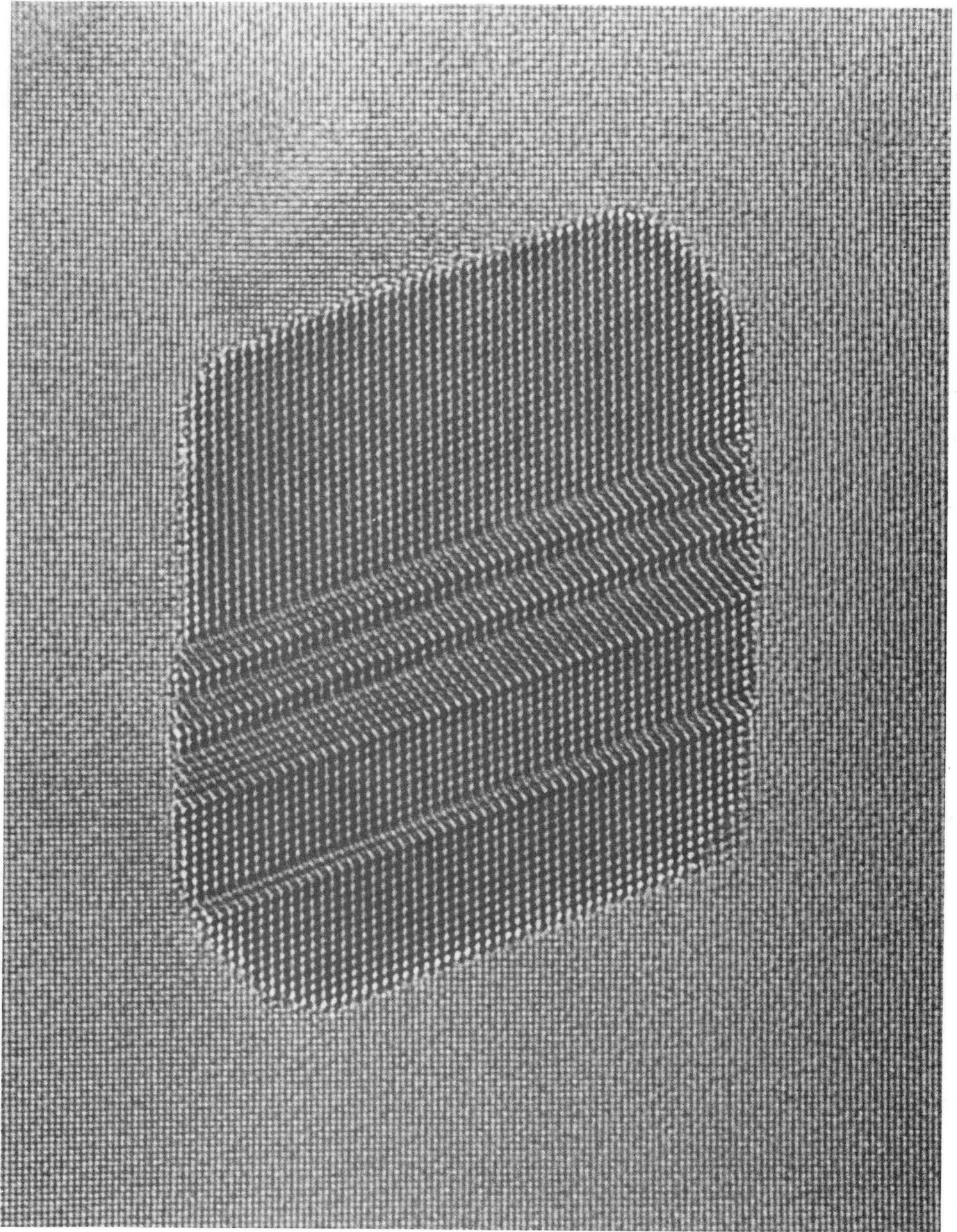
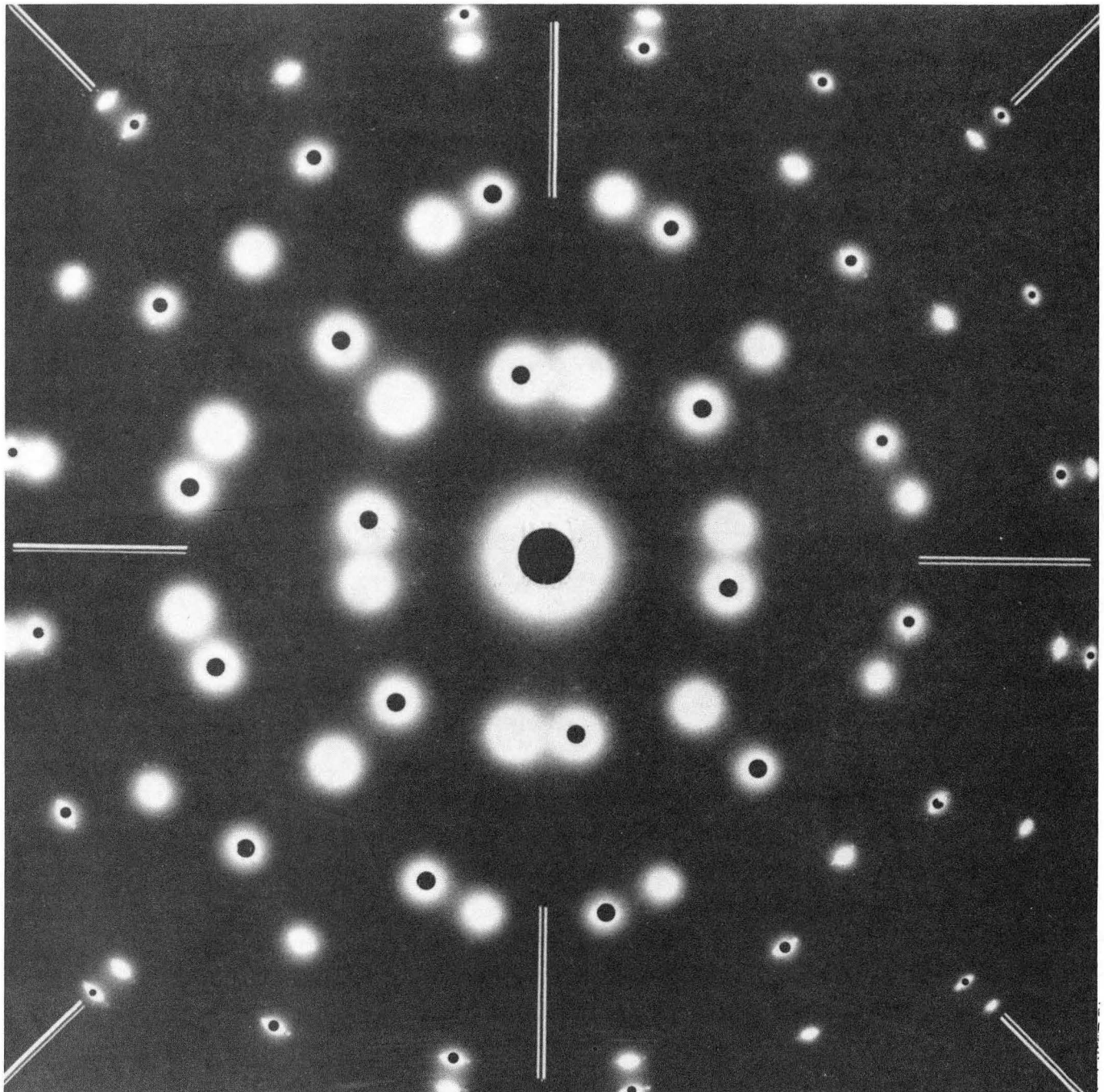
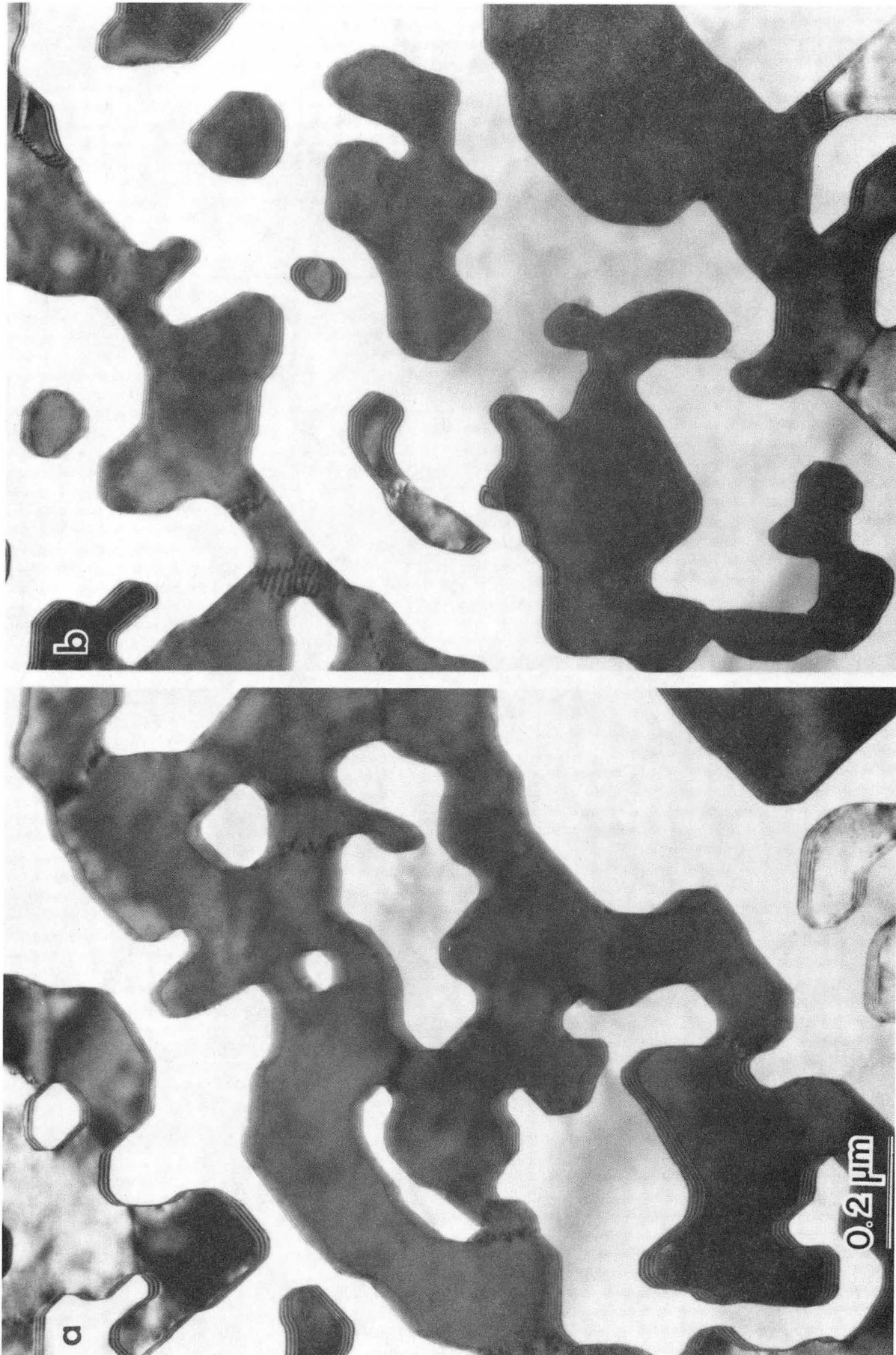


FIGURE 12



XBB 889-8861A

FIGURE 13



XBB 880-10325

FIGURE 14

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