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SOME CRYSTALLOGRAPHIC TECHNIQUES FOR THE STUDY OF MECHANICAL
TWINNING AND THEIR APPLICATION TO SHOCK-INDUCED
TWINNING IN COPPER *

(presented at A.I.M.E. - I.M.D Conference on Deformation
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by

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Some Crystallographic Techniques for the Study of Mechanical
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INTRODUCTION

If there is a definite relation between two macroscopic crystals of the same composition in contact across a surface, this relation must be consistent with the symmetry operations of the two dimensional structure of the interface. That is, the two parts of the structure can be related by 1-, 2-, 3-, 4 or 6 fold rotation and or by reflection or glide. If such a relationship is found to exist in several specimens, the crystals are said to be twinned⁽¹⁾ and the relationship determines a twin law for the substance. In order to characterize the shearing process producing a mechanical twin, four crystallographic elements relating the twin and the matrix are needed^(2,3). As shown in Fig. 1, these are the two undistorted planes, K_1 and K_2 (K_1 is the twin plane) and the two undistorted directions n_1 and n_2 (n_1 is the shear direction). The angle, 2ϕ , between K_1 and K_2 specifies the amount of shear. Because a lattice is centrosymmetric, directions in the lattice of the mechanical twin and parent are related to each other either by reflection across K_1 which is equivalent to 180° rotation around the normal to K_1 (Type I twin) or reflection across the plane perpendicular to K_1 and n_1 or 180° rotation around n_1 (Type II twin). When both relations are present, the twin is called a compound twin. If the unit cell contains atoms not at lattice points, after the shear these may not be in the same

positions relative to the unit cell in the twin and additional "shuffling" of the atoms* may be required to establish the original structure. It is even possible that the unit cell in the sheared region will be different compared to the unit cell of the matrix^(3,4).

It is important to see whether atomic shuffling does indeed occur and to what extent. Generally this has not been done, but it has been assumed that the structures are identical and the amount of shuffling is described in terms of that required to produce the original structure. We will be concerned with methods for investigating these features of mechanical twinning in Parts I and II. In Part III we will use known elements to examine to what extent twinning has occurred and on what systems. In this third section, special emphasis will be given to some of our recent results concerning shock-induced mechanical twinning in copper.

We will be dealing with techniques that generally combine diffraction (electron and x-ray) and direct observation. The methods to be described have, in many instances, appeared in the literature, but some of these will be new. A comprehensive treatment of these is appropriate to the subject of this book.

I. EXPERIMENTAL METHODS FOR DETECTING TWINS.

The available experimental methods needed in what follows can be broken into two groups:

- 1) Examination of the twin on one or two surfaces of a crystal (or grain).
- 2) Orientation of the lattice of the twin with respect to that of the matrix.

*The term "shuffling" used by Cahn (Ref.3) seems quite appropriate for the motions as the directions may vary from atom to atom.

** Such a case is a "grey area" where it is not clear that the transformation should be classified as twinning or martensitic.

The first of these needs no elaborate discussion. If the specimen is optically anisotropic, an examination of the specimen in polarized light may reveal twinning.

The tilt of a surface, as determined from reflected light may be useful in determining the orientation relations between the twinned regions^(7,5) (see Part II). This second group however often involves the use of diffraction methods, electron or x-ray. The situation is illustrated schematically in Fig. 2 for a hexagonal crystal. The orientations are shown of the axes of the crystal ($\vec{a}_1, \vec{a}_2, \vec{c}$) and twin ($\vec{a}'_1, \vec{a}'_2, \vec{c}'$) and the axes of their reciprocal lattices ($\vec{a}_1^*, \vec{a}_2^*, \vec{c}^*$; $\vec{a}'_1^*, \vec{a}'_2^*, \vec{c}'^*$);

$$(a_1^* = \frac{\vec{a}_2 \times \vec{c}}{V_{\text{unit cell}}}, a_2^* = \frac{\vec{c} \times \vec{a}_1}{V_{\text{unit cell}}}, c^* = \frac{\vec{a}_1 \times \vec{a}_2}{V_{\text{unit cell}}}).$$

Although the real twin and matrix are adjacent in the material the reciprocal lattices of both start at the same origin and are interleaved. In general, the two reciprocal lattices do not completely overlap, although this is possible and indeed probable in certain cases⁽⁶⁾. With diffraction methods, a picture of the reciprocal lattice is obtained as a result of the intersection of the sphere of reflection, of radius $1/\lambda$, with the reciprocal lattice. Using electron diffraction, with thin films, the small wavelength(λ) results in essentially a planar section of this lattice perpendicular to the incident beam. An x-ray Laue pattern involves the intersection of spheres of reflection of radii which range over the wavelengths available from the tube. When taking an x-ray oscillation or rotation photograph one wavelength is used and the intersections come from the rotation or oscillation of the crystal and its associated reciprocal lattice, resulting in layers of spots on a cylindrical film. Workers in the field of structural analysis find twins in a

crystal a considerable annoyance because of the extra spots that arise on a diffraction pattern and have mainly been interested in avoiding specimens with such twins⁽⁶⁾. The metallurgist or materials scientist on the other hand normally knows the structure of the untwinned material and is often concerned with ways for detecting and characterising twins. Knowing the structure, the appearance of a diffraction pattern from a crystal without twins can be anticipated and the twin reflections readily identified. "Extra" spots will occur on the diffraction patterns if the incident beam hits both the twin and the matrix, or, just different patterns if two films are taken, one with the beam on the twin and one with the beam on the matrix, so that one can distinguish the twin spots from those from the parent.

The choice of technique among these is not completely arbitrary. If the twins form as a result of deformation and the specimen is moderately distorted (the twin is often more distorted than the matrix) the spots on a Laue pattern spread out and the intensity may be so low that only a few spots from the matrix can be found on the typical flat Laue film. The accuracy is reduced and if the twin is small, the spots from the twin may be too weak to be visible. Considerable improvement in the results can be obtained in these cases by using a cylindrical film instead of a flat film. A greater number of spots are collected. There is also some focusing in the horizontal plane with a cylindrical film for the few Laue spots involving characteristic radiation. (Nets are available for reading such films similar to the familiar Grevinger net for the flat Laue⁽⁸⁾). Ordinary commercial rotation cameras are ideal for this purpose. If one can place the x-ray beam at a sample edge, the patterns can be examined in transmission, and there is always less effect of distortion

in the forward regions. When the sample is severely distorted, the rotation or oscillation pattern is particularly useful. All spots are formed from characteristic radiation; they all are focused by the cylindrical geometry. Furthermore the spots from the twin are immediately obvious because they are generally not on the layer lines from the untwinned crystal. The extra spots will also be obvious in an electron diffraction pattern. Back reflection Laue oscillation and rotation photographs of a shock-loaded crystal containing twins are shown in Fig. 3. The distortion is so severe in this case that there are hardly enough spots on the Laue pattern for a determination of orientation, but the twin spots are clear on the oscillation or rotation pattern. The distortion can be sufficient even in a tensile test to preclude detection of twinning with a Laue⁽⁹⁾. With a rotation or oscillation technique it is possible to detect twinning even if the twins are well below the size necessary for optical examination as we will show later.

Oscillation can be carried out with a goniostat on a diffractometer, as in structural work, where one 'hunts' for reflections in reciprocal space. This basic concept has been used by Blewitt et al⁽⁹⁾, who have designed a simple automatic attachment to a diffractometer to "search" for twin spots.

It should be emphasized that due to the motion of the crystal in an oscillation photograph, the exact orientation of the crystal cannot be obtained, except by special variations of the method⁽⁶⁾. However, what we are interested in are twin spots; the orientation should in fact be determined in the usual ways, and the oscillation carried out about a simple axis, for ease in interpretation.

Moving x-ray film methods, such as the Weissenberg

technique, are not useful for this work because the motion of the film smears out the spot, which is often already smeared out if the twins form in a deformation process. A precession camera can, however, be arranged to give good focusing⁽¹⁰⁾. A fine beam or a microfocus tube can be especially helpful, by allowing examination of thin twins, or of twins inside a grain in a polycrystalline specimen⁽¹¹⁾.

II. DETERMINATION OF THE TWINNING PROCESS.

We will consider first the twin elements, and then the atomic "shuffling". As can be seen in Figure 1 we do not need to experimentally determine all four twin elements, K_1 , n_1 , K_2 and n_2 ; from K_1 and n_2 or K_2 and n_1 it is possible to determine the other two⁽³⁾. For example, if n_2 and K_1 are rational (Type I twin) and known, n_1 is the direction in K_1 that takes n_2 in the twin to a position of a mirror image across K_1 of n_2 in the parent. The rotation of n_2 , $\pi-4\phi$, allows calculation of the shear.

The most general method for determining the twin elements is due to Greninger and Troiano⁽¹²⁾. The tilt of two surfaces of a crystal from one twin must be measured optically. A stereographic projection is made on to K_1 (determined by two surface analysis for the twin or habit plane), as shown in Fig. 4. The shear direction is given by the line joining points a to b, the poles common to both surfaces of the crystal in the parent and twin respectively. K_2 (and hence 2ϕ) is then located as the great circle which is symmetrical to XY before and after the shear and carries poles from their initial orientations to their twinned orientations i.e. poles such as c to d and e to f; the rotations c-d, e-f should be equal. Finally, one should check that the angular relationships between poles are identical before and after the shear.

It is more often than not difficult to obtain twins of a suitable size and location to use the above method; logical paths to the solution of the problem can often be found by considering the nature of twinning. However, before proceeding in this direction, it should be pointed out that to prove that markings are twins it is only necessary to show that there is a repeatable relationship between the regions which is one of the permissible types. For mechanical twinning this relation must be 180° rotation around the normal to K_1 or around n_1 , or both. All four elements are not necessary. This is why diffraction methods are so useful in studying twinning. Often on a Laue, the twin law can be determined directly from the symmetry. Thus for example, if a cubic crystal forms Type I twins by shear on a $\{111\}$, a Laue along a cube diagonal where the beam strikes twin and matrix will show 6 fold rather than 3 fold symmetry, because the twin relationship is 180° around $\langle 111 \rangle$. When such observations are not feasible, we can proceed in the following way.

The Laue spots from the twin, or those from an oscillation photograph, can be used to plot the poles of the planes causing the reflection. In a stereographic projection, the poles of the twin should index with the same angular relationships as the poles of the parent phase. Furthermore, the rotation of the poles should be 180° about the same axis. Indexing a twin spot in an oscillation photograph (for a twin in a crystal of copper) is illustrated in Fig. 5. The angles between the reciprocal vector to the twin spot and the reciprocal vectors 100, 010 and 001 are the angles between these poles of the parent and the pole of the planes in the twin giving the spot. The fractional indices are an immediate clue as to the type of the pole and serve as an aid in indexing a stereogram; the values of $\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$ for twin reflections should correspond to similar values for possible matrix reflections. Thus, in

the case shown the spot must be from a $\{200\}$. (If the unit cell changes during twinning some extra spots will appear which cannot be indexed in this manner, or there will be some missing; these will be useful in establishing the new cell).

After the poles are located in a stereogram from either Laue patterns or oscillation photographs, the axis of rotation can be determined in the following way. As the rotation is 180° , the poles of the twin, matrix and the rotation axis all lie on a great circle. Great circles connecting the corresponding poles of the twin and matrix should all intersect in a common pole. The plane belonging to this pole is the mirror plane of the twin (K_1 for a Type I twin, or the plane perpendicular to n_1 and K_1 for a Type II twin). An example of this procedure is shown in Fig. 6. The twin is compound and there are two 180° rotation axes. In order to ascertain which is the pole of K_1 , K_1 must be determined. As it is the habit plane, there are three methods available:

- 1) Two surface analysis
 - 2) One surface analysis in several grains.
The loci of poles of possible planes from the various grains placed in one orientation will intersect or cluster around the pole of K_1 ⁽¹³⁾.
 - 3) If there is some known reference point on the surface such as two intersecting slip lines or intersecting twins of a known type, the change in position of the unknown twin relative to this point on etching can be used to determine the twin plane⁽¹³⁾. If the twin plane can be observed on the two surfaces of a thin section (either with the optical or transmission microscope), it is possible to determine the indices of the plane⁽¹⁴⁾. (When the twin does not have straight sides, the mid-rib must be used in this work).
- If there is a plane of symmetry perpendicular to K_1 , its

intersection with K_1 must be n_1 . n_2 and K_2 can be then "guessed" keeping in mind that for reasonable shears 2ϕ will be of the order of $70 - 80^\circ$ so that the pole of K_2 must be close to n_1 (see Fig. 1). These possible choices can be checked by examining surface tilts (preferably on two surfaces); even if these can be measured only approximately they may serve to isolate the choice⁽¹³⁾. Such a procedure is especially helpful if K_1 is irrational, for in this case K_2 and n_1 must be rational and lie on a great circle with the pole of K_1 .

Alternately, if K_1 and n_1 are determined, an accurate measurement of tilt on one surface will fix K_2 and n_2 reasonably well, using the procedure illustrated in Fig. 4⁽¹⁵⁾. Sometimes one can guess at reasonable poles for K_2 and n_2 by examining the actual crystal structure.

If both K_1 and n_1 are rational, K_2 and n_2 must be also. When K_1 is known, it is often worthwhile to hunt for a rational 180° rotation axis in K_1 that will produce the determined poles of the twin; this direction would be n_1 , and the twin would be a compound twin.

In structures with high symmetry there are special problems. For example for twins on $\{111\}$ in the face-centered cubic system, a stereographic projection constructed from x-ray data has four 180° rotation axis, one $\langle 111 \rangle$ and the three $\langle 112 \rangle$ perpendicular to it. A separate determination of K_1 will indicate which of the four is the pole of K_1 . To determine the actual operative n_1 from the three possible, one surface tilt must be measured or etch pits examined in the twin and matrix on a plane perpendicular to K_1 ⁽¹⁶⁾.

Most of these methods have proved fruitful in previous investigations. We now will turn our attention to the

actual atomic motions.

As was mentioned in the introduction, the twinning shear may not produce the same unit cell as in the untwinned crystal. In some cases, small additional atomic shuffles are required, but in some systems the shuffles appear to be quite large such as : in ordered alloys containing atoms not at lattice points. Although they are usually assumed to occur, it may well be that these shuffles do not take place, or are incomplete in mechanical twinning and it would be quite useful to examine if they occur by doing a structural determination inside the twin. With x-ray micro-beam tubes and electron diffraction this is now quite feasible; it may even be possible to determine the needed information from extra lines on a powder pattern which arise from the twinned structure, if the unit cell is sufficiently different. The authors know of only one case where this has been done. Just recently E. J. Rapperport et al⁽¹⁷⁾ reported such a study with Mg_3Cd . They found that the twinned regions definitely had a different unit cell. More of this type of work is definitely needed for a complete picture of the twinning process.*

Dislocation configurations may help to explain the atomic shuffling, when this is required. For example Freise and Kelly⁽¹⁹⁾ found that for twins in graphite, the plane of K_1 was essentially a wall of dislocations; this wall provides the needed motions as the twin expands. They also found that the twin was mobile normal to K_1 , i.e. the formal shear parallel to K_1 was not the actual shear to produce or expand the twin.

*It is interesting to note that matrix methods, which have proved so interesting in examining martensitic transformations⁽¹⁸⁾, are not applicable to twinning, unless the unit cell changes type, simply because the initial and final unit cells are identical. However, they may prove useful in systems where the unit cell does change.

Finally, by using the Berg-Barrett technique⁽²⁰⁾, it is possible to learn something of the distortion of the parent in the immediate vicinity of a twin, and in the twin itself. The broadening of twin spots relative to the matrix can be useful in determining whether a twin is distorted more than the matrix⁽²¹⁾. The twins should be large enough to be examined individually so as to avoid any confusion from particle-size broadening of twin spots produced by twins too small to see; it is possible to separate out the effects of mosaic size and tilts and strains by using multiple orders⁽²²⁾.

III. USES OF KNOWN TWINNING ELEMENTS.

A. Operative twin systems during deformation.

During the shear (see Fig. 1), all vectors originally to the left of the plane perpendicular to K and n_1 are shortened, while those ahead of this plane are lengthened⁽²³⁾. Therefore if a K_1 projection is made, for any given twin system, the stress axes suitable for twinning that system in tension or compression can be determined, and the resolved stress can be ascertained, or conversely, if the direction of the stress is known, the resolved stress on various systems can be determined, as well as whether they will operate in tension or compression⁽²⁾. As an example of the results of such considerations, the $\{111\}$ twin systems in a f.c.c. crystal with the maximum resolved shear stresses in tension and compression at the corners of the stereographic triangle are presented in Fig. 7. (The systems operative in tension have appeared incorrectly or incompletely in the recent literature⁽²⁴⁾).

When there is distortion at the interface between the twin and parent, secondary twinning systems may become operative, to relieve the strains, and even if the applied stress is tensile these may be systems which would be expected to operate only in compression⁽¹⁵⁾.

B. Prediction of the twin reflections for a given twinning system

If the twinning elements have been determined, it is possible to calculate the actual positions of possible twin reflections. A stereographic projection is made on K_1 for a Type I twin or along n_1 for a Type II twin. The poles of the 100, 010 and 001 are plotted. These are rotated 180° around the pole of K_1 , or n_1 , and indexed relative to the axes of the matrix. Then a solution is found for the elements of the matrix a_{ij} :

$$\begin{vmatrix} h \\ k \\ l \end{vmatrix}_T = \begin{vmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{vmatrix} \begin{vmatrix} h \\ k \\ l \end{vmatrix}_P$$

With this matrix it is a simple matter to obtain indices of diffraction spots due to the twins. (A computer can quickly generate these) The resulting reciprocal spaces for twinning in the f.c.c. and b.c.c. systems are presented in Fig. 8 and 9. With these and any given diffraction technique, it is possible to quickly determine when twins are present and on what planes. With electron diffraction from thin films, a model based on these drawings can be especially helpful as with this method one sees a planar section through reciprocal space, normal to the incident beam.

C. Quantitative estimates of amount of twinning.

We have been continuing the classic studies by Professor C. S. Smith of shock-induced twinning in copper^(25,26). He found that twin-like markings could be seen in the optical microscope after different pressures with different orientations to the shock. Correspondingly, in polycrystalline specimens, not all grains showed markings and the hardness of a grain did not seem to depend on the presence of these markings. A brief discussion of our results is appropriate to this section. Single crystal and polycrystalline specimens of copper were subjected to planar compressive shock waves as described by Dieter⁽²⁷⁾. Single crystals were always

orientated within 2° of the desired direction. [100], [111] [110] and a point inside the stereographic triangle were the directions of loading. A specimen was mounted in a polycrystalline copper support such that the four sides and bottom of the frame were closely fitted to the specimen but were not attached to the specimen, or to each other. In this way reflected tensile waves from the sides and bottom were not allowed to enter the sample; the frame separated from the specimen when the tensile reflections from the outer sides of the frame reached the interface between it and the specimen. Only the compressive shock wave and the rarefaction trailing it were "felt" by the specimen when a driver plate was impelled (by the explosive) to strike it. The specimen was caught in a tank of water to cool it quickly. Pressures from 75 to 435 Kb were obtained by varying the amount of explosive in the plane wave generator the thickness of the driver plate and the distance between the driver plate and the specimen.

The first step was to prove that the markings observed by Smith were indeed indicative that the crystals had twinned as this has been questioned⁽²⁸⁾. Rotation and oscillation photographs similar to those shown in Fig. 3 confirmed that shocked single crystals did contain twins⁽²⁹⁾.

In Fig. 10 the density of twins in shock-loaded polycrystalline copper (measured metallographically) is seen to increase rather sharply at about 250 Kb. However the rise is suspicious because the width of the twin increases sharply at about the same pressure; the increase in apparent density may well be due to the limits of resolution with the optical techniques! The problem is illustrated further in Fig. 11;

Photomicrographs are shown of four single crystals shock loaded in $[100]$ to increasing pressures. At all pressures, there appear to be markings; it is difficult to decide which are twins. In Fig. 11(c) there are faint sharp lines which might be twins near the "dotted" lines; these are absent at the lowest pressure. Only broad markings which appear more like twins are apparent at the highest pressure. (All the markings were identified by two-surface analysis as $\{111\}$ traces.) The absence of these wide markings at 300 Kb in a crystal shot in a $[100]$ and in certain grains in a polycrystalline specimen led Prof. Smith to conclude that there were no twins in the crystal shot in $[100]$, and also that there was no correlation between hardness and the presence of these markings.

In contrast to the resolution of a few thousand Å possible with an optical microscope, twin diffraction spots can be recognized from twins of a few tens of Angstroms. X-ray oscillation photographs, such as that shown in Fig. 3(b), show twins in all crystals illustrated in Fig. 11, except at the lowest pressure. Oscillation photographs taken by us of Prof. Smith's crystal, shot in $[100]$ at 300 Kb, also indicated the presence of twins. In fact, it is not even possible to make too much out of the appearance of the markings, when they are seen. A crystal shot in $[100]$ at 435 Kb has finer markings than one shot in $[111]$ at the same pressure. As will be shown later, this does not indicate that the pressure for twinning is greater in the $[100]$.

These results may well be significant in normal deformation studies; after any delayed yield or irregularities in a stress-strain curve, the absence of markings that look like twins does not mean they are not formed; even if there are no discontinuities, an x-ray study could be fruitful, as twins have been found in crystals with smooth stress-strain curves, (see the chapter by Venables).

If there are many small twins in a single crystal or

grain a quantitative estimate of the amount of twinning can be readily made by techniques directly analogous to that used for measurements of the quantitative amount of a second phase. The diffracted power depends not only on the quantity of a phase, but also on the absorption, scattering and volume per unit cell. As these last three factors are the same for the twin and the matrix, the ratio of diffracted power from a twin spot to that of the matrix can be used for a measure of the volume fraction of twinning, and as little as a tenth of a percent of twinned volume can be found. Knowing the twin elements one can calculate the positions of spots (as in part B above). Assuming the twin spots and matrix are brought into reflection in the plane of the goniometer and that filtered radiation is used, this volume fraction (V_T/V_P) is given by :

$$\frac{V_T}{V_P} = \frac{P_T \frac{1 + \cos^2 2\theta}{\sin 2\theta} P |F|^2_P A_P}{P_P \frac{1 + \cos^2 2\theta}{\sin 2\theta} T |F|^2_T A_T}$$

where the P's represent integrated intensities of the spots from the twin (T) and parent (P) respectively and θ is the diffraction angle. The structure factor (F) contains the usual angular dependent Debye temperature factor. The absorption term (A_T) has angular factors in it and varies with the sample geometry⁽⁷⁾. A spherical or cylindrical specimen is most convenient, as the absorption factors for these geometries have been tabulated. To get satisfactory integrations the specimens should be quite small, of the order of 0.005" diameter. A wide open counter should be used in a θ - 2θ scan, with the crystal oscillating rapidly during the scan to get all the contributions from any mosaic. Because of the extensive mosaic structure introduced

by deformation, it is difficult to get high precision (for further details see ref.30).

We will illustrate this for the case of the shock-induced twins in copper. Twinning on each of the $\{111\}$ gives a distinct pattern in an oscillation photograph, although all three twin directions in each plane give the same reciprocal space. Therefore if the twins are small one can measure the volume fraction of twins resulting from twins on each plane by choosing a spot for each twin plane. The data for the crystals illustrated in Fig. 11 and for a series of crystals shot in the $\langle 111 \rangle$ is presented in Table I. A comparison with optical measurements is given. (The range of values represents the error due to variations in the integrated intensities of four 111 's from the matrix.) The total volume fraction obtained from the x-ray measurements is equal to or lower than that observed optically. This is the reverse to what might be anticipated as the x-rays should "see" twins that are not seen optically. However, careful etching revealed that an apparent twin is really made up of many segments as shown in Fig. 12, between which there is untwinned material.

Note in Table I that the volume fraction of twinning in copper increases to a constant level over a narrow pressure range and then levels off. By comparing these results with the microstructures, it appears that the distribution changes with increasing pressure from many fine twins to fewer but thicker twins. Also the ratio of pressures to cause twinning for loading in the $[111]$ compared to loading in the $[100]$ is about 1.5; according to Fig. 7, this means that the twins in the $[100]$ form in the tensile rarefaction wave and those in $[111]$ form in the compressive shock wave.

Hardness seems to correlate well with the x-ray values for twin density, in contrast to Prof. Smith's results based on optical studies; the VHN (at 100 g load), measured on the (110) parallel to the shot direction, $[100]$, rose 46 points in the pressure range 145 Kb to 250 Kb, but was fairly constant with pressures above and below this range.

In polycrystalline or powder samples, twin lamellae of significant thickness will not have any effect on the powder diffraction pattern. The reason for this is shown in Fig. 13 for an f.c.c. material, twinned across a (111). The Debye rings from a powder result from the intersection of the Ewald sphere (radius $1/\lambda$) with a sphere whose radius is the distance from the origin to the diffracting spot in question. Reciprocal lattices of the matrix and twin, shown in Fig. 13, have been drawn, using the equivalent hexagonal unit cell shown in the figure; by using this cell it is easy to show the effect of twinning on reciprocal space by a rotation around the normal to the twin plane. In Fig. 13(b) and (c) it can be seen that the distance from the origin to a spot from the matrix and to its twin are identical, and therefore, there is no effect on the pattern.

However, if there is an appreciable number of very thin twins (of several Å) there is an effect on the diffraction pattern. A rigorous treatment of this problem of twin faults in several structures has been carried out^(31,32,33,34,35,36) and we will only consider the basic concepts here. For a high density of small twins on (111) in an f.c.c. structure there are a great number of faults in the stacking sequence of (111) planes. At any reciprocal lattice point that diffracts, the periodicity of the structure in three dimensions can be thought of as an isotropic compression on that point keeping it small and sharp. If in one direction the periodicity is destroyed, the pressure is released in that direction and the intensity distribution is "squeezed" along this line; for twins on (111) this occurs along the [111] direction in reciprocal space. There is no effect on the spot whose direction from the origin of reciprocal space is normal to the planes which are shifted (the 003 spot) as there is no component of the shift of the (111) planes in that direction. Thus, different peaks on a powder pattern will

show markedly different broadening, depending on whether these rods of scattering pass through the sphere more nearly end-on of length-wise, and how many of the components of a peak are affected. In this example for f.c.c., the 200 peaks should be broader than the 111 as there are fewer components of the 111 affected and also the 111 cuts the Ewald sphere more nearly end-on than the 200. The peaks will also streak toward the twin reflection, resulting in an asymmetry, i.e. the 111 in a powder pattern will tail toward high angles and the 200 to low angles.* The reason for this is that as the fault density increases, the f.c.c. structure converts to a hexagonal close-packed structure, whose reciprocal lattice points are halfway between the horizontal rows in Fig. 13. (The c axis of the hexagonal structure is $2/3$ of the $\langle 111 \rangle$ in the cubic phase, so that the rows in the hexagonal reciprocal space are at $3/2$ the distance shown.) Therefore it is possible to determine the density of twin fault from the extra intensity on one side of a peak, as compared with the other⁽³⁴⁾, or from the sine coefficients of a Fourier series representing the peak⁽³²⁾, or most precisely, from the difference in positions of the maximum of a peak and its center of gravity, $(\Delta C.G.^{\circ}2\theta)^{**}$ ⁽³⁵⁾†. $\Delta C.G.$ in degrees 2θ can be written in terms of β where $1/\beta$ is the average number of $\{111\}$ planes between twin faults on all four of the $\{111\}$ and $\frac{4}{\beta}$ is the number between $\{111\}$ planes of any one twin system:

$$\Delta C.G.^{\circ}2\theta = \beta \frac{360 \ln 2}{\pi^2/3} \frac{1}{u+b} \sum_b \pm \frac{h+k+l}{|h+k+l|} \tan \theta$$

*Residual strains can also cause an asymmetry, but this will be in the same direction for all peaks.

**The first and last methods minimize the effect of asymmetry due to strains in the determination of β . If sine coefficients are used more than two peaks must be examined to separate the effects of twin faults from the effects of strain.

†One layer stacking faults cause the well-known peak shift⁽³²⁾ in the f.c.c. system; we will not discuss these here.

b is the number of components of a peak on a powder pattern which are affected by twins ($h+k+l=3N\pm1$) and u is the number unaffected.

$$\Delta C.G. (^{\circ}2\theta)_{111} = + 11 \beta \tan \theta$$

$$\Delta C.G. (^{\circ}2\theta)_{200} = - 14.6 \beta \tan \theta$$

In any heavily twinned or distorted powder pattern of an f.c.c. material, many peaks, such as the 111 and 200, will tend to overlap and it is better to calculate the combined $\Delta C.G.$ for two such peaks, to minimize problems in extrapolation between peaks. Using the 111 and 200:

$$\Delta C.G. (^{\circ}2\theta)_{111} - \Delta C.G. (^{\circ}2\theta)_{200} = \beta (11 \tan \theta_{111} + 14.6 \tan \theta_{200})$$

In doing this work slits should be used that eliminate the doublet resolution and make the peaks from an annealed sample symmetrical. Some typical conditions are 0.1° receiving slits and 1° divergence for peaks at less than $2\theta = 90^{\circ}$.

For twinning on the $\{112\}$ in a b.c.c. material, the equations are⁽³⁶⁾:

$$\Delta C.G. (^{\circ}2\theta) = - \beta \frac{360}{\sqrt{3\pi^2}} \frac{1}{u+b} \left(\sum_b \pm \frac{(-h-k+2l)}{|-h-k+2l|} \right) \tan \theta$$

or $\Delta C.G. (^{\circ}2\theta)_{110} = - 2.43 \beta \tan \theta$

$\Delta C.G. (^{\circ}2\theta)_{200} = 4.87 \beta \tan \theta$ etc.

b is the number of components for which $-h-k+2l = 3N\pm1$. (The unit cell used in the derivation has an edge normal to $[\overline{112}]$). Note that for a b.c.c., the asymmetry is in the opposite direction to that for f.c.c. for the first two peaks, and that the constant is much smaller. Experience indicates that with

the broad peaks characteristic of faulting it is possible to determine $\Delta C.G.$ for one peak or for a pair, to $\pm 0.01^\circ 2\theta$. This means that to be detected with the 111 - 200 combination, in an f.c.c. materials such as Ag, with copper radiation, β must exceed about 0.002, i.e. a twin fault every 2000 {111} planes. For a b.c.c. material such as iron, for peaks at about $2\theta = 90^\circ$, a minimum density of about one twin every 1800 planes is required on each and every {112}.

With hexagonal close-packed materials and faulting on the basal plane, there is no asymmetry because the twinned reciprocal space is identical to that of the matrix. However, the broadening produced by twins (on 0001) can be used to measure the twin fault density. If it can be proved that the contributions due to strain and mosaic size are small compared with the effects of twins and faults the half breadths (B) are⁽³⁴⁾:

$$h-k=3n\pm 1 \begin{cases} l_{\text{even}} B(^{\circ}2\theta) &= \frac{360}{\pi^2} \tan \theta |l| (d_{hkl}/c)^2 (3\alpha+3\beta) \\ l_{\text{odd}} B(^{\circ}2\theta) &= \frac{360}{\pi^2} \tan \theta |l| (d_{hkl}/c)^2 (3\alpha+\beta) \end{cases}$$

where $1/\alpha$ is the average number of planes between stacking faults, in the c direction. Thus if hhl peaks are sharp in comparison with other peaks, the broadening is due to faulting. From say, a 101 peak and a 102, α and β can be determined. When α is 0 the 102 will be three times as broad as the 101, after correcting for $\tan \theta$.

As the breadth can be determined to about $\pm 0.012^\circ 2\theta$, with a peak in the region of $2\theta = 90^\circ$ such as the 102 from Co with Fe radiation, we can detect a β of ≈ 0.0015 . There must be at least one twin every 700 basal planes.

Some typical values for β are given in Table II. No

faults could be found in filings of the b.c.c. metals, Fe, Nb, Ta, and β brass, even when we filed the metals in liquid nitrogen.

It must be emphasized that besides the fact that high densities are required, these methods are best applied when the twin systems are known. However, if there is a pattern of unusual broadening or asymmetries it is possible to assume certain twin planes, construct the reciprocal space and try to find a model that fits the data. There is always the possibility of twinning on different types of systems, and any such model should be carefully checked by examining the twins with the single-crystal methods described above.

Some asymmetry can be produced by segregation to stacking faults⁽⁴¹⁾, but this effect is too small to measure. If there are appreciable numbers of extrinsic double stacking faults, these will produce an asymmetry opposite to that predicted for twin faults⁽⁴²⁾, so that one is measuring the net concentration of twin faults from the asymmetry. Whether or not these are present can generally be decided if the fault density is high by seeing if β and α account for the apparent mosaic size, determined from Fourier analysis⁽³⁴⁾. (The true mosaic size makes little contribution when the fault density is high.)

* * * * *

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TABLE I.

Volume Per Cent of Twins in Shock Loaded Copper vs. Pressure

a) [100] shock direction*

Twin Plane	100 Kb	145 Kb	200 Kb	250 Kb	435 Kb.
$\{111\}_1$	0	$0.3 \pm 0.06\%$	0.05%	$3.0 \pm 0.6\%$	$1.0 \pm 0.2\%$
$\{111\}_2$	0	$1.0 \pm 0.2 \%$	0.017%	$2.0 \pm 0.4\%$	$2.1 \pm 0.42\%$
$\{111\}_3$	0	0.014%	0.088%	$0.2 \pm 0.04\%$	$1.0 \pm 0.2 \%$
$\{111\}_4$	0	0.011%	$0.4 \pm 0.08\%$	$0.1 \pm 0.02\%$	Not detect- able
TOTAL %	0	1.3 ± 0.3	0.5 ± 0.1	5.3 ± 1.1	4.1 ± 0.8

Volume per cent determined by cutting out
(and weighing) twins on a photomicrograph
of a (110) surface. 10.5%

*Volume per cent determined from integrated x-ray intensities of the
 $1/3\{115\}_T$ and $\{111\}_P$ as described in the text.

b) [111] shock direction*

Twin Plane	145 Kb	200 Kb	250 Kb	435 Kb.
$\{111\}_1$	0	$0.5 \pm 0.1\%$	$0.4 \pm 0.1\%$	$6.2 \pm 1.2\%$
$\{111\}_2$	0	0.03%	$1.3 \pm 0.3\%$	$0.7 \pm 0.1\%$
$\{111\}_3$	0	$0.1 \pm 0.02\%$	$0.2 \pm 0.04\%$	$5.1 \pm 1.0\%$
TOTAL %	0	0.6 ± 0.1	1.9 ± 0.4	12.0 ± 2.3

Volume Per Cent determined by cutting out
(and weighing) twins on a photomicrograph
of a (110) surface. 11.7% 12.5%

*Volume Per Cent determined from integrated x-ray intensities of the
 $1/3\{442\}_T$ and $\{200\}_P$ as described in the text.

TABLE II
Some Typical Values of the Twin Fault Probability β^*

Material	Treatment	Reference	$\beta \times 10^3$
Ag	Electrodeposited from argento-cyanide solution at R.T	37	1
Ag	Electrodeposited at R.T. + 0.34 mg. potassium ethyl xanthate	37	11
Ag	Filed at R.T.	38	11
Ag - 9 pct. Sn	Filed at R.T.	38	37
Cu	Filed at R.T.	39	9.5
α -Brass	Filed at R.T.	39	49
Co	Filed at R.T. and annealed 2 hrs. at 300°C.	40	33

*All the values for f.c.c. metals and alloys were determined by the $\Delta C.G.$ method described in the text.

FIGURE CAPTIONS.

Fig. 1. Formal crystallographic elements of mechanical twinning. Shear is in the direction n_1 . K_1 is the habit or twin plane. K_1 , K_2 , n_1 and n_2 are the undistorted planes and directions. The plane of shear is perpendicular to n_1 and K_1 . The shear, S , is given by $2\cot 2\phi$.

Fig. 2. a) real edges of hexagonal unit cell.
b) reciprocal lattice vectors of a region of a hexagonal crystal which is not twinned.
c) these vectors in twinned region.
d) intersection of the Ewald sphere with reciprocal space of the twin and parent, when the incident beam covers the twin and parent.

Fig. 3. The copper single crystal used for these diffraction patterns was loaded with a planar shock wave of 145 Kb in the $[001]$.
a) Laue back-reflection pattern - 1/2 hr.
b) 15° oscillation pattern to one side of $[100]$. Twin spots are circled. (Filtered Cu radiation). - 7hr.
c) 360° rotation pattern around $[001]$. Twin spots are circled. (Filtered Cu radiation).
(b) and (c) were taken using a small needle cut from the larger crystal. - 12 hr.

Fig. 4. a) Optical measurement of the tilt of surfaces in the twin and parent.
b) Stereographic projection of poles of surfaces determined in (a) onto K_1 . (K_1 is determined from 2 surface analysis).

Fig. 5. Indexing an oscillation pattern from twinned Cu crystal oscillated 15° around $[001]$. $004/3$ layer is shown. (The co-ordinate $1/3$ is determined as the ordinate of the layer along the $[001]$ in which the spot lies.) r_c is the camera radius and r_s is the radius of the sphere of reflection. a) the pattern; ϵ is determined from a Bernal chart⁽⁸⁾. b) construction for indexing.

Fig. 6. Stereographic projection for graphite on (0001) . Poles of twin and parent spots obtained from an $\pm 15^\circ$ oscillation pattern around the normal to the plane of a_2^* and c^* of the parent. (The pattern was kindly given to us by Prof. E. J. Freise; it was taken as part of a recent study of twinning in graphite⁽¹⁹⁾, but was not used.) The 180° rotation axes (in the large boxes) were determined from the intersection of great circles containing poles of the twin and parent spots of the same type of index. K_1 was identified from two surface analysis.

Fig. 7. Twinning systems in an f.c.c. structure with the maximum resolved twinning stress in tension and compression at the corners of the stereographic triangle. Values of $\cos \lambda \cos \chi$ are given in the three corners of the stereographic triangle.

Fig. 8. Reciprocal space for b.c.c. with twinning on the systems:

$$\begin{array}{ccc} \overline{K_1} & \overline{n_1} & \\ \left\{ \begin{array}{c} 11\bar{2} \\ \bar{1}21 \\ \bar{2}11 \end{array} \right\} & [111] & - \bullet \end{array}$$

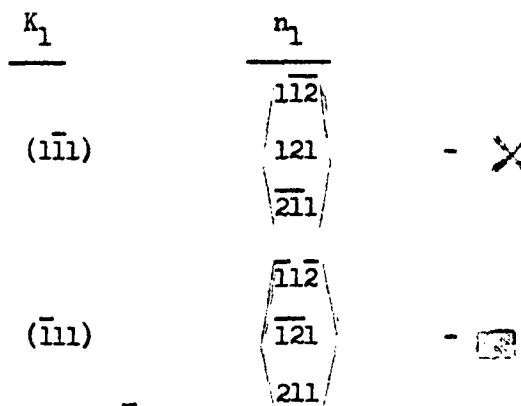
K_1	n_1	
$\begin{Bmatrix} 112 \\ \bar{1}21 \\ 2\bar{1}1 \end{Bmatrix}$	$[\bar{1}\bar{1}1]$	- Δ
$\begin{Bmatrix} \bar{1}12 \\ 12\bar{1} \\ 211 \end{Bmatrix}$	$[\bar{1}\bar{1}1]^*$	- $\times$
$\begin{Bmatrix} 1\bar{1}2 \\ 12\bar{1} \\ 211 \end{Bmatrix}$	$[\bar{1}\bar{1}1]$	- $\square$

To produce a $(00\bar{l})$ layer, rotate the corresponding positive layer 180° about $[001]$ axis.

Open circles are diffraction points of the reciprocal lattice of the matrix.

Fig. 9. Reciprocal space for f.c.c. with twinning on the systems:

K_1	n_1	
	$11\bar{2}$	
(111)	$\bar{1}21$	- $\odot$
	$2\bar{1}1$	
	$11\bar{2}$	
$(\bar{1}\bar{1}1)$	$\bar{1}21$	- Δ
	$2\bar{1}1$	



To produce a (00 $\bar{l}$) layer rotate the corresponding positive layer 180° about [001] axis.

Fig. 10 Density and apparent width of twins vs. pressure in shock loaded OFHC polycrystalline copper.

Fig.11 Single crystals of copper, subjected to planar shock waves in the [001] and sectioned on (110); polished and etched.

Fig. 12. Single crystal of copper shock-loaded in [110] at 300 Kb and sectioned on the (110). Heavily etched with 1M (NH₄)₂S₂O₈; 6M NH₄OH; 0.1M NH₄Cl; x 1000

Fig. 13. a) hexagonal unit cell (A_1) in the f.c.c. structure (a_1) and the transformation matrix for indices and axes.
 b) reciprocal space of an f.c.c. structure indexed with the hexagonal axes in (a). The original f.c.c. indices are indicated in parentheses next to the circles which represent diffraction spots where the structure factor is not zero.
 c) the reciprocal space that results from twinning on the (111), equivalent to 180° rotation around [111]. The asymmetry, when there are many fine twins, is indicated by the shaded "peaks" next to the spots.

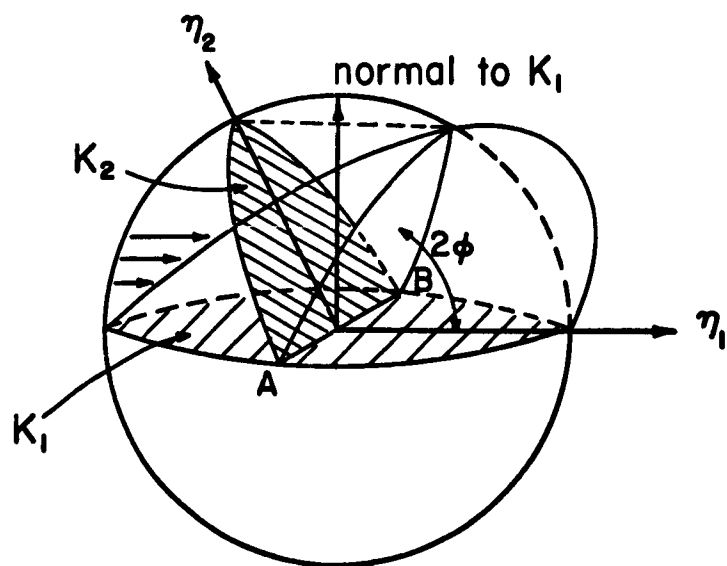


FIG. 1

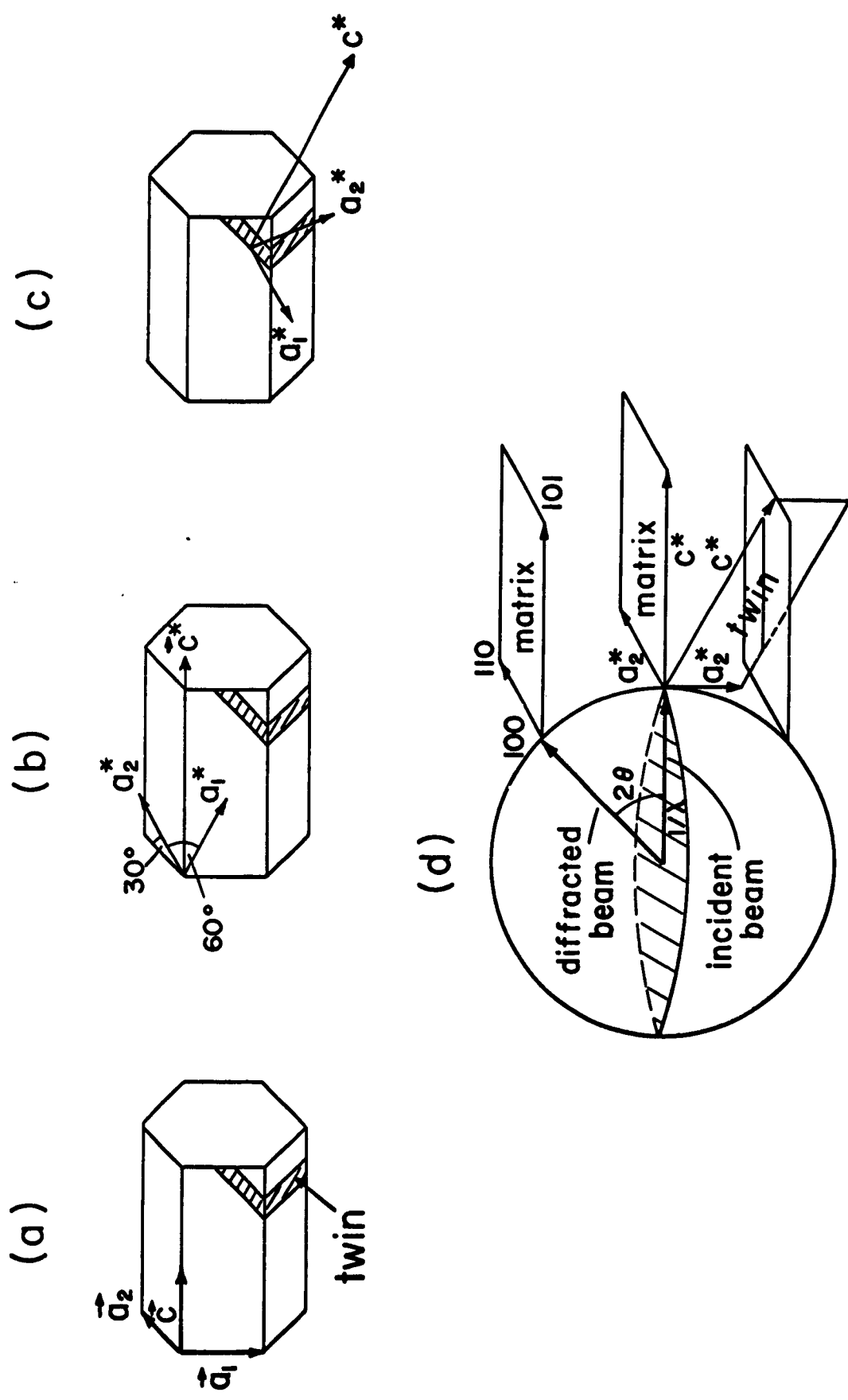
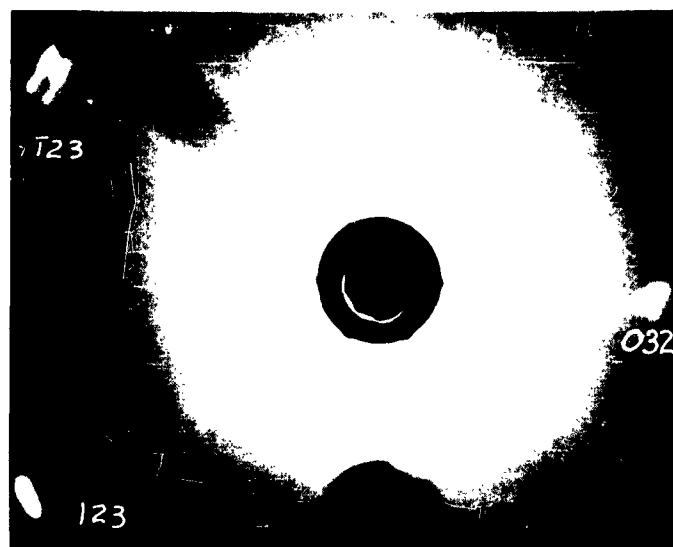
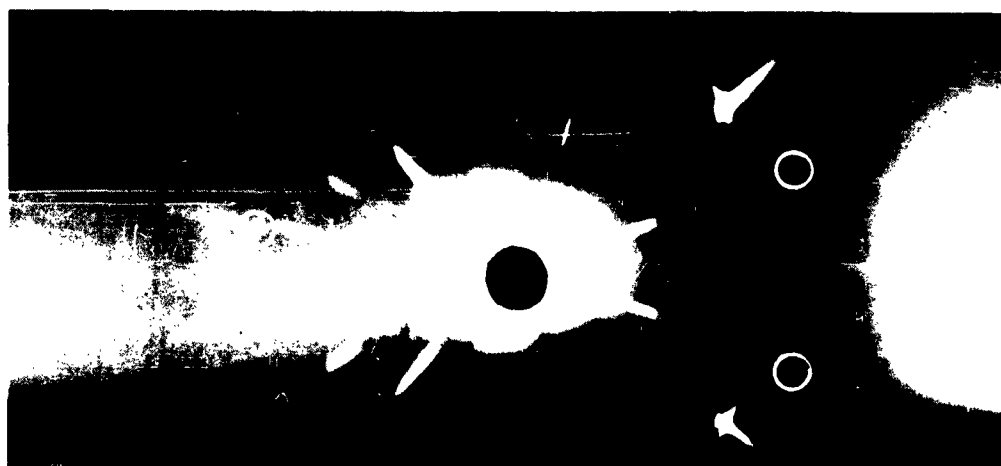


FIG. 2



(a)



(b)



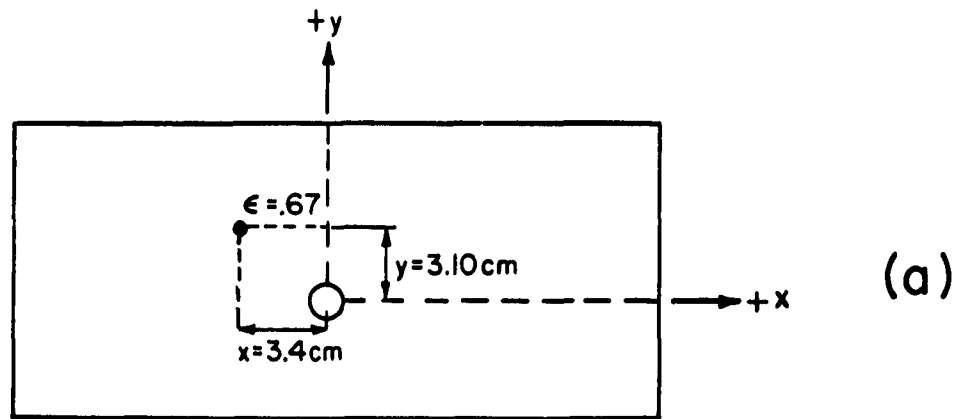
(c)

FIG. 3

A diagram of a rectangular block with a shaded top surface. Four forces are shown acting on the block: P_I and T_I act to the left, while P_{II} and T_{II} act to the right. P_I and P_{II} are applied to the top surface, and T_I and T_{II} are applied to the side surfaces.



FIG. 4



$$\left[\gamma = \frac{x}{r_c} \text{ (Radians)} \quad \begin{array}{l} r_s = 10 \text{ cm.} \\ r_2 = 6.7 \text{ cm} = \\ \epsilon \times r_s \end{array} \right]$$

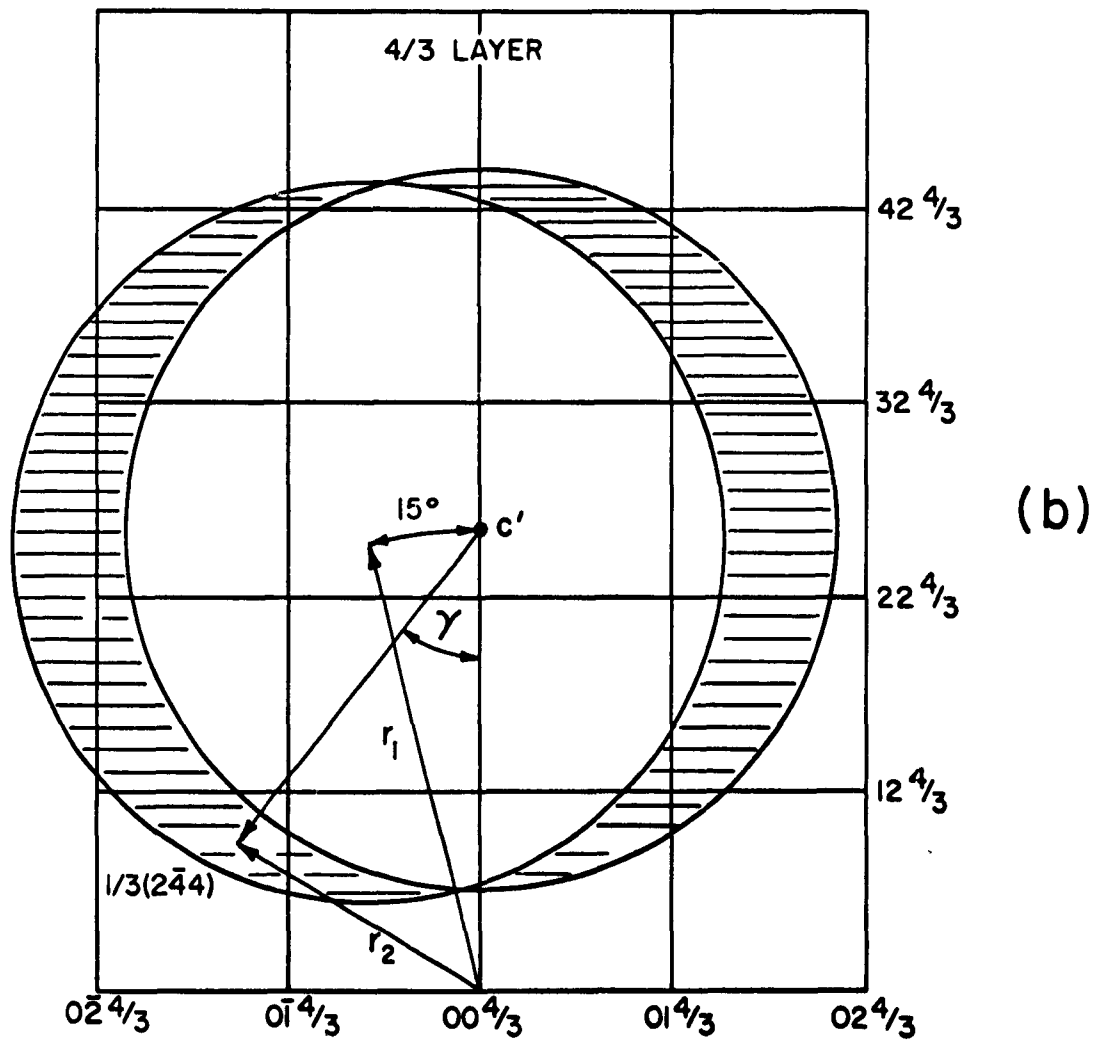
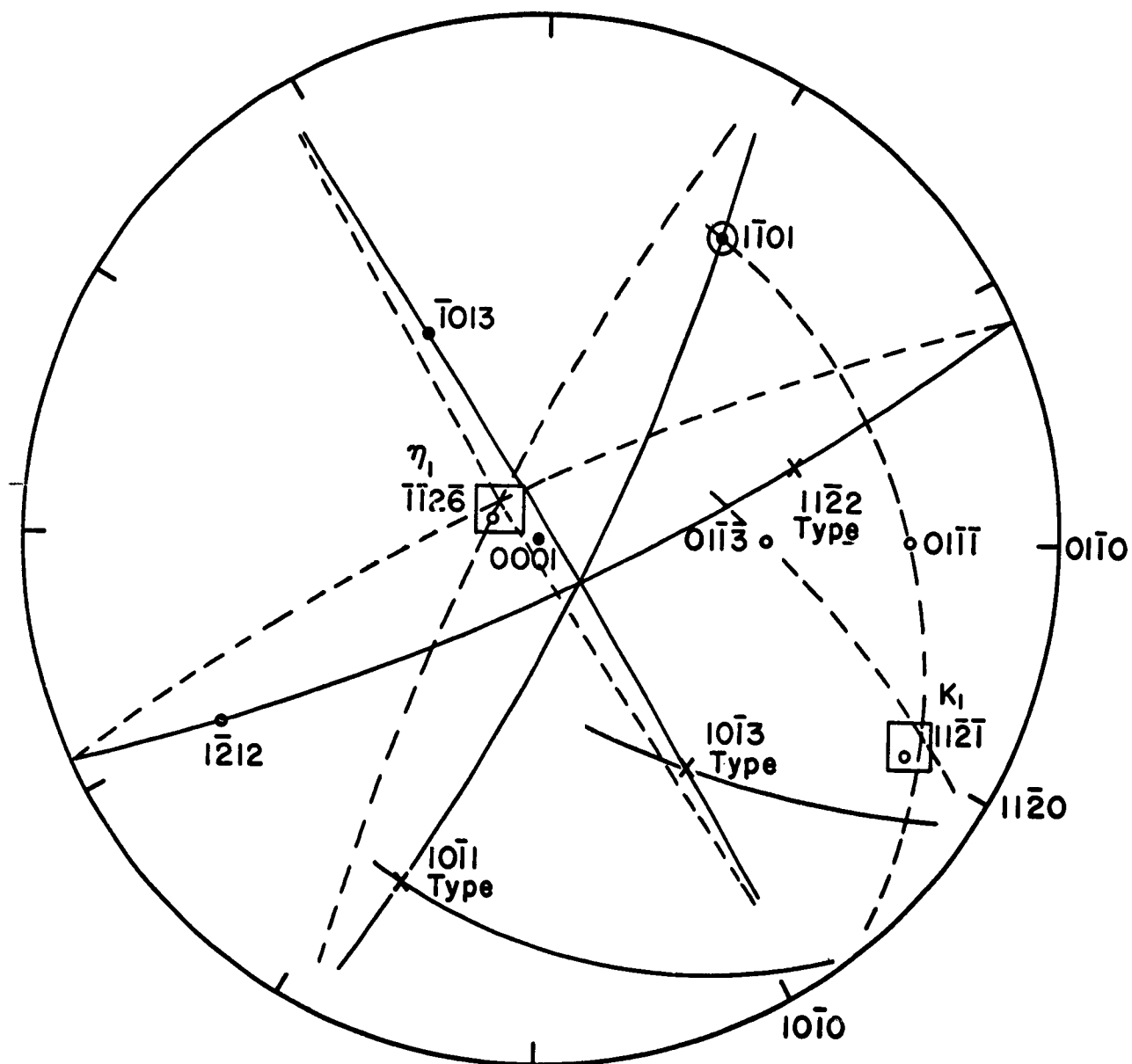


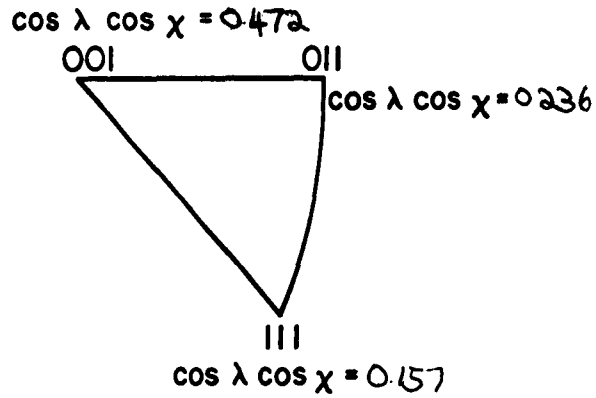
FIG. 5



- x — Twin Poles (Top)
- — Matrix Poles (Top)
- — Matrix Poles (Bottom)

FIG. 6

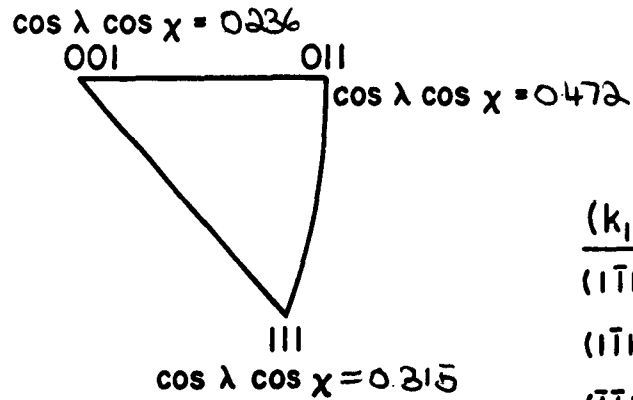
FAVORABLE TWINNING SYSTEMS IN COMPRESSION



$(k_l)[\eta_l]$	STRESS AXIS		
	[001]	[111]	[011]
$(\bar{1}\bar{1})[\bar{1}\bar{2}]$	x	x	
$(\bar{1}\bar{1})[\bar{1}\bar{2}]$	x		
$(11)[1\bar{2}]$		x	
$(\bar{1}1)[\bar{1}2]$	x	x	x
$(11)[1\bar{2}]$	x		x

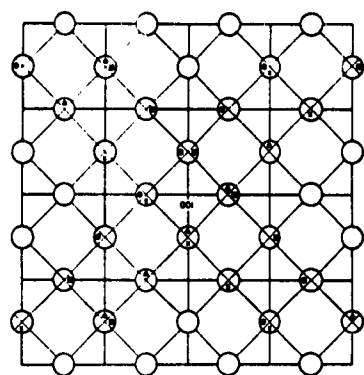
x indicates favorable system

FAVORABLE TWINNING SYSTEMS IN TENSION

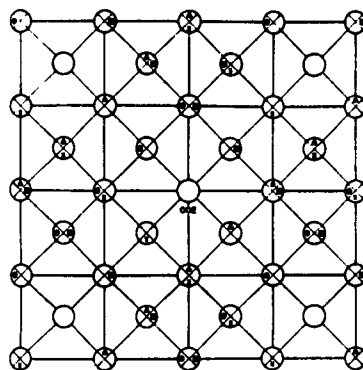


$(k_l)[\eta_l]$	STRESS AXIS		
	[001]	[111]	[011]
$(\bar{1}\bar{1})[12]$	x	x	
$(\bar{1}\bar{1})[2\bar{1}]$	x		
$(\bar{1}\bar{1})[\bar{2}1]$	x		
$(\bar{1}\bar{1})[2\bar{1}]$	x		
$(\bar{1}1)[21]$	x	x	x
$(\bar{1}1)[\bar{2}1]$	x		
$(11)[2\bar{1}]$	x		x
$(11)[1\bar{2}]$	x		
$(11)[12]$		x	

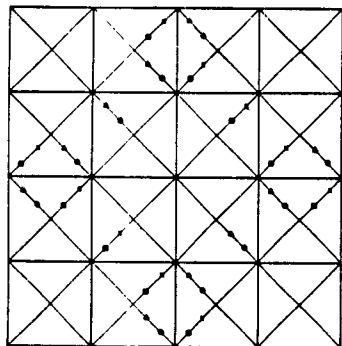
FIG. 7



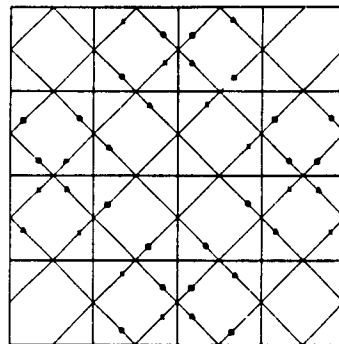
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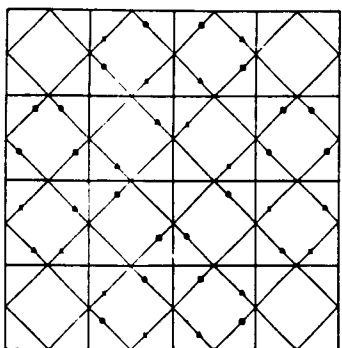
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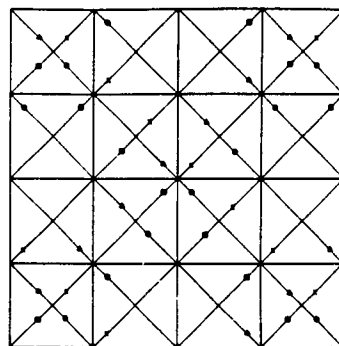
$2/3$



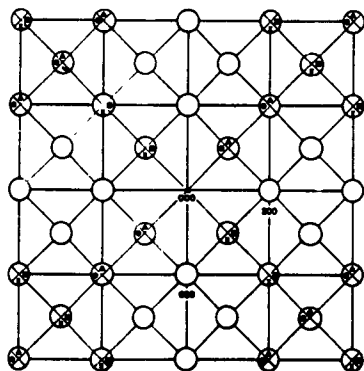
$5/3$



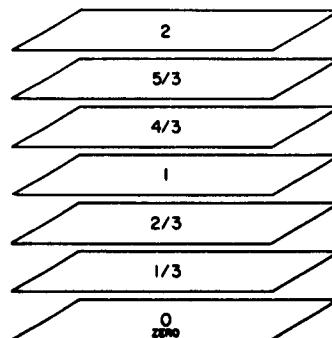
$1/3$



$4/3$



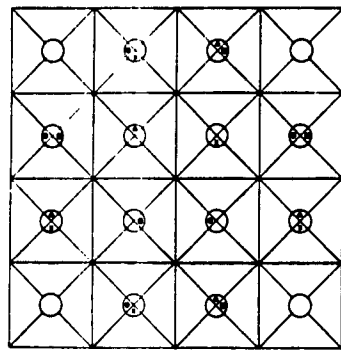
ZERO



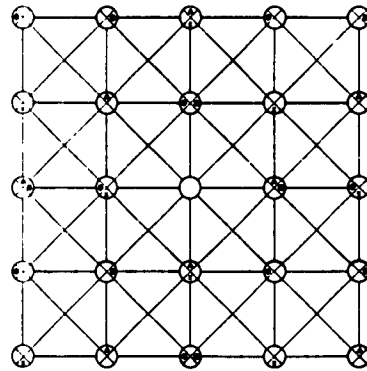
SCHEMATIC OF LAYER ORIENTATION

FIG. 8

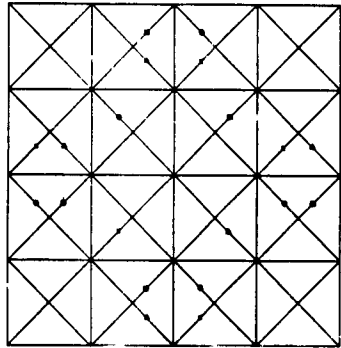
R.J.DeAngelis & J.B.Cohen



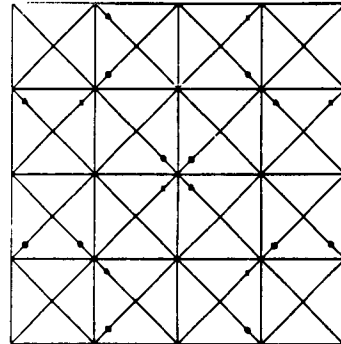
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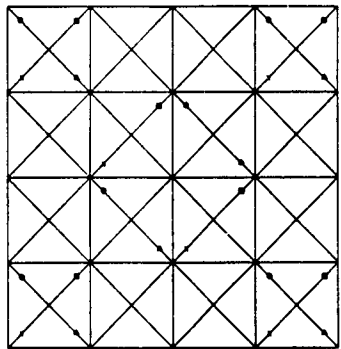
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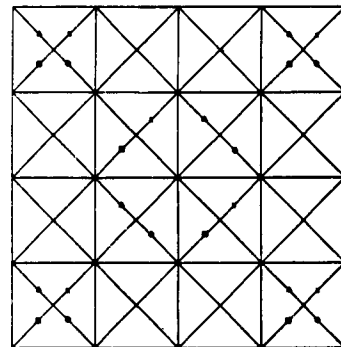
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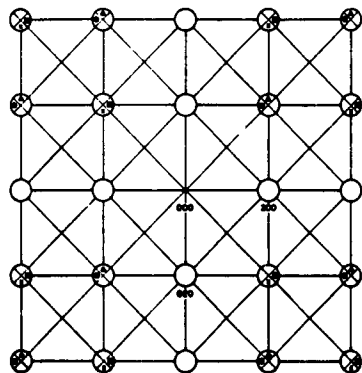
$5/3$



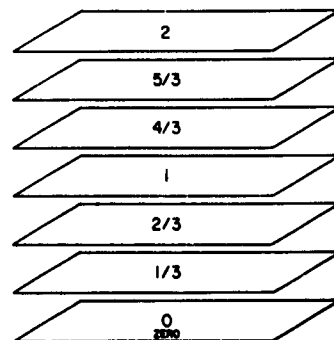
$1/3$



$4/3$



ZERO



SCHEMATIC OF LAYER ORIENTATION

FIG. 9

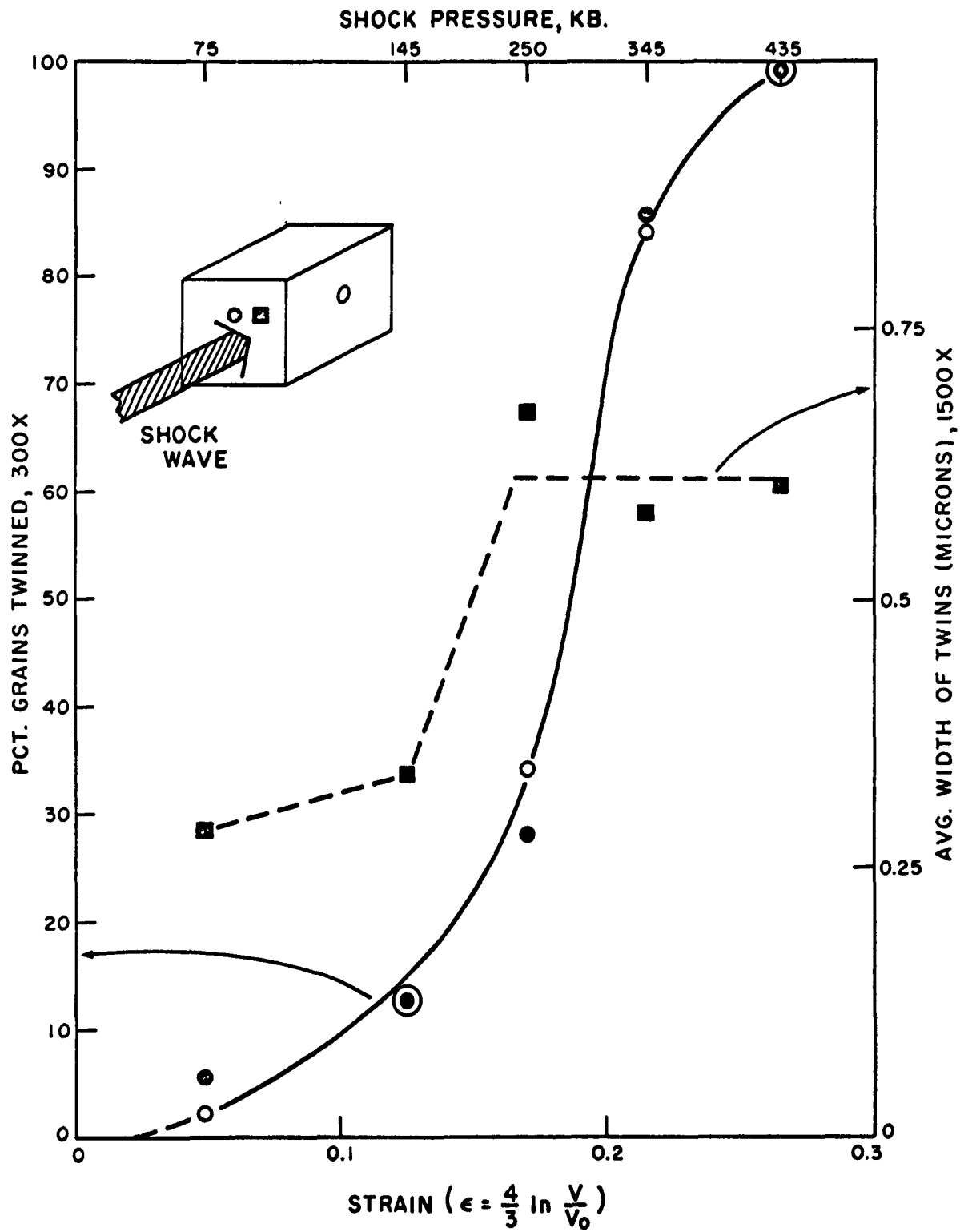
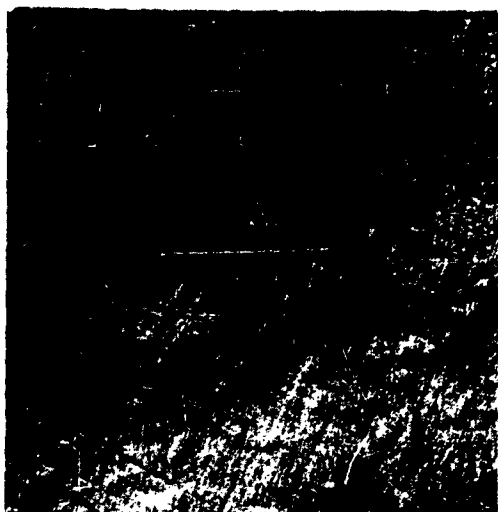
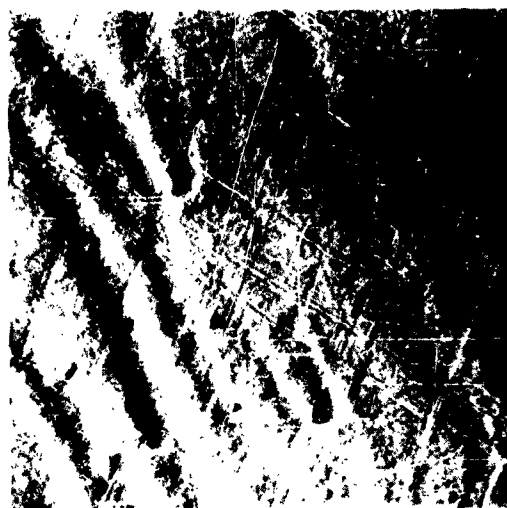


FIG. 10

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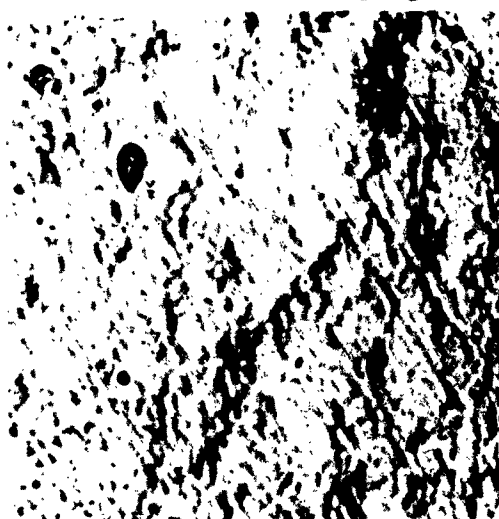
(a) 100 Kb x 250



(b) 145 Kb x 250

↑
Shock
Direction
↓

(c) 250 Kb x 100



(d) 435 Kb x 100

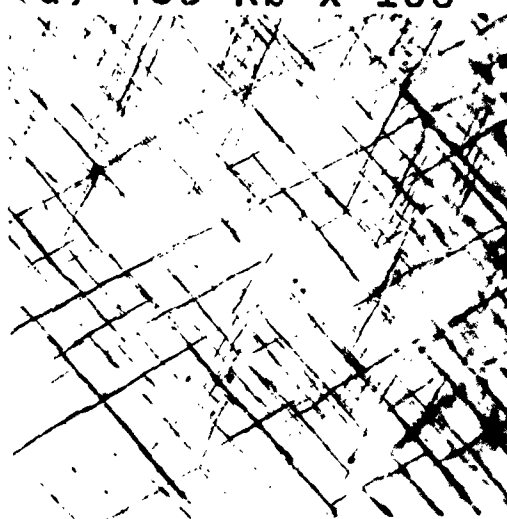


FIG.11

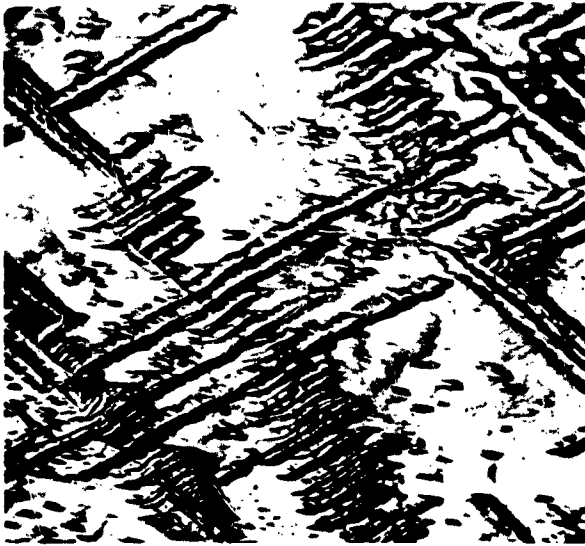
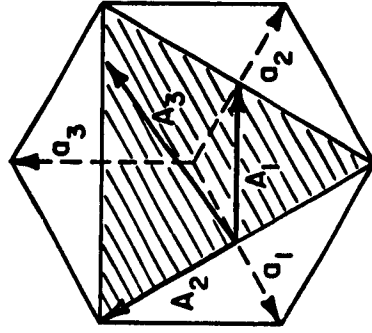


FIG. 12.

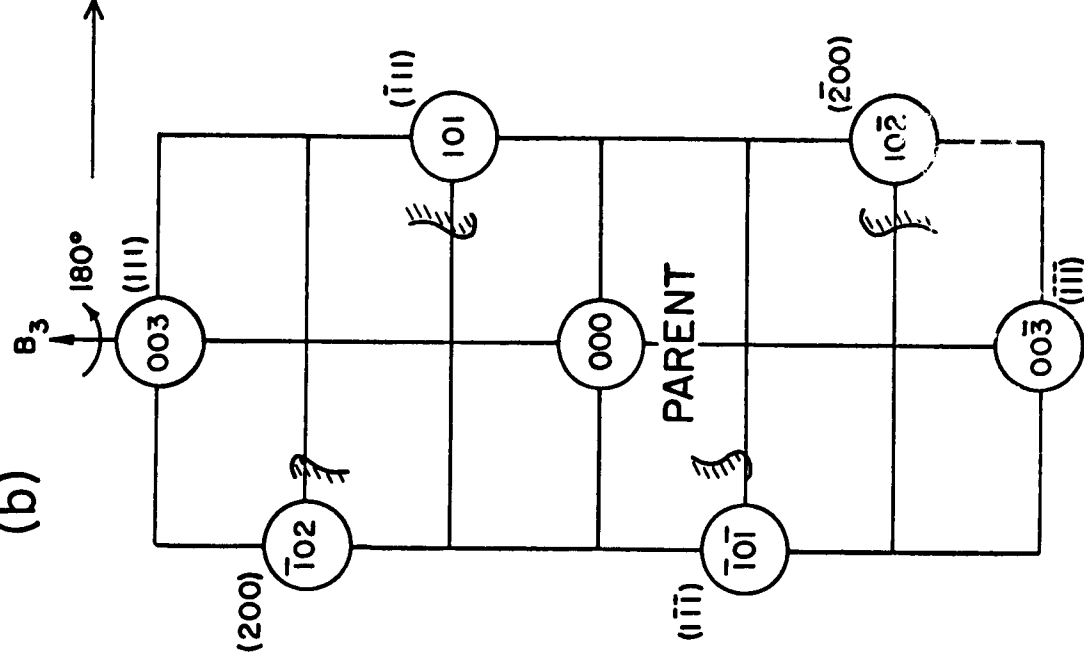
(a)



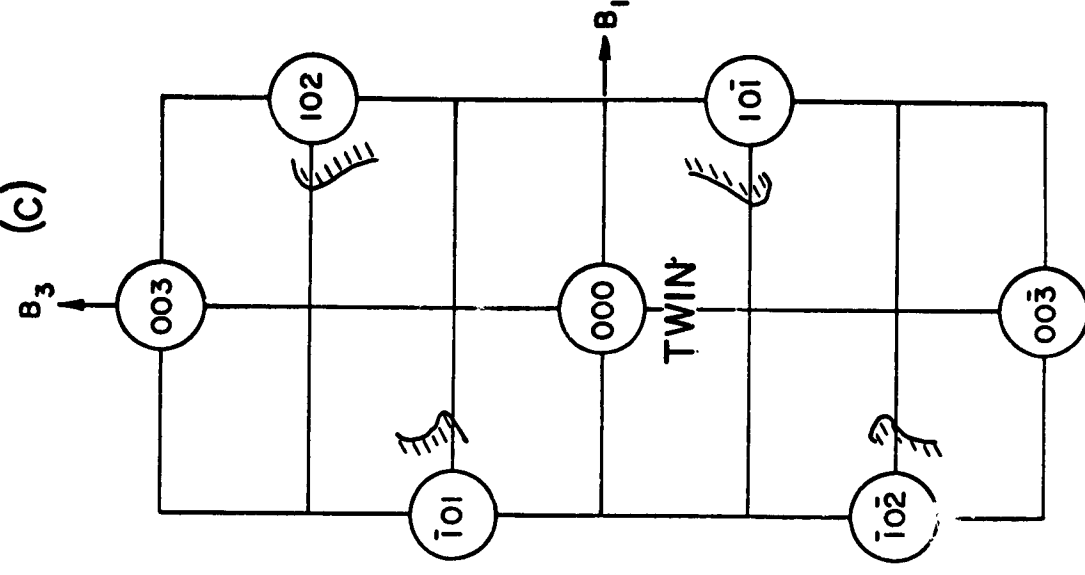
	h	a_1	k	a_2	l	a_3
H, A_1	$-1/2$	$+1/2$	0			
K, A_2	0	$-1/2$	$+1/2$			
L, A_3	1	1	1			

$$B_1 = 1/A_1, B_2 = 1/A_2, B_3 = 1/A_3$$

(b)



(c)



(H O L) PLANE
OF RECIPROCAL SPACE

FIG. 13.

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