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Berkeley, California

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ANALYSIS OF DISLOCATION LOOPS IN QUENCHED FCC METALS

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Master's Thesis

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and contradictions arise when the theory is applied to bright-field images, low-order reflections, thin foils, or defects which are large and not edge-on. In quenched copper it is found that Frank loops are stable below about $125\text{\AA}^2$ and perfect loops are the stable defect above this size.

I. INTRODUCTION

It is now well known that when fcc metals are quenched rapidly from near the melting point large vacancy supersaturations, up to 10^9 , can be retained. The excess vacancies subsequently anneal out to form vacancy clusters, stacking fault tetrahedra, or prismatic dislocation loops (for review, see Ref. 1). In metals of intermediate or high stacking fault energy, e.g., copper and aluminum, dislocation loops are formed. There are three types of prismatic loops possible: (1) Frank loops, which are pure edge dislocations on $\{111\}$ planes with Burger's vectors $b = a/3\langle 111 \rangle$; (2) perfect loops on $\{111\}$ planes with Burger's vectors $b = a/2\langle 110 \rangle$ which normally are formed by the conversion of Frank loops by the nucleation of Shockley partials and are no longer pure edge dislocations, and (3) rhombus loops also with Burger's vectors $b = a/2\langle 110 \rangle$ but formed by the rotation of perfect loops onto planes other than $\{111\}$ so as to minimize dislocation line energy.^{2,3} The rhombus loop appears to be the final stable defect and is commonly found in aluminum after certain quenching and aging procedures.⁴ Rhombus loops are nearly pure edge, as their habit planes are close to, but not exactly, $\{110\}$.

It appears that whilst the stacking fault energy should determine whether or not Frank or perfect loops are formed, in aluminum the quenching conditions seem to be the most important in determining the type of loop that is observed. If quenching is done such that large stresses are set up, mostly perfect loops are formed whereas if little or no deformation occurs, mostly Frank loops are formed.^{4,5} This suggests that Frank loops are formed initially but their conversion to perfect loops by nucleation

of a Shockley partial is stress aided, i.e., the transformation is heterogeneous. Impurities may also play a role in loop nucleation.

There is a fourth type of dislocation loop that may be found in quenched materials but is not dependent upon a supersaturation of vacancies for its formation. This type of loop is a deformation loop with Burger's vector $b = a/2 \langle 110 \rangle$ and is formed by dislocations interacting with other dislocations or impurities or precipitates,^{6,7} or by stress-relief mechanisms such as prismatic punching of loops.⁸

There are distinctly two types of loops possible in fcc metals, the vacancy type which results from the condensation of vacancies to form an intrinsic Frank loop and can be converted to a vacancy-type perfect loop, and the interstitial type which results from the condensation of interstitials to form an extrinsic Frank loop or by prismatic punching of dislocation loops, both which have their Burger's vectors normal to the plane of the loop. Conversion of the extrinsic Frank loop may take place resulting in an interstitial perfect loop on $\{111\}$, but such behavior has not been studied or reported.

Vacancy type loops are normally found in quenched materials and interstitials in irradiated materials. However, Edington and Smallman⁹ have found that both types occur in quenched pure aluminum. From climb rate measurements they estimated that the extrinsic and intrinsic stacking fault energies were about 460 ergs/cm^2 and 280 ergs/cm^2 respectively. Essman and Wilkins¹⁰ found that after neutron irradiation of copper intrinsic, or vacancy, Frank loops were formed. Punched interstitial loops as a result of quenching stresses around impurities are also common in a number

of metals and alloys.^{8,11} However, the primary defect to be discussed here is the vacancy type loop obtained by quenching.

Dislocation loops can be directly observed using an electron microscope, and the loops can be analysed if the microscope is equipped with a goniometric or double tilting device.¹² Dark-field experiments can be carried out if the electron gun is mounted such that it can be tilted from the microscope column by a few degrees in any direction. The problem of image detection involves two contrast mechanisms, diffraction contrast and strain contrast, both of which have been theoretically considered in terms of the dynamical theory of diffraction contrast for electron waves.

Diffraction contrast is the normal mechanism by which defects are observed and depends upon the distortion of the lattice surrounding a defect such as to modify the parameter s , the deviation (in reciprocal space) from ideal Bragg diffracting conditions, and the corresponding displacements introduce a phase change in both the transmitted and diffracted waves.¹³ Thus, locally near a defect, the intensity will deviate from background to give contrast. The position of the image with respect to the true position of the defect depends upon the product $(\bar{g} \cdot \bar{b}) s$,¹³⁻¹⁵ where $\bar{g}$ is the reciprocal lattice vector corresponding to set of crystal planes diffracting and $\bar{b}$ is the Burger's vector of the defect. In the case of a closed loop of dislocation line, the image of the loop lies either inside or outside of the true position of the loop depending upon, for a given loop in a given orientation, the product of these parameters. At $s = 0$ (exact Bragg case) the image coincides with the true position of the dislocation. Diffraction contrast is satisfactory for observing large loops but when the

size of a loop is less than a few hundred angstroms, the images of the sides are no longer resolvable and its strain field changes due to interactions from the various segments of dislocation line surrounding the loop. For small loops, therefore, diffraction contrast is no longer suitable for identifying the nature of the defect and strain contrast imaging must be used.

Strain contrast images are obtained from defects when the crystal is at or near the ideal Bragg condition and the parameter s does not play a role of much importance in the contrast mechanism. The parameter $\bar{g} \cdot \bar{b}$ responsible for phase changes by locally modifying s is still important, however, and very small and localized strain fields can be detected by strain contrast which may not give rise to significant diffraction contrast.

This paper will be concerned with the study of loops of various sizes and will necessitate the use of both contrast mechanisms; diffraction contrast for large loops and strain contrast for small loops. The perfect prismatic loop with the $\{111\}$ habit plane will be of primary interest and a model is proposed to explain the origin of double-arc contrast and the relation of the image to various crystal directions. An outline of the strain contrast theory will be presented and the predictions compared with experimental evidence in quenched aluminum and its alloys and copper. Also consideration will be given to the results obtained when loops large enough to be analysed by diffraction contrast are observed under strain contrast conditions.

A. Analysis of Large Loops

Large imperfect loops are readily distinguishable by their character-

istic enclosed stacking fault if the depth in the crystal covered by the loop is greater than one extinction distance, and vacancy loops can be distinguished from interstitial loops by performing one of a number of tilting experiments which have been well explored in other papers (e.g. Refs. 12,16). The large perfect vacancy loop has a singular distinguishing characteristic in that its image often appears as a discontinuous line image called the double-arc image. The model proposed below explains the origin of the double-arc contrast and relates the image to the crystallography of defects in the material.

Consider an imperfect or Frank loop as shown in top view (Fig. 1a) and in cross-section (Fig. 2a). Upon nucleation of a Shockley partial, the loop is converted to a perfect prismatic loop by the reaction $a/3[111] + a/6[11\bar{2}] = a/2[110]$ as shown in Figs. 1b and 2b. Now at each end of the loop, along the $[11\bar{2}]$ direction, there are two pure edge segments, but the extra half plane lies above the plane of the loop at one end and below this plane at the other end. Since the loop lies along $\langle 110 \rangle$ and is hexagonal in shape, the dislocation segments joining the two pure edge segments will be mixed screw and edge. The extra atoms corresponding to the partial edge character of these mixed segments will be above the plane of the loop on one half and below the plane of the loop at the other half of the loop.

If these extra partial planes are considered as a series of dipoles, the separation of the dipoles will decrease as the intersection of the mixed dislocation segments is approached. The contrast to be expected from any set of dipoles will be such that a cancellation of image intensity occurs to a degree dependent upon their separation. As predicted

for vacancy-type dipoles by Wilkins and Hornbogen¹⁷ and Cass,¹⁸ this cancellation of image intensity will increase with decreasing separation of the dipole segments. Because of this fact, the image to be expected from a perfect loop on {111} is one which has the strongest contrast from the pure edge segments of the loop and the weakest contrast from the intersections of the mixed dislocation components. Thus the double-arc image will be one in which the direction of the line connecting the missing segments of dislocation image lies along a $\langle 110 \rangle$ direction projected onto the surface of the foil and which is parallel to the two pure edge components of the loop.

For an arbitrary foil orientation as interpreted from a diffraction pattern or, more accurately, from a Kikuchi pattern,^{19,20} the projected $\langle 110 \rangle$ directions can be obtained from a stereographic projection oriented to correspond with the foil normal by suitable rotations of all the $\langle 110 \rangle$ poles. The micrograph can then be oriented with respect to the stereogram and the strong edge segments of the double-arc identified. A projection of the Thompson tetrahedron can easily be obtained from the stereogram and is a concise figure containing all the projected $\langle 110 \rangle$ directions.

Since each $\langle 110 \rangle$ direction is perpendicular to only one other $\langle 110 \rangle$ direction, the Burger's vector of any double-arc loop can be identified by inspection, that is, the Burger's vector, one of the $a/2 \langle 110 \rangle$, will lie along the $\langle 110 \rangle$ direction normal to that of the pure edge segments of the perfect loop on {111}. Since each $\langle 110 \rangle$ direction is common to two {111} planes, the identification of the plane of the defect is simplified by the reduction of the number of possibilities from four to two merely by

inspection of the direction of the line joining the missing segments of dislocation arc.

If the sense of the slope of the habit plane is known so that the foil normal can be unambiguously identified,¹² it is usually possible to identify a perfect loop as lying on that plane provided the loop is large enough and inclined far enough from the foil normal so that the directions of the six sides of the hexagonally-shaped loop are easily distinguishable. This is so because in any orientation except $\langle 100 \rangle$ and $\langle 110 \rangle$ all three projected $\langle 110 \rangle$ directions from one $\{111\}$ plane will not coincide with all three projected $\langle 110 \rangle$ directions from another $\{111\}$ plane. Since $\langle 100 \rangle$ and $\langle 110 \rangle$ are axes of two-fold rotational symmetry and thus are normal to mirror planes in the crystal, instances arise in these orientations where the projections of the $\langle 110 \rangle$ directions on adjacent $\{111\}$ planes are parallel, and in these cases the habit plane of a loop cannot be identified.

Figure 3 shows the projected $\langle 110 \rangle$ directions for various foil orientations and the double-arc loops that might be expected from loops on the various $\{111\}$ planes in each of the orientations. These cases are naturally idealized, representing the cases when exact orientations are observed and show the loops both just inside and just outside the true positions of the dislocation line bounding the loops. The actual images may not appear exactly as shown in Fig. 3, e.g., the corners would not be so sharp and the image position would vary with the operating reflection used for diffraction contrast. However, provided the loops are large enough so that both edge components of the images are resolvable, the characteristic skewness of the image enables an analysis to be performed without neces-

situating tilting experiments to find cases where $\bar{g} \cdot \bar{b} = 0$. The important point is that, although for any $\{111\}$ plane (not edge-on) in any orientation the projections of the hexagonally shaped loops are identical, there can result three different double-arc images, each of which is distinct from the other two. In addition, in the $\langle 112 \rangle$ and $\langle 111 \rangle$ orientations, loops on different $\{111\}$ planes are skewed differently and hence the image of the loop can be used to identify the habit plane.

Knowledge of the habit plane and its slope allows the determination of the character of a loop, whether it is a vacancy-type or an interstitial-type, by the analysis of the diffracting conditions giving rise to the image. For example, when $s > 0$ as in normal bright-field operation, if the Burger's vector of the loop is chosen to make an acute angle with the upward normal to the habit plane and thereby making $\bar{g} \cdot \bar{b} > 0$, the image of a vacancy loop will lie inside and that of an interstitial loop outside the true projected image of the loop while the reverse is true if $\bar{g} \cdot \bar{b} < 0$ (for review see Ref. 12).

B. Analysis of Small Loops by Strain Contrast

For very small loops the images from opposite sides of the loop overlap so that the defect appears as a black dot (bright field). In these cases detection must be done by strain contrast methods which image the strain field of the defect and allow conclusions to be drawn about their shape and nature, e.g., it is possible to distinguish between planes and three dimensional defects.¹⁵ The use of dark-field imaging techniques are of particular importance for a number of reasons. The primary reason is that, since strain contrast occurs only at or near the ideal Bragg

diffraction condition (i.e., when $s = 0$), the diffracted beam is more suitable for detecting strain fields because the dark-field intensity is symmetrical about the $s = 0$ position. Furthermore, dark-field images, when obtained by tilting the incident beam so that the diffracted beam travels down the center of the microscope column, have enhanced resolution over the transmitted beam due to the lower amount of background or scattered intensity present. The transmitted beam is not by any means useless, but the uncertainty in the value of s and the difficulty in observing an important field of view at $s = 0$ is not easily overcome. Also it is desirable to use the diffracted beam in order to evaluate results in the light of existing strain-contrast theory as developed by Ashby and Brown.¹⁵

The Ashby-Brown theory predicts the strain contrast image profile along the normal to an edge on circular dislocation loop can be approximated by the image profile of an edge dislocation line parallel to the surface of the foil. The extra half-plane intersects the nearer surface for an interstitial-type defect and intersects the farther surface for a vacancy-type defect. Thus a loop in the top half of the foil is approximated by a dislocation of one sign, and a similar loop in the lower half of the foil by a dislocation of opposite sign. Now based upon the symmetry properties of dislocation images as predicted by the dynamical theory at $s = 0$,^{13,21} the white-black image of a small dislocation loop at a given depth in the top half of a foil will be the reverse, in bright field, of the image of an identical defect in the lower half of the foil an equal distance above the bottom, whereas in dark field the images will be

identical. According to Ashby and Brown¹⁵ the relative contrast between the white and dark halves of the image decreases with increasing distance from the surface of the foil and becomes almost negligible for distances beyond half an extinction distance from either surface.

In summary, the present strain contrast theory predicts that for planar defects within half an extinction distance of either surface, the dark field images will be half-black, half-white elliptical images with the line separating the regions (the line of no contrast) perpendicular to the projection of the Burger's vector of the defects onto the plane of the foil. Vacancy-type planar defects will have the white side of the image on the side of the operating reflection for contrast and interstitial defects will have their dark sides to the direction of the operating reflection. Thicker regions of the foil are preferred to ensure a truly dynamical, rather than kinematical, situation.

The Burger's vector of the defect can be analyzed from the dark field image by observing the direction of the normal to the line of no contrast and comparing that normal with the projections of possible Burger's vectors onto the surface of the foil, e.g., a Frank loop showing strain contrast will exhibit a line of no contrast perpendicular to a $\langle 111 \rangle$ direction lying in or projected onto the plane of the foil. This technique was used by Essman and Wilkins¹⁰ to determine that the black-dot defects (in bright-field) formed by neutron irradiation of copper were primarily Frank sessile vacancy loops.

C. Strain Contrast from Large Loops

Although there is a lower size limit below which diffraction contrast

from loops is not useful for imaging the defects, there is no upper size limit for strain contrast images. At $s \approx 0$ any defect which strains the lattice will appear in strain contrast provided it has resolved strain in the direction of the operating reflection, $\bar{g}$. However, the strain field associated with the larger loops may be more nearly a pure dislocation strain field from the line bounding the loop, so each segment of the loop could contribute its own strain contrast image and the result would not be the expected strain field image of the loop.

An important distinction between the loops useful for observation by diffraction contrast and those useful for observation by strain contrast is that in the former case the loops should be far from edge-on while in the latter case they should be as close to edge-on as possible. Thus, unless care is taken in analysing which type of loop is being observed under strain contrast conditions the conclusions drawn can be misleading. For example consider a large perfect loop lying on a $\{111\}$ plane not normal to the foil surface. Under normal diffracting conditions the loop will appear as a double-arc with the missing segments along a projected $\langle 110 \rangle$ direction. Upon tilting the foil to obtain $s = 0$ for the particular diffracted beam operating, the pure edge segments of the double arc will exhibit strong dislocation strain contrast resulting in an image which might be mistaken for an edge-on loop image (see Ashby-Brown,¹⁵ Fig. 10). The important distinction that can be made is that this type of loop will have a zero-contrast line parallel to a projected $\langle 110 \rangle$ direction (that normal to its Burger's vector) whereas the true strain contrast image of a perfect loop should exhibit this line perpendicular to another $\langle 110 \rangle$

direction (that of its Burger's vector). A second distinction can be made in analysing the direction of streaking of the image associated with strain contrast. While streaking from an edge-on loop should be normal to the habit plane of the loop or along the direction of its Burger's vector, the double-arc loop observed under strain contrast conditions should appear to be streaked in a projected $\langle 112 \rangle$ direction. That is, the $\langle 112 \rangle$ direction in the plane of the loop normal to the pure edge components of dislocation line (see Fig. 3), when projected onto the foil surface, represents the direction of skewness of the image. A $\langle 112 \rangle$ direction can also easily be obtained from the projection of an oriented tetrahedron by connecting the midpoint of the $\langle 110 \rangle$ direction, to which it is normal, to the vertex of the angle opposite this line in the plane of interest.

II. EXPERIMENTAL VERIFICATION

All specimens were observed in a Siemens Elmiskop I electron microscope operated at 100kv and equipped with a double tilting stage. All dark-field micrographs were obtained by tilting the electron gun so that the diffracted beam passed along the optic axis of the microscope. Foils from quenched copper and aluminum or aluminum alloy specimens were prepared in the usual way by electropolishing. Aluminum and its alloys are suitable for investigations of large loops since by adjusting the quenching conditions, Frank, perfect, or rhombus loops can be obtained.⁴ Previous results on copper²² showed that only very small loops are usually produced after careful quench-aging. Thus foils from copper specimens were used to study strain contrast from small loops.

A. Large Loops by Diffraction Contrast

In Fig. 4 are shown a number of various orientations and the projections of the $\langle 110 \rangle$ directions in these orientations. It is seen that the line connecting the missing segments of dislocation arc is in all cases parallel to a projected $\langle 110 \rangle$ direction. Figure 5 shows that Frank loops, diamond shaped rhombus loops and deformation loops do not, as a rule, show double-arc contrast. Rhombus loops, when not lying exactly on the $\{110\}$ plane normal to its Burger's vector, may exhibit a tendency to lose a small amount of dislocation arc exactly as do the perfect loops on $\{111\}$ planes, but since the habit plane is not unique, geometrical analysis permits only the identification of their Burger's vectors. Deformation loops are normally formed such that the Burger's vector is either in the plane of the loop or normal to that plane, the latter also being the case

for a punched prismatic loop. If the loop is very narrow and sharply pointed on the ends, or if rotation on the glide cylinder takes place, some cancellation of image intensity may occur due to dipole relaxation of the strain field at these ends of the dislocations, but the habit plane in this case is also indeterminate.

Figures 6 and 7 illustrate the use of the model to identify defect systems in quenched copper foils. The diffraction patterns have been analysed as near-[111] in each case and the diffraction vectors indexed as shown. All rotations have been performed in comparing diffraction patterns and micrographs. Comparison of the loop in Fig. 6(a) with Fig. 3(c) indicates that, since the line connecting the missing segments of dislocation arc lies along $[1\bar{1}0]$, the loop lies either on (111) or $(\bar{1}\bar{1}1)$ and has as its Burger's vector $\bar{b} = a/2[110]$. Because of its nearly hexagonal shape, the loop must be on (111) . Rough measurements indicate that the image lies outside the true dislocation position, in which case it would unambiguously be a vacancy-type loop. Comparison of the loop in Fig. 6(b) with Fig. 3(c) indicates, by the line connecting the missing segments, that the loop lies on either $(\bar{1}\bar{1}1)$ or $(1\bar{1}\bar{1})$ and has as its Burger's vector $\bar{b} = a/2[\bar{1}01]$. From the geometry it can be seen that, of these two possibilities, only a loop on $(1\bar{1}\bar{1})$ matches closely the image, since the image of a loop on $(\bar{1}\bar{1}1)$ would be skewed in a different direction. There is a weak secondary image on the inside of the loop caused by a second beam, $\bar{g} = [0\bar{4}4]$ acting in a direction almost opposite to $\bar{g} = [22\bar{4}]$, thus this loop also analyses as a vacancy loop. The two loops in Fig. 7 have the same line of no contrast, the projected $[0\bar{1}1]$ direction, and

hence the same Burger's vector $\vec{b} = a/2[0\bar{1}1]$, and lie either on $(\bar{1}\bar{1}1)$ or $(\bar{1}1\bar{1})$. With changing $\vec{g}$, each loop image changes inside or outside the projected dislocation loop position, but does so in an opposite manner from the other. Comparison of these loops with Fig. 3(c) shows that loop A lies on $(\bar{1}\bar{1}1)$ and loop B lies on $(\bar{1}1\bar{1})$. Both loops therefore, are vacancy-type loops, as expected.

B. Small Loops by Strain Contrast

Once the difficulties in obtaining quenched copper specimens with a high supersaturation of vacancies were overcome,²² loops in copper proved to be very informative for the study of strain contrast because only a very small number of loops were observed to grow beyond about 200A in size. Loops as large as those presented in Figs. 6 and 7 were exceptional. Normally a foil oriented for good bright-field intensity revealed little more than a field of black dots, as shown in the right half of Fig. 8(a), which exhibited strain contrast when near bend contours or, as in Fig. 8(b), when the local value of s was reduced nearly to zero by tilting. Dark-field micrographs obtained for various single crystal foil normals and various operating reflections are shown in Figs. 9 through 14.

Since the white side of the strain contrast image lies, in the great majority of cases, to the same side of the zero-contrast line as does the operating reflection vector, $\vec{g}$, for all foil orientations and all operating reflections, the defects are vacancy-type defects. This result is precisely as expected, since quenching can only produce vacancy aggregation because the formation energy of interstitials is very large (about 4 ev).

The fact that the line of no contrast of the image makes various

angles with the operating reflection vectors (they are normal only for $\langle 220 \rangle$ and $\langle 111 \rangle$ reflections, otherwise the zero-contrast line lies perpendicular to $\langle 110 \rangle$ and $\langle 111 \rangle$ directions projected onto the plane of the foil) indicates that the vacancy-type defects are planar rather than spherical, since straining of the crystal only takes place in certain crystallographic directions. A spherical defect would always exhibit a zero-contrast line perpendicular to the operating reflection vector regardless of which vector it was, whereas planar defects must satisfy the $\bar{g} \cdot \bar{b} \neq 0$ criterion for visibility as demonstrated in Fig. 9.

The fact that the lines of no contrast are distinctly perpendicular to either projected $\langle 110 \rangle$ or $\langle 111 \rangle$ directions indicates that the Burger's vectors of the defects are of two types, $a/2\langle 110 \rangle$ and $a/3\langle 111 \rangle$, and thus unresolvable prismatic loops, both perfect and imperfect, are present.

The type of defect observed, whether perfect or imperfect, depends to a great extent upon the operating reflection for contrast. If a $\langle 220 \rangle$ reflection is used, as in Figs. 9(b) and 10, the primary defect observed, or the defect showing the strongest strain contrast, will be one whose Burger's vector lies along the operating reflection, that is, an $a/2\langle 110 \rangle$ type. If a $\langle 111 \rangle$ reflection is used, as in Fig. 11, the defect most likely to be observed will have a Burger's vector of the type $a/3\langle 111 \rangle$ lying along the direction of the operating reflection. If a reflection such as $\langle 200 \rangle$ in a $\langle 110 \rangle$ foil is used, then two $\langle 111 \rangle$ systems are resolved as shown in Fig. 12 and no strong $\langle 110 \rangle$ systems. In any case, the most easily observed defects will have Burger's vectors which lie in the plane of the foil. Defects with Burger's vectors not in the plane of the foil will give

rise to weaker strain contrast and may be entirely invisible if the angle between the Burger's vector and the plane of the foil is large.

When analysing the black-white strain contrast images of the vacancy precipitates it was found that it is important to distinguish between the line of no contrast (the line separating the black lobe from the white lobe) and the so-called quasi-symmetry line (the major axis of the elliptically shaped image) pointing from black to white, i.e., the direction of streaking. These lines may or may not be perpendicular depending on the orientation and type of defect. In Fig. 13(a) there are three distinguishable black-white directions, but there are four zero-contrast lines as shown in Fig. 13(b). Similarly in Fig. 14(a) there are a number of different defect systems as shown in Fig. 14(b). In Fig. 13 cases A, C, and D have zero-contrast lines perpendicular to their black-white directions, but case B has a zero-contrast line which lies normal to the direction of a projected $\langle 110 \rangle$ vector whereas its black-white direction is along a $\langle 111 \rangle$ direction in the plane of the foil. In Fig. 14 cases A, C, and E are all streaked normal to their zero-contrast lines while cases B and D are streaked in $\langle 111 \rangle$ directions but have their zero-contrast lines perpendicular to the $[1\bar{1}0]$ direction. These results indicate that the line of no contrast is always normal to the Burger's vector of the defect while the black-white direction of streaking is normal to the habit plane of the defect if this normal lies in the plane of the foil, otherwise the streaking may occur normal to the zero-contrast line if the loop is not edge-on.

C. Strain Contrast Images of Large Loops

Interesting images appear when large double arc loops are observed

under strain contrast conditions as shown in Figs. 15 and 16. The images appear similar to those predicted for edge-on loops when higher order beams are operating, that is, when the $\bar{g} \cdot \bar{b}$ product is 2 or more. However, the directions of streaking of the images are projected $\langle 112 \rangle$ directions and the apparent line of no contrast is parallel to rather than perpendicular to projected $\langle 110 \rangle$ directions.

Since the skewness is well defined in the strain contrast image, a comparison of the direction of streaking to the projections of the two $\langle 112 \rangle$ directions normal to the zero-contrast line will permit identification of the habit plane of the loop as shown in Fig. 15. If more than one defect system is in the field of view, as in Fig. 16, each can be analyzed separately by its line of no contrast and its direction of streaking.

III. DISCUSSION

A. Double-arc Contrast

The double-arc model fits observations very well and provides a rapid method for identifying the Burger's vector of a perfect loop since the line of no contrast (the line connecting the missing segments of dislocation arc) is very easily identified and compared with projections of $\langle 110 \rangle$ directions onto the plane of the foil. However, unless the loop is large enough to permit identification of the directions of its weak sides, it is difficult to determine the habit plane of the loop since two $\{111\}$ planes have the same common $\langle 110 \rangle$ direction. Due to the high stacking fault energy of aluminum, a perfect loop may rotate away from $\{111\}$ and exhibit less and less the double-arc effect as it approaches a pure edge dislocation configuration on $\{110\}$. Metals of lower stacking fault energy, such as aluminum alloys and copper, should have perfect loops bounded by an extended dislocation line and would therefore, have difficulty in changing their habit planes by rotation.²

Both the habit plane and its sense of slope must necessarily be known before identification of the nature of the defect can be made. The sense of slope can be determined in any of a number of ways such as by tilting the foil and observing the effect upon the size of the loop image²³ or, in the case of single crystals, by x-ray diffraction analysis of the specimen mounted in the specimen holder of the microscope, thereby eliminating the 180° ambiguity present in analyzing electron diffraction patterns.²⁴ Alternately, contrast experiments can be performed on a stacking fault or a slip trace (see Fig. 20) in the foil, comparing dark-field images to

bright-field images, to determine which side of the projected trace represents the top surface of the foil.¹⁶ Once the sense of slope of the $\{111\}$ plane is known, the slopes of the other three can be determined from a suitable oriented stereogram or a tetrahedron.

The calculated image profiles of Wilkins and Hornbogen¹⁷ predict, for the case of screw dipoles, that, when the image lies to the outside of the dipole, greater lessening of image intensity will occur the smaller the separation of the dislocations forming the dipole, and when the image is on the inside of the dipole, enhancement of the image intensity will occur down to very small separations. Upon this analysis a good percentage of randomly distributed perfect loops on $\{111\}$ planes should not exhibit double-arc contrast, but rather images somewhat like that of loop A in Fig. 15. However, since double-arc images are observed both inside and outside the true dislocation positions, this analysis cannot be strictly true for loops on planes inclined far from the foil normal but is qualitatively true for loops such as in Fig. 7, lying on planes making a small angle with the foil normal or for very small loops, that is, the image when inside the projected loop position appears as a single strong image. The treatment of the problem by Wilkins and Hornbogen is based upon the kinematical approximation for diffraction contrast which is really valid only for thin foils and for values of the parameter s far from zero.

On the other hand, Cass¹⁸ has applied the two beam dynamical theory of Howie and Whelan to calculate profiles for edge dislocation dipoles and predicts that dipole diffraction contrast images grow weaker in intensity

with decreasing separation for images both inside and outside the projected dipole position. This is also expected intuitively due to cancellation of the strain field at small separations. The conclusion that can be drawn is that both vacancy and interstitial type perfect loops on $\{111\}$ planes will show double-arc contrast both inside and outside the true projected loop position provided that the loop is large enough and inclined far enough from the foil normal. Perfect prismatic interstitial loops created in aluminum by alpha particle bombardment²³ do, in many cases, exhibit double-arc contrast although their habit planes are uncertain.

There is no reason why the use of the double-arc model should be restricted to fcc metals. Any crystal structure in which the Burger's vector of a loop makes an acute angle with the habit plane of the loop would be as suitable to analysis as is the fcc structure. For example, the bcc system may have loops on $\{110\}$ planes with Burger's vector of the type $\frac{a}{2} \langle 111 \rangle$ making a 55° angle to these planes. Meakin and Greenfield²⁴ have found interstitial loops in irradiated molybdenum which at times exhibit definite double-arc contrast. Projections of the $\langle 111 \rangle$ directions should lie normal to the lines of no contrast of these loops.

It has been implied that double-arc images arise purely by diffraction contrast where the magnitude of the $\bar{g} \cdot \bar{b}$ product determined the type of image, if any, that will be observed. However, in one particular instance, when the Burger's vector of a loop on (111) in a $[110]$ foil lies along the foil normal as interpreted in Fig. 17a, the $\bar{g} \cdot \bar{b}$ product is zero for every reflection in the plane of the foil. Therefore, if any double-arc image appears, it must do so purely by residual contrast caused

by lattice displacements normal to the direction of $\bar{b}$. Contrast from this type of lattice straining is neither unpredicted nor unobserved, being the type of contrast by which Frank loops in fcc or basal plane loops in hcp materials are often observed.^{16,25} Figure 17(b) shows the same loops as in Fig. 17(a) under strain contrast conditions where the line of no contrast is normal to the [002] direction as would be expected.

A pure edge loop in the plane of the foil, observed by residual contrast, may also exhibit double-arc contrast if the residual strain on parts of the loop is normal to the operating reflection. This is a trivial case, however, and is easily distinguished from the double-arc image of a perfect loop by the shape and nature of the image (neither foreshortening nor skewness will occur) and the fact that the line of no contrast is always normal to the operating reflection.

When large perfect loops on {111} are viewed edge-on, the double-arc model presented above would predict that, when the Burger's vector of the loop lies in the plane of the foil, the image will be two spots perhaps separated by a line of weak contrast. That this is indeed the case is illustrated in Fig. 18. Figure 18(a) is a dark-field image with $s = 0$ showing the strain field images (probable iso-strain contours) as thin black lines and Fig. 18(b) shows the same loops at $s \neq 0$ under diffraction contrast in dark-field. It should be noted that this double-spot image is unique to a perfect loop for which the normal to the habit plane and the Burger's vector of the loop both lie in the plane of the diffraction pattern, i.e. only in special instances in $\langle 110 \rangle$ foils will it occur. In all other cases of edge-on loops, whether perfect or imperfect,

the diffraction contrast image will be a single line (see Figs. 4 and 21).

Although Frank loops, when not edge-on, are generally distinguishable by their enclosed stacking fault or by their continuous length of dislocation line bounding the loop, it is possible to obtain images which are similar to double-arc images.⁵ If the loop is small enough so that two black fringes and one white fringe appear and the dislocation line bounding the loop is out of contrast ($\bar{g} \cdot \bar{b} = 0$), or very weak ($\bar{g} \cdot \bar{b} = \pm \frac{1}{3}$) it may appear to be a double-arc image but, in general, there would be no crystallographic relationships satisfied by this image except in special cases. The stacking fault fringes would be parallel to the foil surface and not along the edges of the loop as seen in the loops of Fig. 5(a). In this case, however, if there were any doubt, an operating reflection with a different extinction distance could be used and either more or less fringes would appear and the image would no longer be similar to a double-arc image.

B. Strain Contrast Images of Small Loops

The strain contrast images of small loops, in general, agree with predictions of the Ashby-Brown theory, but there is one assumption in the theory that needs further exploration. That assumption is the inherent one that bright-field micrographs of a foil containing small vacancy loops randomly distributed should have roughly about half the strain contrast images reversed. In investigations on quenched copper foils this has not been found to be the case and bright-field images, such as Fig. 19, are found to be not much different than dark-field images and the incidence of black-white images black on the side of the operating reflection is in

both cases very low. Brandon et al.²⁶ performed very convincing experiments on irradiated foils that verified Ashby and Brown's prediction that bright-field images would be asymmetrical and dark-field images symmetrical for defects near opposite surfaces of the foil. Furthermore, slip traces give rise to strain contrast images at $s = 0$, as shown in Fig. 20, that are the same in dark-field at both surfaces of the foil, while in bright-field the images are opposite at the two surfaces, illustrating the predictions are certainly valid at the foil surfaces.

A possible explanation for the conflicting results obtained in quenched copper is that the nature of the strain field and hence the strain contrast image may depend upon the size of the defect; the strain contrast images of very small defects may depend only on the sense of the displacement around the defect, that is, only on $\bar{g} \cdot \bar{b}$, while large defects may exhibit images which are compounded with diffraction effects and depend on $(\bar{g} \cdot \bar{b})s$.

Dark-field images also exhibit a dependence on s when large defects are observed. Whereas the strain contrast images of the small loops in copper are insensitive to the sign of s in dark-field and can be analyzed as vacancy loops on both sides of a bend contour, dark-field strain contrast images of large rodlike defects in doped silicon show a reversal of the black-white images as the sign of s is changed.²⁷ The dark-field images of slip traces shown in Fig. 20 indicate that strain fields near the top surface are not well resolved for $s > 0$ and those near the bottom are not resolvable for $s < 0$ suggesting that, for a given value of s , the detection of a defect by strain contrast is sensitive to its position in the foil.

It is improbable that defects need be restricted to within half an extinction distance of either surface in order to be detected by strain contrast. The Frank loops shown in Fig. 21 are nearly 1000A in diameter and exhibit remarkable strain contrast with the operating reflection $\bar{g} = [\bar{1}11]$ for which the half-extinction distance is about 325A. The half-extinction distance for the $\langle 111 \rangle$ reflection in copper is about 125A and, since a region denuded of defects is expected near the foil surfaces, only a very small number of very small defects should be observed for this reflection. Figures 11 and 22 show that an appreciable number of defects appear for the $\langle 111 \rangle$ reflection and that defect sizes up to about 200A still exhibit good strain contrast. Furthermore, dislocations observed under strain contrast conditions usually have easily visible black-white contrast at the center of the foil regardless of how the image may be complicated by primary extinction. An example of this is the dislocation at A in Fig. 22.

One feature not covered in the theory is the effect of varying foil thickness upon the black-white contrast. Whereas the calculated image profiles of Ashby and Brown are for a foil an integral number (five) of extinction distances thick, the best relative contrast, as can be seen in Figs. 8 and 13, occurs somewhere between the strongly-white areas and the strongly-dark areas of the foil, that is, for foil thicknesses of odd-quarter extinction distances. This would be the case intuitively, since neither strong white against a white background nor strong black against a dark background would give too much apparent contrast, whereas both would show well against a grey background.

In conjunction with this, the evidence near bend contours, such as in Fig. 22, indicates that the optimum viewing condition for the black-white contrast is not exactly at $s = 0$, but rather slightly to one side of this position. This is also illustrated in Fig. 23 in the vicinity of a small angle boundary. Normally in dark-field the small vacancy-type planar defects show: (a) weak strain contrast or appear as black dots for $s < 0$, (b) fair strain contrast for $s = 0$, (c) very good strain contrast for slightly positive values of s somewhere between the maximum intensity peaks for bright-field and dark-field intensity profiles, (d) very poor strain contrast normally appearing as white dots (black-dots in bright-field) for s positive such that maximum bright-field intensity is obtained.

Also, the black-white contrast generally appears better for foil thicknesses of four or more extinction distances where the background appears grey and there is less fluctuation of maximum to minimum intensity with increasing thickness. This is illustrated in Fig. 22 where the foil gets thicker going from left to right.

In certain instances strain contrast images appear which are the reverse of the vacancy-type image expected. This may be an effect associated with impurities such as prismatic punching as observed by Clareborough et al.¹¹ in copper quenched in an argon atmosphere and as illustrated in Fig. 24 for vacuum-quenched copper, or there may be particular depths in the foil at which the strain-contrast image is reversed, an effect predicted by the theory for defects at one-half an extinction distance from either surface. The incidence of this type of

image is highest for $\langle 111 \rangle$ reflections (see Figs. 11 and 22), and is almost non-existent for higher-order reflections such as $\langle 220 \rangle$ and $\langle 311 \rangle$. Therefore, this reverse image effect could be associated with defects near the foil surfaces where the dynamical theory becomes less applicable with lower-order beams. This last analysis is in complete contradiction to the theory since good strain contrast behavior is predicted only for defects near the surfaces of the foil, but the dynamical theory, and hence the strain contrast theory, should be modified to exclude a certain amount of the foil near the surfaces where there can be no dynamical interaction between the transmitted and diffracted beams. The distance $y = d/\theta$ is the depth below the top surface of a foil that radiation, scattered according to Bragg's law from the first scattering center on one plane of atoms, must travel before striking a scattering center on an adjacent plane. Dynamical interaction between the transmitted and diffracted beams is expected only at the scattering centers and not in the regions between, hence the dynamical theory, which treats the crystal as the continuum of scattering centers, is really not applicable in the first depth y of the foil. Similar considerations hold near the lower or exit surface of the foil, whence the dynamical theory is limited in its applicability to the region of the foil between a distance y of either surface. Since d gets smaller and θ larger with increasing h , k , and l , higher-order beams have a smaller distance y associated with them. Higher order reflections also have larger extinction distance so the ratio of d/θ to $t_0/2$ decreases with increasing order of reflection. Thus, the contradiction can be resolved by basing analyses on results obtained from higher-order

reflections. This attitude is commensurate with the suggestion of Ashby and Brown that higher-order beams be used, although they recommend it for reasons of enhanced resolution.

The defects observed in the quenched copper specimens were vacancy-type planar defects caused by quenching in a supersaturation of vacancies; such defects are not observed in unquenched or well annealed copper foils. The defects cannot have been caused by damage to the foil in the microscope by the electron beam since Howe et al.²⁸ demonstrated in ion bombardment experiments on copper that spots and loop-like features were produced only when a coated filament was employed and that no damage occurred when the microscope was operated under normal conditions. The strain contrast images shown in Figs. 8 through 14 cannot be due to end-on dislocations²⁹ because the number of different orientations used preclude the dislocations being end-on in all cases, and any result derivable from one image is also derivable from an image of a foil in a different orientation.

The size range of the Frank loops as identified by their strain contrast images appears to be from less than 60A to about 125A. Loops smaller than 60A in diameter are generally too small to give other than very weak strain contrast and loops larger than 125A almost always have the zero contrast line of the strain field image normal to a projected $\langle 110 \rangle$ direction. The size range of perfect loops as determined from their strain contrast images is between about 100A and 200A. Thus, the probable sequence followed for the condensation of vacancies after quenching copper is: single vacancies $\rightarrow$ divacancies¹¹ $\rightarrow$ clusters

(spherical or oblatelly ellipsoidal)^{30,31} → Frank loops → perfect loops at about 100-125A. That the perfect loop is the stable defect at large size is supported by the fact that almost all large loops observed, although of considerable less density than those small loops observed by strain contrast, have been of the perfect type as indicated by their double-arc appearance. Figure 25 illustrates the relative size of some of the larger double-arc loops to that of the loops observed by strain contrast.

When loops become larger than about 200A, the strain contrast images seem to be no longer of a simple white-black type. Instead a small arc of white appears on the black side of the image and a corresponding small arc of black on the white side of the image as witnessed in most of the strain contrast images from aluminum and the larger strain contrast images from copper (see Figs. 24 to 26). Although this is most obvious for high-order operating reflections, it sometimes occurs for the $\langle 111 \rangle$ reflections, so that this effect cannot be entirely associated with $\bar{g} \cdot \bar{b} > 1$ where such intensity variations are expected from theoretical dislocation intensity profiles.¹³ It may be that there is a superposition of diffraction contrast effects from the dislocation line bounding the loop and strain contrast effects from the strain field of the loop as a whole.

A feature exhibited in Fig. 18 is that the strain contrast images for large edge-on perfect loops are exceptionally weak for loops as large as 500A in diameter while Fig. 21 shows that imperfect loops up to 1000A in diameter still exhibit remarkably good strain contrast. An idea of the approximate upper size limit for the usefulness of strain contrast for identifying the Burger's vector of a perfect loop can be obtained from Fig. 26 where loop B, about 250A in size, appears as a

white dot on either side of the $[2\bar{2}0]$ bend contour (in a bright field image it would appear as a black-dot defect) and shows good strain contrast with the line separating the black and white areas normal to the $[220]$ direction. Loop A, about 300A in size, appears first as a strain contrast image with its apparent line of no contrast normal to the $[1\bar{1}\bar{1}]$ rather than the $[1\bar{1}0]$ direction and then exhibits the characteristic doubly-spotted image expected from an edge-on perfect loop (Fig. 18). Therefore, while the size of an imperfect loop is unimportant as long as it is nearly edge-on, the size of a perfect loop on a $[111]$ plane seems to determine whether its line of no contrast is normal to its Burger's vector or appears to be parallel to the plane of the loop. Thus, there is a maximum size beyond which strain contrast is no longer useful for identifying the Burger's vector of a perfect loop. This size is estimated to be about 200A to 250A in both copper and aluminum. This is indicated in Fig. 26 for aluminum and 200A defects in copper show good strain contrast with the line of no contrast normal to projected $\langle 110 \rangle$ directions while perfect loops about 250A in size exhibit double-arc contrast as seen in Fig. 8, loop A.

C. Strain Contrast from Large Loops

Large double-arc loops observed under strain contrast conditions exhibit also the type of image that has arcs of white on the black lobe or arcs of black on the white lobe, but there are a number of important distinctions that can be made to differentiate them from the more complicated images of small loops. The first is that the line of no contrast lies parallel to, not normal to, a projected $\langle 110 \rangle$ direction. The second

is that the direction of streaking lies along a projected $\langle 112 \rangle$ direction and not along the projection of the Burger's vector or along the projection of the normal to the defect plane. The third is that the lobes may not be always black on one side of the zero contrast line and white on the other, but may be black on both sides or white on both sides, with the characteristic smaller arc of opposite color enclosed within the lobe. This last effect is most probably due to dislocation image variations with foil depth and contrast conditions such as the sign of the parameters $\bar{g}$, $\bar{b}$ and s , and defect size and inclination to the incident beam. The important fact is that erroneous conclusions can be arrived at if the strain contrast image is considered to be the image of the strain field of an edge-on planar dislocation loop, i.e. the habit plane and the Burger's vector can be completely misinterpreted.

IV. CONCLUSIONS

Perfect prismatic dislocation loops on $\{111\}$ planes in fcc materials exhibit double-arc contrast in electron microscope images because the displacement field responsible for diffraction contrast is weakened by the dipole-like interaction of the edge components of dislocation line at the intersections of the mixed screw and edge segments of the loop. Double-arc contrast disappears as the loops rotate away from $\{111\}$ to $\{110\}$ planes where they become pure edge dislocation loops. This double-arc model is supported by observations and serves as a useful tool in analysing the Burger's vector of the loop.

Although this model was applied toward vacancy loops, it should be equally as valid for interstitial loops. Also, a metal with a different crystal structure, such as bcc, should display loops exhibiting double-arc contrast if these loops lie on planes which are not normal or parallel to their Burger's vectors.

In metals with intermediate stacking fault energies, where loops are confined to the $\{111\}$ habit planes, the model is further useful in allowing the habit plane of a loop to be established in most cases, provided that the loop is large enough to allow easy observation of the directions of its sides and further provided that the sense of slope of the habit plane can be determined by tilting or contrast experiments.

The predictions of the Ashby-Brown strain contrast theory are valid provided care is taken to obtain data under the conditions specified by the theory. These are: (a) use dark field images, preferably obtained by tilting the electron gun in such a manner that the diffracted beam

passes down the center of the microscope column, (b) maintain diffracting conditions close to $s = 0$, preferably slightly positive, (c) utilize thicker regions of the foil to eliminate strong background fluctuations and eliminate ambiguity in identifying the character of a defect, (d) use high order reflections both for enhancement of resolution and to minimize the region near the foil surface where the inapplicability of the dynamical theory may cause conflicting results to be obtained, (e) limit the use of strain contrast images to defects which are impossible to analyse by diffraction contrast.

Further features that would be desired in the theory are: (a) an analysis of strain contrast image intensities with respect to background intensities with varying foil thickness and varying values of s , the deviation parameter, (b) an analysis of defect images as a function of defect size, i.e. the effect of changing from the strain field of a small loop to the strain field of a dislocation line, (c) an analysis of when the parameter s plays an important role in determining the arrangement of a black-white image, (d) an analysis of bright-field images as a function of defect size and placement in the foil, and as a function of changing s , (e) an analysis of whether or not strain contrast images are obtainable only from defects near the surfaces of the foil and exactly what type of images would be expected from defects so near the surfaces that there is no true dynamical interaction between the transmitted and diffracted beam for a given set of diffracting planes.

Based upon the strain contrast images of loops in quenched copper foils it is concluded that perfect loops are the stable defect for loop

sizes greater than about 125A and Frank loops are stable below this size and down to less than 60A. Diffraction contrast results revealing double-arc loops as the primary defect support this conclusion and indicate, by the low density of these large loops that the quenched-in vacancy concentration is not nearly as high as that obtainable in quenched aluminum or that annealing out of vacancy clusters takes place at an early stage.

Lastly, strain contrast images of large loops can easily be obtained, but care must be exercised in analysing such loops by the strain contrast theory since this theory may not be applicable if the loops are perfect loops or if they lie upon planes which are far from being parallel with the incident electron beam. Perfect loops on {111} planes not parallel to the incident beam will have strain contrast images with a line of no contrast parallel to a projected $\langle 110 \rangle$ direction and appear to be streaked in the projected $\langle 112 \rangle$ direction which is normal to the zero contrast line and lies in the habit plane of the loop. This direction of streaking serves as a further aid in identifying the habit plane of a loop.

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FIGURE CAPTIONS

- Fig. 1 (a) Top view of a Frank loop on (111). Empty spaces indicate the missing layer of B atoms inside the loop. (b) Same loops after nucleation of an $a/6[11\bar{2}]$ partial dislocation and transformation to a perfect prismatic loop. Extra atoms between A and B positions are below the loop plane, those between B and C positions are above the loop plane.
- Fig. 2 (a) Side view of a Frank loop showing hcp stacking sequence across the loop plane. Viewing direction is $[1\bar{1}0]$. (b) Side view of the same loop after being converted to a perfect prismatic loop. The fcc stacking sequence has been restored but there are two extra half planes, one above and one below, at the ends of the loop.
- Fig. 3 Tetrahedra, bounded by $\langle 110 \rangle$ directions for (a) $[001]$, (b) $[11\bar{2}]$, (c) $[111]$ and (d) $[110]$ projections, and appearances of double-arc images for each projected $\{111\}$ plane. Images are shown both to the inside and to the outside of the true loop position for each $\{111\}$ plane except those edge-on. Position and width of an image will vary with contrast conditions and operating reflections.
- Fig. 4 Double-arc images of loops and projected tetrahedra for the foil normals: (a) $[013]$ (about 18° from $[001]$), (b) $[325]$ (about 6° from $[112]$), (c) $[345]$ (about 11° from $[111]$), and (d) $[47\bar{1}]$ (about 17° from $[110]$). Figures (a) and (b) are from pure aluminum quenched from 550°C into water at 20°C and aged 24 hours at room temperature. Figures (c) and (d) are from aluminum-7% magnesium alloys quenched from 550°C into iced brine and aged 100 hours at 100°C .

Fig. 5 Various loops which do not exhibit double-arc contrast are: (a) Frank loops as obtained in pure aluminum quenched from 550°C into silicone oil at 20°C and aged 24 hours at room temperature. Frank loops, in addition to exhibiting continuous dislocation line images, are characterized by their enclosed stacking fault fringes which are parallel to the foil surface rather than the $\langle 110 \rangle$ directions of the dislocation lines bounding the loops. (b) Rhombus loops as obtained in aluminum-5% magnesium quenched from 550°C into silicone oil at room temperature. The rhombus loops are perfect prismatic loops but have rotated so that they lie nearly normal to their Burger's vectors. (c) Deformation loops caused by the interaction of moving dislocations during bending or other deformation of the specimen. The loop shown is from a copper specimen quenched in vacuum by liquid nitrogen-cooled helium.

Fig. 6 Illustrations of the use of the double-arc model to identify defect systems in quenched copper: (a) The line of no contrast of the loop is parallel to $[1\bar{1}0]$, indicating the Burger's vector is $\bar{b} = a/2[110]$ and that the loop is either loop A on (111) or loop B on $(\bar{1}\bar{1}1)$. The shape of the loop proves that it is loop A. (b) The line of no contrast of the loop is parallel to the projection of $[101]$, indicating that $b = a/2[\bar{1}01]$ and that the loop is either loop A on $(\bar{1}\bar{1}1)$ or loop B on $(1\bar{1}\bar{1})$. The skewness of the image is used to determine that it is loop B.

Fig. 7. The lines of no contrast of the loop shown are normal to the operating reflections in quenched copper, thus $b = a/2[0\bar{1}1]$. (a) The skewness of loop A indicates that it lies on $(\bar{1}\bar{1}1)$. (b) The skewness of loop B indicates that $(\bar{1}1\bar{1})$ is its habit plane.

Fig. 8 Bright-field images of defects in vacuum-quenched copper: (a) Defects appear primarily as black dots for $s > 0$ except near an equalthickness contour. (b) The same defects exhibit black-white strain contrast when $s \approx 0$. Loop A, about 250A in size, exhibits double-arc contrast. Better strain contrast images are observed in the grey areas separating the white and black regions.

Fig. 9 Dark-field images of quenching defects in copper indicate that the defects observed are planar rather than spherical. (a) The operating reflection is normal to the Burger's vectors of a large number of defects, hence $\bar{g} \cdot \bar{b} = 0$ and no strain contrast is observed. (b) The operating reflection is parallel to the Burger's vectors $\bar{b} = a/2[01\bar{1}]$, $\bar{g} \cdot \bar{b} = 2$, and the defects are observable.

Fig. 10 The primary defects observed in dark-field using a $\langle 220 \rangle$ operating reflection in a $[111]$ foil are those which have their Burger's vectors along the operating reflection, i.e. perfect loops.

Fig. 11 The primary defects observed in dark-field using a $\langle 111 \rangle$ operating reflection in a $[110]$ foil are Frank loops normal to that reflection. However, those loops at F have their zero-contrast lines normal to $[\bar{2}20]$, indicating that they are perfect prismatic loops. Most loops are vacancy-type and have their white sides on the side of the operating reflection but the loops at G are reversed.

Fig. 12 In dark-field, the use of the $[002]$ operating reflection in a $[110]$ foil reveals two defect systems, H and J each with their zero-contrast line normal to a different $\{111\}$ direction while no $a/2\langle 110 \rangle$ defect systems are observed.

Fig. 13 Defects which have their lines of no contrast normal to the direction of streaking of the strain contrast image such as cases A, C, and D of this dark-field image are the most common. However, if the normal to the habit plane of a perfect loop lies in the plane of the foil, the direction of streaking of the image will be along this $\langle 111 \rangle$ direction as in case B, while the zero-contrast line is normal to the projection of the $a/2 \langle 110 \rangle$ Burger's vector of the loop onto the plane of the foil. Note that the best strain contrast images occur in the grey regions (thicknesses of oddquarter extinction distances) rather than in the strongly white or black regions.

Fig. 14 In a $[110]$ foil, the dark-field strain contrast images of Frank loops, A and E, are streaked normal to their lines of no contrast, while the images of perfect loops on the same habit planes, B and D, have zero-contrast lines normal to $[\bar{2}20]$ but are streaked normal to their habit planes. The defect at C may be a perfect loop which has rotated to become normal to its Burger's vector $\bar{b} = a/2[1\bar{1}0]$.

Fig. 15 A perfect prismatic dislocation loop on $\{111\}$ will appear as: (a) a double-arc image under normal bright-field diffracting conditions as seen in water-quenched aluminum, and (b) a complicated strain contrast image near $s = 0$. The lines of no contrast of the strain contrast image and the double-arc image are the same projected $\langle 110 \rangle$ direction, the dotted line on the projected tetrahedron. The projected $\langle 112 \rangle$ direction in the habit plane of the loop which is normal to this line of no contrast is the direction of skewness of the image. The projections of the two $\langle 112 \rangle$ directions normal to the line of no contrast are shown and

comparison with the image shows that the $\{111\}$ plane on the lower side of the dotted line is the habit plane of the loop. The loop at A shows an enhancement of its image intensity at the ends rather than a decrease.

Fig. 16 (a) and (b) Dark-field strain contrast and diffraction contrast images of double-arc loops in aluminum. The edges of the projected tetrahedron are parallel to the zero-contrast lines of the images. The three most obvious directions of streaking are parallel to the projected $\langle 112 \rangle$ directions shown.

Fig. 17 Dark-field images of double-arc loops in a $[110]$ aluminum foil. (a) $s \neq 0$ and the lines of weak contrast are parallel to $[\bar{2}20]$, indicating that the Burger's vectors of the loops lie parallel to the foil normal, whence $\bar{g} \cdot \bar{b} = 0$ for diffraction contrast. The loops are observed by residual contrast caused by the displacements of the lattice, normal to $\bar{b} = a/2[110]$, in the $[001]$ direction (see Fig. 2(b)). (b) $s \approx 0$ and the strain contrast images show sharp lines of no contrast normal to $[002]$.

Fig. 18 Dark-field images of edge-on double-arc loops in a $[110]$ aluminum foil. (a) $s \approx 0$ and the strain contrast images of loops A and B appear as iso-strain contours which relate the two dots within each image. (b) $s \neq 0$ and the images appear as white diffraction contrast images consisting of two sharp dots, indicating that the lines of no contrast of the loops lie parallel to the foil normal and the Burger's vectors of the loops lie in the plane of the foil, i.e. the two strong edge components of the loops are end-on as in Fig. 2(b).

Fig. 19 Representative bright-field image of quenched-in vacancy defects in copper. The foil normal is $[111]$. Few if any of the strain contrast images are white on the side of the operating reflection. If the vacancy-type loops are randomly distributed throughout the thickness of the foil, approximately half should show a reversal of contrast, assuming the Ashby-Brown strain contrast model to be valid for these defects.

Fig. 20 Slip trace images in an $[001]$ aluminum foil. (a) Bright-field images of the traces at either surface are asymmetrical. The $s = 0$ position is in the region where the images indicate that cross-slip has occurred and s is positive to the left. The strain contrast images are roughly equal for all values of s at both surfaces. (b) The corresponding dark-field images at exactly the same diffracting conditions are symmetrical, that is, the same at either surface. The slip-trace images are therefore exactly as predicted by the dynamical theory for an edge or screw dislocation (Burger's vector parallel to the foil surface) of one sign at the top surface and a similar defect of opposite sign at the bottom surface. The top surface of the foil is where the images are the same in both bright-field and dark-field and is therefore well defined by the upper image of the slip-trace (such an analysis is useful in determining the sense of slope of a $\{111\}$ slip plane). Although near $s = 0$ the dark-field images are about the same at both surfaces, for $s > 0$ strain contrast at the lower surfaces is excellent while that at the upper surface is very poor and vice versa for $s < 0$. (c) The dark-field image after the $s = 0$ position has

been moved to the far right by tilting the foil so that the entire region has a positive value of s . The image of the slip-trace at the bottom surface of the foil has thus been brought into sharp strain contrast.

Fig. 21 Dark-field images of large Frank loops in quenched aluminum appear as: (a) dislocation line images for $s \neq 0$, and (b) strain contrast images about the dislocation line image for $s \approx 0$. The sizes of the loops range from about 700Å to about 1200Å in diameter. Since the half-extinction distance for the reflection shown is about 325Å, the centers of the loops cannot be within half an extinction distance of the surfaces, yet they show very good strain contrast in contradiction to the predictions of Ashby and Brown.

Fig. 22 Small black-white images in quenched copper near a bend contour. s is negative in the upper right corner and positive in the lower left; the best strain contrast images are observed for values of s slightly positive. The foil becomes thicker from left to right; better strain contrast images are observed in the thicker regions. The dislocation line at A, nearly at $s = 0$, shows black-white contrast throughout its entire length. Note also the number of black-white images black on the side of the operating reflection.

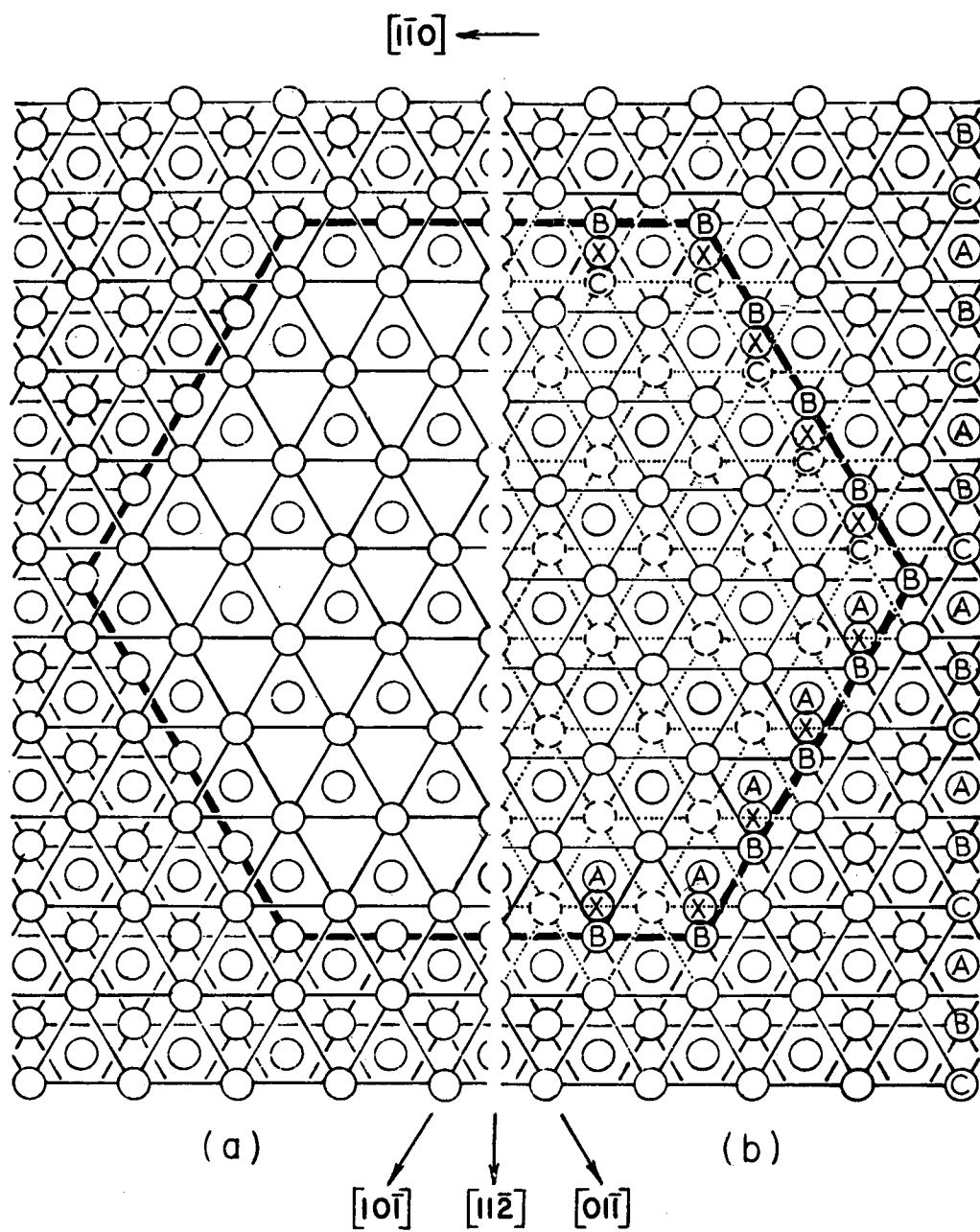
Fig. 23 The effect of changing s upon dark-field strain contrast images is illustrated near a small angle boundary in a quenched copper [110] foil. (a) $s \approx 0$ on the left and is changed to a somewhat positive value on the right by the tilting effect of the boundary on the diffracting planes. (b) The specimen has been tilted so that $s > 0$ on the left and the region on the right of the boundary shows diffraction

contrast images consisting of white dots.

Fig. 24 Punched interstitial loops in a quenched copper $[110]$ foil lie in rows along $\langle 220 \rangle$. (a) The dark field strain contrast images are black on the side of the operating reflection and the loops are interstitial in character. (b) The bright-field image consists of line images caused by diffraction contrast.

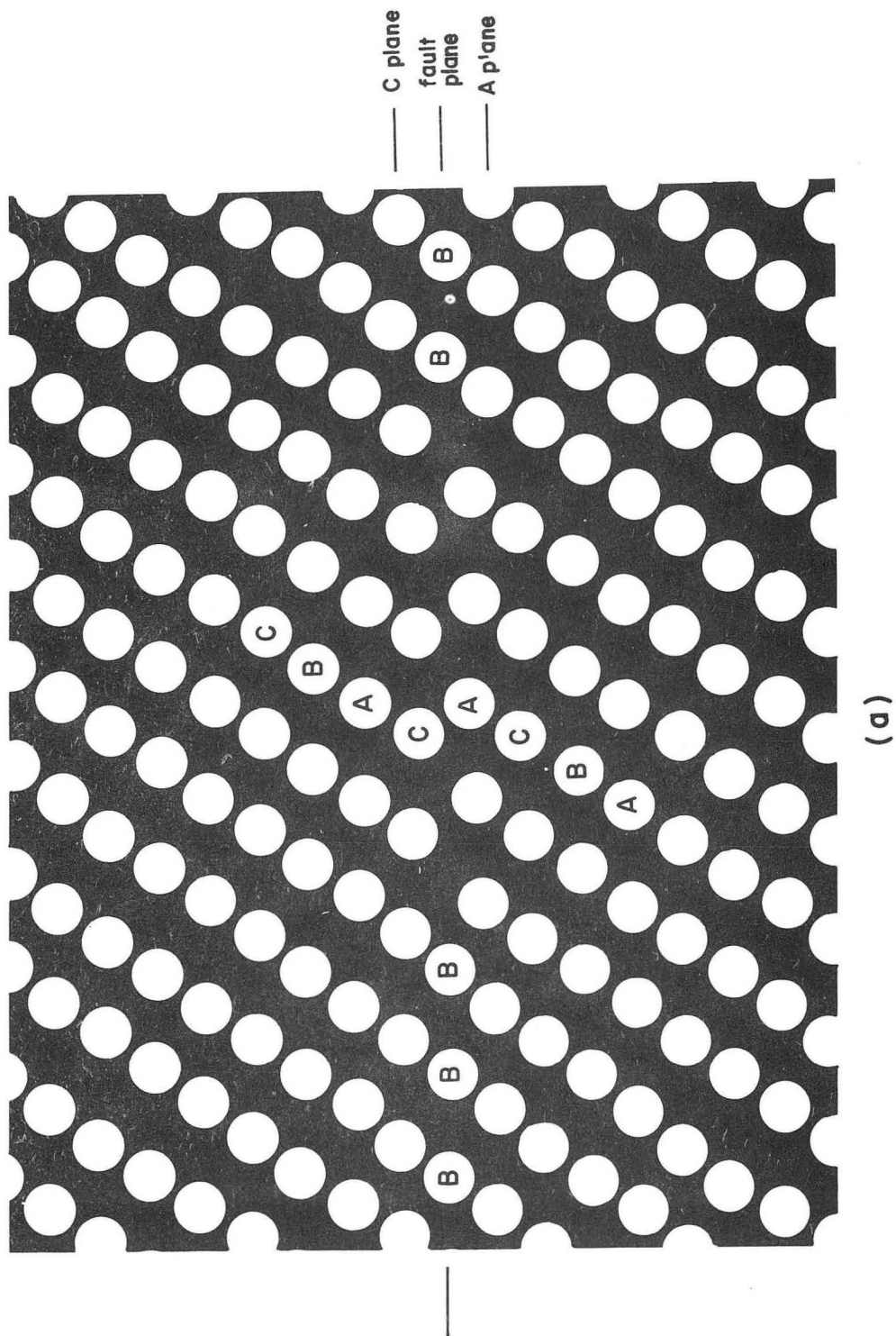
Fig. 25 A bright-field image of vacancy defects in quenched copper showing the smaller loops by strain contrast and the larger loops by double-arc contrast.

Fig. 26 Dark-field images of small loops in a quenched aluminum $[110]$ foil indicate that although a perfect loop may be observed by strain contrast, its line of no contrast may not be normal to its Burger's vector if the size of the loop is much greater than about $200\text{\AA}$. The center of the figure is at: (a) $s > 0$, (b) $s = 0$, and (c) $s < 0$. The double-dot diffraction contrast image of loop A indicates that it is a perfect loop of about $300\text{\AA}$ in size but the line of no contrast of its strain contrast image appears to lie parallel to its habit plane rather than normal to its Burger's vector. Loop B is about $250\text{\AA}$ in size and is small enough to appear as a white dot in diffraction contrast and shows a strain contrast image with a line of no contrast normal to its Burger's vector $\vec{b} = a/2[\bar{1}10]$.



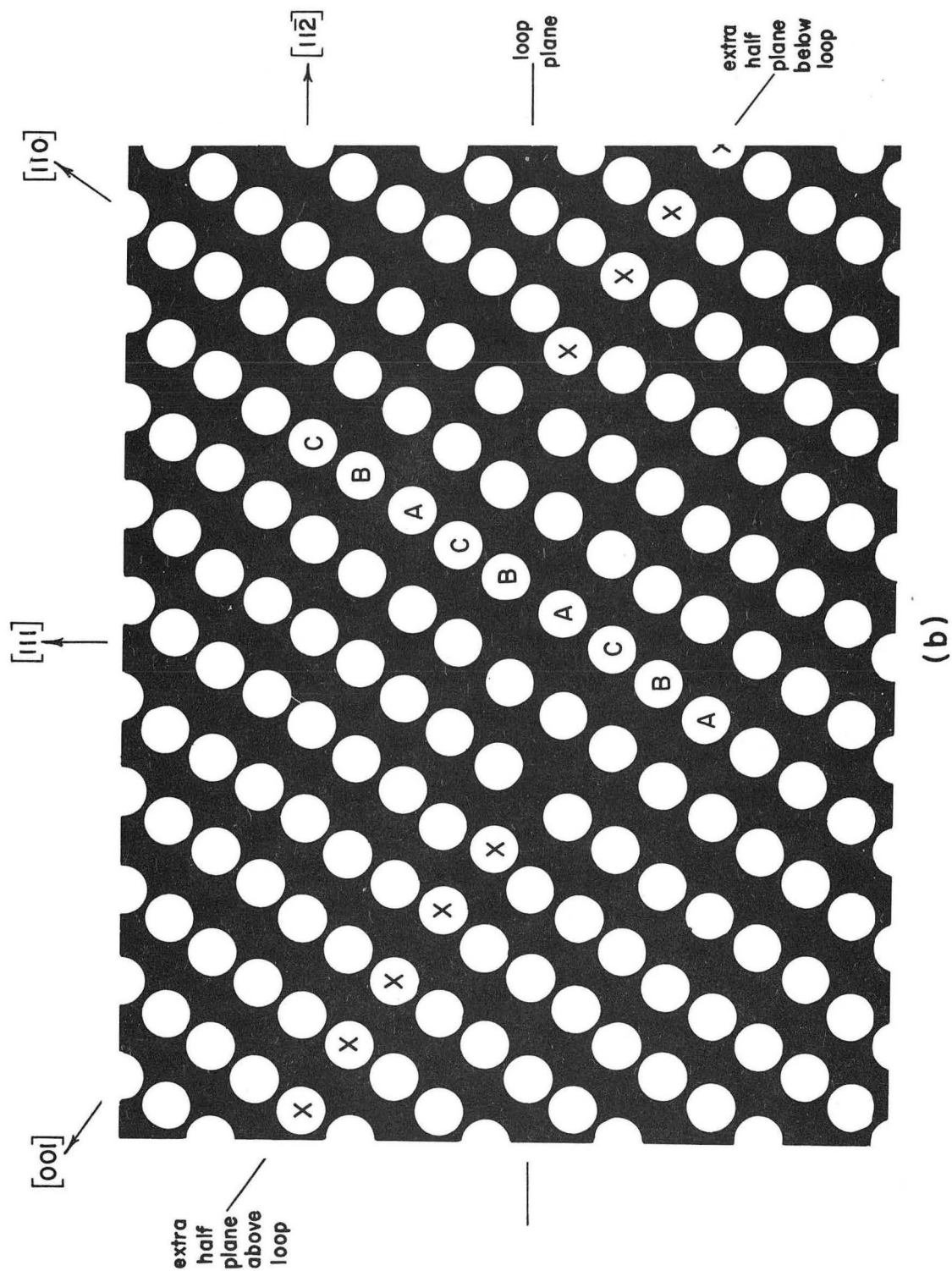
MUB-5618

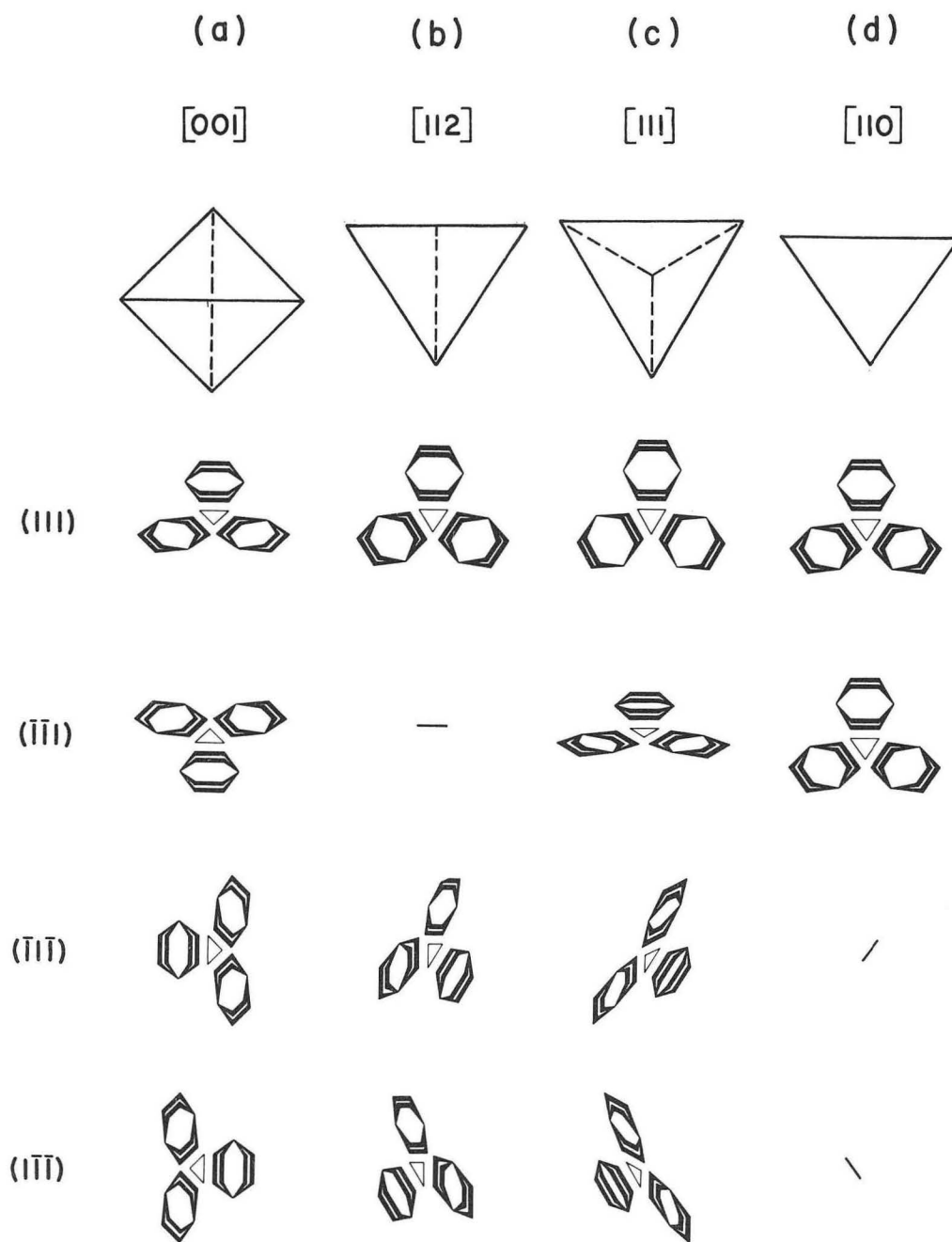
Fig. 1



MUB-5619

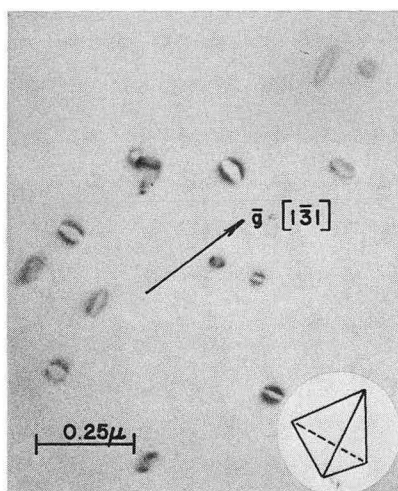
Fig. 2a



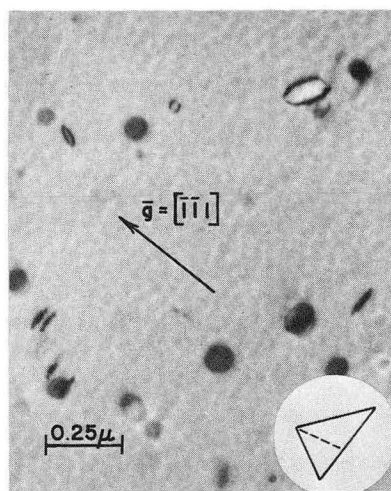


MUB-5621

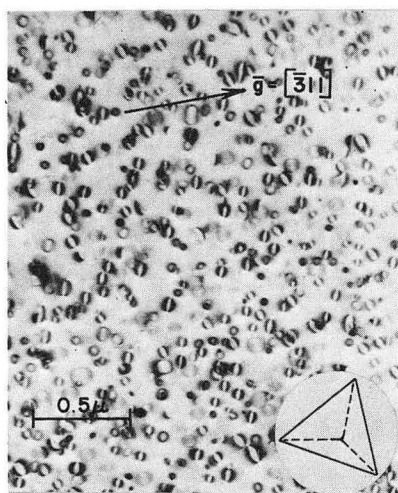
Fig. 3



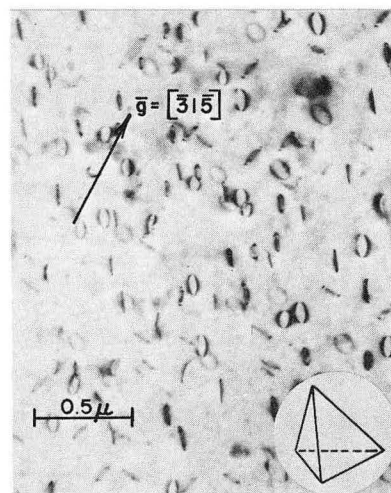
(a)



(b)



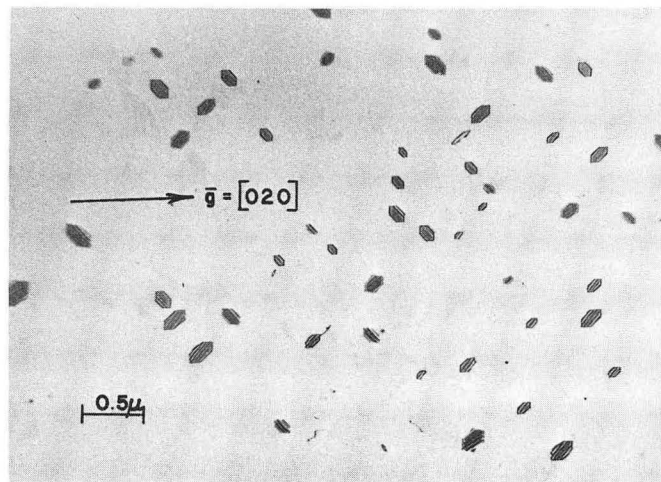
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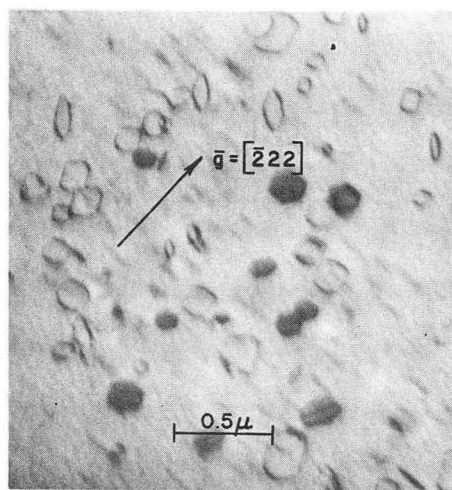
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ZN-4795

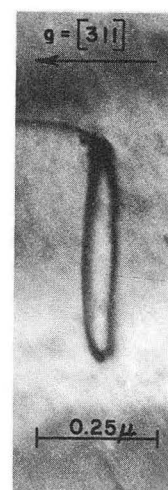
Fig. 4



(a)



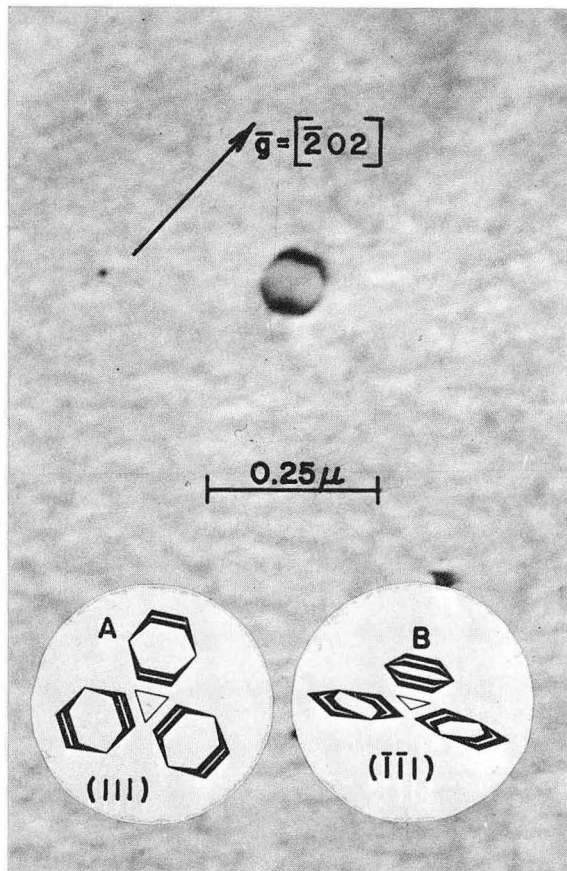
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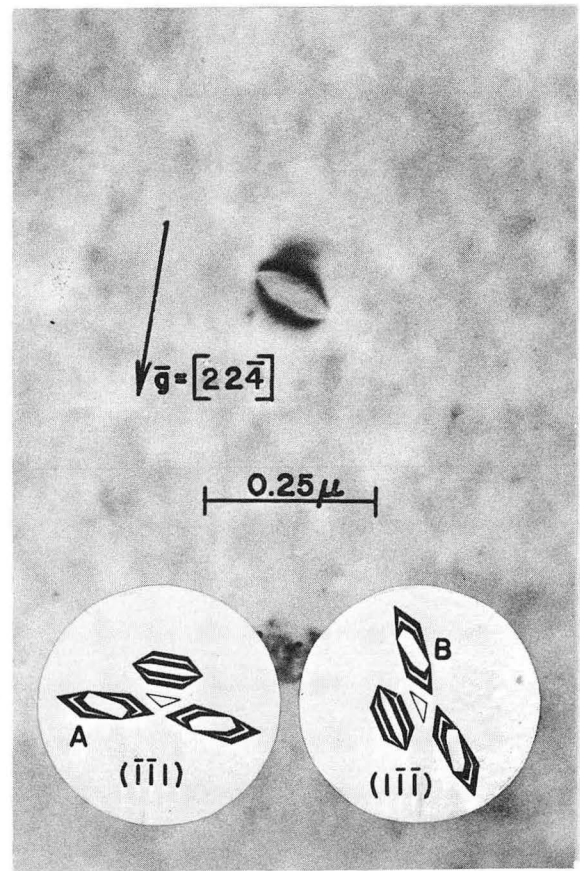
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ZN-4801

Fig. 5



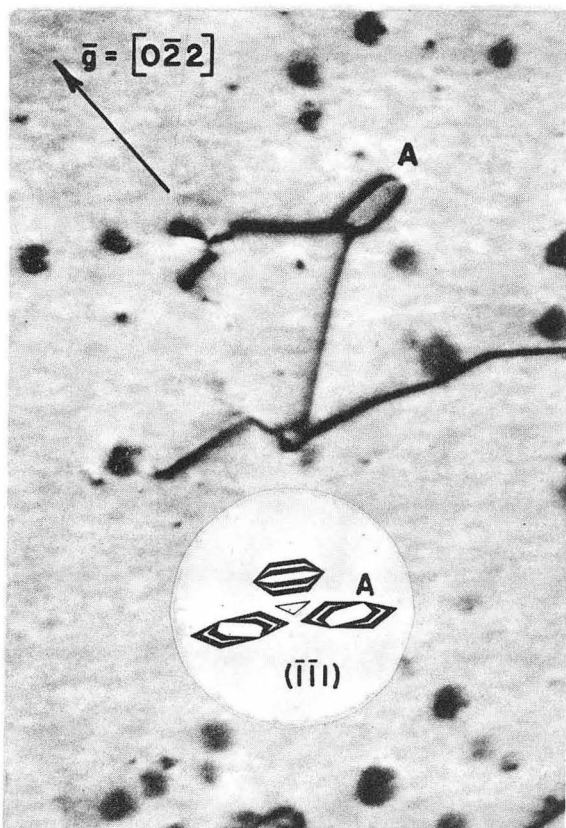
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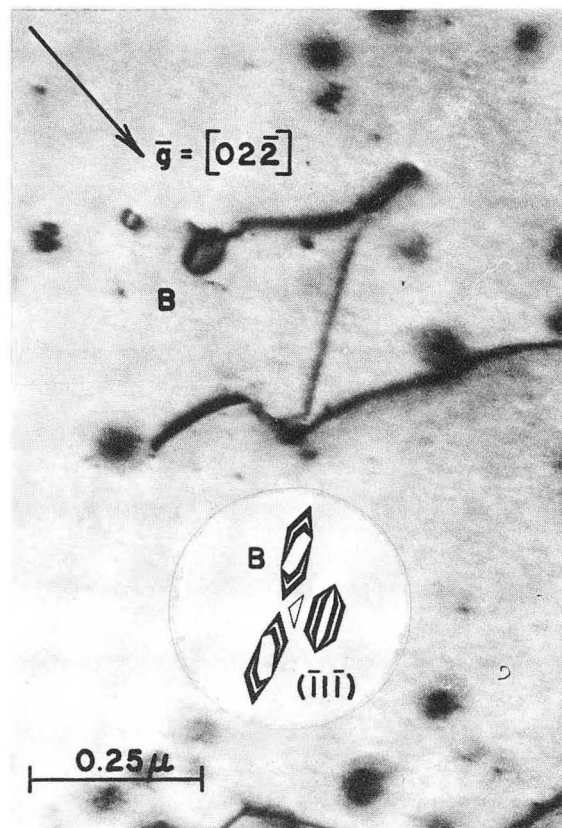
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ZN-4808

Fig. 6



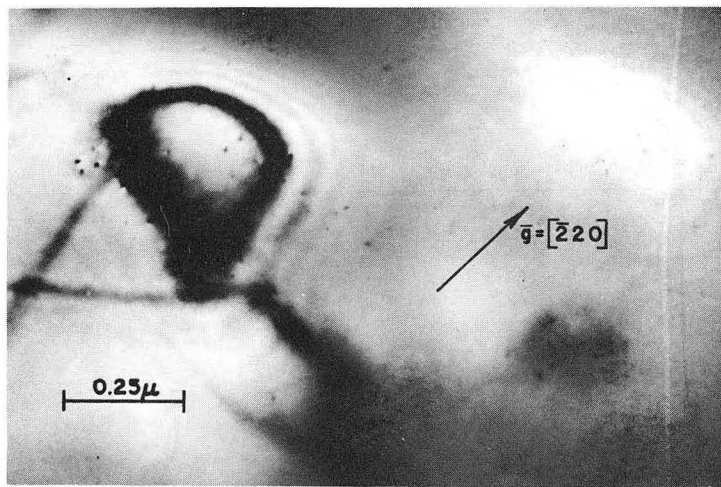
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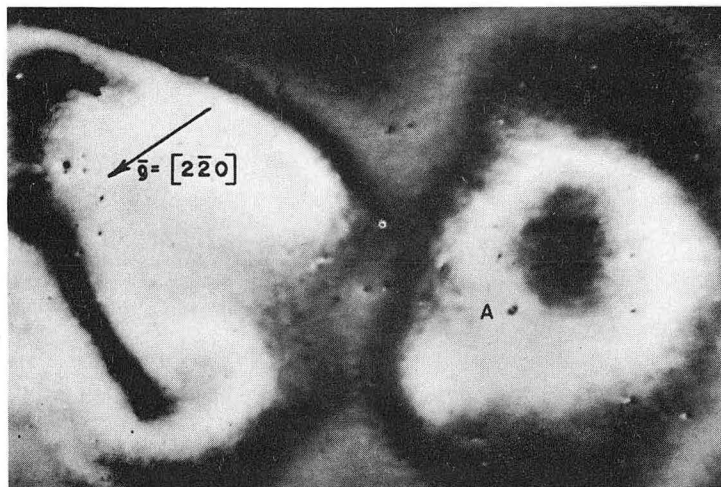
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ZN-4794

Fig. 7



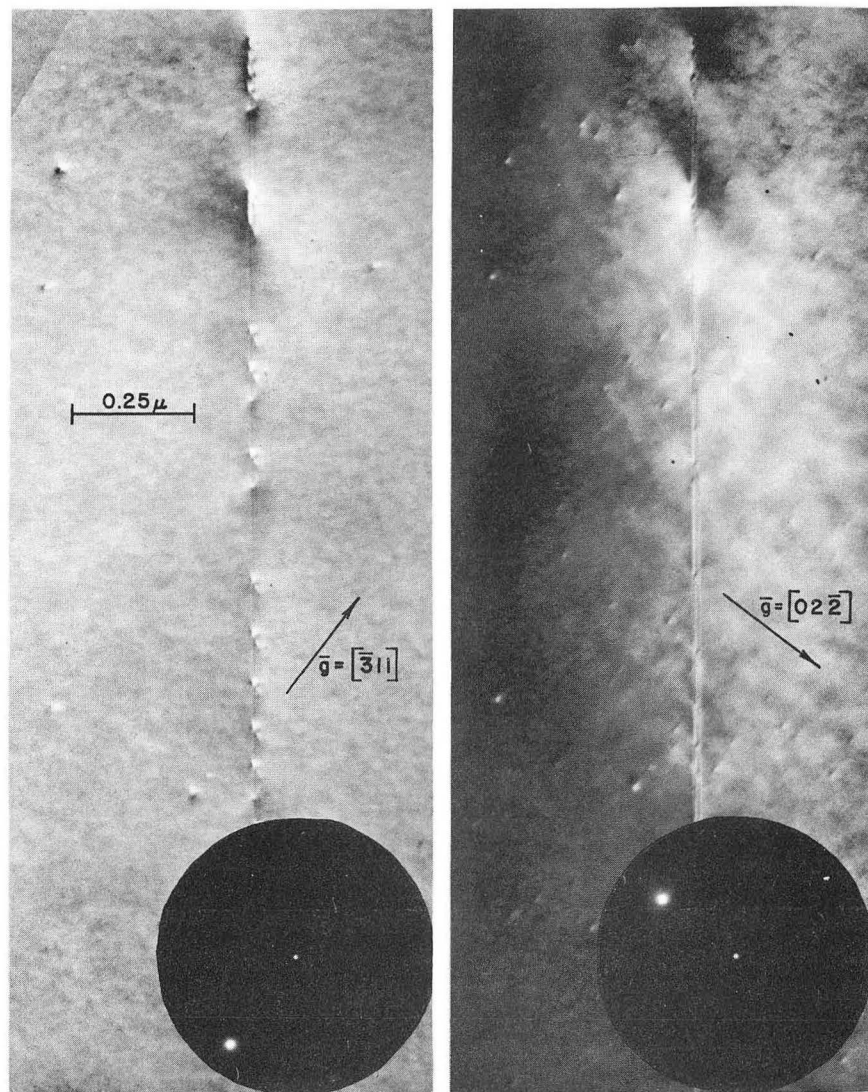
(a)



(b)

ZN-4796

Fig. 8

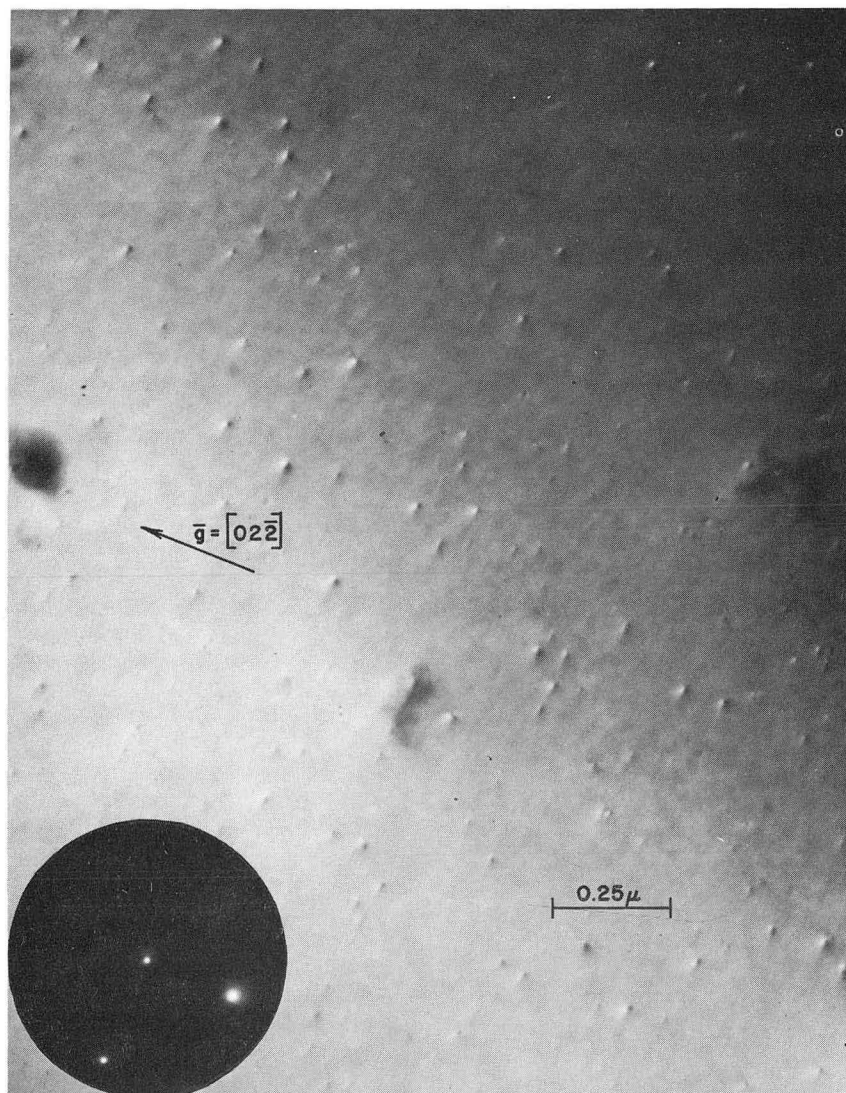


(a)

(b)

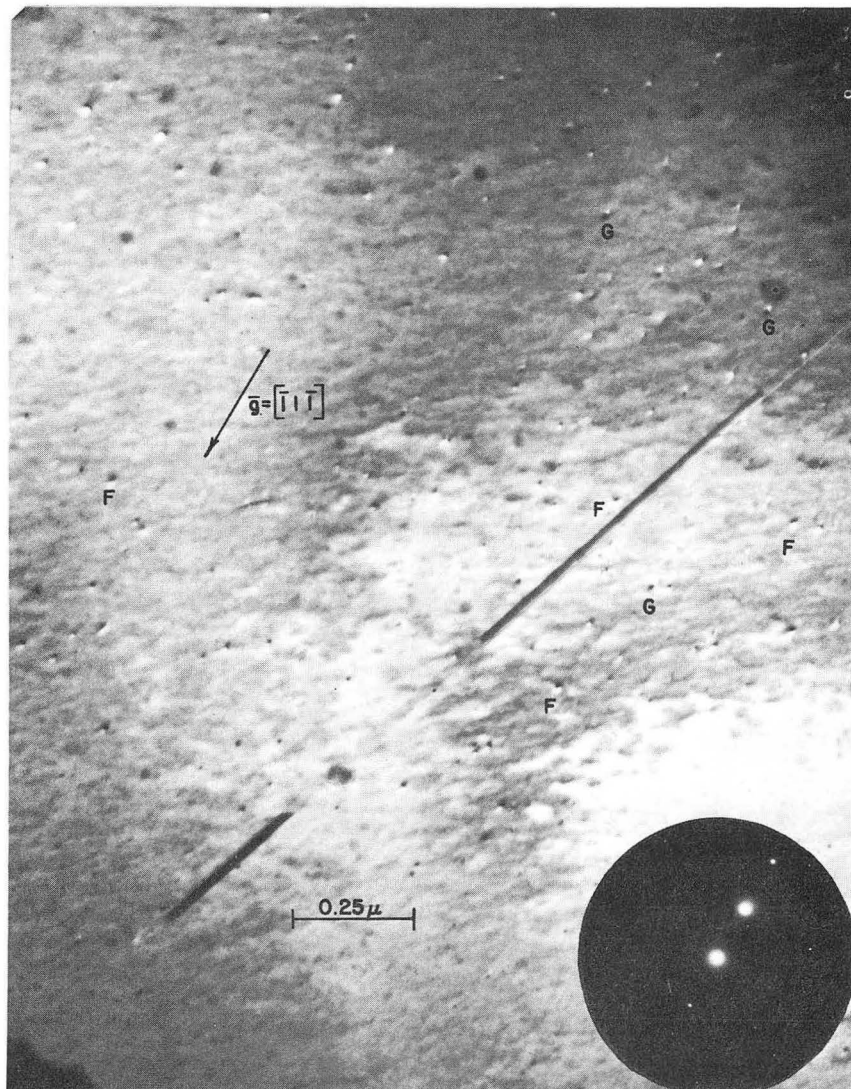
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Fig. 9



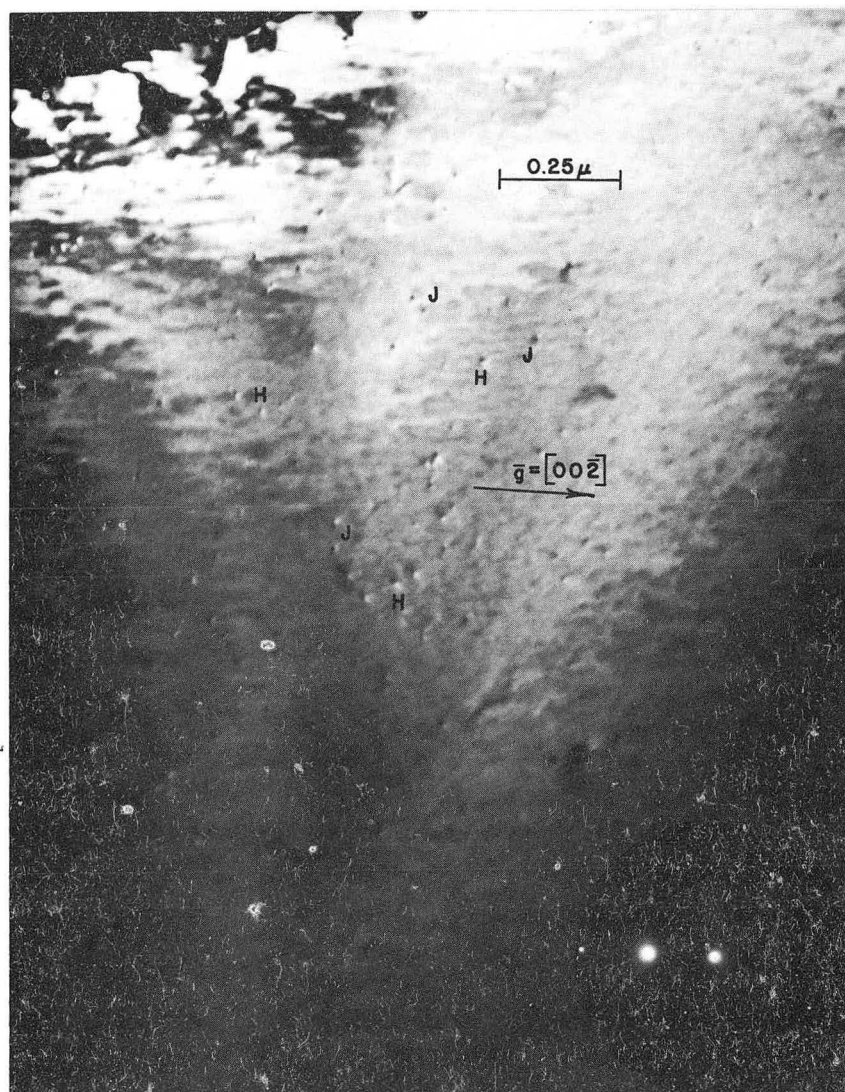
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Fig. 10



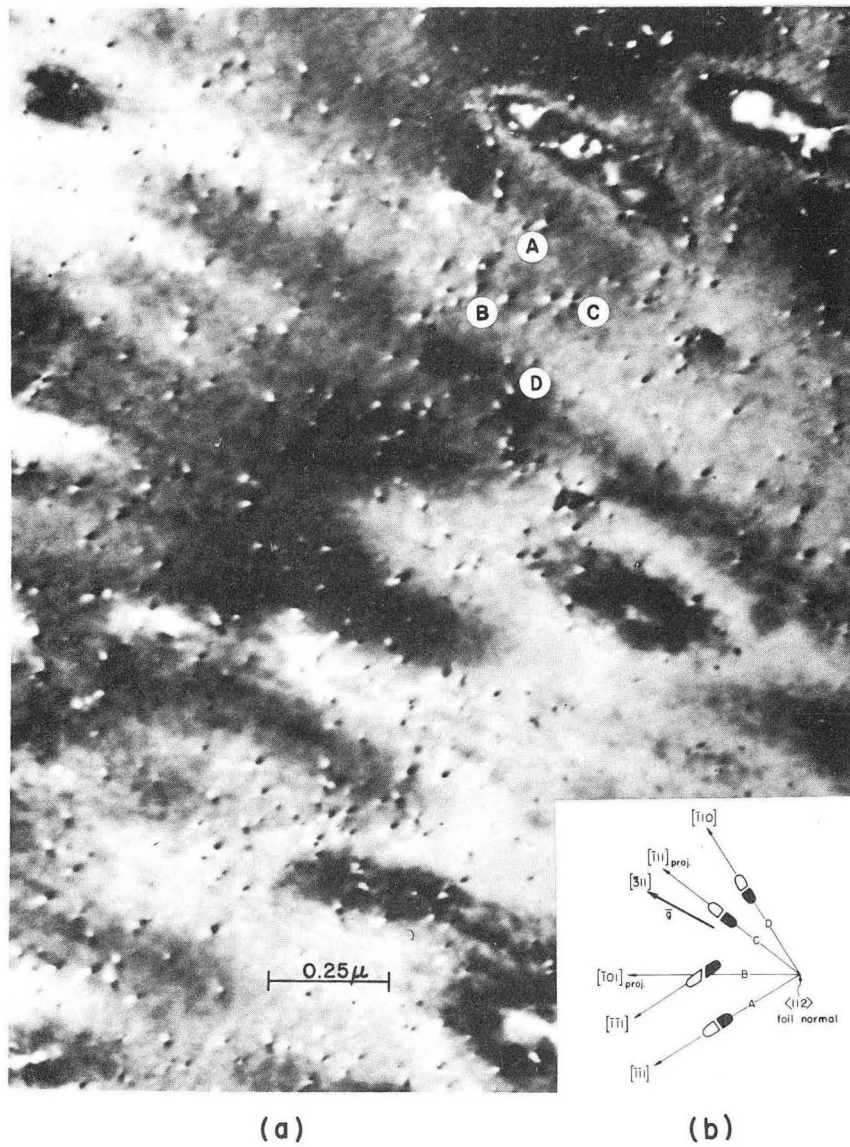
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Fig. 11



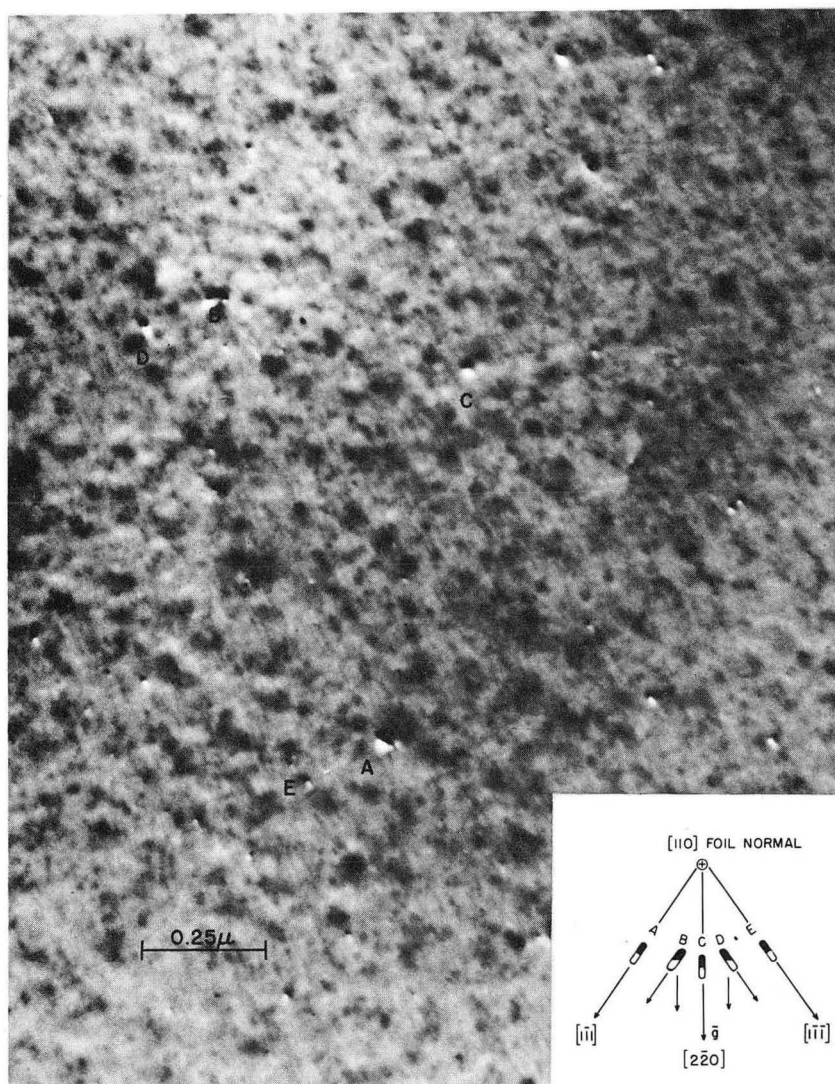
ZN-4792

Fig. 12



ZN-4802

Fig. 13

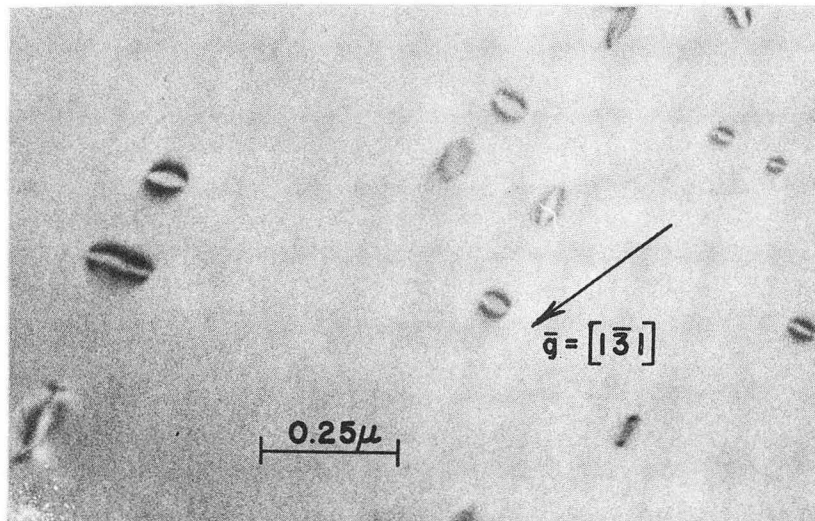


(a)

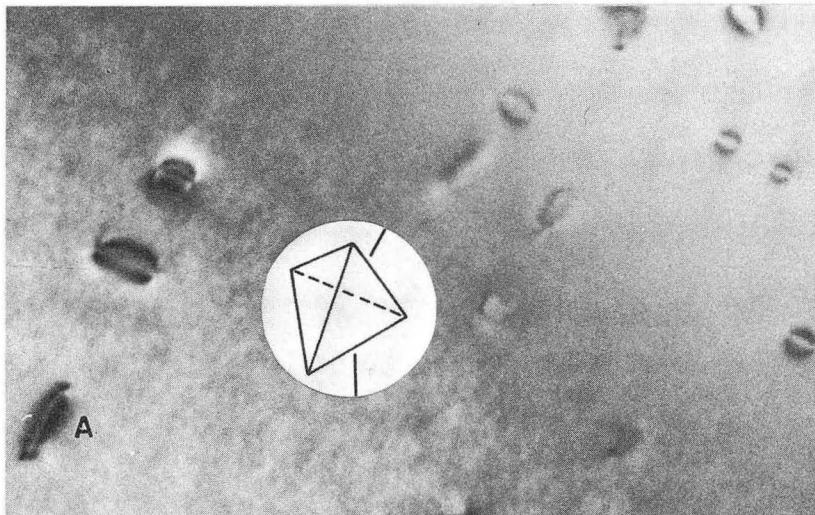
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ZN-4807

Fig. 14



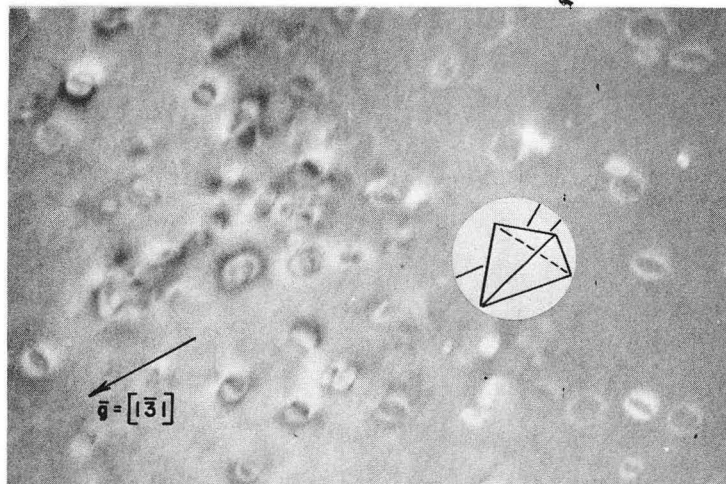
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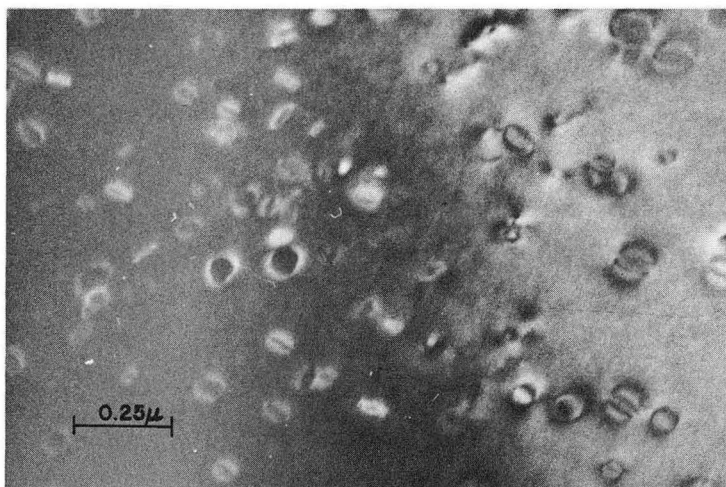
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ZN-4810

Fig. 15



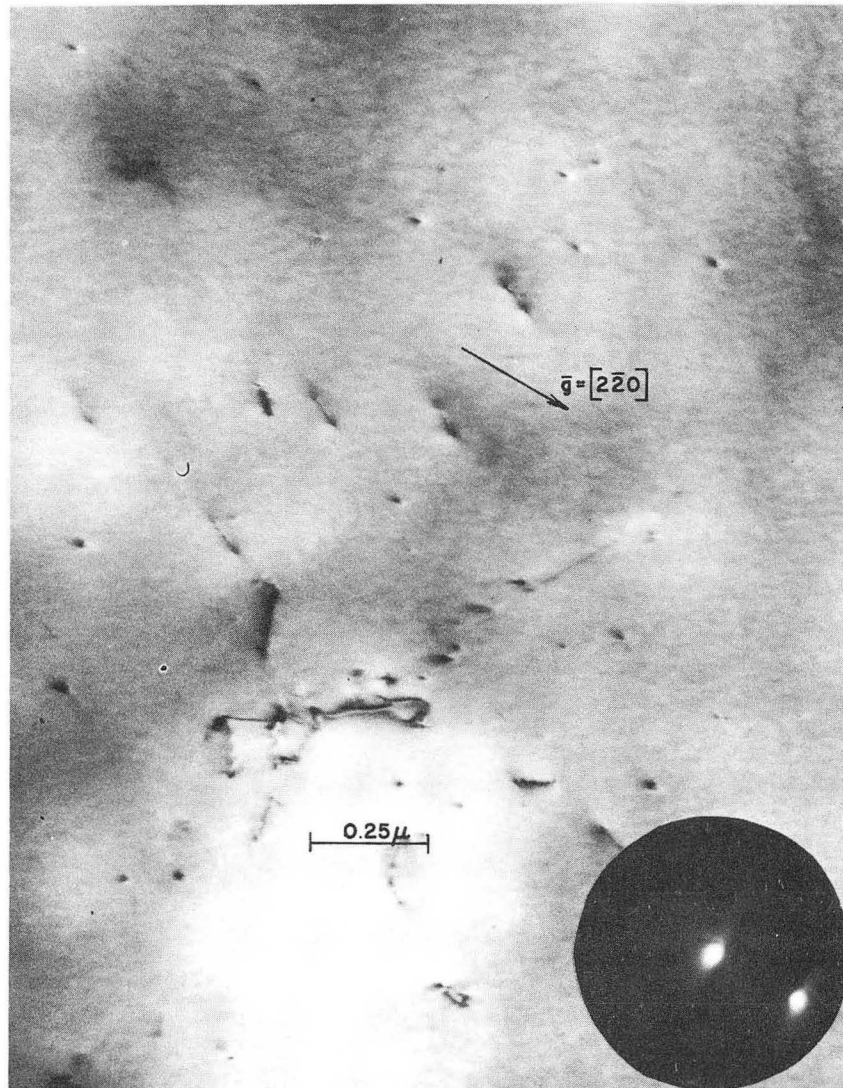
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(b)

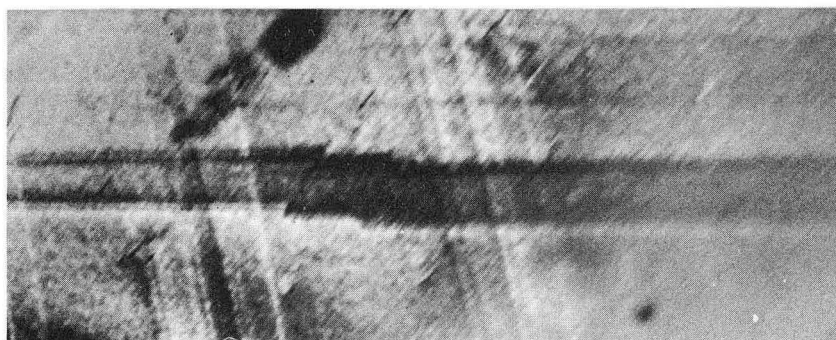
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Fig. 16

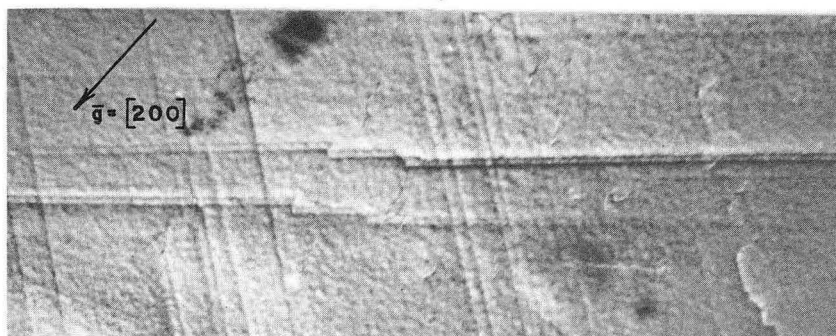


ZN-4812

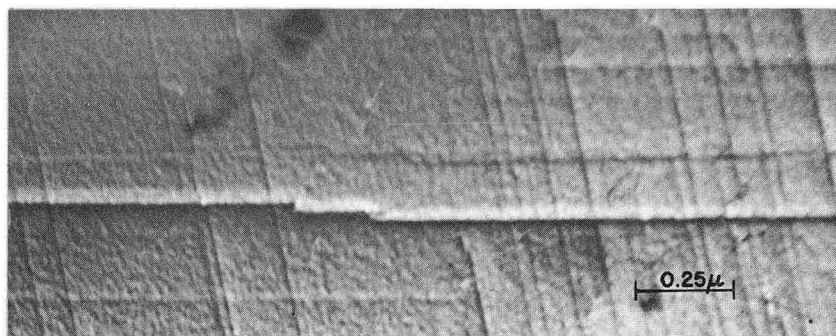
Fig. 19



(a)



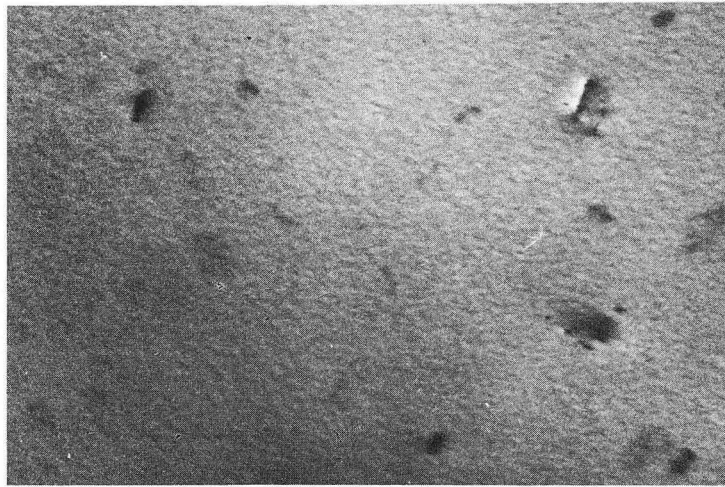
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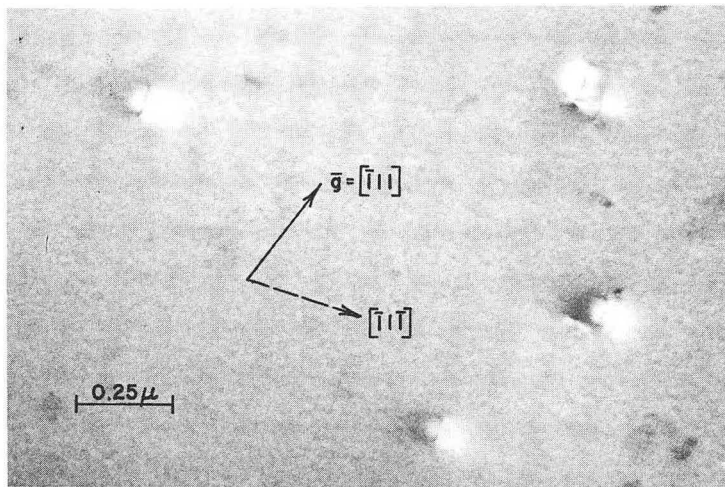
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ZN-4804

Fig. 20



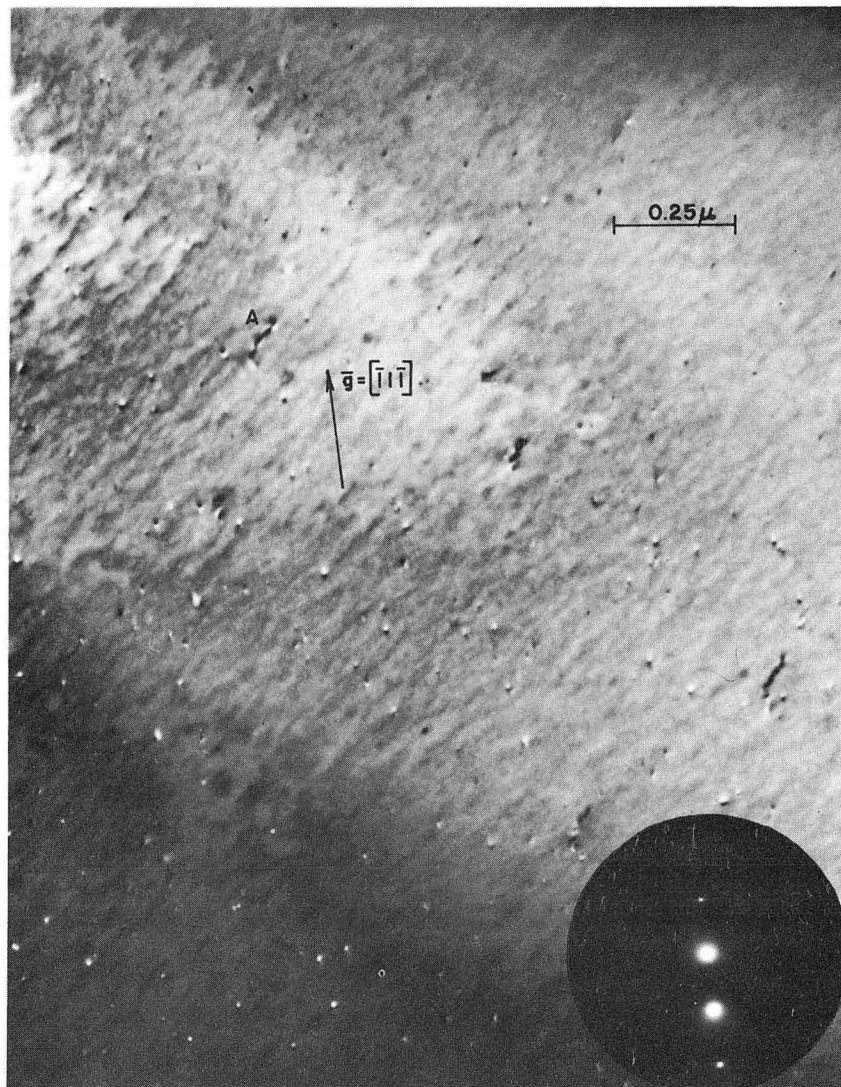
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(b)

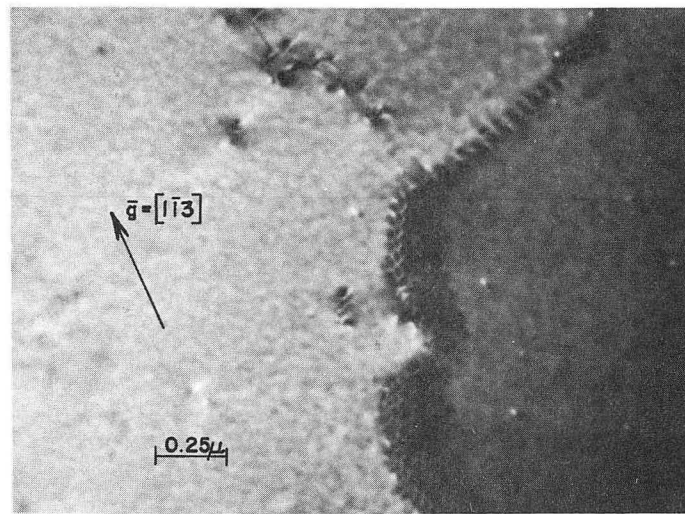
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Fig. 21

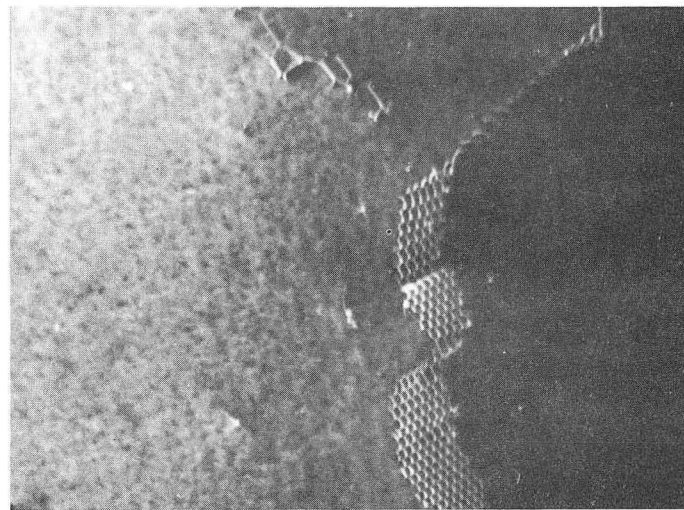


ZN-4809

Fig. 22



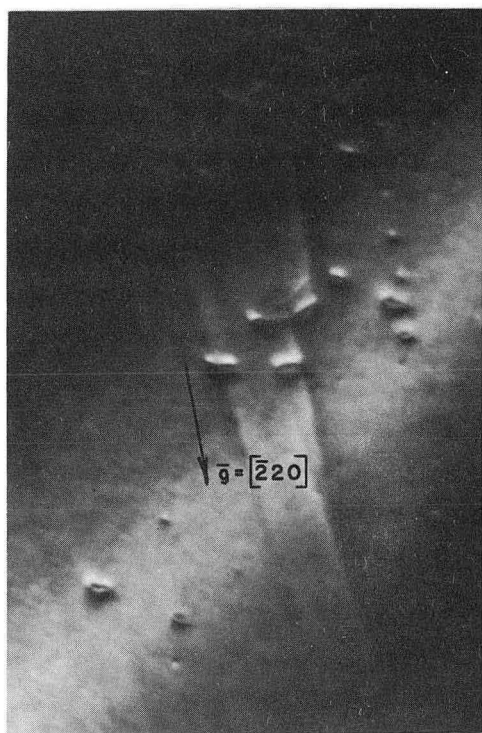
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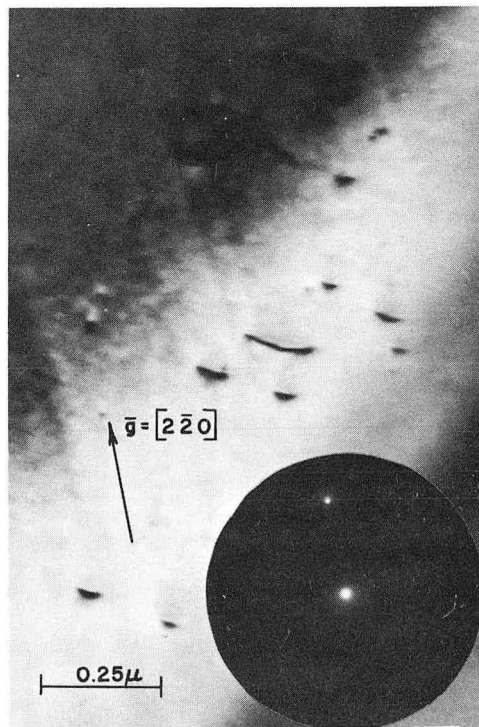
(b)

ZN-4813

Fig. 23



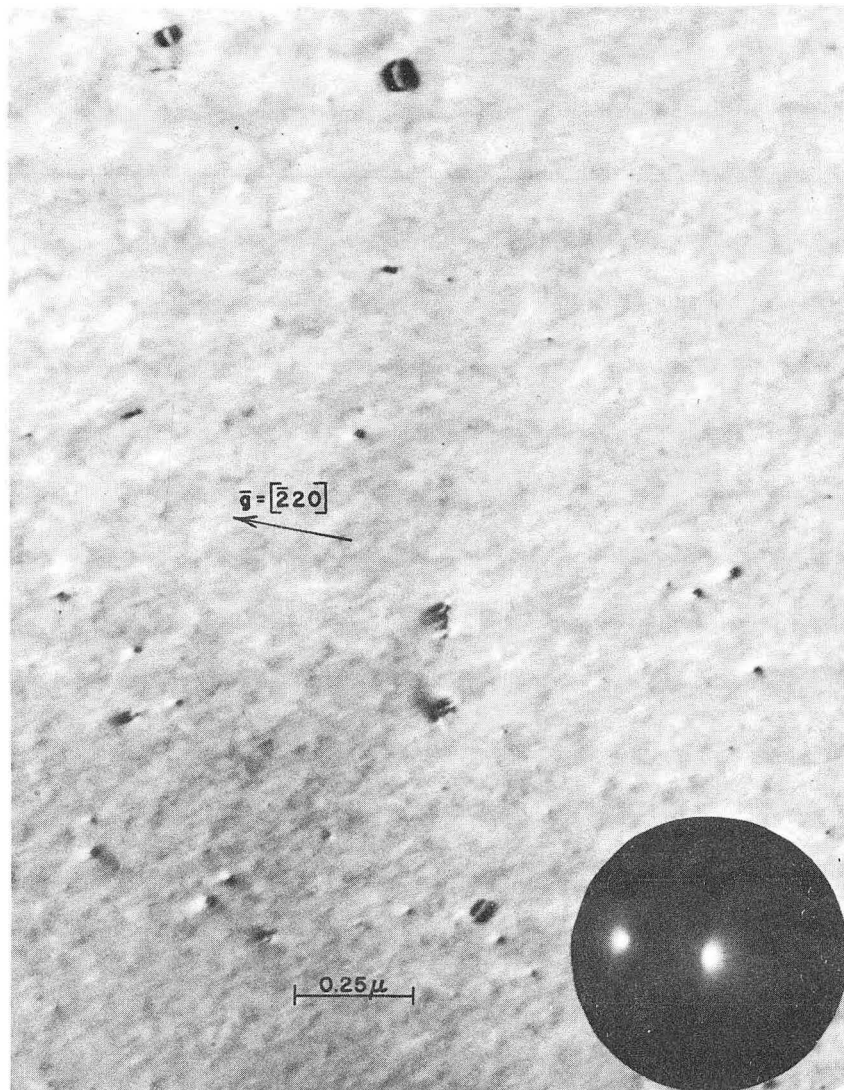
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(b)

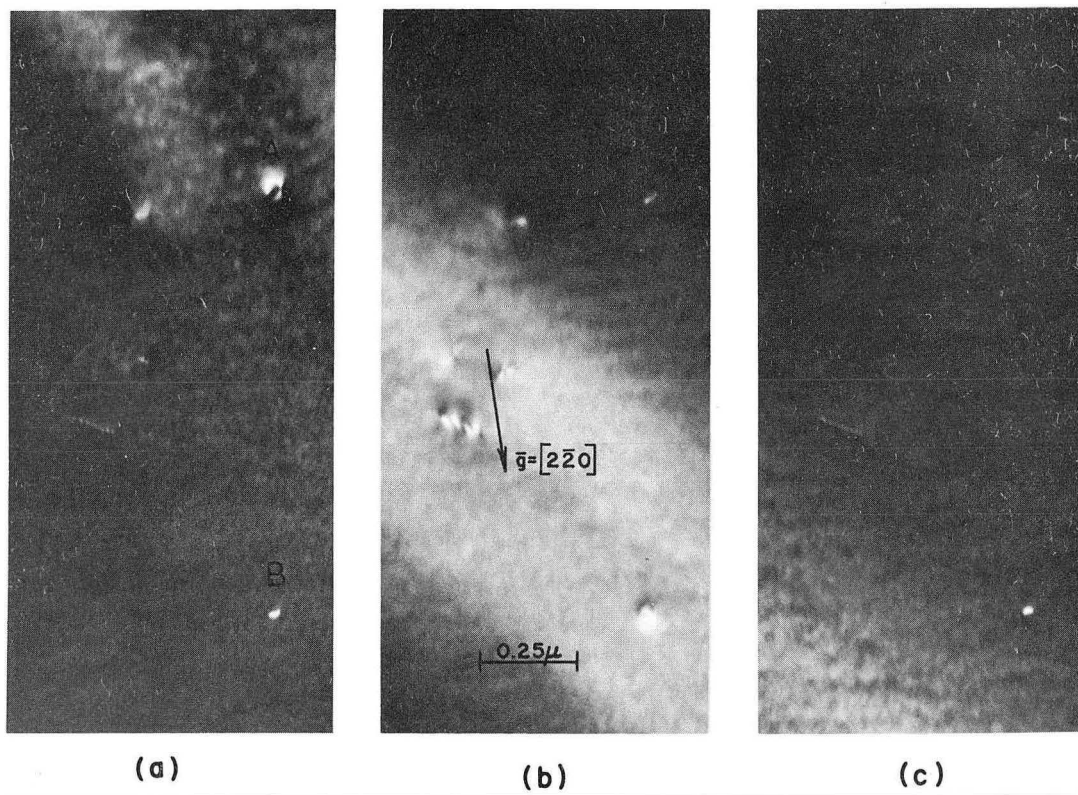
ZN-4791

Fig. 24



ZN-4805

Fig. 25



ZN-4798

Fig. 26

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