

First

PROGRESS REPORT

(Project SR-111)

on

**LOW TEMPERATURE EMBRITTLEMENT MECHANICS
DEDUCED FROM ZINC SINGLE CRYSTAL FRACTURE STUDIES**

by

Peter Harlow Morton

and

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CASE INSTITUTE OF TECHNOLOGY

Transmitted through

**NATIONAL RESEARCH COUNCIL'S
COMMITTEE ON SHIP STEEL**

Advisory to

SHIP STRUCTURE COMMITTEE

Division of Engineering and Industrial Research

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Washington, D. C.

May 1, 1953

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Dear Sir:

As part of its research program related to the improvement of hull structures of ships, the Ship Structure Committee is sponsoring a study of transition temperature phenomena in metals other than iron at Case Institute of Technology. Herewith is a copy of SSC-58, Technical Report No. 1, of the investigation, entitled "Low Temperature Embrittlement Mechanics Deduced from Zinc Single Crystal Fracture Studies", by P. H. Morton and W. M. Baldwin, Jr.

The project is being conducted with the advisory assistance of the Committee on Ship Steel of the National Academy of Sciences-National Research Council.

Any questions, comments, criticism or other matters pertaining to the Report should be addressed to the Secretary, Ship Structure Committee.

This Report is being distributed to those individuals and agencies associated with and interested in the work of the Ship Structure Committee.

Yours sincerely,



K. K. COWART
Rear Admiral, U. S. Coast Guard
Chairman, Ship Structure Committee

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W. M. Baldwin, Jr.

under

Department of the Navy
Bureau of Ships
Contract NObs-50303

with the

Case Institute of Technology
Bureau of Ships Project NS-011-078

for

SHIP STRUCTURE COMMITTEE

ABSTRACT

The most important conclusion of this work is that low temperature brittleness in zinc single crystals is not due to the cessation of slip below a certain temperature. On the contrary slip is quite active, and it is its activity that induces low temperature brittleness: slip causes rotation of the lattice which in turn sets up orientation gradients adjacent to constricted and unslipped regions. At high temperatures these orientation gradients are accommodated by kink-planes, at low temperatures, by cleavage.

These observations are important, for they bear directly on the problem of low temperature brittleness of polycrystalline zinc (because of the orientation gradients always found in polycrystalline metals when deformed) and on the reasons for the lack of low temperature brittleness in face-centered cubic metals (which kink differently from hexagonal close packed crystals).

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INTRODUCTION

Purpose of the Investigation

This is an investigation into the effects of temperature, orientation, and specimen shape on the mode of fracture and the ductility accompanying fracture of zinc single crystals. The research was undertaken to understand the reasons why those metals not crystallizing in the face-centered cubic system become brittle at low temperatures, high strain rates, or tensile mean stress states.

Steels, in the practical sense, are the most important metals in this group, but it was felt that a study of zinc was quite useful--even in the solution of the steel problem--for the following reasons: both zinc and steel become brittle under the test or service conditions mentioned above, both metals twin, and both can fracture by cleavage. Furthermore, zinc is readily obtained in reasonable purity, it is readily fabricated into single crystals, and its transition temperature from ductile to brittle behavior under the usual tensile testing conditions is conveniently near room temperature.

Previous attempts to explain the part-time brittle behavior of non-face-centered cubic metals have usually centered on the concept of "Technical Cohesive Strength" as developed by Kuntze, McAdam and others. It is supposed

that as the temperature is lowered, the stress required to cause deformation rises more rapidly than does the stress required for fracture or technical cohesive strength, thus leading to brittle failure. The embrittling effect of triaxial tension or stress concentration can be easily explained in these terms. However, the determination of cohesive strength proves to be difficult as it is affected by the stress condition at the moment of fracture, the strain-rate, the temperature and the amount of previous deformation. The latter two variables are particularly difficult to separate; the theory makes no prediction as to the effect of these two variables, so that although it may be useful in describing the phenomenon, it does not lead to any further basic understanding of the problem. In particular it offers no explanation of why face-centered cubic metals do not show low temperature brittleness, nor any prediction of the precise temperature at which the non-face-centered cubic metals should become brittle.

Flow of Zinc Single Crystals

Zinc single crystals commonly slip on the system (0001)

$[11\bar{2}0]$ ⁽¹⁾* and twin on the $10\bar{1}2$ plane by shear in the direction perpendicular to $[1\bar{2}10]$ (see Fig. 1).

In a tensile test crystals whose (0001) basal poles lie less than 70 to 75° to the tensile axis ($\phi_0 < 70\text{--}75^\circ$) (see Fig. 2(a)) deform by slip (primary slip), the crystal rotating thereby such that in a standard stereographic projection the tensile axis migrates along a great circle toward the $[11\bar{2}0]$ slip direction (Fig. 2(b)). Because the crystal rotates during a tensile test and because it is constrained from rotating in the grips, there must be a change in orientation somewhere between the gripped and free portions of the specimen. This change was observed ^(1, 7) as a sharp bend in the crystal lattice (see Fig. 2(b)). Hess and Barrett ⁽⁸⁾ demonstrated that these bends were the same as Orwan's kink-planes ⁽⁹⁾. A kink-plane is defined as a plane lying between two portions of crystal that have slipped by different amounts and consists of a regular array of parallel edge-type dislocations. It is the form of

*Other slip systems must also act at high temperatures. Crystals strained at 100 to 200°C show markings that could not be explained on the basis of (0001) $[11\bar{2}0]$ ⁽²⁾. Kolesnikov ⁽³⁾ on rather weak evidence, it is true, suggests the slip system $(1\bar{1}00)$ $[11\bar{2}0]$ to operate in crystals having the (0001) basal plane nearly parallel to the tensile axis when tested above 300°C. Washburn ⁽⁴⁾ finds good evidence for the slip system $(110X)$ $[11\bar{2}0]$ for crystals of similar orientation tested at 400°C. All of these slip systems (including the usual system) involve a common slip direction $[11\bar{2}0]$ but different planes zonal to this direction.

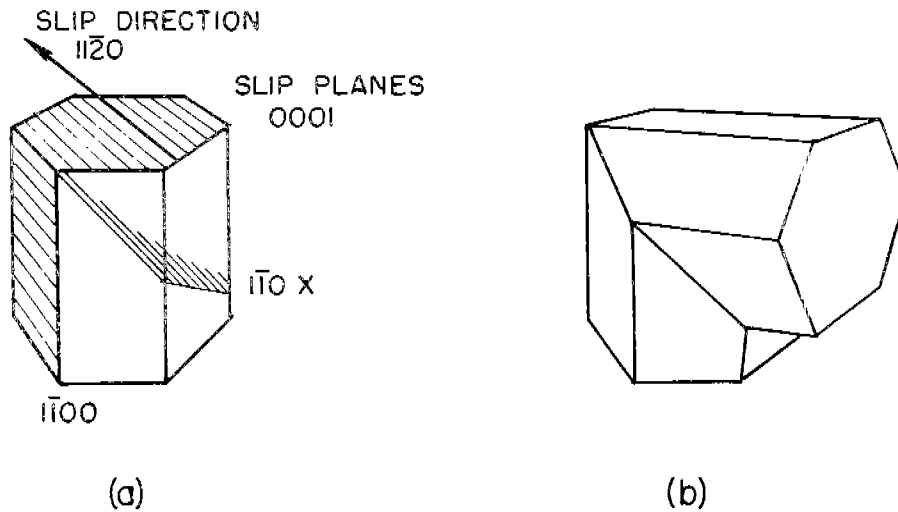


FIG. 1: DEFORMATION MECHANISM OF ZINC

- a) VARIOUS SLIP PLANES HAVE BEEN REPORTED ALL OF WHICH HAVE THE $11\bar{2}0$ SLIP DIRECTION IN COMMON. THE MOST USUAL SLIP PLANE IS THE (0001) BASAL PLANE.
- b) TWINNING OCCURS ON THE $10\bar{1}2$ PLANE. IT RE-ORIENTS THE CRYSTAL SUCH THAT THE TWINNED BASAL PLANE IS 86° REMOVED FROM THE ORIGINAL BASAL PLANE.

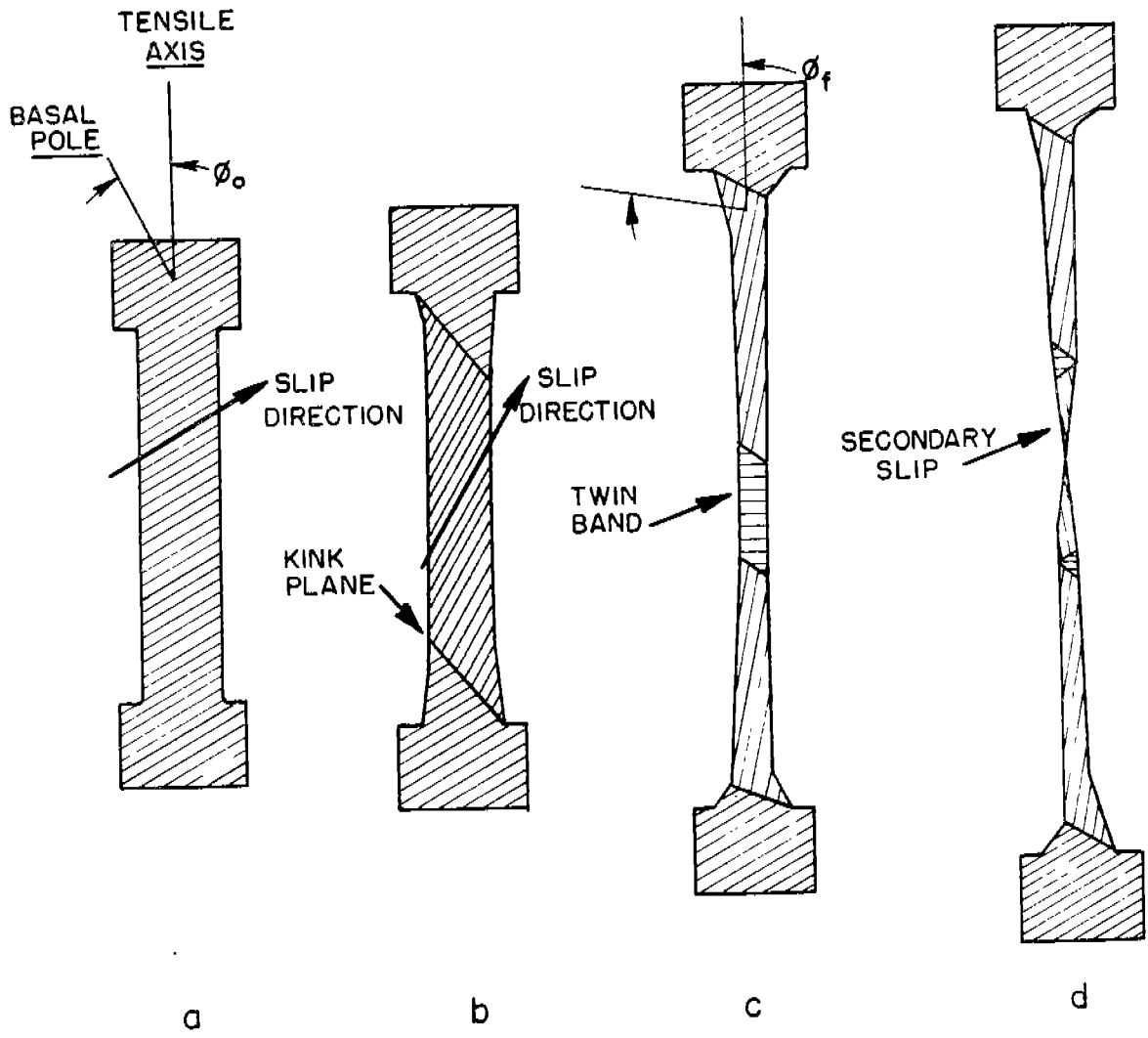


FIG. 2: STEPS IN THE FLOW AND FRACTURE OF A ZINC CRYSTAL

bend in a crystal lattice which has the lowest energy⁽¹⁰⁾ and a more gradual bend in the lattice tends to "polygonize" into these discrete bends^(11,12). Rotation of the crystal proceeds until at a critical point the crystal twins (Fig. 2(c)) yielding bands of a different orientation in which further secondary slip occurs (Fig. 2(d)). There is some confusion as to the criterion setting the critical point at which primary slip terminates. In early work^(1,5) it was reported that both zinc and cadmium twinned when the angle between normal to the (0001) basal plane and the tensile axis reached a critical value, ϕ_f , (see Fig. 2(c)), dependent upon temperature (Fig. 3). In subsequent work⁽⁶⁾ it was reported that both metals twinned when the shear strain resolved on the slip plane reached a critical value \underline{a} , also dependent upon temperature (Fig. 4). Actually, for $\phi_o < 70$ -- 75° and for high values of \underline{a} , (such as were reported cf. Fig. 4) the two criteria are very much the same (cf. Fig. 5). It is of interest to note moreover that the values of resolved shear strain reported as critical for zinc⁽⁶⁾ convert to the same range of values of ϕ_f reported as critical for zinc⁽¹⁾ as shown in Fig. 6.

Gilman⁽¹³⁾ notes that crystals in which $\phi_o > 80^\circ$ deformed at room temperature by twinning "mostly" and by

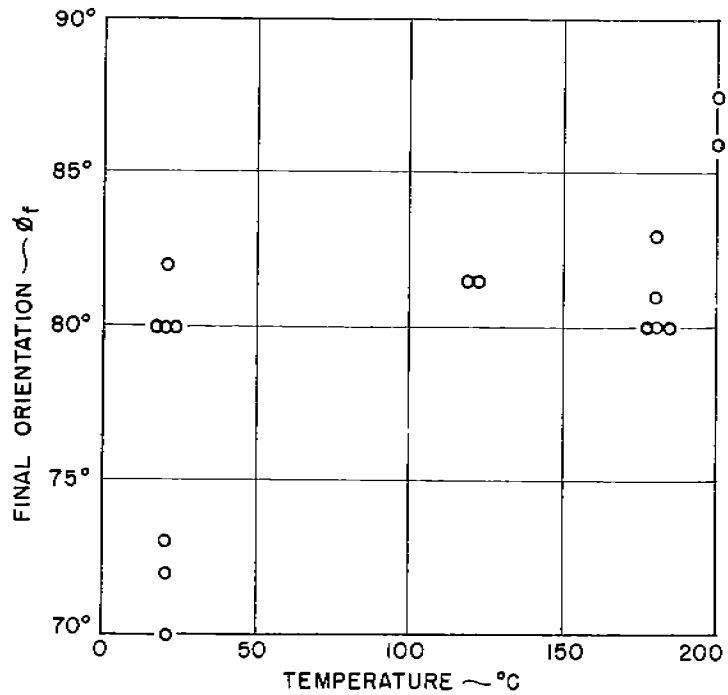


FIG. 3: EFFECT OF TEMPERATURE ON THE ORIENTATION JUST BEFORE TWINNING (1)

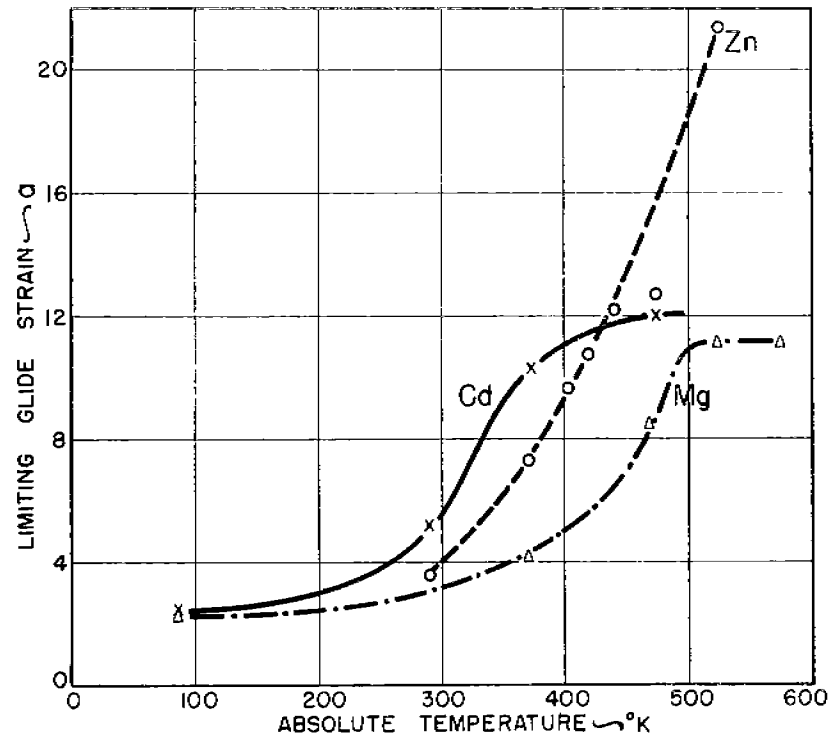
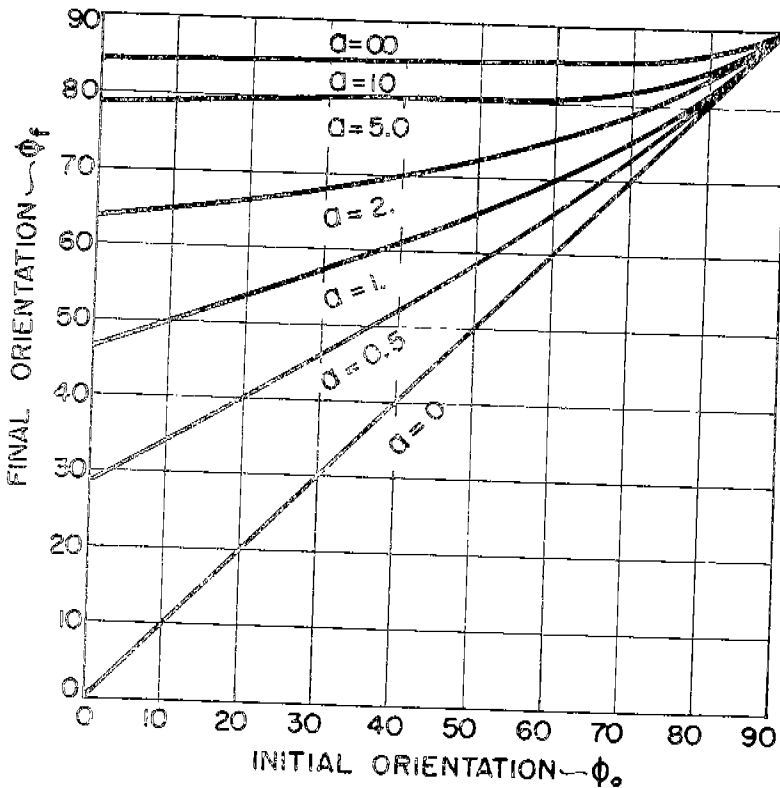
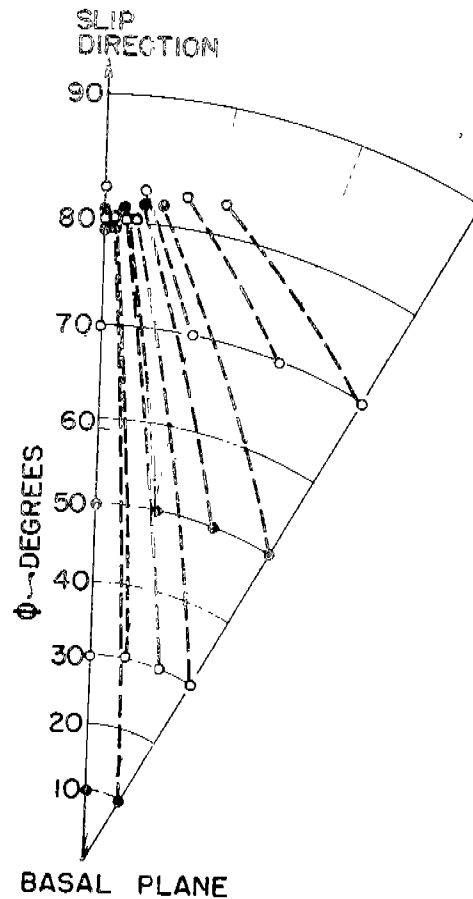


FIG. 4: EFFECT OF TEMPERATURE ON THE TOTAL SHEAR, α BEFORE TWINNING OCCURS (6).



(a)



(b)

FIG. 5: (a) THIS GRAPH SHOWS THE ORIENTATION ϕ_f AT WHICH PRIMARY SLIP IS TERMINATED BY TWINNING FOR ANY STARTING ORIENTATION, ϕ_o , PRESUMING THAT THE CRITERION FOR TERMINATION OF PRIMARY SLIP IS A CONSTANT VALUE OF RESOLVED SHEAR STRAIN, α . FOR HIGH VALUES OF α THE VALUE OF ϕ_f IS ALMOST CONSTANT FOR ANY VALUE OF ϕ_o . LESS THAN 70 TO 75°, THE RANGE OVER WHICH THE CRITERION IS SUPPOSED TO HOLD. FOR $\alpha = 5$, FOR EXAMPLE, ϕ_f RUNS FROM $\sim 79^\circ$ WHEN ϕ_o IS 0 UP TO 82° WHEN ϕ_o IS 70° . THIS SMALL DIFFERENCE IS ABOUT EQUAL TO THE ACCURACY OF ORIENTATION DETERMINATION WHENCE IT IS EASY TO SEE THE DIFFICULTY IN DECIDING WHETHER TWINNING OCCURS AT A CONSTANT VALUE OF ϕ_f UNDER THESE CONDITIONS, OR A CONSTANT VALUE OF α .

(b) THIS IS A STEREOGRAPHIC PLOT OF THE FINAL ORIENTATIONS OF CRYSTALS JUST BEFORE TWINNING OF CRYSTALS WITH VARIOUS INITIAL ORIENTATIONS ASSUMING THAT THE CRITERION FOR TERMINATION OF PRIMARY SLIP IS A RESOLVED SHEAR STRAIN OF 5.

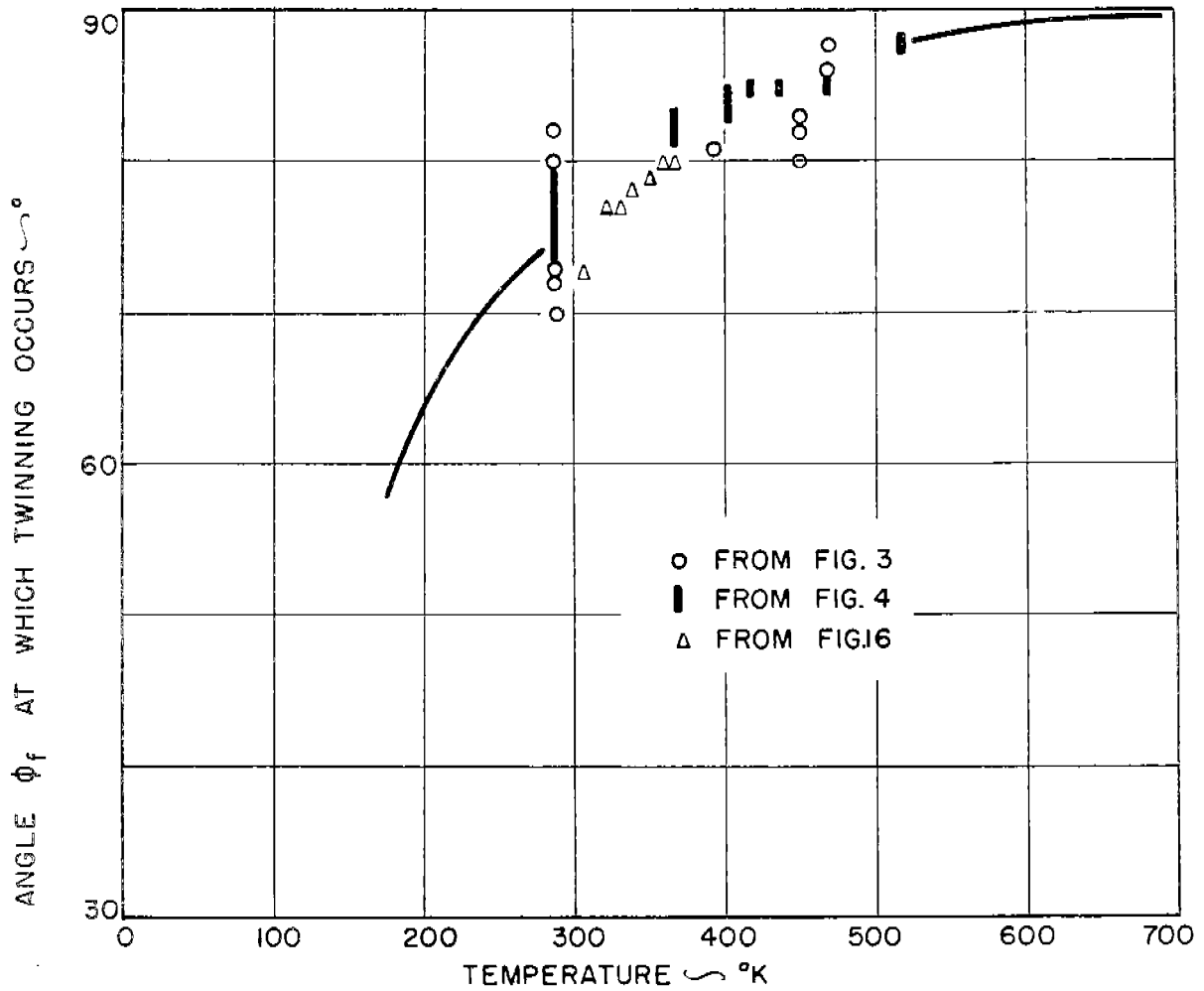


FIG. 6: THIS GRAPH IS A COMPARISON OF THE REPORTED VALUES OF ORIENTATIONS AT WHICH TWINNING TERMINATES PRIMARY SLIP. THE CIRCLES ARE RETAINED DIRECTLY FROM FIGURE 3. THE BARS REPRESENT THE RANGE OF VALUES OF ϕ_f FOR ALL VALUES OF ϕ_o FROM 0 TO 70° FOR THE VALUES OF \underline{a} GIVEN IN FIGURE 4, THE CONVERSION BEING MADE BY MEANS OF FIGURE 5. THE TRIANGLES ARE VALUES OBTAINED IN THE PRESENT REPORT (cf. FIG. 16).

twinning only when $\phi_0 > 85^\circ$, i.e., crystals which were originally in the condition of Fig. 2(c) twinned without primary slip.

The Fracture of Zinc Single Crystals

At temperatures of 18°C and over, zinc is ductile; secondary slip within the twinned lamella "leads to a marked necking of the crystal ribbon and to final rupture" (Fig. 2(d))⁽⁶⁾.

At temperatures of -80°C and lower, Fahrenhorst and Schmid^(14,6) found that single crystals of zinc fractured by cleavage. For low values of ϕ_0 , cleavage was on the basal plane at a critical value of normal stress on that plane (Sohnke's law). The amount of shear preceding fracture was of course no longer constant, but it is of interest that one crystal underwent a total shear of 200 per cent at -185°C . At that temperature the normal stress for fracture was only slightly dependent on the amount of prior slip (i.e., on the initial orientation). At -82°C the critical normal stress was raised by prior slip, and at -253°C it was lowered, but in neither case was the effect very great, and there was considerable scatter in the data. The mean value of the normal stress at cleavage was about

200 gm. per sq. mm. For crystals with high values of ϕ_0 cleavage occurred only after twinning had taken place*^(1,4) and the critical normal stress for cleavage was considerably greater than for simple basal cleavage, being about 1800 gm. per sq. mm. The fracture stress for this type of cleavage at -185°C was very little affected by prior slip at the same temperature⁽¹⁴⁾. On the other hand it was strongly affected by prior slip at room temperature; the critical normal stress rose approximately proportionally to the amount of prior elongation, and an elongation of 500 per cent at room temperature increased the normal stress for cleavage at -185°C by a factor of about 3.7. These are the only quantitative data published on the cleavage of zinc of which the authors are aware.

Since apparently all crystals tested at 100°C parted after localized necking whereas at -80°C they failed by cleavage, it is evident that a transition temperature exists. Schmid and Boas⁽⁶⁾ state that at 20°C zinc crystals did not "as a rule" fracture by cleavage, so that the transition temperature (as evidenced by a change in the type of fracture) is presumably near but below room temperature.

*The early workers described the fracture as prismatic cleavage, but it has since been shown by Mathewson and Philips to be in fact cleavage on the basal plane in the twinned crystal⁽¹⁵⁾.

MATERIAL AND PROCEDURE

The zinc was three-quarter inch extruded rod from the National Lead Company. The nominal purity was 99.99% Zn.

A polycrystalline zinc specimen of the shape required* for the single crystal was used as the pattern in an investment mold, which was heated at 1000°F until the zinc melted. It was then removed from the furnace and directional cooling was produced by a quenched copper rod acting as chill. A single crystal nucleated at the point of contact of zinc with copper, and grew into the rest of the melt.

The crystal was given an acid polish in a 10 per cent solution of HNO_3 and then etched for five to ten seconds in a 50-50 solution of HCl. Any grain boundaries were clearly revealed. The crystals were made in batches of eight to twelve, and the yield varied, but an average of about 80% of the investments gave good single crystals.

*Preliminary experiments had shown that the manner in which the specimen was gripped was of pronounced influence on the ductility of the specimen, fracture frequently being caused by and located at the orientation difference at the juncture of the free test length (which had rotated during testing) and the shoulder or head of the specimen (which had not rotated). The double shoulder on the specimen shapes finally adopted (Fig. 7) placed a reasonably reproducible restraint on the ends of the free test length, and at the same time insured fracture at a point not concealed by the grip.

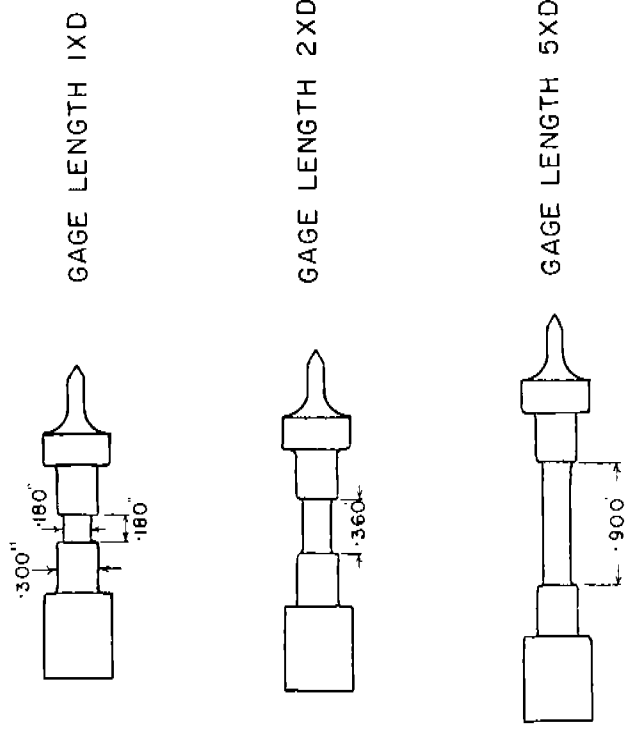


FIG. 7: SHAPE OF SINGLE CRYSTAL SPECIMENS.

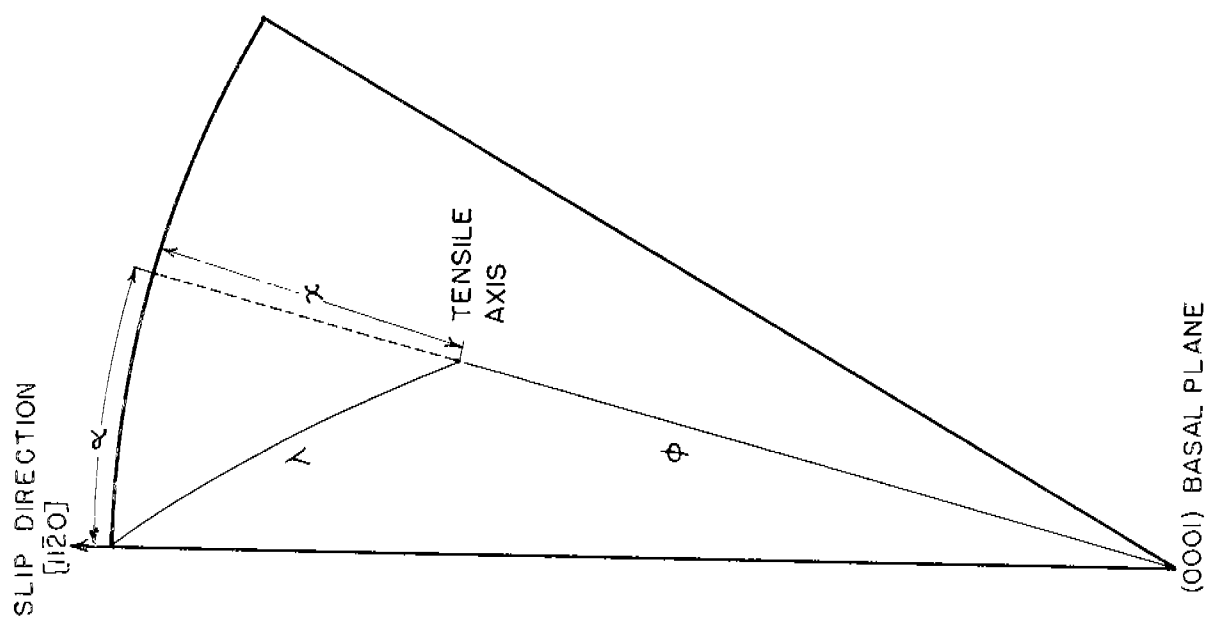


FIG. 8: STANDARD PROJECTION SHOWING ANGLES USED IN ORIENTATION DETERMINATIONS.

The orientation of each crystal was determined by means of an X-ray back-reflection photograph using the Greninger method.

From the measurement of the film, three angles representing the orientation of a crystal were determined. These were ϕ , λ , and α as shown in Fig. 8. (The λ used here is the same as that used by Schmid and Boas⁽⁶⁾. The angle, χ , which they used is $90^\circ - \phi$, and their angle k is the same as α .)

Extreme care was required in the handling of the crystals as some even deformed under their own weight at room temperature. Damage during handling was revealed by an overall bending of the crystal or by blurred spots on the Laue photograph. If an overall bend was observed, the crystal was not tested.

Handling of the crystal was reduced to a minimum by inserting the specimen in the test rig before putting the rig into the temperature bath. The temperature medium used was water from 5°C to 95°C and a transparent mineral oil at 105° and 115°C . The different environment appeared to have no effect on fracture properties. Thus the crystal could be observed directly throughout the test.

Although the rate of movement of the tensile machine head was kept constant at about 0.2 in. per min., the rate of straining was different for the three series (because of their different free length) and also changed as soon as slip

became localized by necking or by the opening of a cleavage crack. The rate of shear, resolved in the slip direction on the slip plane, was of course different for different orientations.

RESULTS AND DISCUSSION

A comprehensive presentation of the dependence of ductility (as measured by reduction in area) on temperature and orientation is given in Fig. 9. Each of the surfaces in this model are discussed below:

The Ductile Plateau (Surface A in Fig. 9)

In the case of specimens tested at 95°C, for example, all crystals showed 100 per cent contraction in area irrespective of their starting orientation, although the elongation values were sharply dependent upon this variable (cf. Fig. 10(h)).* This behavior is a result of the various processes by which zinc deforms from its initial orientation.

In the region of $\phi_0 = 80$ to 90° , specimens twinned with little or no preceding slip, and twinning and secondary slip (slip within the twinned region) led to a highly necked specimen. Fig. 13 shows such a specimen and illustrates the low elongation and high contraction in area typical of crystals with this starting orientation.

*In no case was any consistent variation of ductility with α observed. Therefore in most of the following discussion orientation will be treated as if it were governed by ϕ_0 alone.

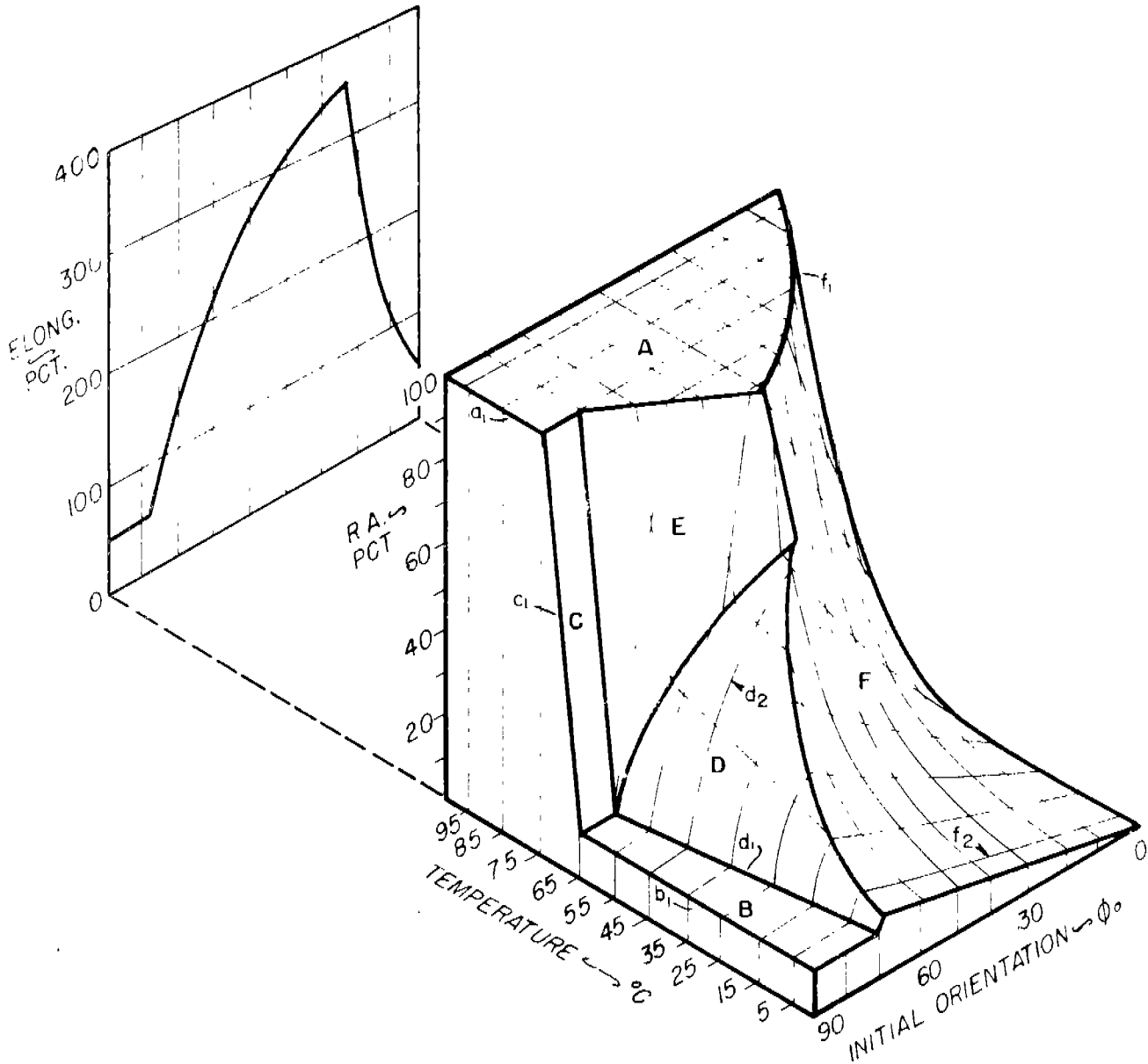


FIG. 9: SIMPLIFIED REPRESENTATION OF DUCTILITY (REDUCTION IN AREA) AS A FUNCTION OF ORIENTATION AND TEMPERATURE FOR 5D SPECIMENS. EACH SURFACE A,B...F, IS DISCUSSED IN DETAIL IN THE TEXT. SECTION a_1 , c_1 , b_1 , IS TAKEN FROM FIG. 24, LINE d_1 FROM FIG. 6, LINES d_2 FROM FIG. 10, LINES f_2 FROM FIG. 27.

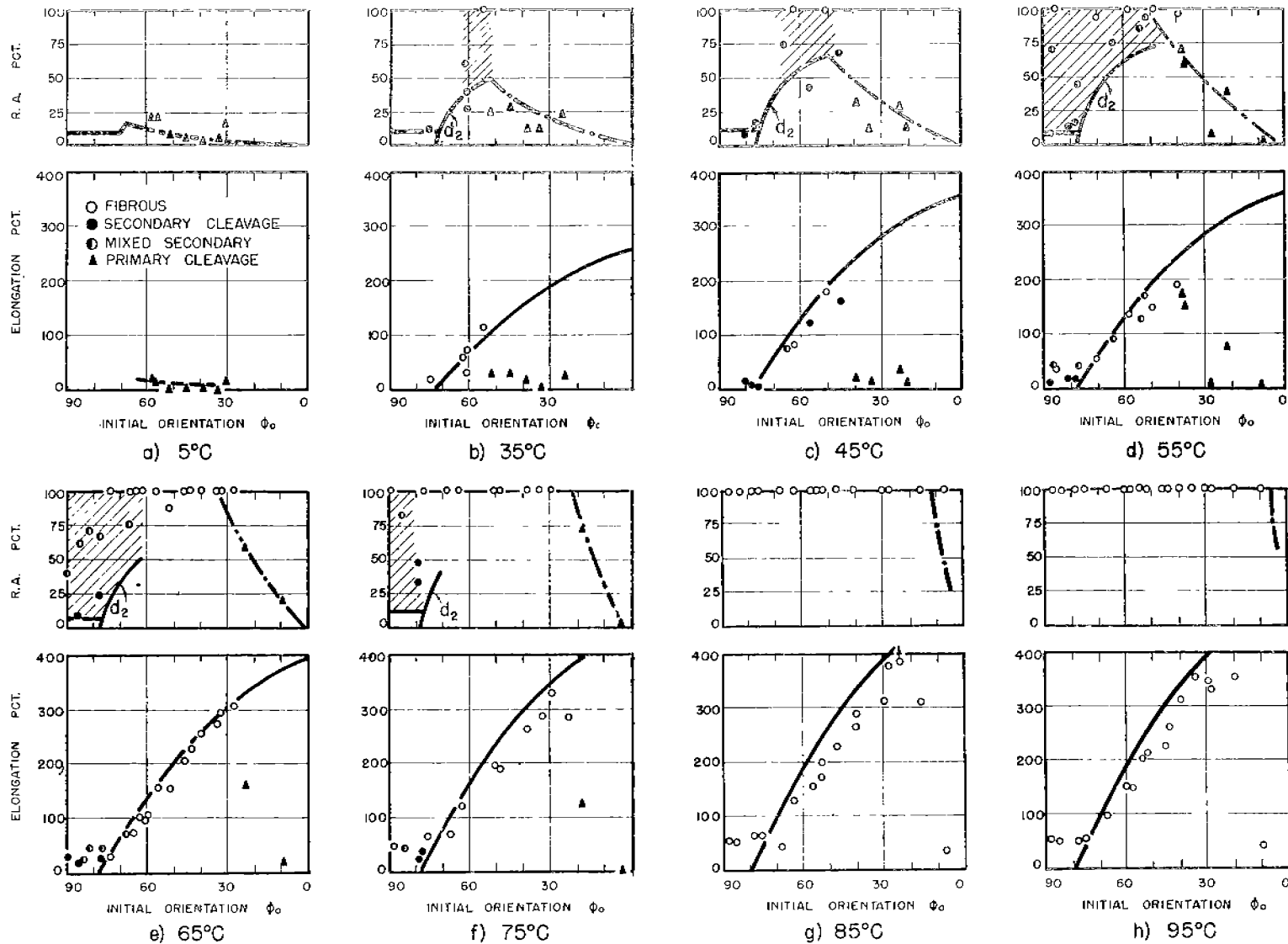


FIG. 10: DUCTILITY (REDUCTION IN AREA AND ELONGATION) AS A FUNCTION OF INITIAL ORIENTATION, ϕ_0 , AT THE TEMPERATURES INDICATED FOR 5D SPECIMENS. SOLID LINES REPRESENT IDEAL DUCTILITIES AS COMPUTED BY EQUATIONS (2 OR 3) OF THE TEXT. IN THE CASE OF ELONGATIONS DATA CONFORM TO THESE CURVES WHETHER FRACTURE WAS BRITTLE OR DUCTILE SINCE LOCALIZED NECK OF DUCTILE FRACTURE CONTRIBUTED LITTLE TO ELONGATION VALUES. IN THE CASE OF REDUCTION IN AREA ONLY THE BRITTLE FRACTURES CONFORM TO THESE CURVES, THE DUCTILE FRACTURES FALLING ABOVE THE CURVE TO AS HIGH AS 100%. SHADE AREAS REPRESENT TRANSITION TEMPERATURE ZONE WHERE FRACTURES MAY BE EITHER DUCTILE OR BRITTLE AND REDUCTION IN AREA VALUES WILL FALL BETWEEN THESE TWO LIMITS. DOT-DASH CURVES ARE TAKEN FROM FIG 27, REPRESENT PRIMARY CLEAVAGE.]

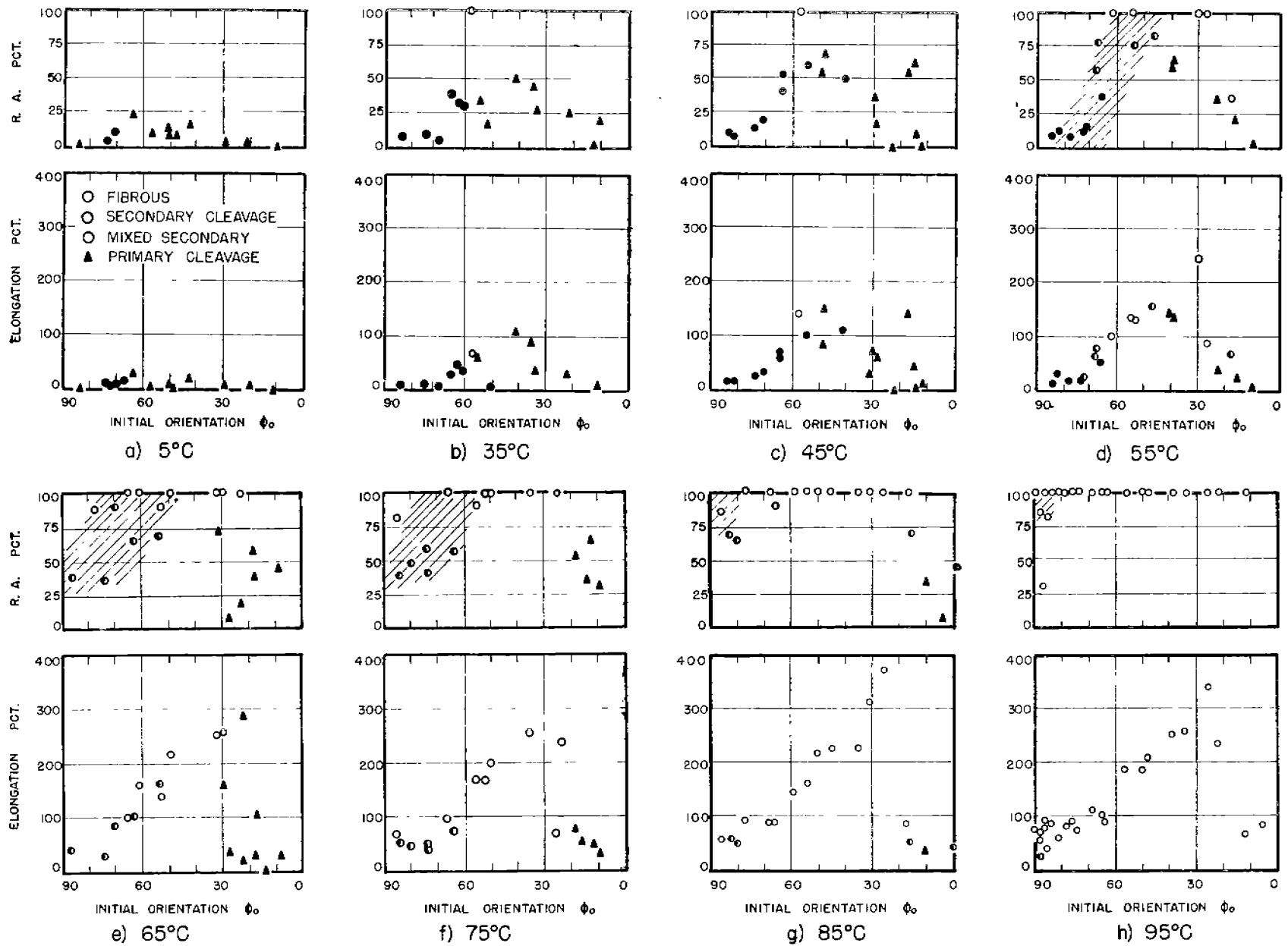


FIG. II: DUCTILITY (REDUCTION IN AREA AND ELONGATION) AS A FUNCTION OF INITIAL ORIENTATION, ϕ_0 , AT THE TEMPERATURES INDICATED FOR 2D SPECIMENS.

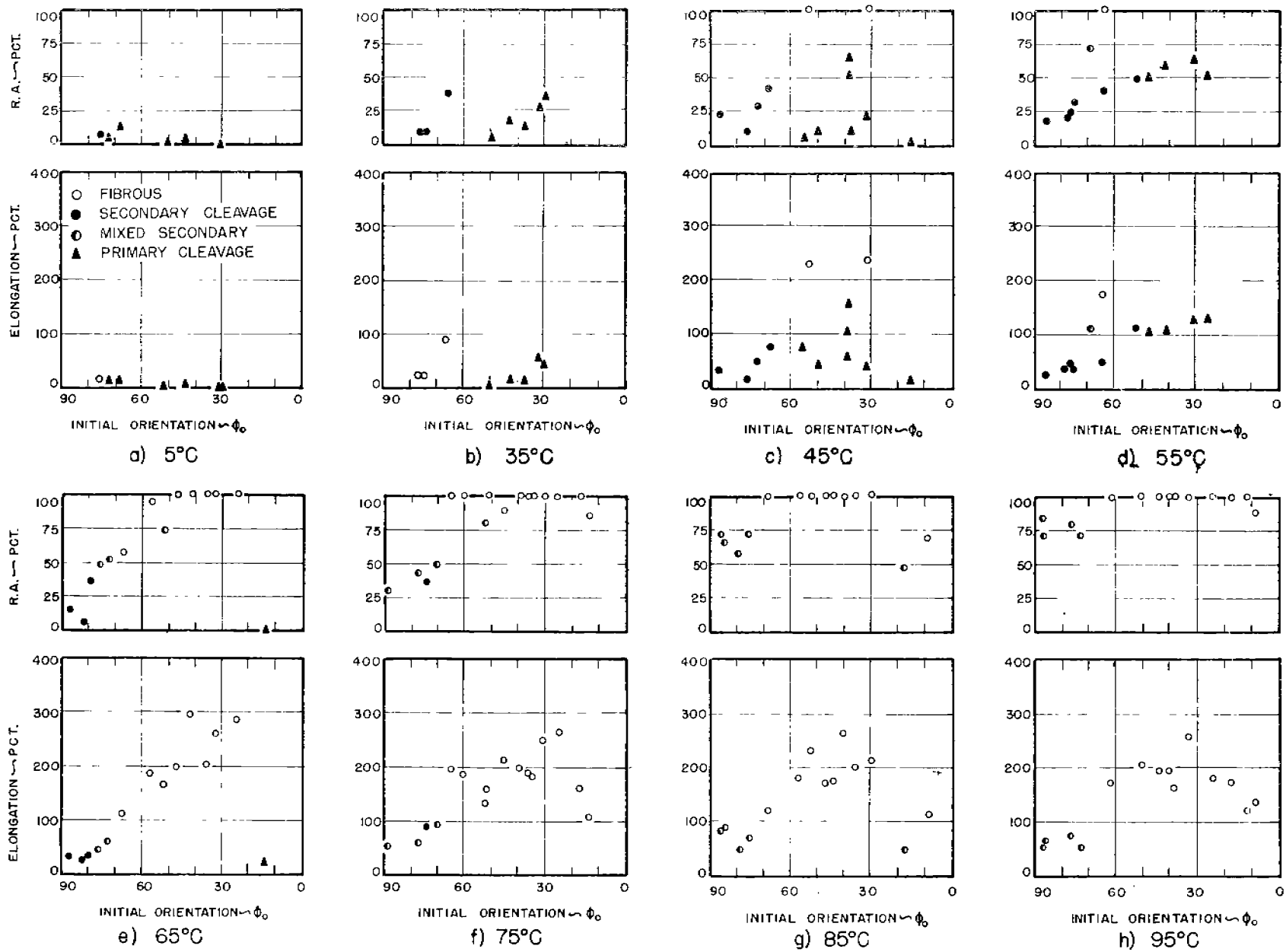


FIG. 12: DUCTILITY (REDUCTION IN AREA AND ELONGATION) AS A FUNCTION OF INITIAL ORIENTATION, ϕ_0 , AT THE TEMPERATURES INDICATED FOR ID SPECIMENS.

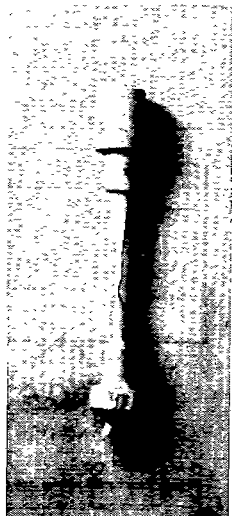


FIG. 13: PHOTOGRAPH OF ZINC SINGLE CRYSTAL WITH $\phi_0 > 80^\circ$ TESTED AT 95°C SHOWING LOW ELONGATION BUT HIGH CONTRACTION IN AREA DUE TO LOCALIZED NECKING RESULTING FROM TWINNING AND SECONDARY SLIP.

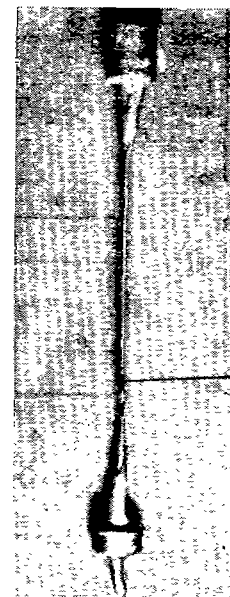


FIG. 14: PHOTOGRAPH OF ZINC SINGLE CRYSTAL WITH $\phi_0 = 44^\circ$, TESTED AT 95°C SHOWING HIGH ELONGATION AND HIGH CONTRACTION IN AREA DUE TO OVERALL PRIMARY SLIP FOLLOWED BY LOCALIZED NECKING RESULTING FROM TWINNING AND SECONDARY SLIP. MINOR AXIS OF ELLIPSE AT A WAS USED TO DETERMINE ϕ FOR FIG. 16.

The portion of crystal next to the fracture had a fibrous appearance, as did all fractures involving very high contraction in area. This portion was observed to recrystallize at room temperature, but although the precise mechanism of parting is not known, it is believed that recrystallization did not occur until after the fracture process was complete.

Crystals with starting orientation about 30° to 80° deformed first by primary slip and thereby elongated. Twin bands finally appeared and secondary slip within the twin band led immediately to necking and rupture (cf. Fig. 14). The contribution which the necked band made toward the elongation was minor and elongation data therefore may be taken as a first approximation to decide what is the proper criterion for the termination of primary slip. The shear strain, \underline{a} , resolved on the slip plane, and the final orientation ϕ_f , corresponding to these elongation values, δ , were computed using equations 26/3 and 26/2, respectively, of Ref. 6,

$$\underline{a} = \frac{1}{\cos \phi_0} \sqrt{(1 + \delta)^2 - \sin^2 \lambda_0} - \cos \lambda_0 \quad (1)$$

$$\cos \phi_f = \frac{\cos \phi_0}{1 + \delta} \quad (2)$$

and plotted in Fig. 15(a) and (b). The data clearly favor

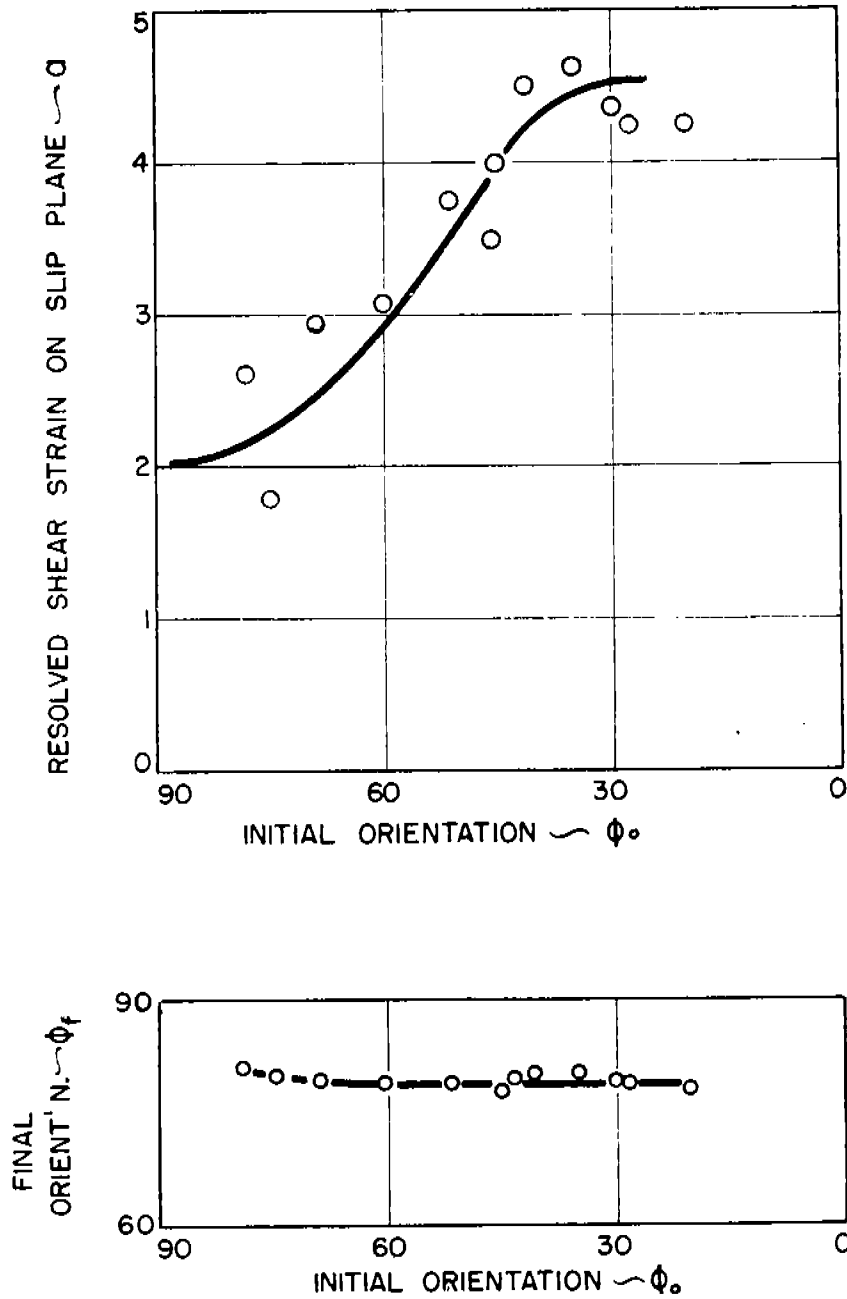


FIG. 15: —a) RESOLVED SHEAR STRAIN ON SLIP PLANE, α , CORRESPONDING TO THE ELONGATION VALUES GIVEN IN FIG. 10h AS COMPUTED BY EQUATION [1] OF THE TEXT.
b) FINAL ORIENTATION, ϕ , COMPUTED FROM SAME ELONGATION VALUES BY EQUATION [2] OF THE TEXT.

the constancy-of-final-orientation criterion. If that small portion of the elongation due to secondary slip and necking were subtracted from the total elongation values that were used, the decision would be even more clear cut, for the values of \underline{a} would drop considerably in the range of $60 < \phi < 80^\circ$, whereas the values of ϕ_f would drop only a degree or two at high values of ϕ_o . All other elongation data reported in this paper give plots similar to Fig. 15, but are not reproduced here in the interest of economy.*

A more precise determination of the value of ϕ_f can be made from the ratio of the cross-sectional area when primary slip ceases, a_f , to the initial cross-sectional area, a_o , through the equation:

$$\frac{a_o}{a_f} = \frac{\cos \phi_o}{\cos \phi_f} \quad (3)$$

*The choice between the two criteria considered here is of more importance than merely the choice between two criteria that in the manner in which they are observed are closely similar. The implication of a constancy-of-slip criterion is that twinning is set by a single standard: at a constant value of resolved shear strain or relatedly (because of the invariance of the resolved shear stress-resolved shear strain diagram) at a constant value of resolved shear stress or at a constant value of deformational energy (cf. Ref. 16 and 17). The constancy-of-final orientation implies that twinning is set by a comparative standard, that the ratio of stresses on two different deformation systems is critical or is brought into a critical state by virtue of the particular final orientation (cf. Ref. 18).

(which follows directly from equation (2) under the assumption of constancy of volume). In the present case in place of the ratio of areas, the minor axis of the elliptical section of the uniformly deformed portion adjacent to the necked twin band was divided by the original diameter. Although this is not strictly true unless $\alpha = 0$, the error involved was less than half a degree in the great majority of cases and in no case exceeded two degrees. The values of ϕ_f so determined are plotted on a standard stereographic projection in Fig. 16, where it has been assumed that the tensile axis rotated along a great circle as demanded by simple slip.

The shorter crystals--2D and 1D gage lengths--of this orientation range, $\phi_o = 30 - 80^\circ$, behaved in a similar way to the longer ones just described. They deformed by primary slip up to a constant final angle ϕ_f . Here the reduction due to necking however occupied a greater proportion of the gage-length and it was difficult to determine the amount of deformation due to slip alone, but measurements were taken and the values of ϕ_f calculated were slightly lower for the 2D and 1D crystals than for the 5D crystals.

In the range of initial orientations $\phi_o = 0$ to 30° , crystals with gage-length five times their diameter deformed by primary slip up to a constant angle ϕ_f of 80° , twinned

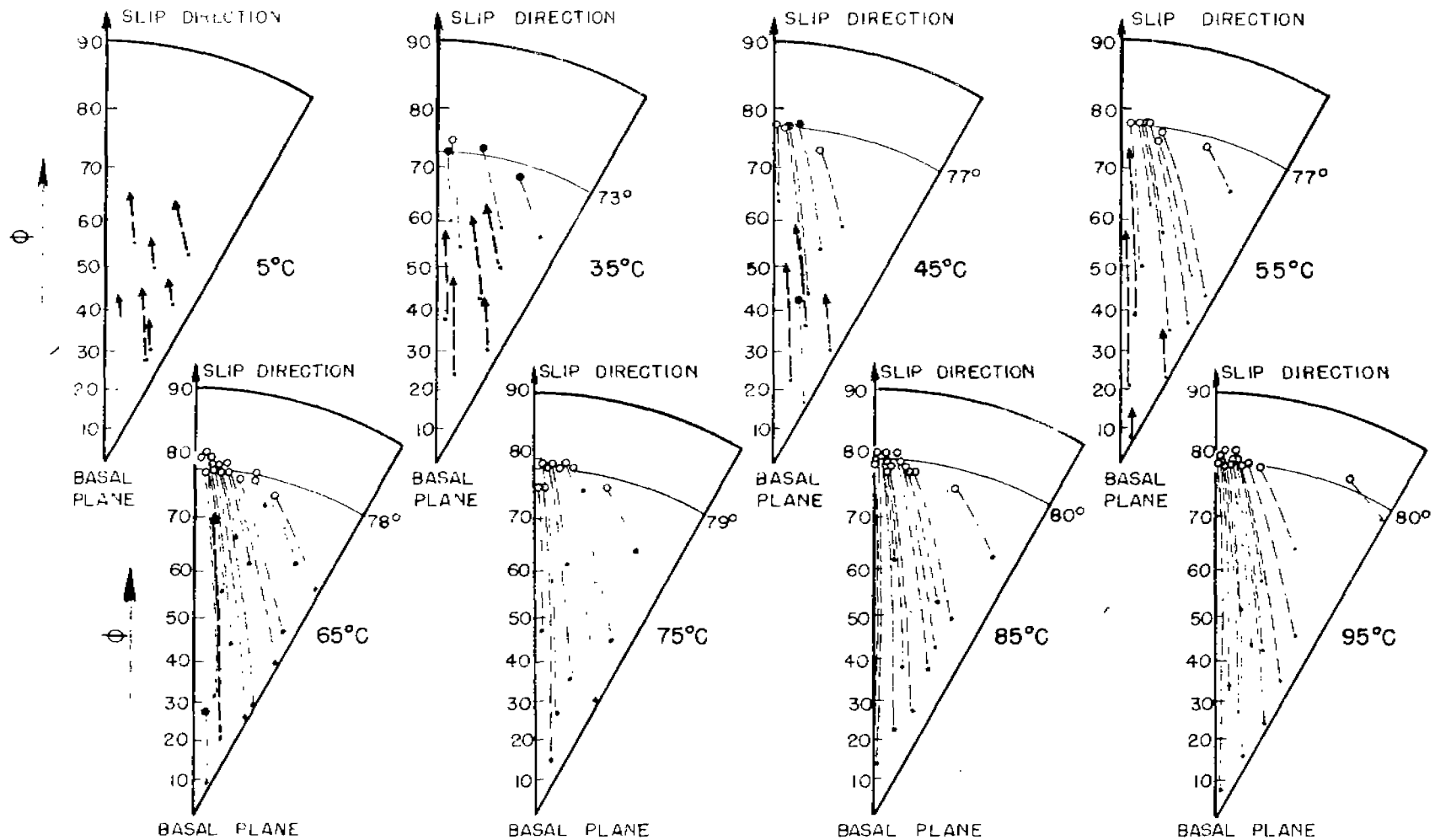


FIG.16: INITIAL AND FINAL ORIENTATIONS OF 5D SPECIMENS PULLED AT THE TEMPERATURES INDICATED. STARTING ORIENTATIONS ARE GIVEN BY THE DOTS. FINAL ORIENTATIONS GIVEN BY OPEN CIRCLES INDICATE SPECIMEN TWINNED AND NECKED TO GIVE DUCTILE FRACTURE. MINOR AXIS OF CROSS-SECTIONAL ELLIPSE OF SPECIMEN NEXT TO TWIN (AS AT POINT A IN FIG. 14) WAS USED IN EQUATION (3) OF TEXT TO DETERMINE FINAL ORIENTATION. FINAL ORIENTATIONS GIVEN BY SOLID CIRCLES INDICATE SPECIMEN TWINNED AND CLEAVED AND HERE MINOR AXIS OF CROSS-SECTIONAL ELLIPSE NEXT TO FRACTURE WAS USED TO DETERMINE FINAL ORIENTATION. FINAL ORIENTATIONS GIVEN BY SOLID TRIANGLE INDICATE SPECIMEN FAILED BY PRIMARY CLEAVAGE. MINOR AXIS OF CROSS-SECTION ELLIPSE WAS TAKEN MIDWAY ALONG TEST LENGTH.

and failed by fibrous fracture with 100 per cent reduction in area. However in these crystals the deformation was usually localized (see Fig. 17) to a quite short length of the crystal.

In the shorter crystals, the necked region again occupied a greater portion of the gage-length. In addition one or two crystals failed by an unusual "double-cup" fracture. Apparently, the crystal first separated on the basal plane near the core of the test section, leaving a hollow cylinder of material around it which drew apart in an almost viscous manner. Indeed the surface of the basal plane, visible at the bottom of the cup, had a remarkable matte, viscous-like appearance, quite unlike the familiar basal cleavage.

The operation of primary slip in all crystals with ϕ_0 less than 80° led to an orientation gradient near the shoulder and this was accommodated in every case by kinks of the type shown in Fig. 18(b). This was particularly striking for crystals with low values of ϕ_0 and gage length lD . In these crystals a kink band could not form immediately since its plane would be almost parallel to the tensile axis and would not traverse the test section. However, in practice the grip supports were not entirely rigid, and some lateral movement was possible. Consequently deformation commenced by shear along the slip-plane and the grips

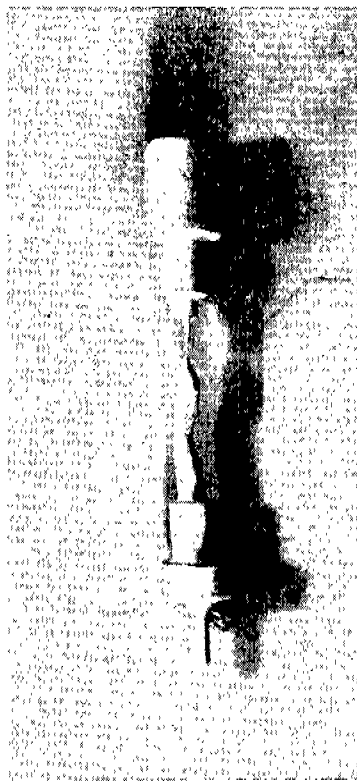


FIG. 17: PHOTOGRAPH OF A ZINC SINGLE CRYSTAL WITH $\phi_0 = 10^\circ$ TESTED AT 85°C SHOWING LOW ELONGATION BUT HIGH CONTRACTION IN AREA DUE TO LOCALIZED PRIMARY SLIP FOLLOWED BY LOCALIZED NECKING RESULTING FROM TWINNING AND SECONDARY SLIP.

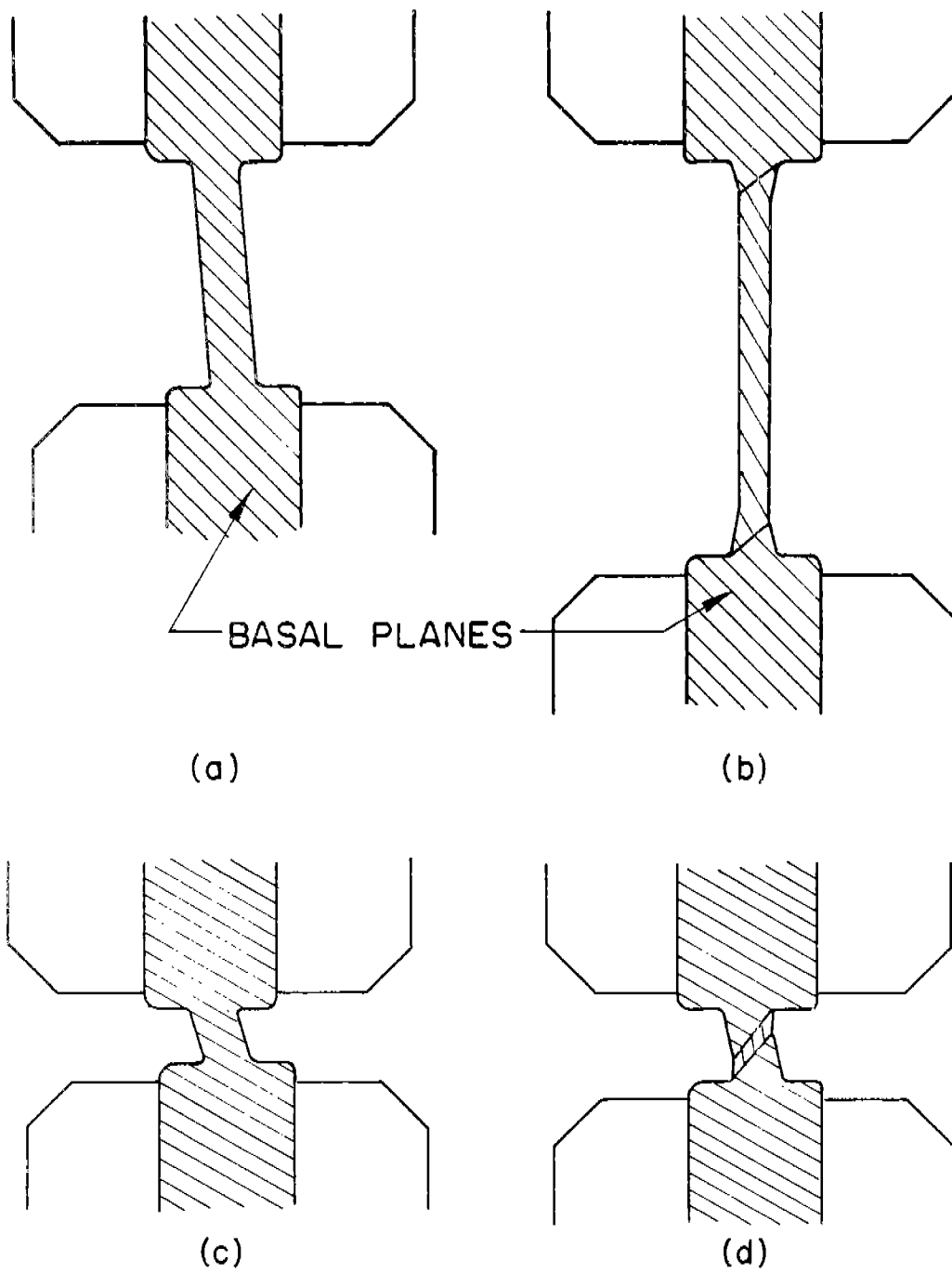


FIG. 18: (a, b) FORMATION OF A KINK BAND IN A LONG CRYSTAL.
(c, d) FORMATION OF A KINK BAND IN A SHORT CRYSTAL WITH LOW ORIENTATION ANGLE, θ_0 .

became offset by elastic bending of the central steel guide-rods, as shown in Fig. 18(c). However this imposed a bending moment on the crystal tending to straighten it, so that after a certain amount of shear the crystal straightened rather suddenly with the formation of a kink-band, see Fig. 18(d). This released the elastic energy in the guide-rods and shear within the kink-band took place rapidly, leading to fibrous fracture. It is important to realize that the formation of such a narrow kink-band gave rise to an overall movement of the crystal which was tantamount to slip on a plane at right angles to the basal plane. However no slip lines were observed on that plane, and the mechanism was almost certainly simply localized basal slip.

During deformation of the shorter crystals (1D and 2D), longitudinal ridges near the shoulders were seen as shown in Fig. 19. Fig. 20 shows diagrammatically the slip lines visible before necking commenced. The upper set of ridges are those shown in Fig. 19. This specimen was cleaved by inserting a razor blade parallel to the slip planes at the temperature of liquid nitrogen, and the sections obtained are shown in Fig. 21. It is seen that the ridges produced a number of "gable roofs" on these slip planes.

These ridges apparently form as a consequence of the following chain of events. Deformation takes place largely

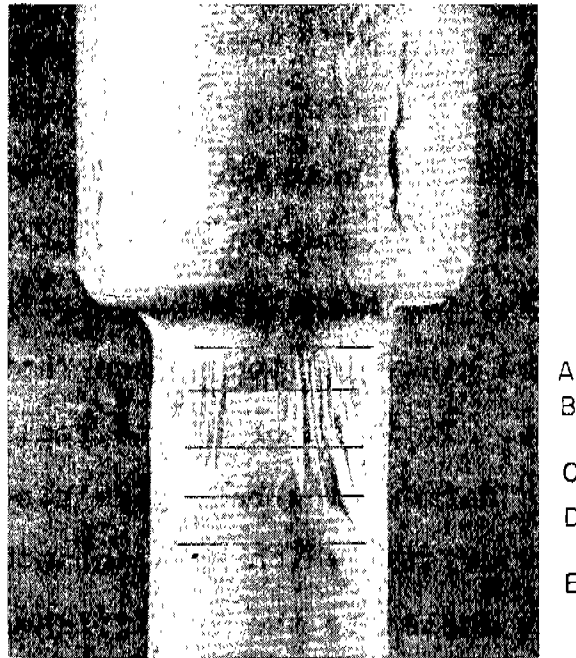


FIG. 19: RIDGES FORMED AT HIGH TEMPERATURE ON SECOND SPECIMEN WITH LOW ORIENTATION ANGLE $\phi_0 = 25^\circ$, WHEN PULLED AT 75°C . MAGNIFICATION ABOUT 7X. LETTERS a,b...e REPRESENT THE SECTIONS SHOWN IN FIG. 21 (a-e).

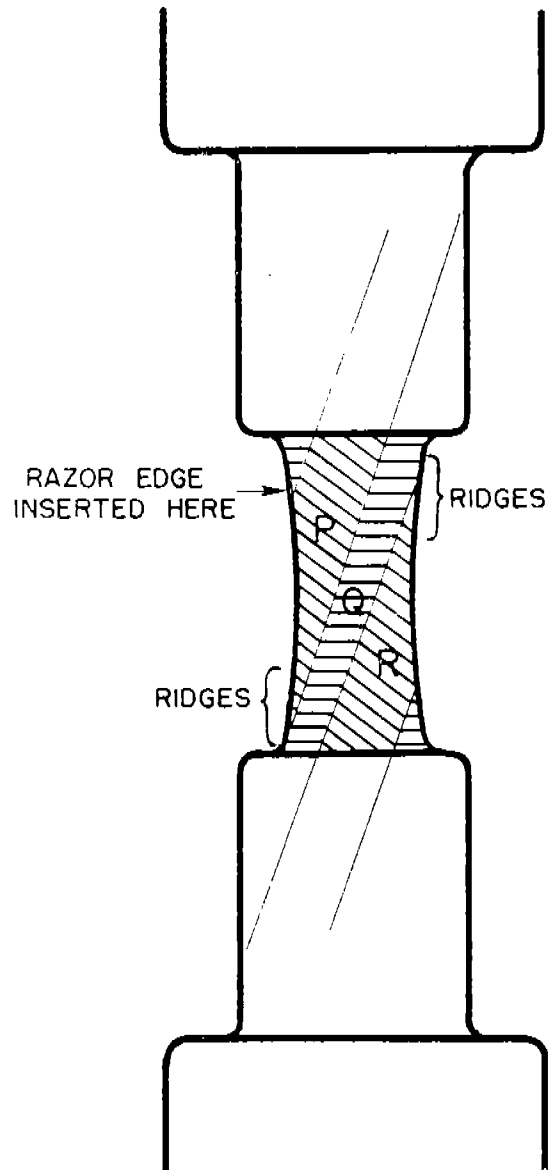


FIG. 20: SCHEMATIC DRAWING OF SLIP LINES VISIBLE ON SPECIMEN SHOWN IN FIG. 19 BEFORE NECKING TO FRACTURE. UPPER SET OF RIDGES ARE RIDGES VISIBLE IN FIG. 19. RAZOR WAS INSERTED PARALLEL TO SLIP LINES TO GIVE SECTIONS SHOWN IN FIG. 21.

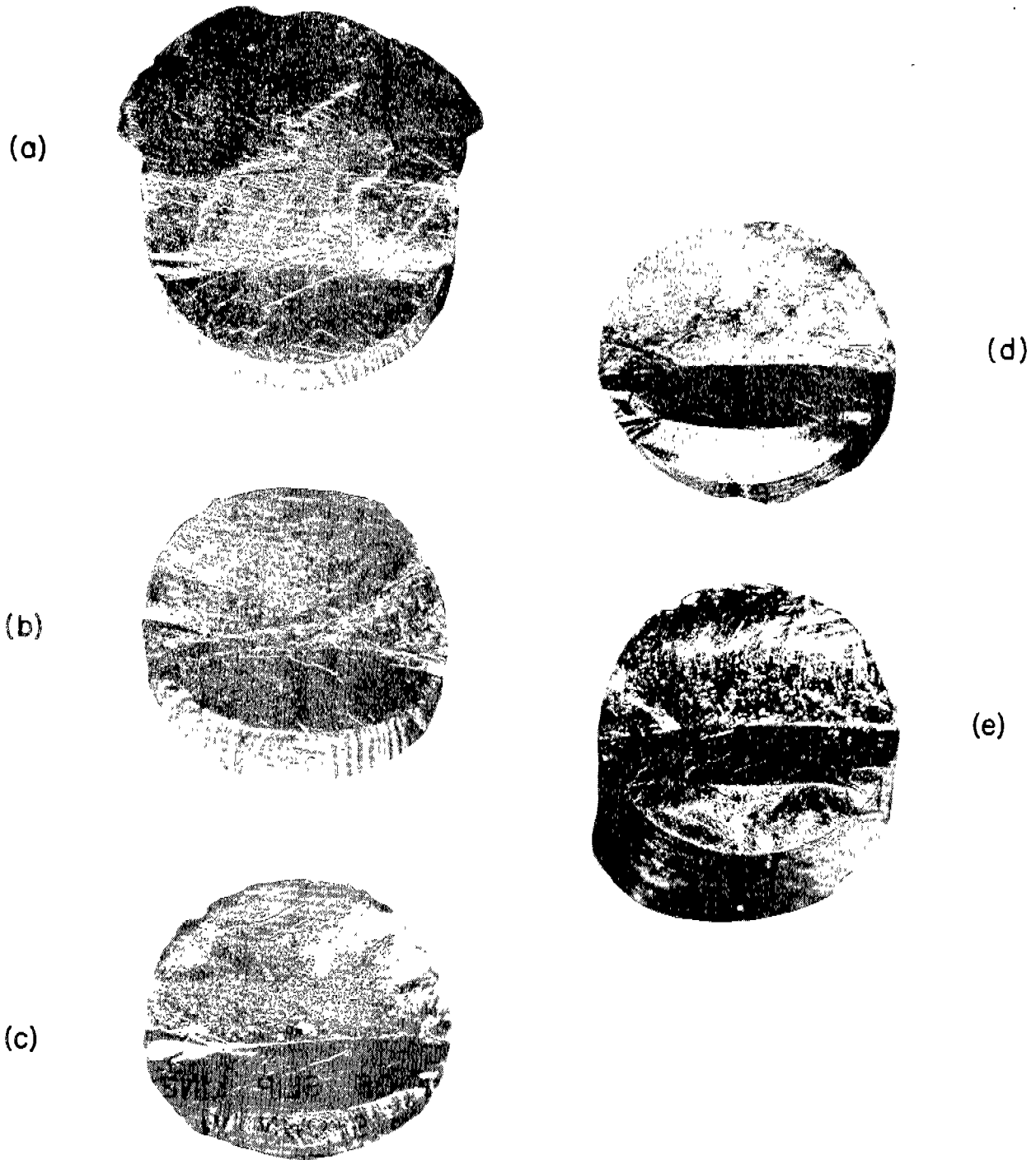


FIG. 21: CLEAVED SECTIONS THROUGH RIDGED REGION OF SPECIMEN SHOWN IN FIG. 19.

in the two band-shaped regions, P and R in Fig. 20. Prior to the ridge formation a section near the top of the deformed region is as in Fig. 22(a). The plane ABCD is a kink-band boundary or kink-plane. If it assumes the position of lowest energy it bisects the angle between the slip-planes on either side of it⁽⁸⁾. As further slip takes place, the kink-plane tends to rotate in a clockwise direction (Figs. 20 and 22). The appearance of the sections in Fig. 21 seems to indicate that this movement is resisted at the surface of the crystal; the points A and C are effectively anchored*, so that the movement of B which is comparatively free gives rise to a curved kink-plane as shown in Figs. 21(b), (c) and in Fig. 22(b). But such a curved kink-plane is in a fairly high energy state, so that B will also reach a limit of movement dependent on the magnitude of the force tending to straighten AB'C. Then line B'E' is unable to rotate further (clockwise about the fixed point B') as a result of slip, without shortening the length E'C on the surface of the crystal. This shortening can only be accommodated by means of a buckling process as shown in Fig. 22(c). The fact that ridges were observed only where the kink-plane was curved (cf. Fig. 21) is consistent with this theory. The buckling process need involve only basal slip (though in directions other than that of the primary operative slip). In that case the edge FGH should lie in a

*Hess and Barrett⁽⁸⁾ report a similar effect in the compression of zinc single crystals.

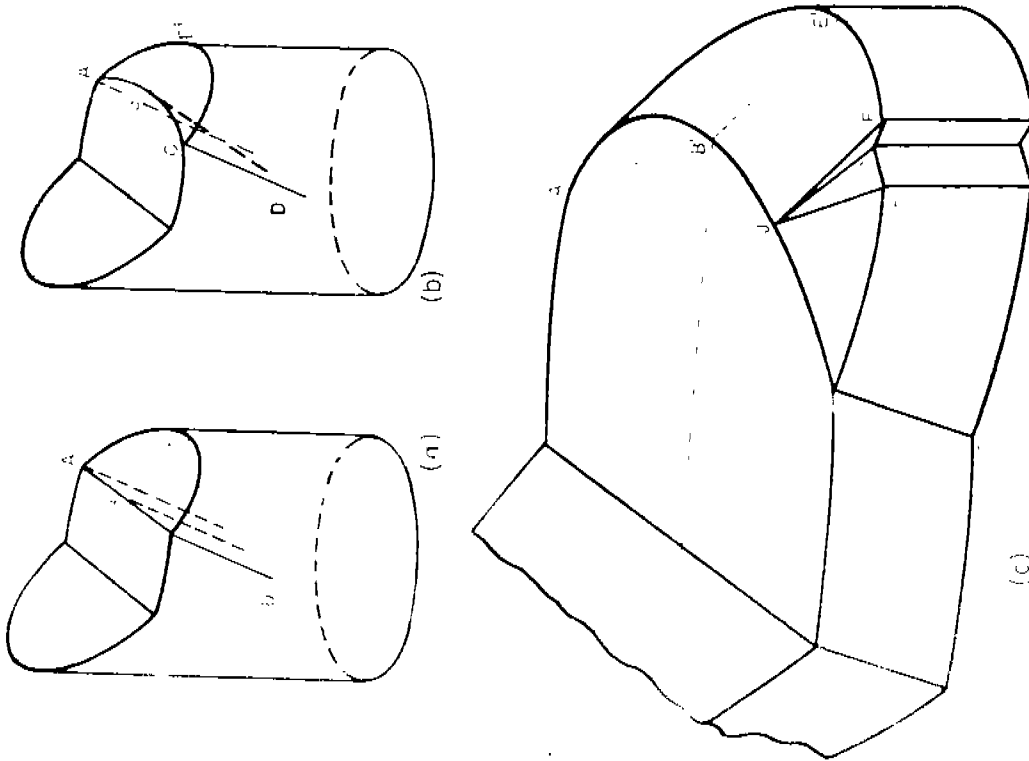


FIG 22: THE FORMATION OF A RIDGE.

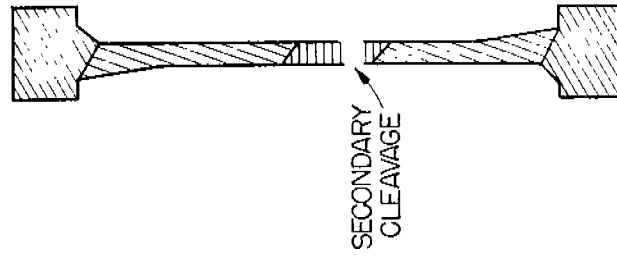


FIG.23: SCHEMATIC CROSS-SECTION OF CRYSTAL THAT FAILED BY SECONDARY CLEAVAGE.

plane perpendicular to the surface FHJ. The ridges in Figs. 21(b) and (c) were examined under a binocular microscope and such appeared to be the case.

The Brittle Floor Due to Secondary Cleavage (Surface B in Fig. 9).

As test temperature was lowered, all of the above phenomena underwent changes. As may be seen in Figs. 10 to 12, the crystals with initial orientation $\phi'_0 = 80$ to 90° became embrittled in the sense that their contraction in area dropped from 100% to about 10%. These crystals, which at higher temperatures necked by twinning and secondary slip, at lower temperatures simply twinned and cleaved on the basal plane of the twinned metal, generally midway along the gage-length (Fig. 23). That the cleavage surface was the basal plane of the twinned metal and not the original crystal could be deduced not only from its orientation, but from the fact that it always showed parallel striations which were in fact shallow steps marking basal cleavage in small interleaving sheets of untwinned crystal. Cleavage in a crystal which has twinned will be designated as "secondary cleavage" as distinguished from "primary" basal cleavage which occurs in untwinned material.

The Transition Cliff (Surface C in Fig. 9)

The cliff (C) in Fig. 9 which denotes a transition between ductile and brittle behavior for crystals with $\phi_0 > 80^\circ$ is a simplification of the facts. From the plots of actual contraction in area data for 5D specimens in Fig. 10 for example, it is seen that secondary cleavage can occur at a temperature as high as 75°C , while in a statistical manner typical of transition behavior in general, ductile fractures can occur at temperatures as low as 55°C . In this temperature range the fracture was often mixed secondary cleavage and fibrous.

Fig. 24 shows the contraction in area as a function of temperature for crystals with $\phi_0 > 80^\circ$. The transition for crystals with 5D gage-length was sharp covering the range 75° to 55°C , whereas for the 2D crystals it was more gradual, and for gage-length 1D even more so, covering the range about 95° to 60°C .

The Brittle Zone D in Fig. 9

In this region of temperature and orientation the crystals would deform by primary slip to a critical terminating orientation, ϕ_f , twin, and then fracture by secondary cleavage, usually near one end of the test section.

The critical orientation, ϕ_f , at which primary slip ended,

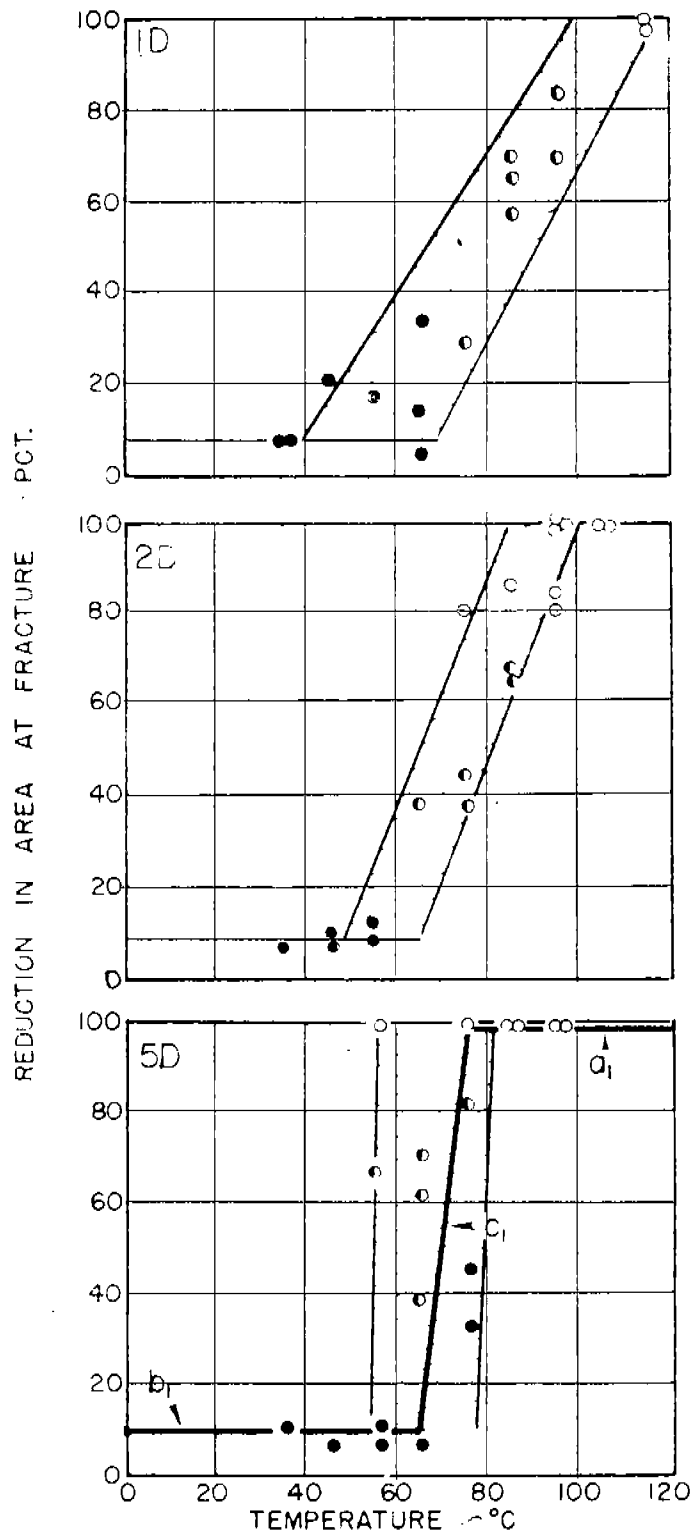


FIG.24: REDUCTION IN AREA AS A FUNCTION OF TEMPERATURE FOR SPECIMENS WITH INITIAL ORIENTATION, $\phi_0 \geq 80^\circ$. THESE GRAPHS ARE CROSS-PLOTS OF THE DATA (FOR SPECIMENS $\phi_0 \geq 80^\circ$) GIVEN IN FIGS. 10,11 AND 12. LINE a_1, c_1, b_1 , DRAWN AS A TREND CURVE THROUGH THE DATA POINTS FOR 5D SPECIMENS IS THE SAME AS SECTION a_1, c_1, b_1 , IN FIG.9. THE SHADED AREAS CORRESPOND TO THE SHADED AREAS AT $\phi_0 \geq 80^\circ$ IN FIGS.10,11, AND 12.

though constant for a given temperature, decreased slightly with decreasing temperature as indicated by the skew course to line d_1 in Fig. 9. The experimentally determined values of ϕ_f are shown in the stereographic projections in Figs. 16 and the mean values which were obtained are plotted as a function of temperature in Fig. 6 together with those of Mark, Polanyi and Schmid⁽¹⁾ and Boas and Schmid⁽⁶⁾.

Since specimens that broke in secondary cleavage showed no necking, equation (3) may be used to relate the reduction in area with initial orientation. Lines "d₂" in Figs. 9 and 10 are computed by this equation using the average values of ϕ_f given in Fig. 16.

Transition Cliff (Surface E of Fig. 9)

- As in the case of crystals with $\phi_o = 80 - 90^\circ$ there was a considerable temperature range where failure could be either ductile or brittle, but in the present case this range was displaced downward as ϕ decreased. This trend can be seen in Fig. 10, for example, where crystals with orientations $\phi_o > 80^\circ$ have started to embrittle at 75°C whereas crystals with orientations $\phi_o < 80^\circ$ are still ductile; and again at 45°C where crystals with high ϕ_o are all brittle, while crystals with lower $\phi_o \approx 60^\circ$ are by and large relatively ductile. This is an interesting point; it means that while a crystal whose orientation, ϕ_o ,

was initially greater than 80° would be brittle at a given temperature, one that rotated by primary slip from a lower value of ϕ_0 into the range $\phi > 80^\circ$ remained ductile. In other words primary slip or some concomitant of primary slip allayed embrittlement due to secondary cleavage.

Primary Cleavage Slope (Surface F of Fig. 9)

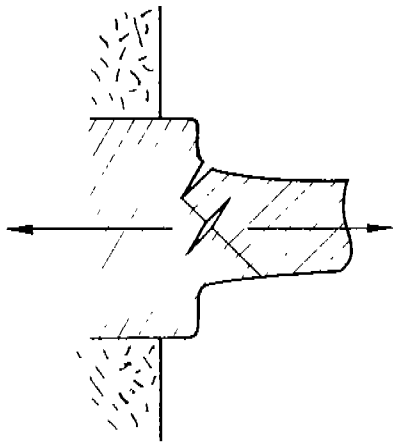
The crystals in this region would commence to rotate by primary slip, the orientation gradient developed at the shoulder would be accommodated at first by kinking, but soon the crystal would cleave on its basal plane in the vicinity of the kink as shown in Fig. 25*. These cleavages

*The type of crack shown in Fig. 25 at "A" would result when a kink-plane becomes fixed in one position. To maintain the position of lowest energy the kink-plane should rotate during extension of the crystal so as to bisect the slip planes on either side of it. Should it fail to do so, large stresses will be developed as slip proceeds on one side of it (cf. 19). Evidence of such stress can be judged from Burke and Hibbard's work on magnesium single crystals⁽¹⁸⁾. These authors found kink-planes near the grips of a tensile specimen where constraint was greatest and further observed twins on the relatively unslipped side of the kink-plane which twins terminated precisely on the kink-plane. Magnesium can only form twins if there is extension in the direction normal to the basal plane. This indicates that on the unslipped side of the boundary, there is a tensile stress perpendicular to the basal plane which can cleave the sample as shown in Fig. 25.

Thus it appears that the accommodation of orientation gradients by kink-planes involves not only the formation of the plane but its ability to rotate also.



(a)



(b)

FIG. 25: PRIMARY CLEAVAGE CRACKS ALWAYS SPREAD FIRST ACROSS THE BASAL PLANE AT RIGHT ANGLES TO THE SLIP DIRECTION, AND IN MANY CASES PENETRATED ACROSS THE CRYSTAL IN THIS DIRECTION WITH HARDLY ANY SPREAD IN OTHER DIRECTIONS. SUCH CRACKS CAN BE SEEN IN THE DEFORMED PORTION OF THE CRYSTALS IN FIG. 25a (AT POINT A), AND ALSO IN THE OUTER SHOULDERS OF THE SAME CRYSTALS. FIG. 25b SHOWS SCHEMATICALLY HOW SUCH A CRACK CAN OCCUR, AND ILLUSTRATES THE FACT THAT PRIMARY CLEAVAGE IS CLOSELY ASSOCIATED WITH BENDING OF THE LATTICE.

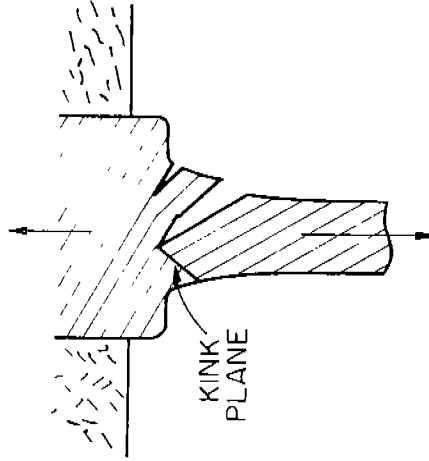


FIG. 26: SKETCH SHOWING PRIMARY CLEAVAGE CRACK PARTIALLY PENETRATING SPECIMEN CROSS-SECTION. KINK-PLANE FORMED AT ROOT OF CRACK ACCOMMODATES ORIENTATION GRADIENT DEVELOPED BY PRIMARY SLIP IN TEST LENGTH OF CRYSTAL.

would enlarge until sometimes the crystal was held together by only a thin skin, yet the load on the crystal continued to increase. During this growth of the crack a kink-plane would spread out from the root of the crack (see Fig. 26) which permitted further rotation of the crystal before the cleavage crack would completely sever the crystal*.

The reduction in area was still taken on the test length of the crystal (even though the fracture was in the shoulder) and is a measure of the amount of rotation which the test length suffered before cleavage completely severed the crystal. A more fundamental value to have recorded might have been the reduction in area (and relatedly the rotation) at the point where cracks first appeared but it is extremely difficult to observe the first small cracks.

At values of ϕ_0 and temperature adjacent to line f_1 the amount of rotation before the appearance of a crack and during its growth was considerable which accounts for the relatively high values of ductility in this region. At relatively low values of ϕ_0 or temperature, primary cleavage occurred with

*In a few borderline cases (just over the line f_1 on the ductile plateau in Fig. 9,) the crystal would rotate while held together by a thin skin until the crystal twinned along the test length, necked in the twin band and ruptured with 100 per cent contraction in area.

very little rotation or reduction in area either before or after the crack started. Indeed as temperature was lowered the deformation was so reduced (see Fig. 9, 16 and 27) that at 5°C no crystal with $\phi_0 < 70^\circ$ could rotate to the critical orientation for twinning and then fail by secondary cleavage which explains the virtual disappearance of surface D in Fig. 9 at this temperature. It is felt that in the absence of primary cleavage (i.e., in tensile tests in which rotation in the grips is not prevented) surface F would not appear in Fig. 9 and surfaces A, E, and D would be extended to lower temperatures as suggested in Fig. 28.

The inability of kink-planes to accommodate orientation gradients in zinc as temperature is lowered and the appearance of primary cleavage is of enormous importance, for it at once gives a clue to a) why secondary cleavage appears, b) a reason for the temperature at which it occurs, c) an explanation for the skew course to surface E in Fig. 9, d) a reason for the temperature at which polycrystalline zinc becomes brittle, e) and finally an explanation of why hexagonal crystals are prone to low temperature brittleness but face-centered crystals are not.

- a) Secondary cleavage and primary cleavage both occur on the basal plane. They both occur in regions where slip is restricted: primary cleavage in the

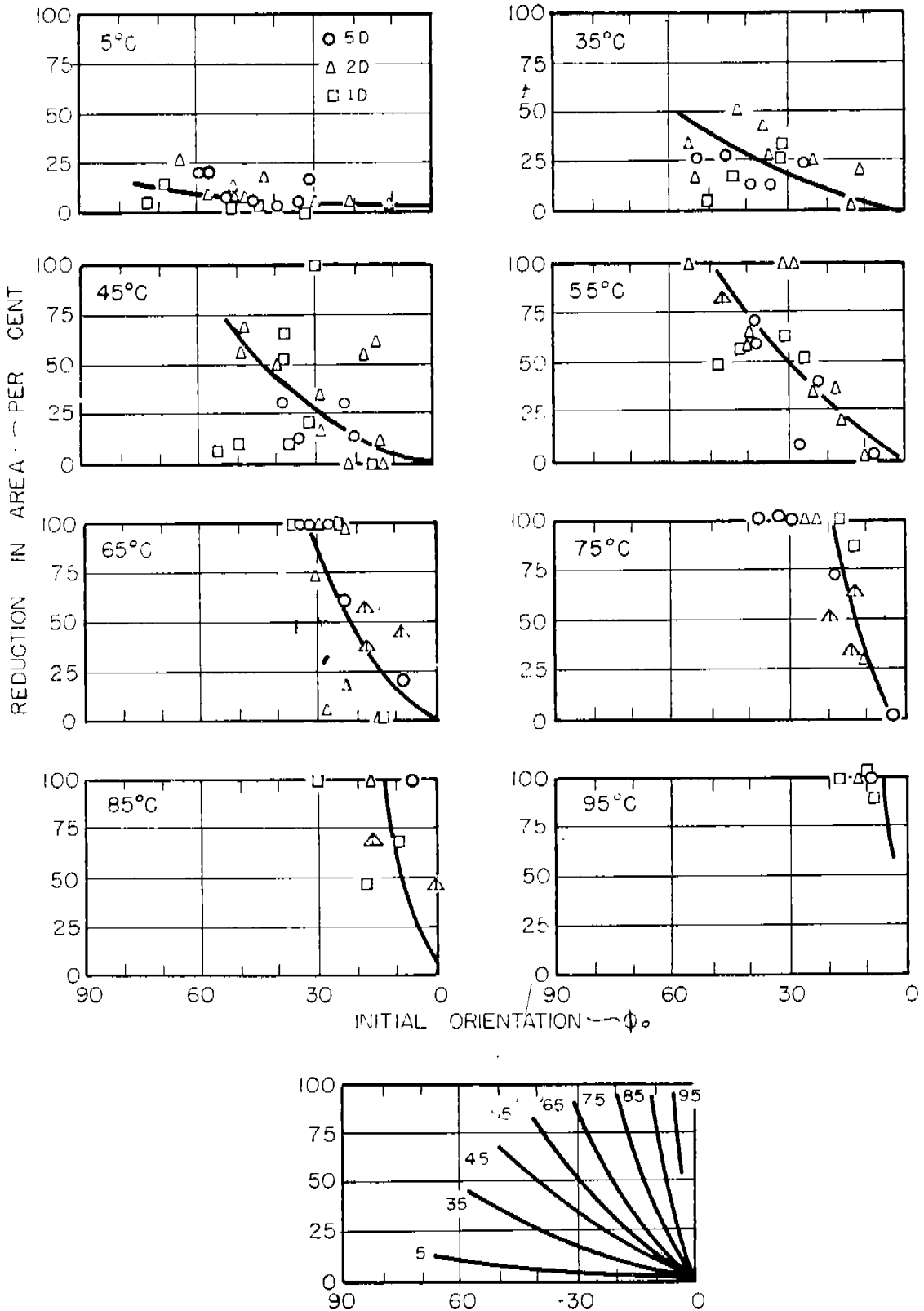


FIG. 27: A COMPOSITE PLOT OF REDUCTION IN AREA VALUES FOR 5D, 2D AND 1D SPECIMENS WHICH FAILED BY PRIMARY CLEAVAGE. THE DATA TAKEN FROM FIGS. 10, 11 AND 12 SHOW THE NEGLIGIBLE EFFECT OF GAGE LENGTH ON THE VALUES OBTAINED.

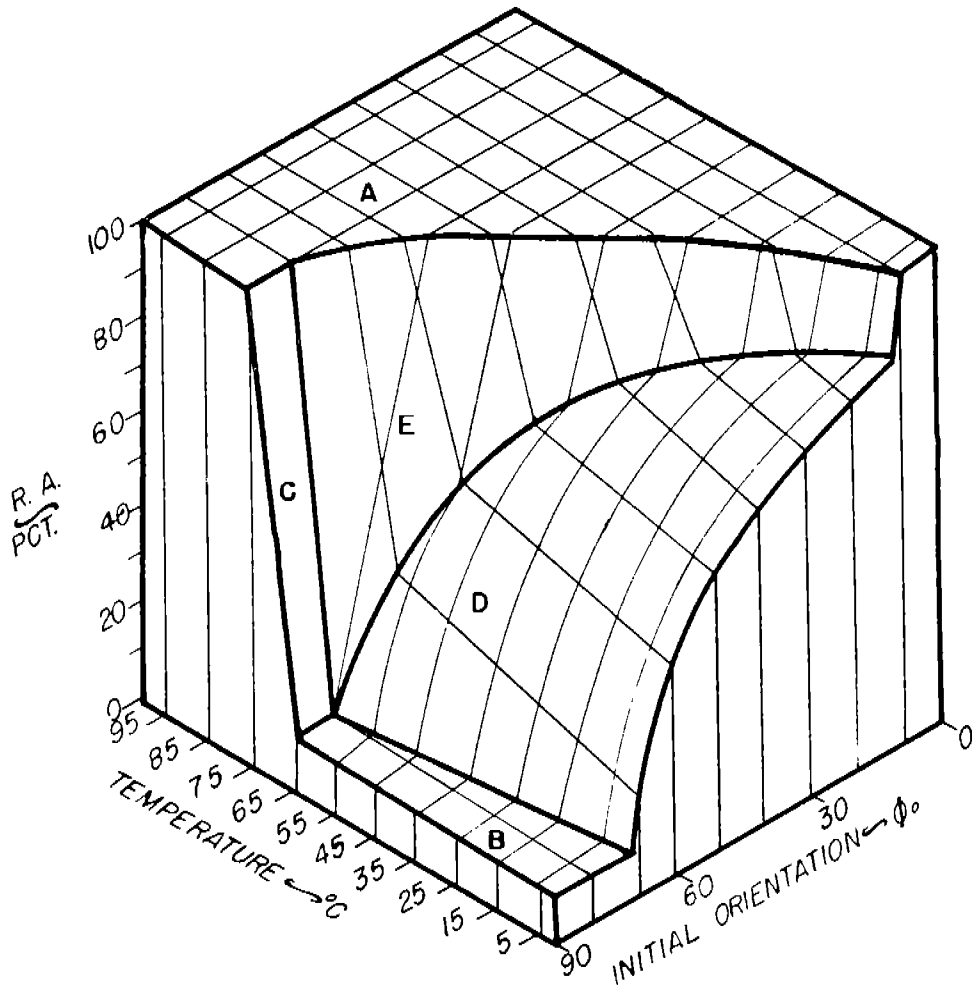


FIG. 28: SUGGESTED COURSE TO FIG. 9 IN THE ABSENCE OF PRIMARY CLEAVAGE INDUCED BY CONSTRAINT OF SHOULDERS OR GRIPS.

shoulder where slip is restricted in the broader portion of the crystal, secondary cleavage in a twin band where orientation gradients must develop adjacent to the twin boundaries to maintain compatibility (cf. Ref. 33). It is proposed therefore that secondary cleavage is fundamentally the same as primary cleavage, the only difference being the manner in which the orientation gradient which induces basal cleavage is developed.

- b) Twinning reorients the crystal. The angle ϕ_t between the tensile axis and the pole of the basal plane of the twinned material was measured (by recording the inclination of the cleaved surface) for all crystals of 5D gage length which failed by secondary cleavage. The values, recorded in Table I, are clustered around a low average value (17.5°). It was seen from either Fig. 9 or 27 that crystals with low values of ϕ_0 suffered primary cleavage at higher temperatures than those of high values of ϕ_0 . In fact primary cleavage appeared at 65 to 75°C at $\phi_0 = 17.5^\circ$, a temperature range which compares quite closely with the transition temperature of surface C.
- c) The fact that kink-planes could not accommodate orientation gradients at the shoulder in full

cross-section, but could after cleavage partially had reduced the cross-section (cf. Fig. 26) indicates that this accommodation is a function of size, being favored by smaller cross-sections*. This is compatible with the skew course of surface E: crystals with relatively lower values of ϕ_0 would possess a smaller cross-section on arriving at the twinning orientation ϕ_f (cf. Equation 3) and therefore accommodation of the orientation gradients in the twin band would be favored.

- d) The deformation of polycrystalline metals involves orientation gradients (21--29). If these gradients cannot be accommodated by kinking, cleavage may be expected to result.

It is to be expected that the conditions for cleavage in different grains will be reached at different stages in a polycrystalline aggregate. Homes and Gouzou (30) have shown by direct observation of the process of fracture that this is the case for zinc below the transition temperature. It was not until a large number of "premature" cracks had formed that rapid cleavage cut through the remaining grains. Nevertheless, it is obvious that cleavage will occur at much higher temperatures

*Dislocation theory teaches this is so (cf. pp. 92--94 Ref. 20).

on the average because of orientation gradients than in their absence. It is significant that the transition temperature for the polycrystalline zinc rod from which the single crystals were made approximates the average transition temperature of Fig. 9 rather than that of Fig. 28.

- e) The development of kink-bands in hexagonal metals and face-centered metals is quite different. A tensile test on aluminum produces a high density of microscopically small (0.05 mm.) kink-bands throughout the gage length of the metal (31, 25, 32) which offers a much greater degree of freedom to accommodate orientation gradients than does the one band formed in zinc (i.e., the orientation difference on either side of a kink-plane in aluminum is small compared with that on either side of the one kink-plane at the shoulder in zinc).

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APPENDIX

TABLE I

ORIENTATION OF THE BASAL PLANE OF TWINNED CRYSTALS
AS MEASURED AT FRACTURE FOR ALL CRYSTALS GAGE-LENGTH
5D WHICH CLEAVED ON THAT PLANE

<u>Specimen Number</u>	<u>Temperature</u>	<u>Orientation of Twin ϕ</u>	<u>Fracture</u>
18	75	10	Mixed
29	75	37	"
5	65	17	Cleavage
90	65	11	"
63	55	21	"
84	55	6	"
41	45	8	"
51	45	15	"
54	45	10	"
30	45	38	"
89	35	19	"
Average		17.5	