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ELEVENTH TECHNICAL REPORT

EFFECT OF THE STRUCTURE OF DISLOCATION BOUNDARIES ON YIELD STRENGTH

BY

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Eleventh Technical Report

Effect of the Structure of Dislocation Boundries on Yield Strength

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Office of Naval Research Project

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ABSTRACT

The contributions of three types of dislocation array to the strength of zinc crystals were studied. They were: (1) a pair of pure edge dislocation boundaries, (2) a complex array of pure tilt boundaries, (3) an array of screw dislocations in the slip plane produced by a twist about the c-axis.

The strengthening effects of all these substructures were found to be dependent on the temperature to which the crystal had been heated subsequent to introduction of the array of dislocations by plastic bending or twisting. An annealing temperature close to the melting point was necessary to develop an appreciable strengthening effect. In all cases the yield strength was raised; the sharpness of the yield was decreased; but the slope of the linear part of the stress strain curve characteristic of hexagonal crystals was unchanged by the introduction of dislocation arrays.

Quantitative measurement of strengthening effect as a function of boundary angle and angle of twist showed a rapid increase in yield strength for very small angles followed by decreasing increments in yield strength for additional increments of boundary angle or twist angle. As a tentative explanation for these quantitative results it was suggested that small angle boundaries may act as nuclei for the pile up of dislocation groups the stress fields of which would be sufficiently long range to interact with one another over the observed distances between neighboring slip lines.

Introduction

Small angle boundaries are present in nearly all crystals; this is particularly true for metal crystals. The idea that most crystals are made up of small relatively perfect blocks which are slightly rotated one with respect to another first arose from the comparison of observed and calculated x-ray diffraction intensities by Darwin(1) and Ewald(2). The high integrated intensities of diffraction maxima could be explained only by assuming that the diffracting crystals were mosaic crystals with a network of small angle boundaries spaced at intervals of approximately 10^{-4} centimeters.

More recently, due to improvements in x-ray and metallographic techniques it has become recognized that most metal crystals also contain small angle boundaries on a microscopic or even macroscopic scale(3)(4)(5). Such large mosaic structures in crystals may be formed during growth(6)(7) and may also be introduced by nonuniform plastic deformation(8)(9).

A dislocation model for the structure of such boundaries originally was proposed by Burgers(10) and Bragg(11) and later developed in more detail by Shockley and Read(12). Experimental work has subsequently borne out the predictions of the theory as regards mobility(13) and energy(14)(15) of dislocation boundaries, thus leaving little room for doubt that the dislocation model is at least qualitatively correct.

Dislocation theory appears to possess the necessary versatility to be able to account for the complex plastic properties of crystals. However, this same versatility frequently makes it possible to devise several dislocation models which can satisfactorily explain any isolated

observation; it is often difficult to make a satisfactory choice between possible alternatives because of the limited information available. The dislocation theory, while twenty years old, is still not capable of establishing a unique detailed model for the most important plastic phenomena yield strength, strain hardening, recovery, and creep. The basic concepts are undoubtedly sound but they have not yet been woven into a completely coherent structure. It is felt that a deeper insight will be possible when the results from more discriminating experiments become available.

Two important difficulties exist with most experimental measurements in crystal plasticity: (1) it is not yet possible to determine with sufficient accuracy the initial or final structural state of the crystal being tested (distribution and density of crystal imperfections), (2) it is difficult to decide to what extent measured properties are intrinsically those of the crystal or are associated with the particular test conditions. For example, until quite recently it was thought that the parabolic stress strain curve for single crystals of cubic metals was always associated with slip in cubic crystals. However Kochendorfer[16] has shown that the shape of the curve is dependent upon the method of testing; Honeycombe[7] found that tension testing of aluminum crystals causes formation of microscopic kinks and the development of asterism during the initial rapidly rising part of the stress strain curve. Therefore the initial rapid strain hardening observed in the tension test now appears to be due to the development of strain inhomogenities produced during the early stages of plastic extension. These inhomogenities

are on a scale five or six orders of magnitude larger than atomic dimensions. When cubic crystals are deformed in simple shear, which avoids the development of small angle boundaries during extension, nearly linear stress strain curves similar to those ordinarily observed for hexagonal metals are obtained. Thus is exposed the danger of interpreting macroscopic measurements in terms of an atomic model. Taylor(18) as far back as twenty years ago set up a dislocation model which explained the parabolic stress strain curve. This is a good illustration of the versatility of the theory since it is almost a certainty that the assumed array of dislocations was not at all the distribution which actually existed in the strained crystals.

The above interpretation of the initial rapid increase in flow stress during tension testing of cubic crystals suggests that the presence of dislocation boundaries may represent an important factor in the shear strength of a crystal. Further evidence that dislocation boundaries are effective barriers to slip is also available. Tate and McLean(18) showed that development of a network of dislocation boundaries within the grains of polycrystalline aluminum had a marked strengthening effect in creep. Hazlett and Parker(20) have found that the presence of dislocation boundaries greatly increases the resistance to plastic deformation of nickel and nickel solid solution alloys both in an ordinary tension test and in creep at high temperature. A simple experiment(21) with a zinc single crystal illustrates even more directly the role of dislocation boundaries as barriers to slip. Fig. 1 shows a crystal of rectangular cross section in which the basal planes are horizontal and at right angles to the plane of the paper. The slip direction, is in the plane of the paper. The specimen originally contained a

FIG. 1 MACROGRAPH SHOWING A 15° BOUNDARY DEVELOPED FROM A 1° BOUNDARY BY TRAPPING DISLOCATIONS GENERATED WITHIN THE CRYSTAL DURING RAPID LOADING AT 375°C. 4X.

one degree pure tilt boundary located at right angles to the long axis of the crystal approximately at its center. It was mounted as a cantilever beam and a load suddently applied to the free end. The crystal bent at the position of the small angle boundary and under the line of application of the load. If the boundary had not been present the bending would have occurred at the grip and under the point of application of the load (positions where a sudden drop in shear stress occurs). The dislocation wall therefore was able to stop all of the dislocations moving toward the gripped end of the specimen in spite of the fact that no drop in shear stress was present at this position. Another experiment with a zinc single crystal also confirmed this strengthening effect. Fig. 2 shows two stress strain curves. The first is for a nearly perfect crystal. The second is for the same crystal after introduction of a network of dislocation boundaries into the gage length. A 20% increase in the yield stress was observed.

Although the effectiveness of small angle boundaries as barriers to the motion of dislocations through a crystal seems well established it is not at all clear from the theoretical point of view why this should be so. The interaction forces between dislocations forming a small angle tilt boundary do not appear to be great enough to explain the magnitude of the observed strengthening effect. It appears that a refinement of the model will be necessary to account for the size of the effect.

The object of the following series of experiments was to determine the conditions under which dislocation boundaries could substantially increase the shear strength of a crystal. A number of variables appeared

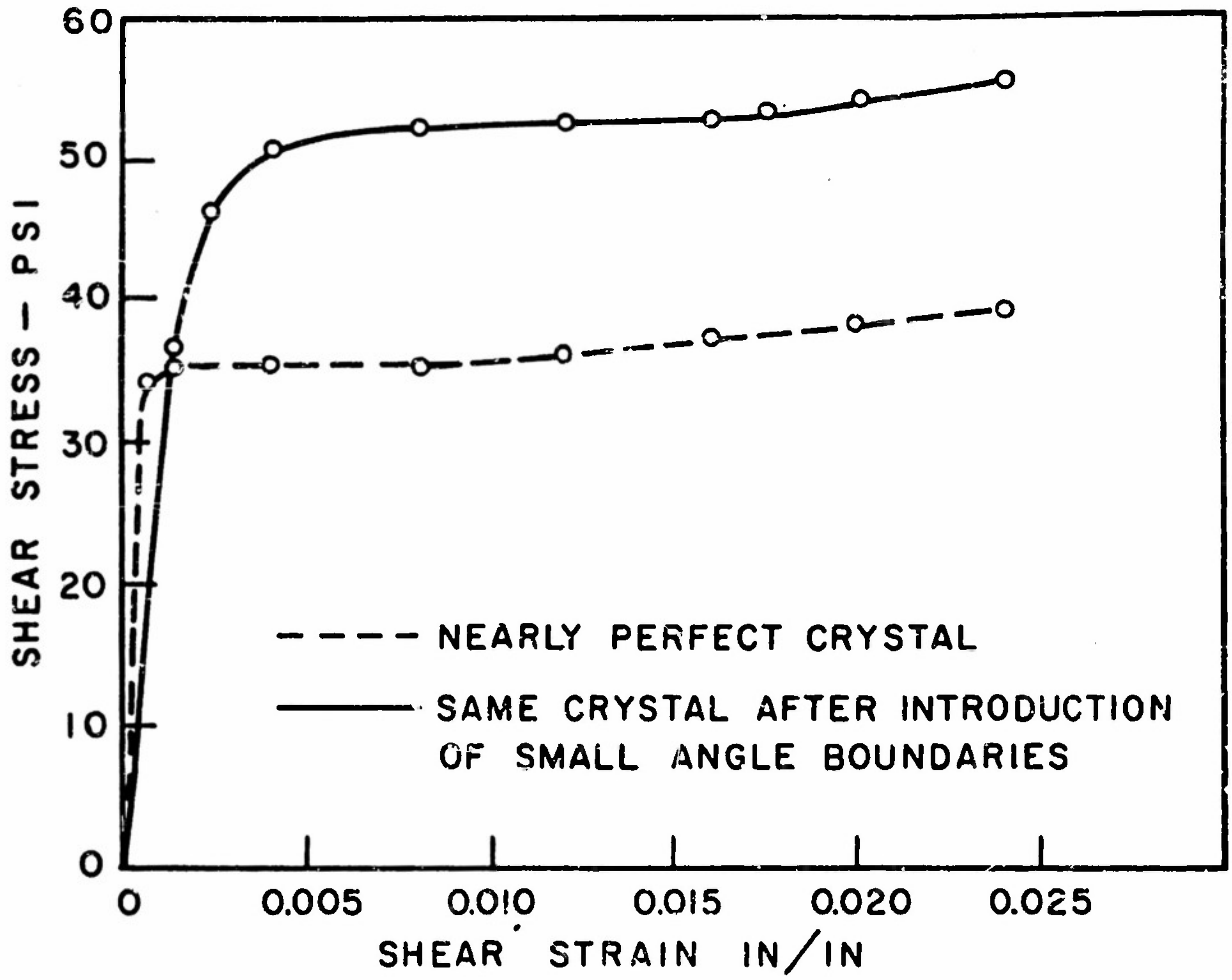


FIG 2 STRESS STRAIN CURVES OF A 99.99% PURE ZINC CRYSTAL SHOWING THE EFFECT OF INTRODUCING A SUB-STRUCTURE OF SMALL ANGLE BOUNDARIES.

to be of potential importance, only a few of which could be studied with any degree of thoroughness in the present investigation. These were:(1) boundary angle, (2) orientation of the boundary relative to the active slip direction, (3) intersections between boundaries, (4) structural details of the boundary, (5) interactions between impurity atoms and small angle boundaries. Unfortunately all of these variables plus others not listed are probably interrelated making the problem extremely complex.

Experimental Procedure

Stress strain curves of hexagonal metals are generally nearly linear to very large strains[22]. At the low end of the strain scale the linearity persists to small strains, the magnitude of which depends upon the uniformity of applied stress and upon the structural perfection of the crystal in the gage section. In a tension test the shape of the initial portion of the stress strain curve is extremely sensitive to the straightness of the specimen and to the axiality of loading. (See for example the curves obtained by Jillsen[23]). Other objections to the tensile testing of soft single crystals arise from the rotations accompanying extension[24]. Some of these difficulties can be avoided by testing of single crystals in shear. Previous work[24] has shown that a particularly uniform distribution of slip throughout the gage section of a specimen can be obtained by this type of loading. A section through a shear specimen showing the method of application of the load is shown in Fig. 3. Unfortunately this simple specimen was not suitable for some of the desired tests because simple tilt boundaries could not

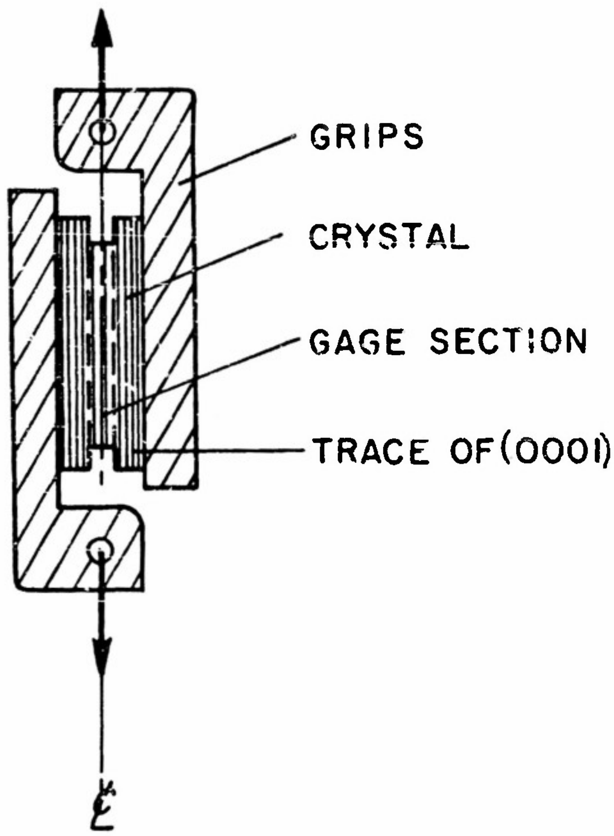


FIG 3 NORMAL METHOD OF LOADING IN SHEAR – SHEAR STRESS IS APPLIED IN THE SLIP PLANE ALONG A SLIP DIRECTION.

be introduced into the gage section of the specimen without causing complications in gripping.

The following were considered to be the desirable features of a specimen to be used for investigating the strengthening effect of dislocation boundaries: (1) the loading method should be capable of producing sharp yields and substantially linear stress strain curves at small strains; (2) the deformation should be as nearly as possible a pure slip deformation, making possible complete recovery of mechanical properties on annealing[24]; (3) the specimen should be of such a form that the grips would not interfere with the introduction of simple tilt boundaries in the gage section; (4) the gage section should be constrained in such a way that the angle of a boundary that had been introduced would be forced to remain constant during subsequent deformation of the gage section (in contrast to the behavior of the boundary in the specimen of Fig. 1); (5) the gripping arrangement should be suitable for use at liquid nitrogen temperature without damage to the crystal; (6) the crystal should be easily removable from the grips for annealing and for introduction of boundaries.

Fig. 4 shows the type of specimen finally adopted, indicating the orientation of crystal axes and the estimated elastic shear stress distribution on the slip plane in the slip direction. When a shearing load is applied to such a specimen the crystal deforms by slip on the (0001) planes. The active gage section is indicated in Fig. 4 by the crosshatching. The boundary of the slipped volume, instead of being a free surface on four sides as in the usual type of shear specimen, consists of a small angle tilt boundary on two of the faces. Figs. 5 and 6 are photographs of a specimen before and after a 25%

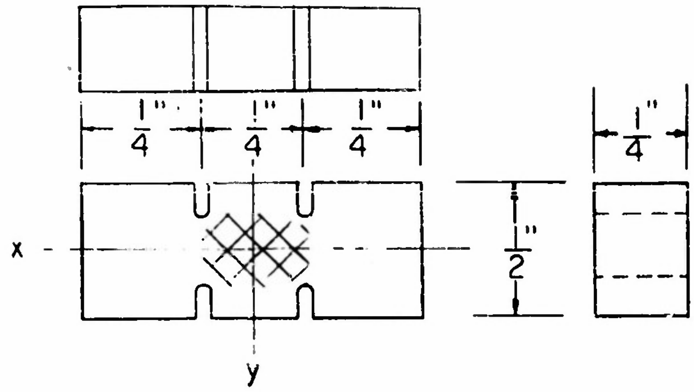


FIG 4 (a) SKETCH SHOWING SPECIMEN DIMENSIONS TEST SECTION CROSS HATCHED SLIP PLANES OF THE CRYSTAL CORRESPOND TO THE y = A CONSTANT PLANES OF THE SKETCH

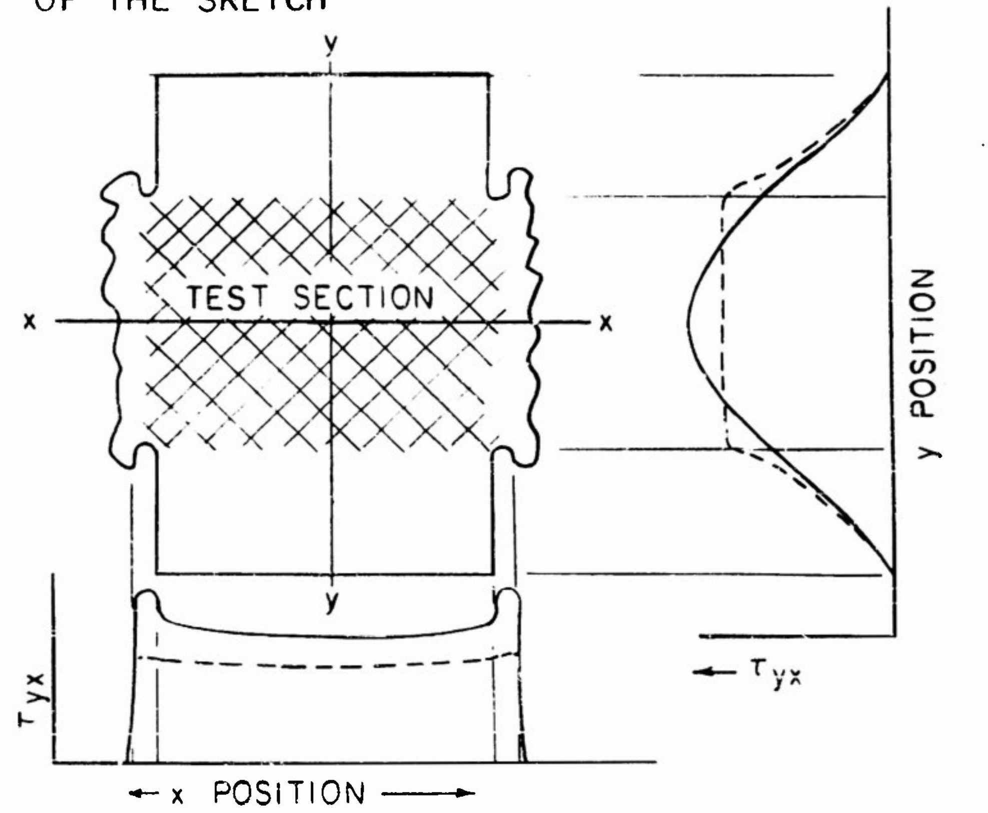


FIG 4 (b) ENLARGED VIEW OF TEST SECTION SHOWING ESTIMATED ELASTIC SHEAR STRESS DISTRIBUTIONS IN THE SLIP PLANE ALONG x AND y AXES (BEFORE START OF PLASTIC DEFORMATION - SOLID LINES, AFTER START OF PLASTIC DEFORMATION - DASHED LINES)

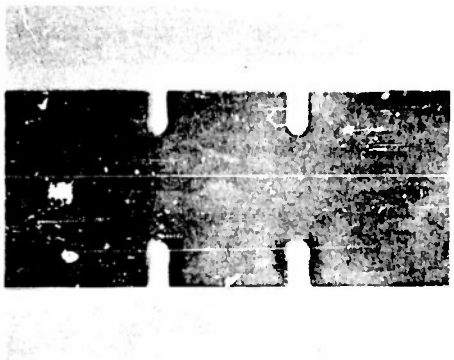


FIG.5 UNDEFORMED KINK SPECIMEN

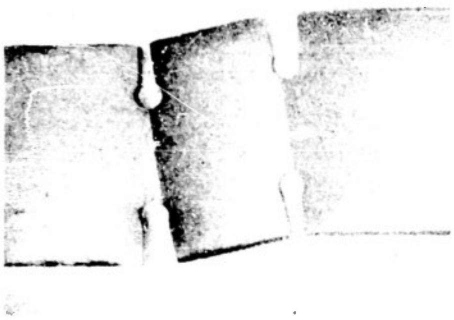


FIG.6 KINK SPECIMEN AFTER A SHEAR STRAIN OF 25 %

FIG. 7 KINK SPECIMEN CLEAVED THROUGH THE CENTER AFTER A 1 % STRAIN SHOWING THE SMALL ANGLE BOUNDARY ON BOTH CLEAVAGE AND SIDE SURFACES

shear strain. The tilt boundaries at the extremities of the gage section are visible on the side surfaces because of the change in intensity of reflected light (due to the relative rotation of the etch pit faces across the boundary). Fig. 7 shows a specimen which had been cleaved through the center after a 1% strain. The small angle boundary is visible on both the cleavage surface and the side surface of the specimen. To distinguish it from the normal type of shear specimen this new specimen will hereafter be referred to as a kink specimen.

The high c-axis coefficient of thermal expansion in zinc crystals always causes gripping troubles. Since all measurements were made at the temperature of liquid nitrogen (to avoid recovery during the test), it was necessary to have a crystal mounting which could be aligned at room-temperature and then could be cooled to -196° C without having the crystal become loose in the grips. To obviate this difficulty, the clamping force was applied through springs so that the c-axis contraction would not cause loosening during cooling. The cleavage surfaces which were in contact with the grips were seldom perfectly flat; they usually had a few small steps where the cleavage shifted from one plane to a nearby parallel plane. It was found that a rubber pad placed between the crystal and the grip surface distributed the load evenly. The rubber pads easily conformed to the surface contour at room temperature but became hard and unyielding at -196° C; they therefore afforded rigid mountings for the specimen at the test temperature.

The strain in the specimen was determined by measuring the relative displacement between the two grips. Since it was desired to measure displacements to an accuracy of .000025 inch, rigid mounting was essential. The critical load for slip in a specimen of about .22" x .22" cross section was of the order of one pound. Therefore the extensometer had to be capable of measuring to .000025 inch and had to respond to a force of less than .004 pound. A roller extensometer was designed to meet these requirements; it is shown in the schematic drawing of Fig. 8 and in the photograph of grip and extensometer assembly in Fig. 9. The .013 inch diameter rollers were held between flat rectangular stainless steel rods .10 inch by .03 inch. The rod on which the dial was supported was permanently mounted to one side of the grips. The other rod which ran between the two rollers at the top end was attached to the other side of the grips by a small drop of rubber cement. The rubber cement formed a strong rigid bond at the test temperature; after testing, when the assembly was again at room temperature, it could be easily removed. The rods were surrounded by a glass tube which extended far enough above the liquid level to be above the dew point so that water vapor would not condense on them. The inside of the glass tube was filled with a pure nitrogen atmosphere supplied by evaporation of the cooling liquid and therefore no condensation occurred internally. In order to keep the size of the dial small but still achieve sufficient total magnification, the position of the pointer on the scale was observed through a low power microscope. The total magnification of the extensometer was approximately 10^3. Fig. 10 is a curve of extensometer reading versus load obtained with a steel

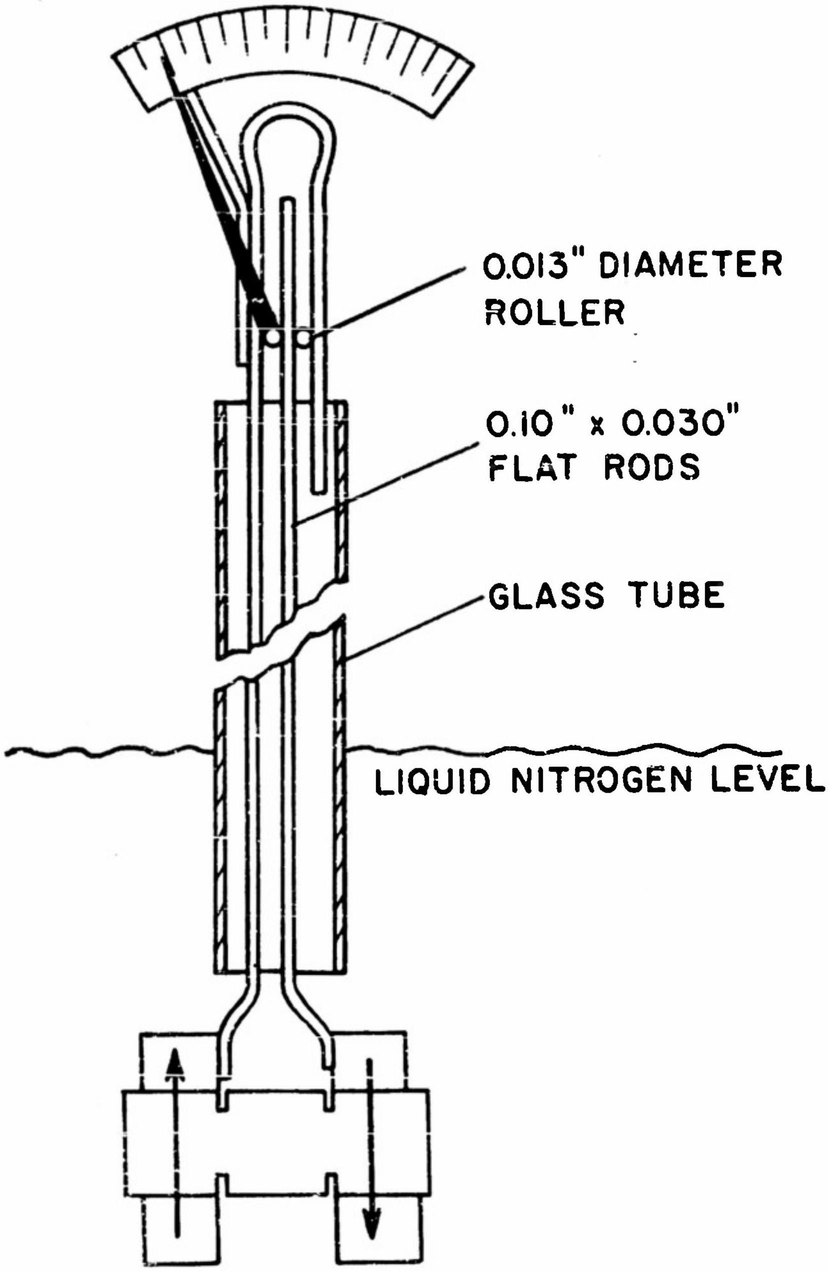


FIG. 8 SCHEMATIC DIAGRAM OF ROLLER EXTENSOMETER

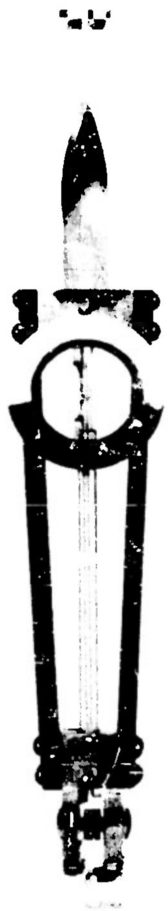


FIG.9 LOADING AND STRAIN MEASURING ASSEMBLY
EMPLOYED FOR TESTING OF KINK SPECIMENS

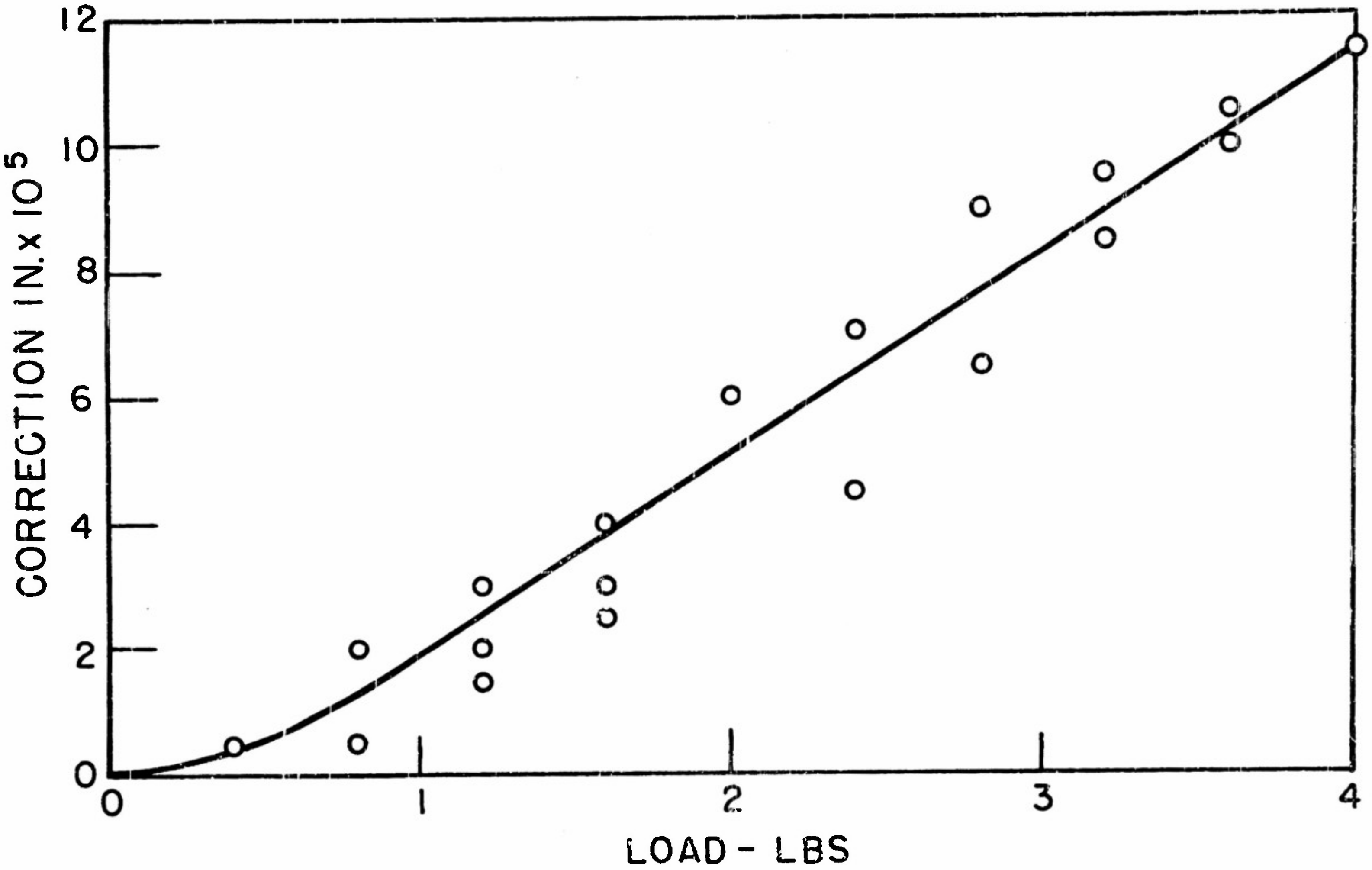


FIG. 10 EXTENSOMETER CORRECTION FOR ELASTIC SPRING IN THE GRIP ASSEMBLY

bar in place of the crystal specimen. This curve indicates the amount of elastic distortion of the grip and extensometer assembly and was used to correct the readings when stress strain curves were plotted.

The pulling head (Fig. 11) was actuated through a 900 to 1 worm gear reduction unit driven through a pair of external gears by a one rpm synchronous motor which caused the head to move .0005 inch per minute. The load was applied to the specimen through a load measuring beam, the curvature of which was indicated by S-R-4 strain gages. Two gages fastened to the compression and tension sides of the beam respectively formed two arms of a balanced resistance bridge; the other two arms were contained in the standard S-R-4 strain indicator. The least division on the scale of the measuring resistance represented .04 pound. Readings could be easily interpolated between divisions to .01 pound. The load measuring beam was linear up to a total load of 5 pounds, which was ample range for the specimens being tested.

The crystals were made with Horse Head Special Zinc (from the New Jersey Zinc Company) which had a nominal purity of 99.99%. Long rectangular crystals were grown from which the desired specimen blanks could be cut. The orientation of crystallographic axes in the large crystal was controlled by a seed crystal having the desired orientation relative to specimen directions. Three steps involved in the preparation of specimen blanks are shown in Fig. 12. A polycrystalline blank was cast into the desired shape by allowing a weighed charge to melt in a helium atmosphere and run into a graphite mold, as shown in Fig. 13. The cast bar, which was about six inches long, in contact with a two inch long single crystal seed having the same cross section were placed in

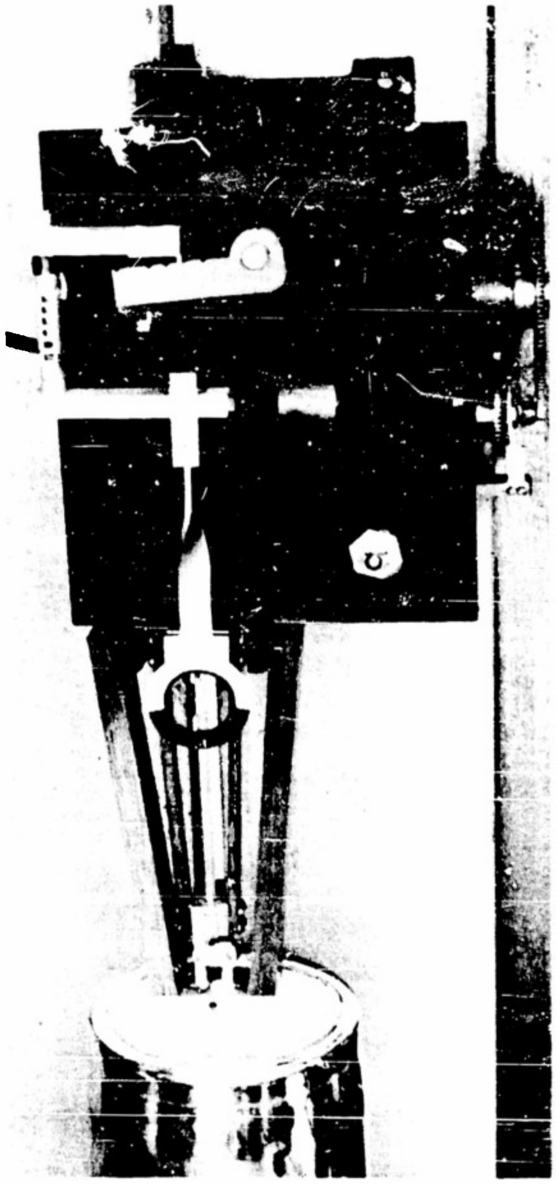


FIG. 11 COMPLETE TESTING MACHINE SHOWING S-R-4 LOAD MEASURING BEAM

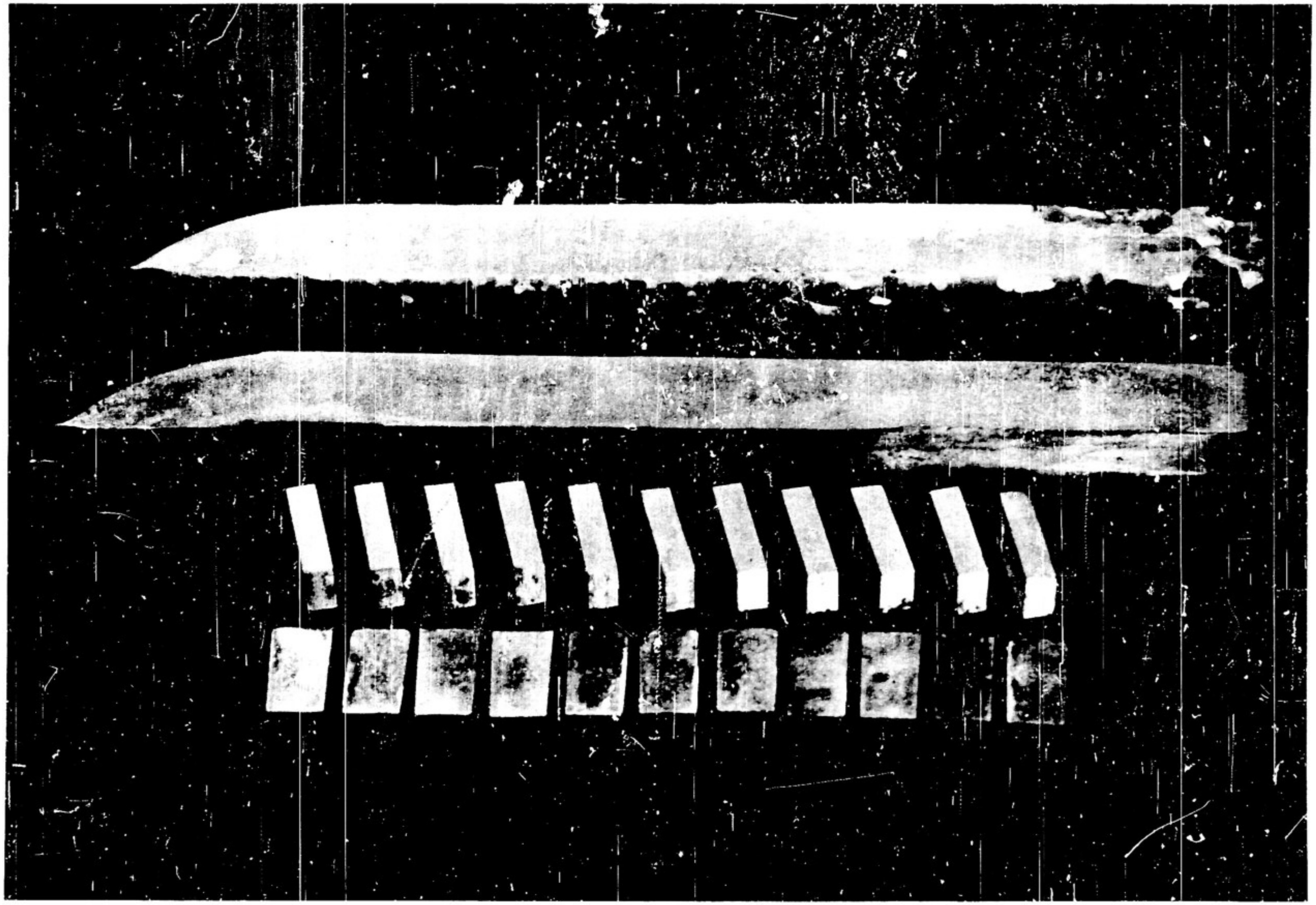


FIG. 12 THREE STEPS IN THE PRODUCTION OF SPECIMEN BLANKS (TOP) CAST POLYCRYSTALLINE BAR (CENTER) LARGE SINGLE CRYSTAL GROWN FROM SEED CRYSTAL (BOTTOM) SPECIMEN BLANKS CUT FROM LARGE CRYSTAL

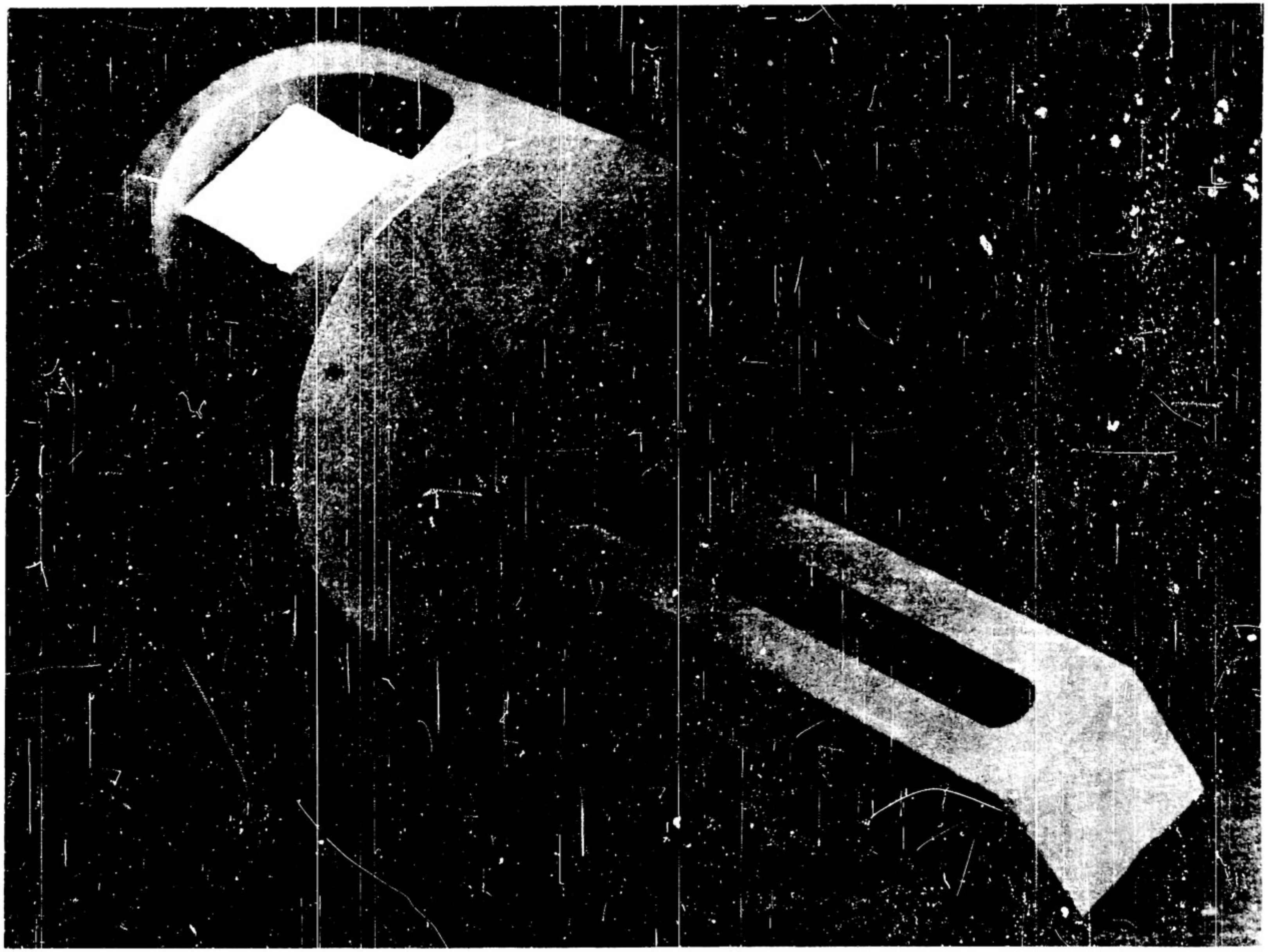


FIG.13 MOLD FOR CASTING POLYCRYSTALLINE BLANK

the graphite mold shown in Fig. 14. The mold was placed inside a silica tube and a helium atmosphere was introduced. Outside the tube on a sliding carriage was a short heating coil. The current input to the coil was adjusted so that under equilibrium conditions a molten zone approximately three inches long was obtained under the coil. By changing the position of the coil it was possible to control the position of the liquid solid interface accurately since it was at all times visible through the silica tube. To start the growth of the crystal, about one-half to one inch of the seed crystal was melted. The oxide skin at the junction between seed and polycrystalline blank was dispersed and caused to float to the surface by rocking and jarring the mold after the seed was partially melted. The entire rod was then solidified into a single crystal having the orientation of the seed by moving the heating coil at a rate of one-half inch per hour away from the seed end of the mold.

Slicing of the long crystal into specimen blanks, without causing any plastic deformation in the crystal, was accomplished by acid sawing(25). The device pictured in Figs. 15 and 16 was designed to make six cuts simultaneously. The crystal was held with a force of a few grams against stainless steel wires which carried a thin film of concentrated nitric acid over the crystal. The specimen was carried by a moving table supported at a fulcrum, shown at the right in the picture. The weight of crystal and table were just slightly over-balanced by the counter weight (partially visible in the picture); thus a small upward force was maintained against the wires. The wires moved about 12 inches in one direction then 12 inches back in the

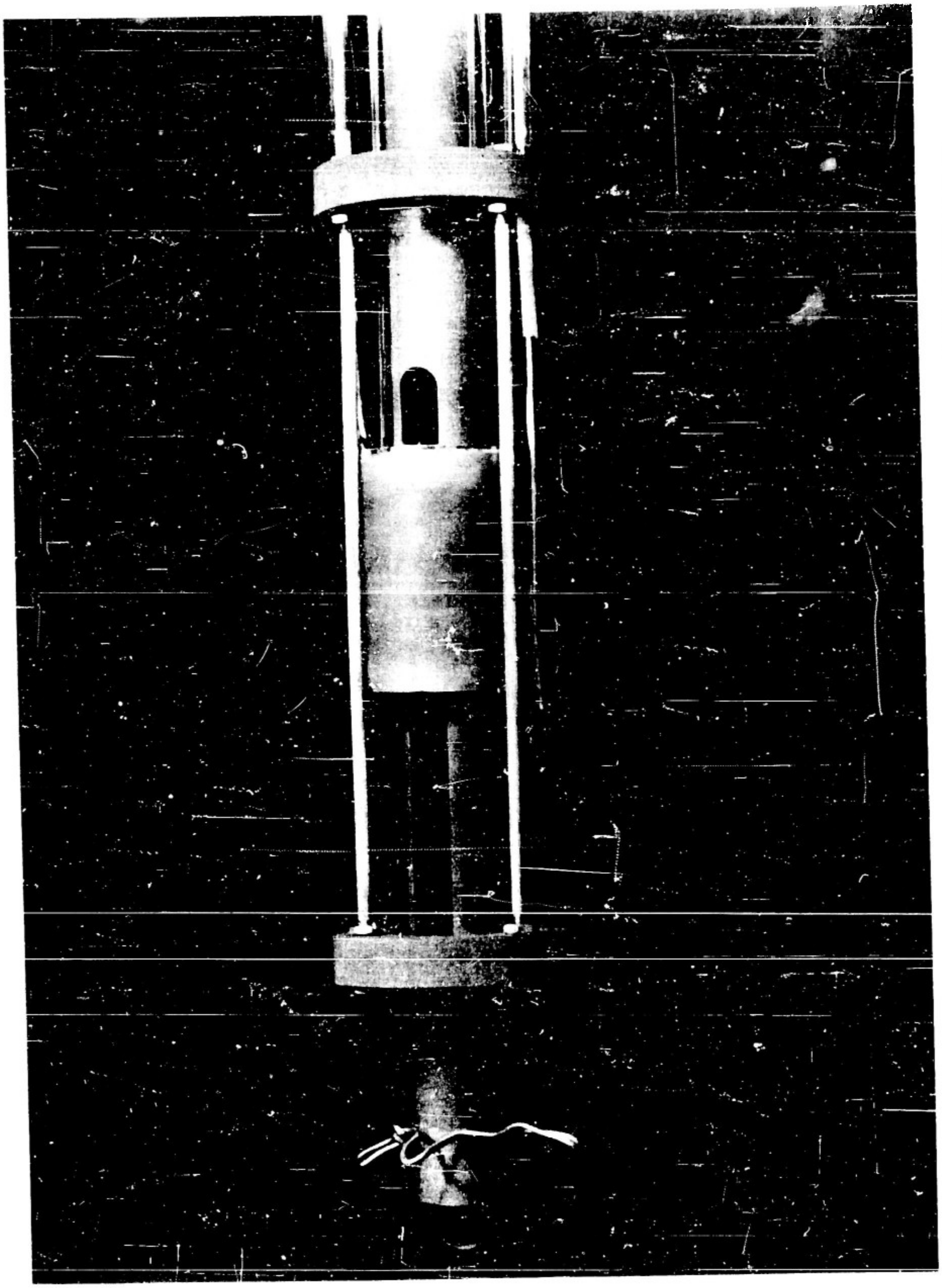


FIG. 14 TRAVELING FURNACE AND MOLD USED FOR GROWING LARGE RECTANGULAR CRYSTALS OF CONTROLLED ORIENTATION

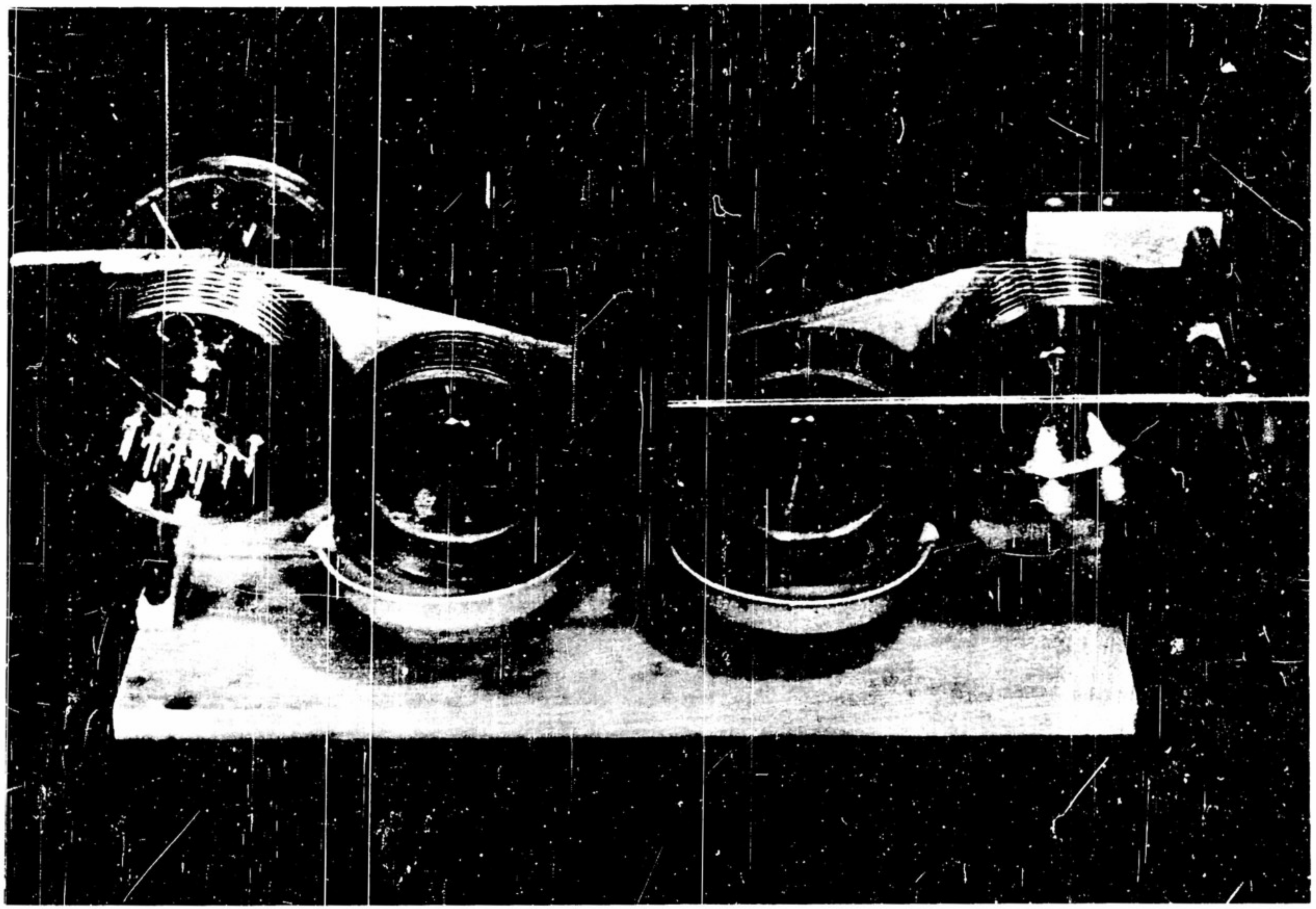


FIG.15 ACID SAW USED FOR CUTTING CRYSTALS WITHOUT DAMAGE TO THE CUT SURFACE

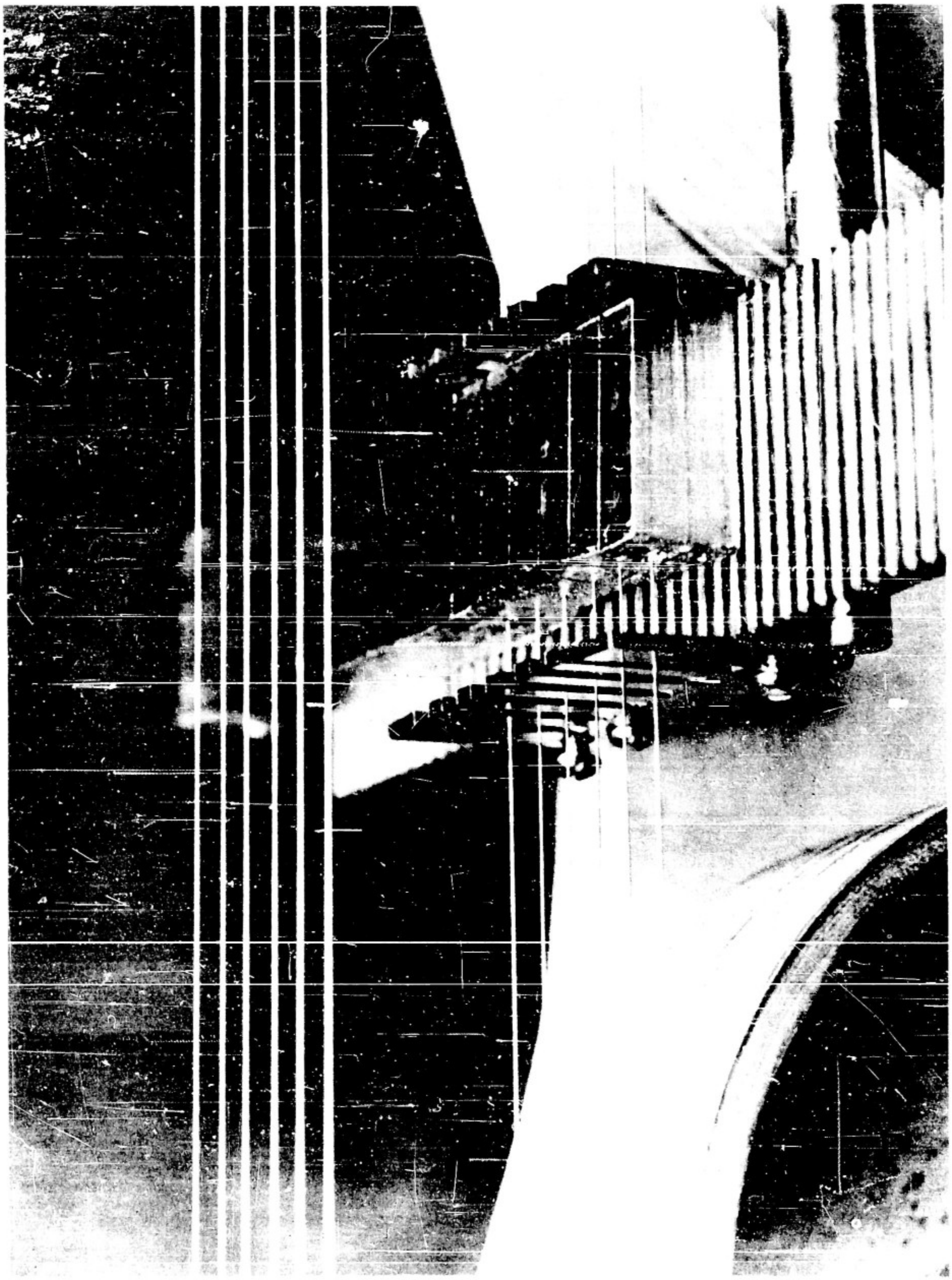


FIG. 16 CLOSE UP VIEW CF FARTIALLY CUT CRYSTAI

opposite direction, which made it unnecessary to have endless loops. This procedure also produced a more uniform cut since fresh acid was brought in alternately from each direction. The time required to cut through a crystal one-half inch thick was approximately twenty hours.

The next step in the preparation of specimens of the form shown in Fig. 4 was to cleave a wafer about 1/32" thick from top and bottom surfaces, thus obtaining faces which were flat and accurately parallel to the (0001) plane in the crystal. The last step was cutting of the four slots which defined the gage length. This was also done with the acid saw, set up with only two wires instead of six. A stop was arranged to limit the cuts to the desired depth.

As mentioned previously, a kink specimen is characterized by the formation during plastic flow of a pair of pure edge dislocation boundaries at the extremeties of the gage section. The angle of the boundaries is directly related to the relative displacement of the two grips, which can be accurately measured. This specimen geometry suggested a way of introducing boundaries of controlled angle into the gage section of the specimen. Fig. 17 shows the type of specimen used for introduction of a pair of edge dislocation boundaries across the gage section. At any stage during a series of tests the specimen could be gripped at the inner pair of notches and a pair of boundaries of known angle could be introduced. The accuracy within which the boundary angle could be controlled was $\pm$ .01°.

Circularly symmetrical arrays of dislocation boundaries could be introduced through the gage section by impact of a small hemispherical indenter on one of the (0001) surfaces. Fig. 18 describes the experimental conditions and shows schematically a section through a conical dent. The presence of high internal stresses in these conical

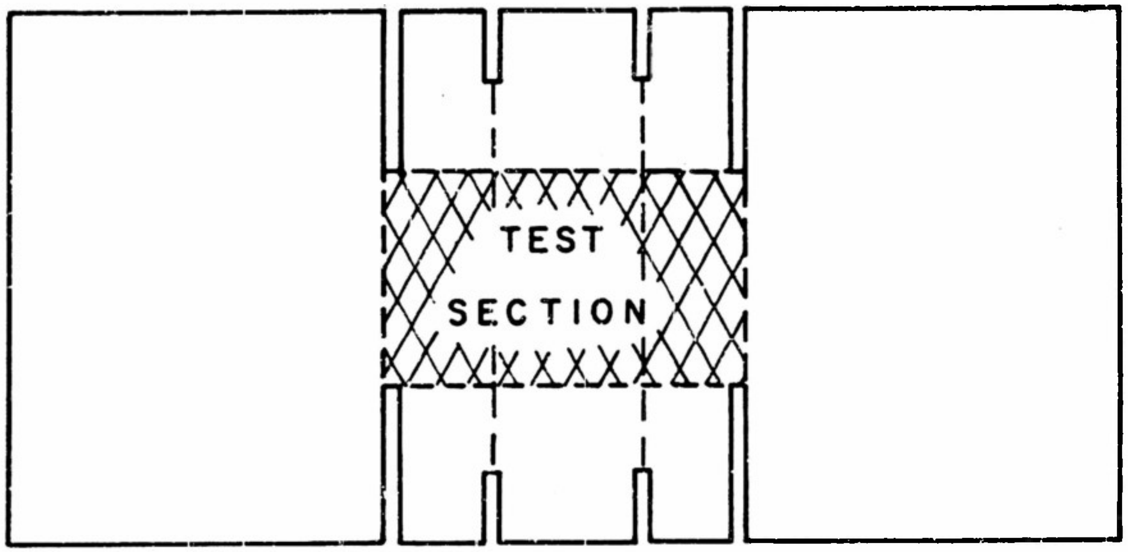


FIG.17 TYPE OF SPECIMEN USED FOR INTRODUCTION OF A PAIR OF SIMPLE TILT BOUNDARIES OF CONTROLLED ANGLE THROUGH THE TEST SECTION OF A KINK SPECIMEN

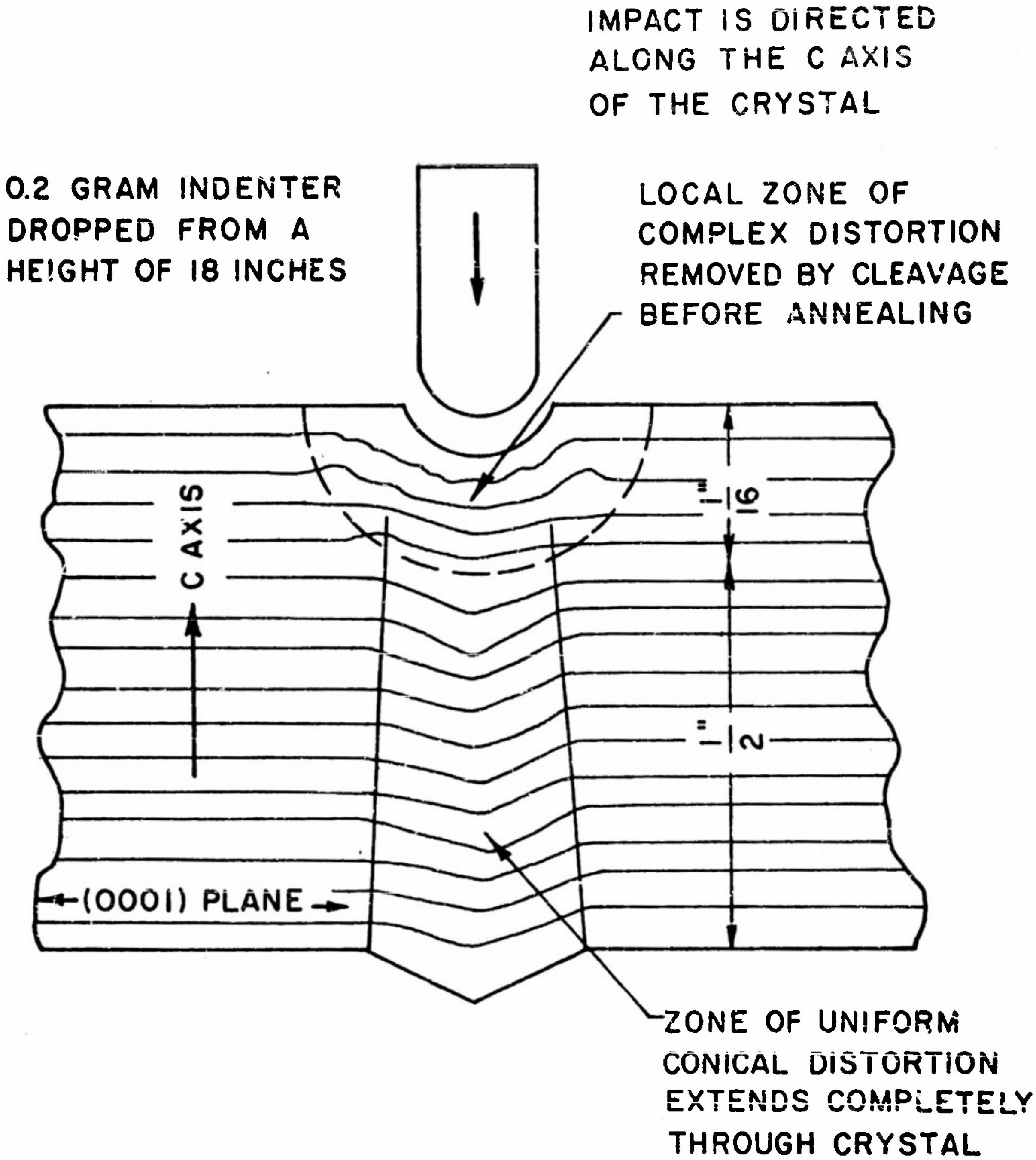


FIG 18 METHOD OF OBTAINING CIRCULARLY SYMMETRICAL ARRAYS OF EDGE DISLOCATION BOUNDARIES EXTENDING THROUGH THE GAGE SECTION OF KINK SPECIMENS.

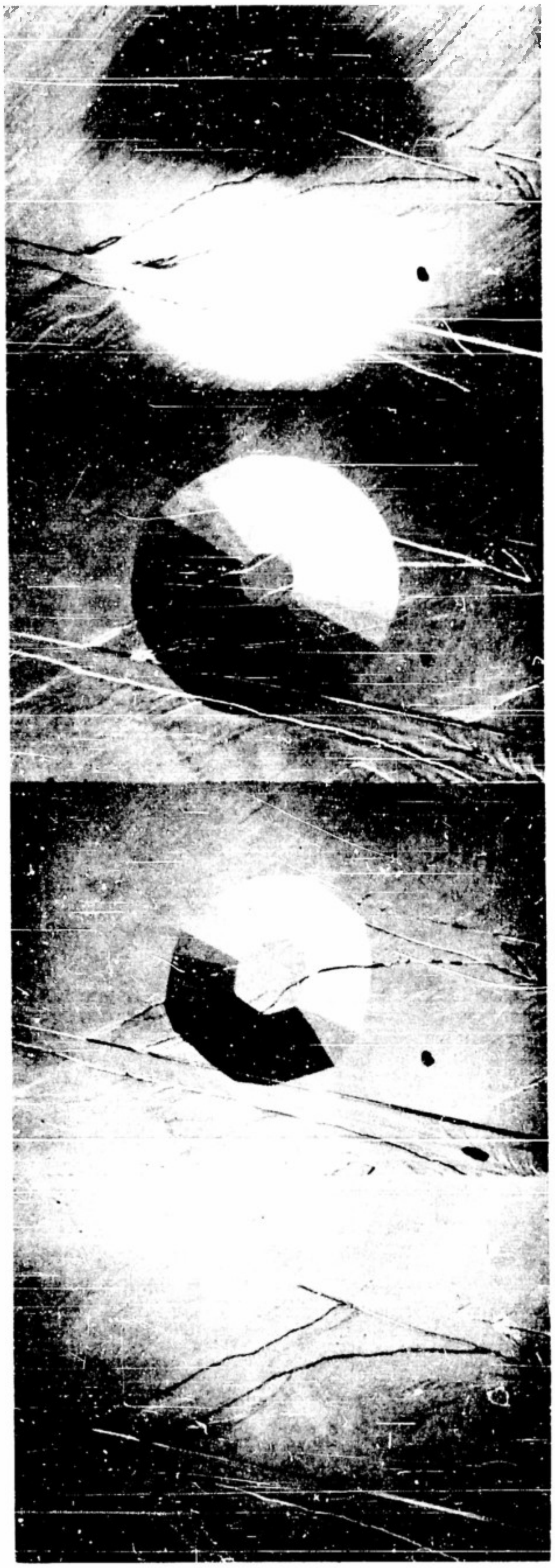
indentations is indicated by the rapidity with which the whole network of boundaries disappears when the crystal is heated. The structural changes observed when such a specimen is heated are shown in the series of photographs in Fig. 19. They were taken on the cleavage face on the convex side. The angle between the flat face of the crystal and the sloping sides of the dent was approximately 1°. As can be seen from the last picture, heating even momentarily to 400° C resulted in complete disappearance of the network of boundaries. Fig. 20 shows the final form of a dent just prior to complete disappearance. Nearly all the remaining boundaries were at right angles to $\langle 2\bar{1}\bar{1}0 \rangle$ directions.

The third type of substructure which was studied was a network of screw dislocations introduced into the (0001) planes by a twist of the specimen about the [0001] axis. Conventional shear specimens were used for these tests since the cylindrical gage section was ideally suited for the introduction of a pure twist deformation. The angle of twist per mm of length along the c-axis could be controlled to $\pm .01^{\circ}$.

All specimens were annealed in a helium atmosphere in the furnace shown in Fig. 21. Two standard annealing cycles were used, 300° C for two hours, and 400° C for one hour. The crystals were always furnace cooled to room temperature after annealing; the maximum rate of cooling, which occurred immediately after turning off the power, was 10° C per minute.

Experimental Results

Cottrell states in his book Dislocations and Plastic Flow in Crystals, "Soft single crystals are not the easiest materials to consider from the theoretical point of view because the process of making them involves the deliberate removal, as far as is possible,



1. DIRECTLY AFTER IMPACT ROOM TEMP.

2. HEATED TO 300°C AND IMMEDIATELY COOLED TO R.T.

3. HEATED TO 350°C AND IMMEDIATELY COOLED TO R.T.

4. HEATED TO 400°C AND IMMEDIATELY COOLED TO R.T.

FIG. 19 COMPLETE RECOVERY OF A CONICALLY DISTORTED VOLUME OF CRYSTAL DURING ANNEALING
50X MAGNIFICATION

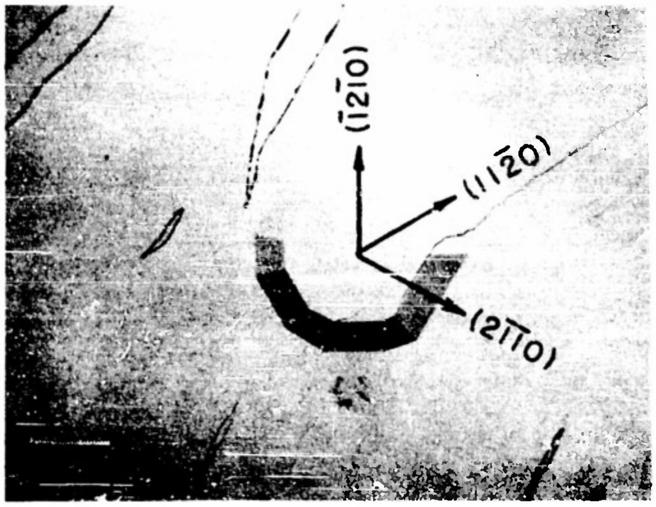


FIG.20 FINAL STAGE IN THE DISAPPEARANCE OF AN INDENTATION DURING ANNEALING. THE MOST STABLE BOUNDARIES ARE THOSE AT RIGHT ANGLES TO $[2\bar{1}\bar{1}0]$ DIRECTIONS.

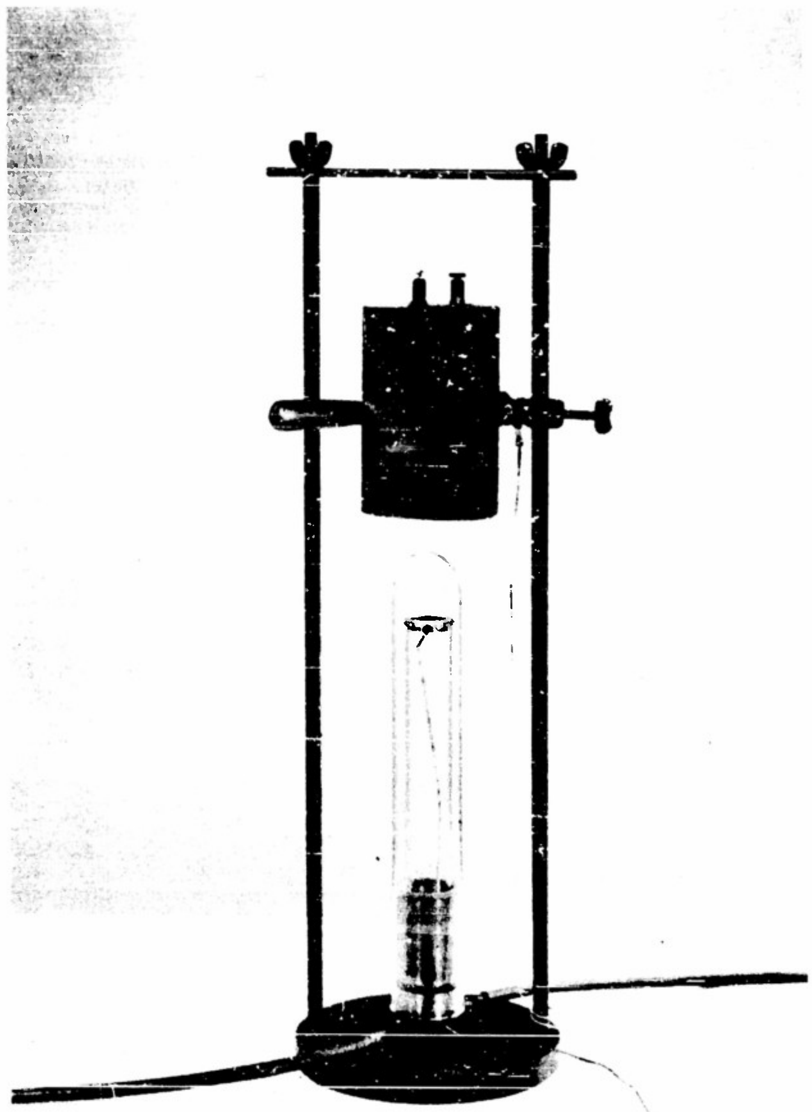


FIG.21 FURNACE USED FOR ANNEALING SPECIMENS IN A HELIUM ATMOSPHERE

of all known sources of hardening and thereby makes the cause of their residual hardness uncertain." Introducing known crystal imperfections and observing the changes in plastic properties caused thereby may eventually lead to a more detailed model of the annealed state of a crystal.

To study the effect of controlled arrays of dislocation boundaries on the yield strength of zinc crystals, the most straightforward approach seemed to be to prepare a series of specimens in which simple arrays of boundaries had been introduced and then measure the yield strength of each specimen. Unfortunately this procedure involved the assumption that the initial properties of all specimens were identical. Previous experience had shown that this is seldom a good assumption. Even when the greatest care was taken to prepare specimens with identical histories, it was not unusual to have a 20 percent scatter in the value of the yield stress. The only way of avoiding this difficulty was to measure the yield stress of each specimen initially and select those with lowest yield strength, assuming them to be the most perfect; all comparisons could thus be made on the basis of results from a single specimen. In order to measure the yield stress, however, it was necessary to plastically deform the crystal. Therefore when a series of tests had to be made, it was essential that the strain hardening could be completely removed by heating the crystal near its melting point. Previous work had shown that with shear specimens it is possible to reproduce the stress strain curve over and over again if the crystal was heated to 400° C after each test. The scatter in yield stress observed in a series of such tests was usually less than half as much as would be obtained from the same number of care-

fully selected separate specimens.

It was first necessary to determine whether similar reproducibility could also be obtained with kink specimens; in this kind of specimen the gage section was terminated by a pair of edge dislocation boundaries which would be continuously increasing in angular magnitude throughout the series of tests and might exert an influence on the form of the stress strain curve. The shaded areas of Figs. 22, 23, 25 and 26 show the scatter observed during several tests of four different crystals. No regular change in successive stress strain curves was observed. The magnitude of the yield stress (as computed from the minimum cross-section between the bottoms of the notches) and the rate of strain hardening in kink specimens were found to be comparable with those obtained with conventional shear specimens.

From previous work[26] it was suspected that the strengthening effect of dislocation boundaries might be greatly influenced by the annealing procedure employed. Since it was desirable to determine the change in yield strength obtained by annealing at different temperatures, it was necessary to know whether the base yield stress was a function of annealing temperature. It was found that for a good crystal (one having a yield of about 30 psi) a change in annealing temperature from 300° C to 400° C resulted in a negligible shift in the level of the stress strain curve. It was therefore possible to determine the base yield stress employing a 400° C annealing temperature and be reasonably certain that nearly the same value would have been obtained if a 300° anneal had been used instead.

Results for Simple Edge Dislocation Boundaries

A pair of pure edge dislocation boundaries lying at right angles to the active slip direction was the first type of substructure to be studied. In this case the dislocations forming the boundary array all had the same Burgers vector as the dislocations contributing to the strain. Fig. 22 shows the effect of introducing a pair of such boundaries on the stress strain curve of a kink specimen. The base curve, as represented by the shaded area, was obtained before the boundaries were introduced; the crystal was annealed at 400° C between tests. The boundaries were then formed by gripping the specimens at the inner set of notches and shearing the crystal a pre-determined amount at liquid nitrogen temperature; the specimen was then annealed at 300° C. The stress strain curve fell exactly in the center of the shaded area enclosing the initial stress strain curves. There was no apparent strengthening effect produced by the boundaries*. The same specimen was then annealed at 400° C and a second stress strain cruve was obtained. A 22% increase in yield stress was observed. The experiment showed that a pair of edge dislocation boundaries has an almost negligible strengthening effect unless the crystal containing the boundaries has been heated to a temperature close to the melting point prior to testing.

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* However, the base yield stress for this crystal was a little higher than that obtained for the most perfect specimens tested. It is likely that a 300° anneal before introduction of the boundaries would have resulted in a slightly lower set of base curves. Therefore it is probable that the pair of one degree boundaries annealed at 300° C did have a small strengthening effect.

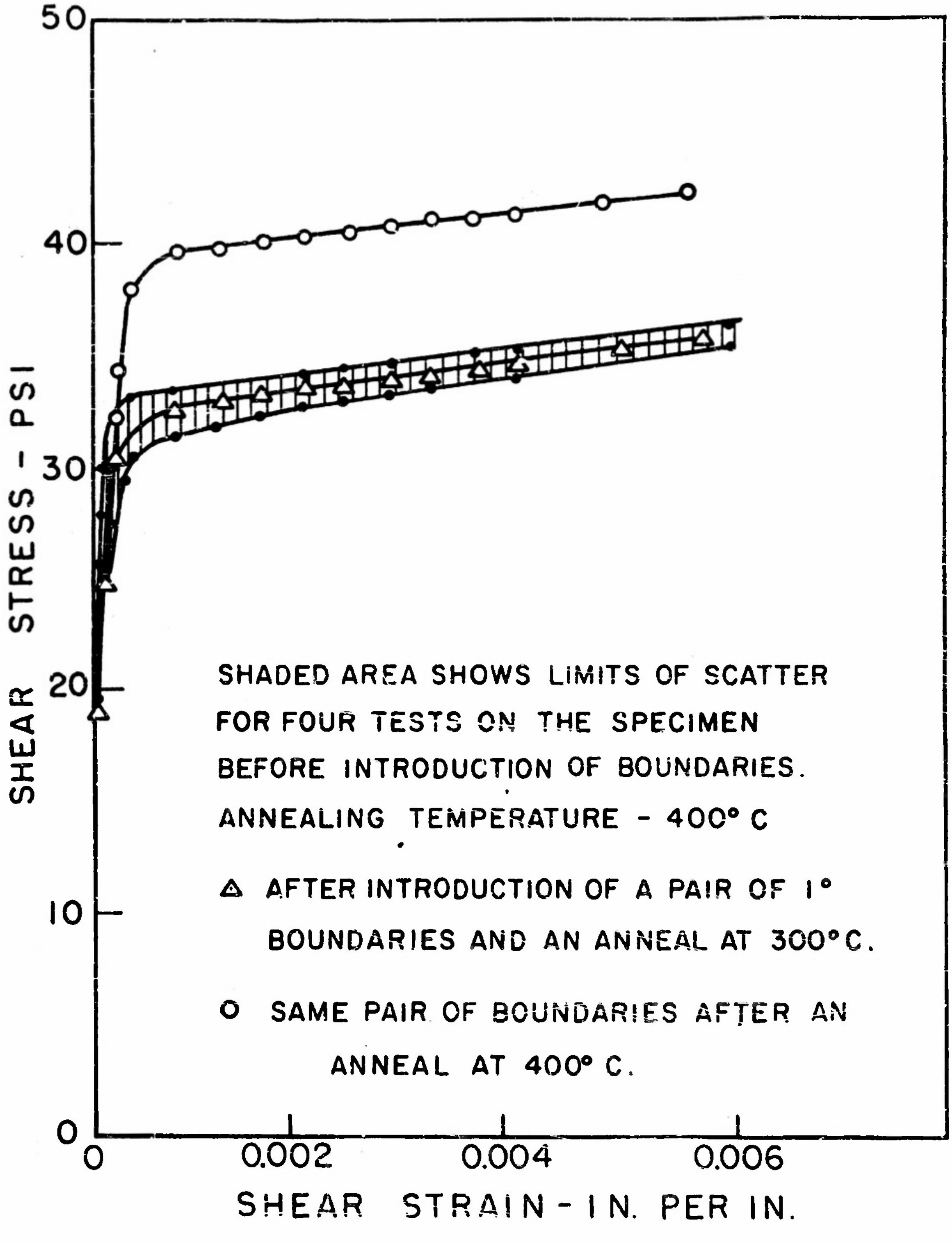

FIG. 22 EFFECT OF ANNEALING TEMPERATURE ON THE STRENGTHENING DUE TO A PAIR OF 1° EDGE DISLOCATION BOUNDARIES

Another interesting feature of these curves was the constant value of the strain hardening coefficient; the presence of substructure affects only the yield stress.

The relationship between boundary angle and strengthening effect was investigated with the same type of kink specimen. Fig. 23 shows the results obtained with boundaries of various angles. All of the tests were preceeded by an anneal at 400^{o} C. The crystal used for this series of tests was initially quite perfect as evidenced by the relatively low base yield stress and the small amount of scatter for the first three tests. Yield stress increase as a function of boundary angle is plotted in Fig. 24. The yield stress rises rapidly at very small boundary angles but appears to saturate by the time an angle of one degree has been reached.

Results for a Complex Array of Pure Tilt Boundaries

In the first set of experiments the dislocations in the boundaries had the same Burgers vector as those contributing to the strain. More complex arrays of boundaries as produced by a conical indentation were also investigated. Boundaries made up of dislocations having all of the three possible Burgers vector directions were present in an indentation such as that shown in Figs. 19 and 20. Dislocation loops in all three slip systems were probably formed by the stress concentration under the indenter used to form the hexagonal array. The dent was smoothly rounded at the top and joined the flat undeformed part of the crystal in a smooth cruve before annealing, as shown in Fig. 19.1.

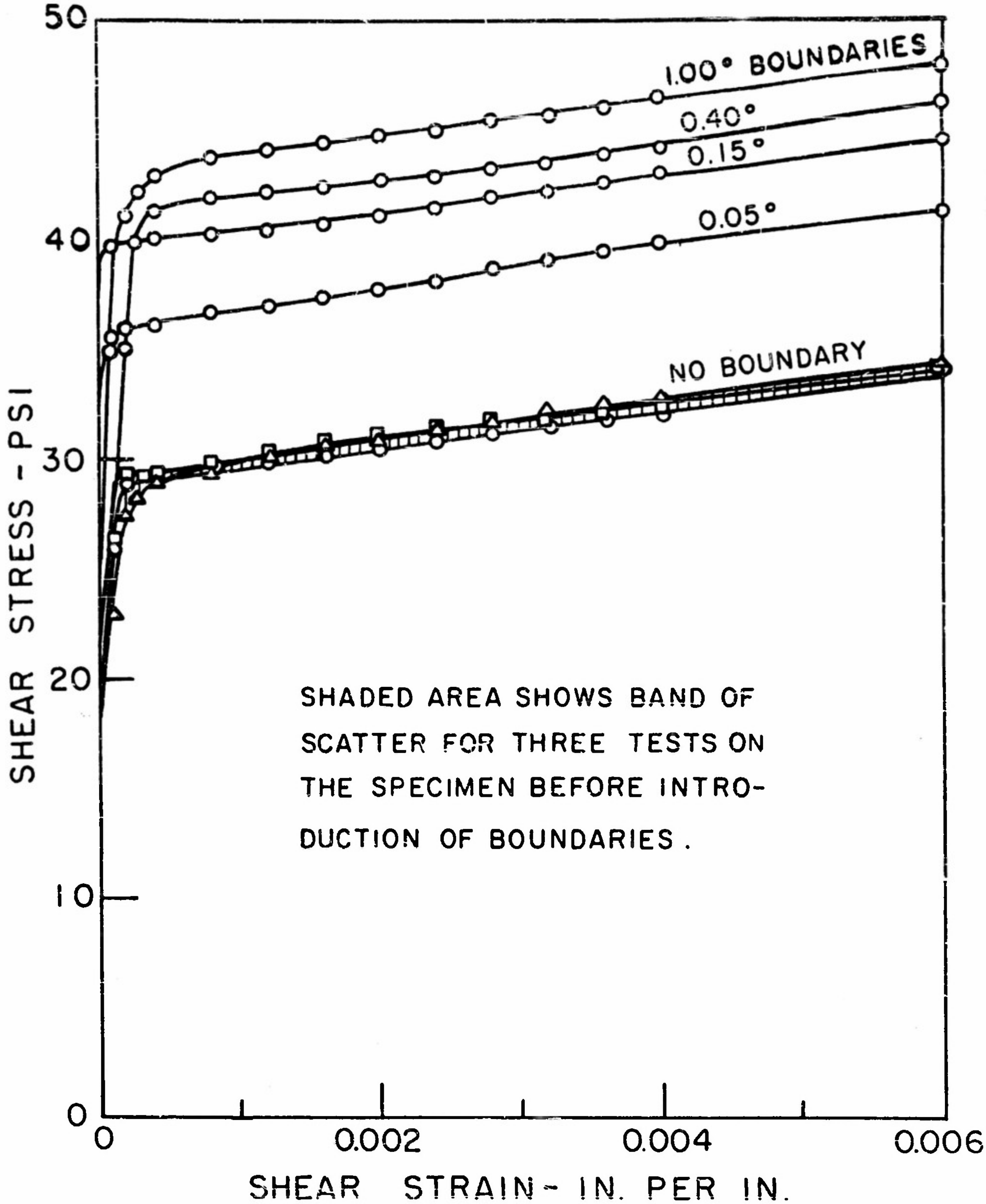

FIG.23 STRESS STRAIN CURVES FROM A SINGLE SPECIMEN SHOWING THE EFFECT OF INTRODUCING A PAIR OF PURE TILT BOUNDARIES OF CONTROLLED ANGLE - CRYSTAL ANNEALED AT 400° C BEFORE EACH TEST TEMPERATURE OF TESTING - 196° C.

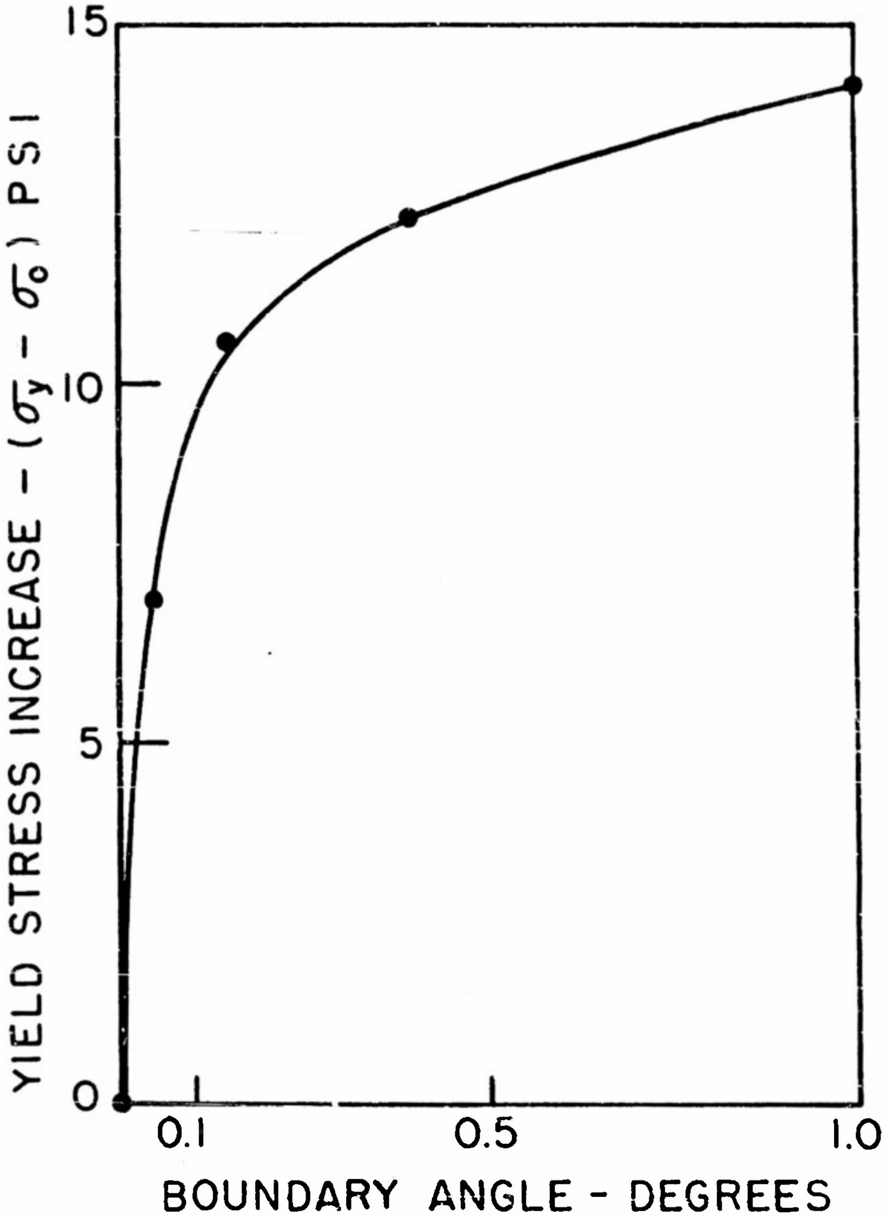

FIG.24 INCREASE IN YIELD STRESS AS A FUNCTION OF BOUNDARY ANGLE FOR A PAIR OF SIMPLE EDGE DISLOCATION BOUNDARIES ANNEALED AT 400° C.

The geometry of the dent could be explained on the basis that a random circumferential distribution of dislocation loops of all three possible slip vectors was formed, each one passing through a point near the center of the dent and being tangent to the outside circumference. When a dent of this sort was heated, polygonization occurred; radial and circumferential boundaries formed most of which were not perpendicular to the a-axes directions. The dislocation structure of most of the boundaries was therefore complex. A boundary which is not perpendicular to one of the a-axis directions contains dislocations of two different Burgers vectors each with some screw and some edge character. In a pure tilt boundary following a non-crystallographic direction the screw components of the various dislocations in the boundary cancel one another so there is no over-all twist in the plane of the boundary. On further heating the dislocation lines all tended to shorten themselves. A flat surface formed at the top and grew in size and the outside circumference moved in. In the meantime, wherever two radial boundaries touched one another at the inner ring the junction moved rapidly down until it met the outer ring. The two boundaries thus became one whose angle was the sum of the two component boundaries. Such a combination resulted in a decrease in total boundary energy as can be seen from the theoretical equation for energy of small angle boundaries derived by Read and Shockley:[27]

$$E = E_0\theta(A - \ln\theta)$$

where E is the boundary energy, E_o and A are constants and θ is the boundary angle. At a later stage it became apparent (Fig. 20) that the most stable boundaries were those lying closest to the directions at right angles to the three close packed directions, thus demonstrating clearly that boundaries composed of identical dislocations in pure edge position have the lowest boundary energy. Finally, the most spectacular feature of these dents was their quick disappearance when heated to 400° C. In the series shown in Fig. 19, the furnace was heated at its normal rate until it reached 400° C and then immediately shut off. Although the crystal was in the neighborhood of 400° C for only a few minutes the indentation had completely disappeared, leaving a cleavage surface that was just as flat as if the dent had never been made.

The effect of such a complex array of edge dislocation boundaries on the stress strain curve is shown by Fig. 25. The base curve before introduction of a dent through the gage section was determined for a 300° annealing temperature since it was impossible to anneal the specimens at 400° C without completely eliminating the array of boundaries. After denting, the crystal was annealed at 300° C, producing an array of boundaries similar to that in the third picture of Fig. 19. The strengthening effect of such an array annealed at 300° C was very small. Thus the boundaries of unlike Burgers vector appeared to be no more effective as a barrier to slip than the simple edge boundaries when annealed at 300° C. Fig. 2, on the other hand, shows that an even more complex

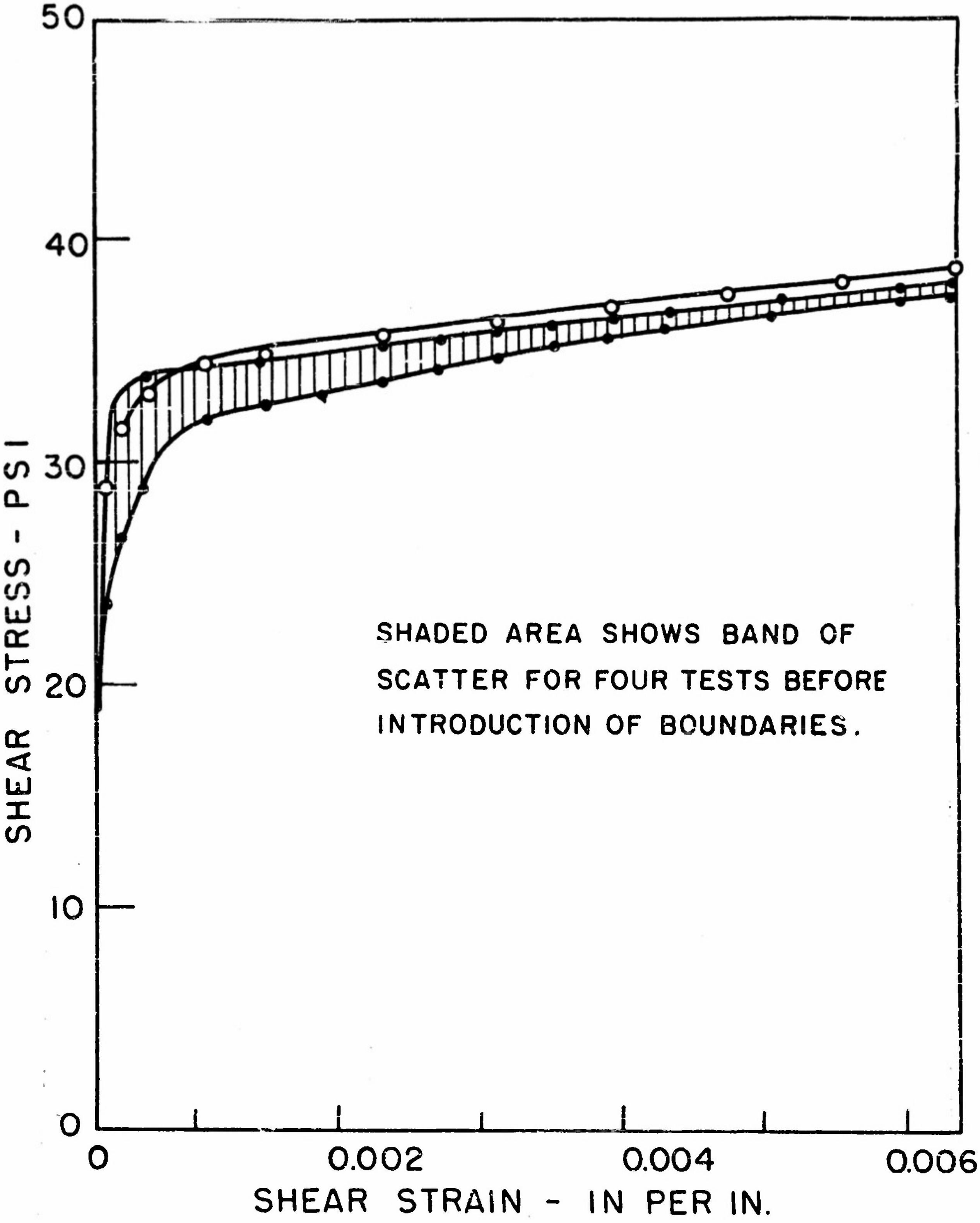

FIG.25 EFFECT OF A CIRCULARLY SYMMETRICAL ARRAY OF COMPLEX TILT BOUNDARIES AFTER ANNEALING AT 300° C.

array of boundaries which was formed by a concentrated static load at high temperatures through the gage section of a normal shear specimen was able to strengthen greatly the specimen when annealed at 400° C. The strengthening effect of complex arrays of edge dislocation boundaries was therefore qualitatively the same as that for pairs of simple edge boundaries.

Effect of Screw Dislocation Arrays

An interface across which a pure tilt of the crystal lattice occurs can be accounted for geometrically by an array of edge dislocations. In the general case the lattices on two sides of a boundary could only be brought into coincidence by a twist about an axis normal to the plane of the boundary in addition to a tilt about an axis in the plane of the boundary. A second simple case therefore, is a pure twist boundary which, as suggested by Burgers,[10] must consist of an array of screw dislocations.

The effect of introducing an array of screw dislocations into a kink specimen by twisting about the c-axis is shown in Fig. 26. The base curve was established and the specimen was twisted 0.16° per mm of length along the c-axis. After annealing at 400° C the yield stress was raised from its base value of about 32 psi to 52 psi and the apparent rate of strain hardening was increased by a factor of 4. Examination of this specimen revealed that the crystal had deformed as shown in Fig. 27. The reason for the apparent high rate of strain hardening was the fact that all the strain had been confined to two narrow bands immediately under the notches. The

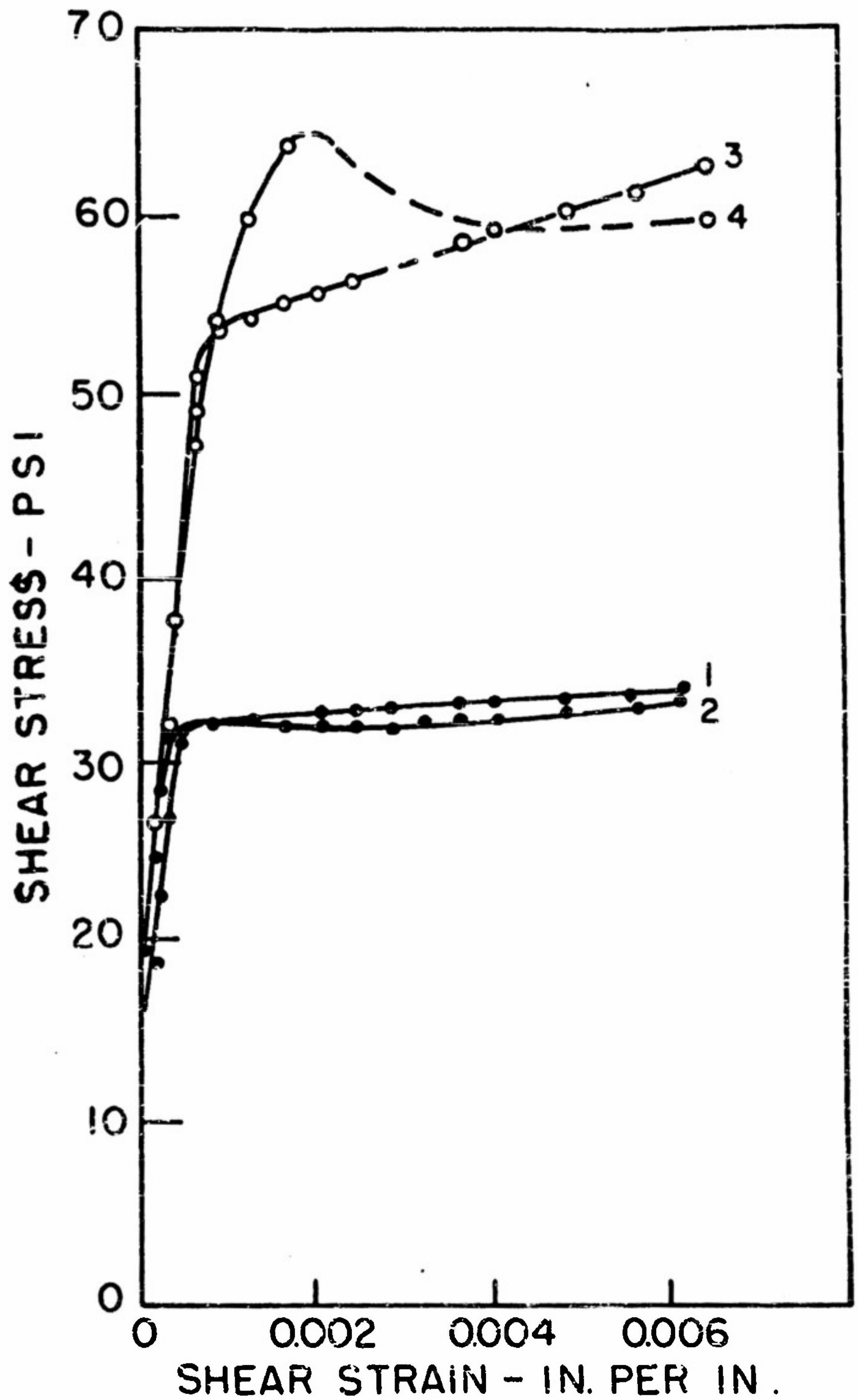

FIG. 26 EFFECT OF A TWIST OF .16° PER mm. ABOUT THE NORMAL TO THE SLIP PLANE - ANNEALED AT 400° C. TEST TEMPERATURE - 196° C.

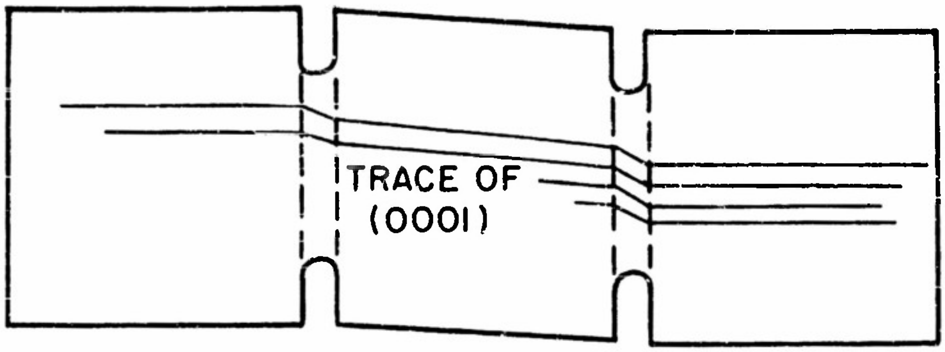

FIG. 27 SKETCH SHOWING MODE OF DEFORMATION IN THE THIRD TEST OF FIG. 26 - DEFORMATION WAS LOCALIZED IN TWO NARROW BANDS UNDER THE NOTCHES.

effective gage length was about one-fourth of the total. Therefore, if the data were to be plotted on the basis of this reduced gage length the curve would have the normal slope. After another anneal at 400° C the yield did not occur until 64 psi at which stress there was a sudden drop in load accompanied by rapid strain. This type of yield is usually explained on the basis of Cottrell locking[29] of dislocations by foreign atoms. These results suggested another way in which a yield of this type could originate. Initially the deformation was probably confined to the narrow bands under the notches. At a critical stress dislocations which had been held up by the two inner boundaries were able to break through, suddenly spreading the strain across the entire gage section. This type of yield was frequently observed with kink specimens on the second and subsequent tests. A more typical example is shown in Fig. 28 where the phenomenon is confined to the range of strain below .001. With the normal type of shear specimen this type of yield is seldom observed even after many cycles of straining and annealing. The conditions of growth and annealing were the same for these kink specimens as for previous work on normal shear specimens. Therefore, it seems unlikely that there was a difference in dissolved nitrogen concentration.

Normal shear specimens were more suited to a quantitative investigation of the strengthening effect due to an array of screw dislocations. Fig. 29 shows the curves obtained from a cylindrical shear specimen before twisting and after twisting .11° per mm and

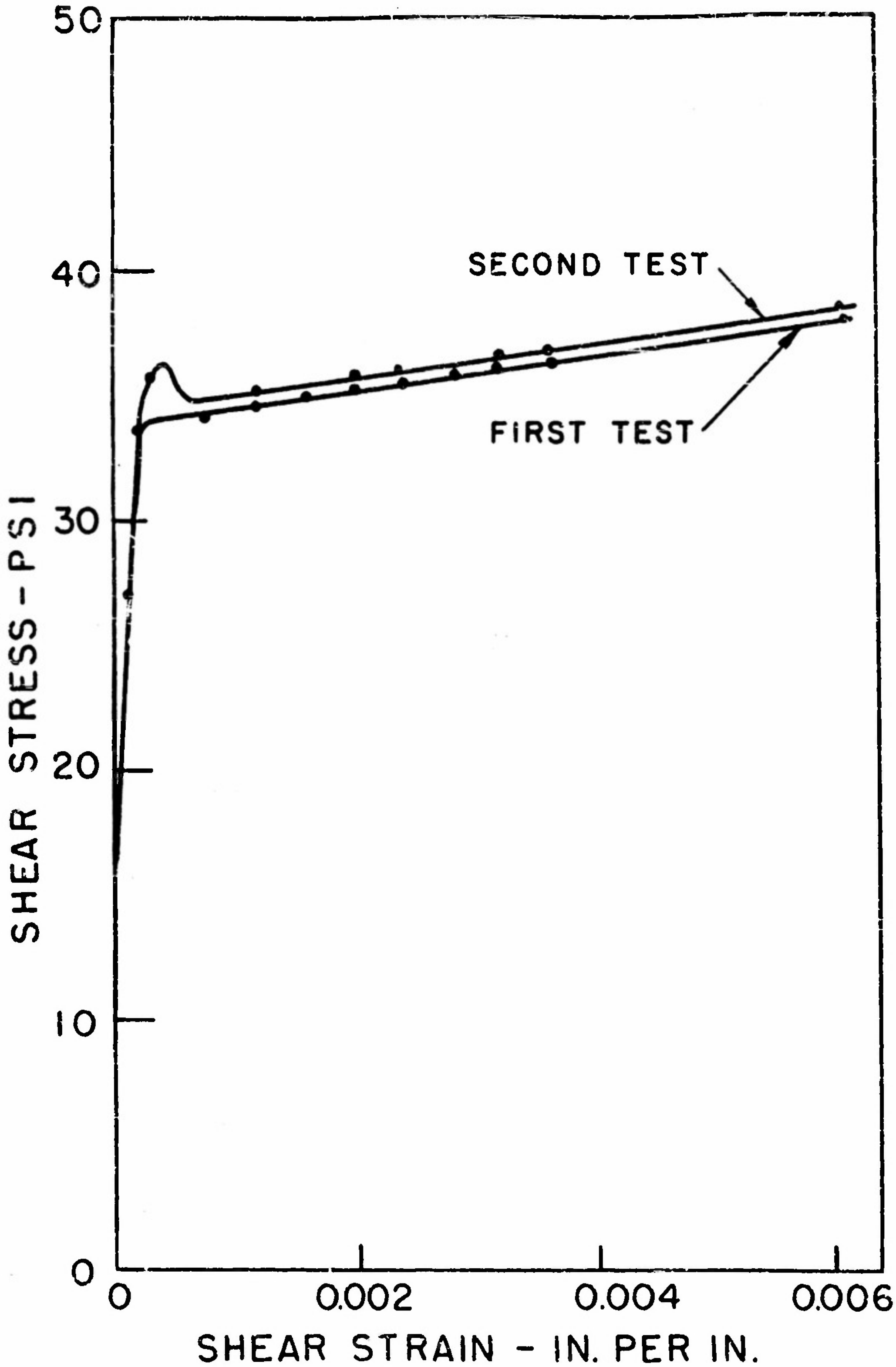

FIG. 28 DISCONTINUOUS YIELDING OF THE TYPE USUALLY ATTRIBUTED TO THE PRESENCE OF DISSOLVED NITROGEN.

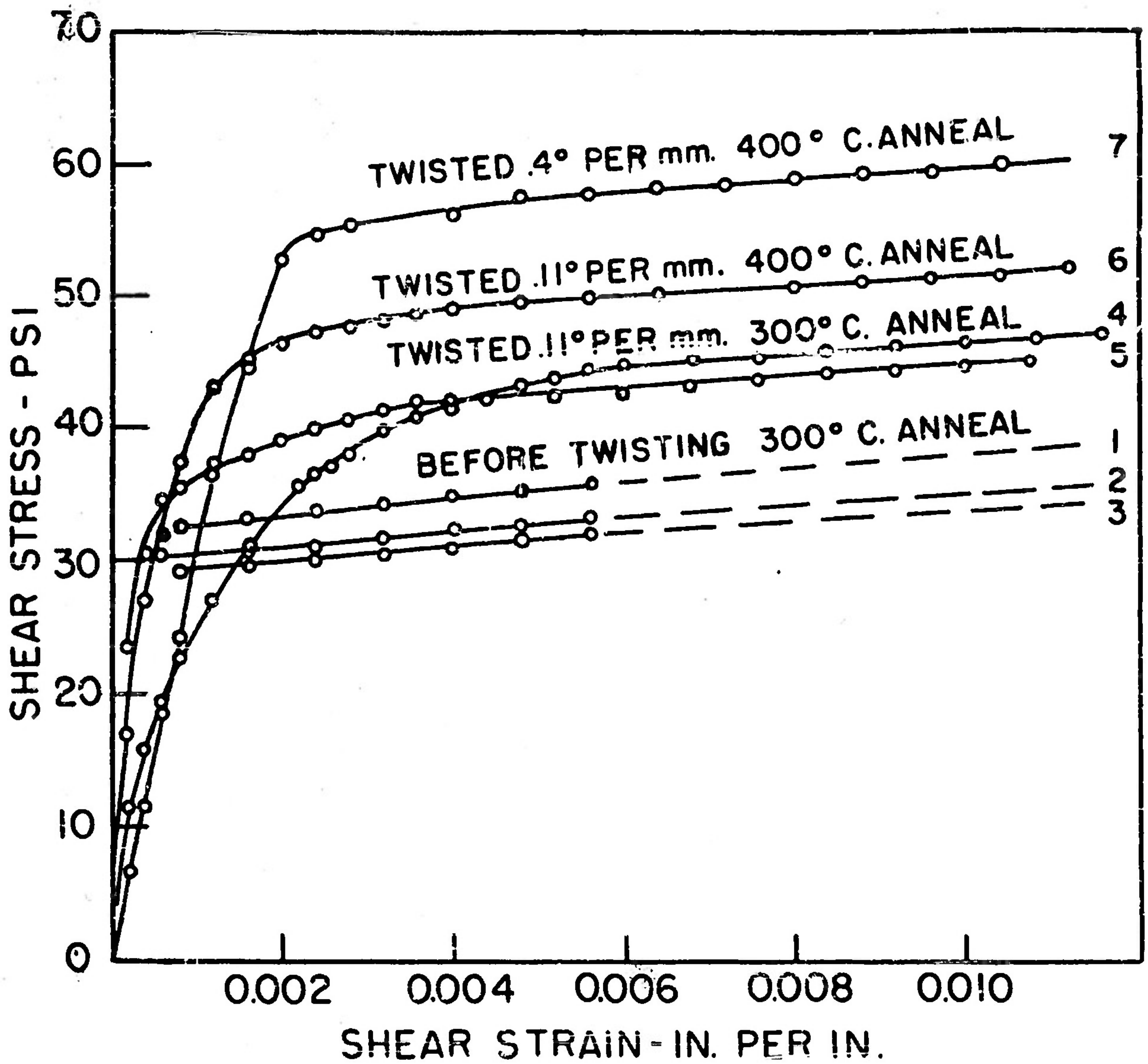

FIG. 29 EFFECT OF TWISTING ABOUT AN AXIS NORMAL TO THE SLIP PLANE ON THE STRESS-STRAIN CURVE

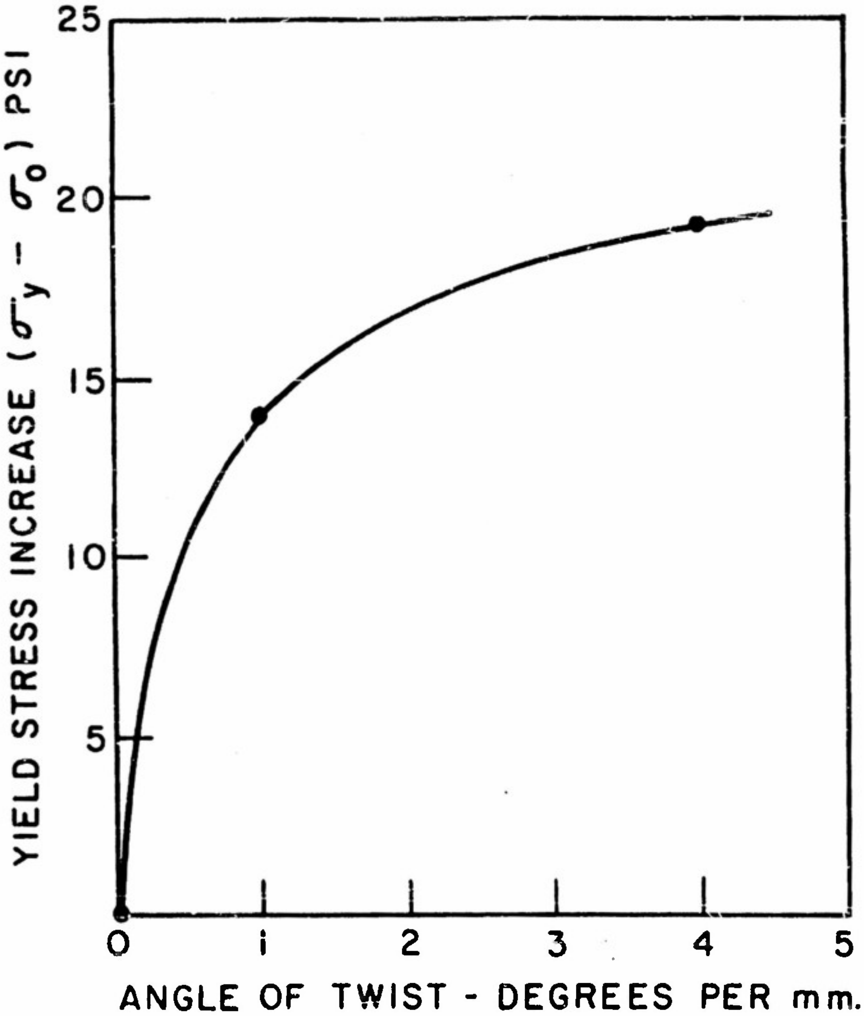

FIG. 30 INCREASE IN YIELD STRESS AS A FUNCTION OF TWIST ANGLE

$.4^{\circ}$ per mm. 300° annealing was used for the base curves and for the first two curves after twisting $.11^{\circ}$ per mm. Unlike the edge dislocation arrays the screw array had a significant strengthening effect even after 300° annealing. After twisting, the yield was poorly defined with the linear part of the curve only being reached after a strain of .006. Again as in the case of edge arrays no change in the slope of the linear part of the curve was observed. 400° annealing produced an additional increment of strengthening as shown by the third curve for the $.11^{\circ}$ twist. The increase in yield stress as a function of twist angle is plotted in Fig. 30. The relation is analogous to that obtained for edge boundaries. A sharp rise for small angles of twist followed by decreasing increments in strengthening effect for additional increments of twist.

Discussion

It is a good assumption that all metal crystals of a size ordinarily used for mechanical tests contain a network of dislocations. The uniformity with which these dislocations are disposed throughout the crystal determines the microscopic and macroscopic perfection of the crystal. X-ray and metallographic evidence indicates that in actual crystal specimens this distribution is usually far from uniform. Concentrations of like dislocations forming dislocation boundaries of macroscopic proportions usually exist.

The yield stress (stress required to cause slip) of a hypothetical crystal which is perfect except for a single Frank-Read source of length l would be given[27] by:

$$\frac{Gb}{l}$$

where G is the shear modulus, b is the Burgers vector of the dislocation and l is the length of the source. Taking G as 10^7 psi, b as 10^{-8} cm and a typical value of the yield strength of zinc crystals as 30 psi then l is about 3×10^{-3} cm. In such an ideal crystal the transition from elastic to plastic behavior would occur suddenly. The yield stress could be defined unambiguously as the critical stress, $\frac{Gb}{l}$, and slip would continue on a single slip plane as long as the applied stress was maintained at or even below this critical value. The dislocation loops spreading out across the slip plane from the single source would encounter no obstacles and therefore no strain hardening would occur.

The behavior of real metal crystals is quite different. Deviations from elastic behavior occur at almost vanishingly small stresses. As the applied stress is raised the non-elastic portion of the strain grows at an ever increasing rate. At larger strains, for single crystals deformed under conditions where a simple slip deformation is achieved, $\frac{d\sigma}{d\varepsilon}$ is small and remains constant with increasing strain. The transition from the initial rapidly rising portion of the stress strain curve to the linear portion may be sudden or gradual depending on the geometrical perfection of the crystal and the test conditions. When a sudden break in the stress strain curve occurs

this is usually referred to as the yield stress. When the transition is gradual it is necessary to make an arbitrary definition of yield stress. For single crystals giving substantially linear stress strain curves at large strains the yield stress is frequently taken as the stress corresponding to the point where an extrapolation of the linear part of the stress strain curve intersects the modulus line. In the following discussion this will be the meaning ascribed to the term: yield stress.

Unlike the ideal crystal cited above, the factors which determine the yield stress in real crystals are not at all clear. In addition to the length of available dislocation sources there are impediments to the free motion of dislocation lines to be considered. According to a recent suggestion by Mott[28] the main impediment to motion of dislocations in the basal planes of hexagonal metals may be a high density of c-axis screw dislocations. The idea of a frictional force due to such localized obstacles was developed by Mott[28] into a theory which predicts non-dynamic operation of Frank Read sources and results in a linear relation between stress and strain provided dislocations are trapped within the crystal. Another effect which appears to be of importance concerning the mobility of dislocations is their interaction with impurities both in the manner suggested by Cottrell[29] in which immobile atmospheres of foreign atoms tend to collect around dislocations and in the way discussed by Mott[30] in which foreign atoms contribute to the frictional force resisting the motion of dislocations. The generation of lattice vacancies

by the crossing of screw dislocations[31] may also contribute to the frictional term. There is still, however, no quantitative theory for plastic flow in soft hexagonal crystals.

The results of this investigation illustrate the fact that the shape of the stress strain curve is determined not only by the submicroscopic distribution of dislocations forming the mosaic structure of the crystal but also to an important extent by concentrations of dislocations on a much larger scale.

Edge Dislocation Arrays

A simplified model assuming interactions only between neighboring dislocations in a small angle boundary can be used to estimate the stress required to propagate a slip line through such a boundary.

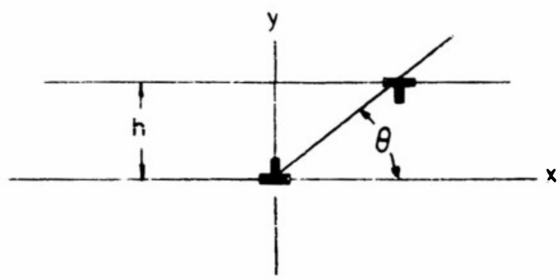

Fig. 31. Interaction between two parallel edge dislocations moving on parallel slip planes.

The shear stress acting along the plane y = h due to the edge dislocation at the origin is given by Read[27] as:

$$\sigma = \frac{Gb}{2\pi(1-\nu)} \cdot \frac{x^3 - h^2x}{(x^2+h^2)^2} = \frac{Gb}{2\pi(1-\nu)\rho} \cos\theta \cos 2\theta$$

$$\sigma_{max}\left(\theta = \frac{3\pi}{8}\right) \cong \frac{Gb}{8\pi(1-\nu)h}$$

Taking the shear modulus $G \approx 10^7$ psi, Poisson's ratio $v \approx .33$, the Burgers vector $b \approx 2 \times 10^{-8}$ cm, and the vertical distance h between dislocations as 10^{-6} cm (about equal to the spacing of dislocations in a one degree boundary), the order of magnitude of the local stress needed before the two dislocations can pass one another is about 10^4 psi. Since the observed flow stress for a crystal containing a one degree boundary was of the order of 50 psi a stress concentration of about 200 would be required on the basis of the above model before the slip line could break through. A pile up of about 200 dislocations in the slip plane would create a local stress of the required magnitude[32]. For a $.1^{\circ}$ boundary the stress to break through would be only about 10^3 psi requiring a pile up of only 20 dislocations.

The simple two dislocation model would be isotropic in the sense that if one of the dislocations was in some way fixed the stress required to move another dislocation past it on the parallel slip plane a distance "h" away would be the same regardless of whether the two dislocations were like or unlike. However, when the stress fields due to second and third etc., nearest neighbors in a dislocation boundary are considered it appears that such a boundary would be a more effective barrier than the simple model for dislocations of opposite sign to the boundary dislocations and a much less effective barrier than the simple model for dislocations of like sign to those in the boundary. In any case considering the fact that a thousand or more dislocations are throught to pass across a single slip plane to produce the observed surface steps[32][33] it seems that the number of piled up dislocations needed before the slip could break through a boundary would be a small fraction of the total number moving in each active slip plane.

Furthermore, since the interaction forces between dislocations vary inversely with the first power of the distance between them, the strengthening effect of a dislocation boundary might be expected to be a linear function of boundary angle for small boundary angles. The experiments showed that this was not the case. The strengthening effect of annealed boundaries increased very rapidly at extremely small boundary angles and reached almost its maximum value for a boundary angle of only one degree.

The observed results might be explained by considering the boundary as merely a sort of nucleus which initiates the formation of a more effective barrier. Perhaps groups of dislocations held up against the boundary in neighboring slip lines are able to build up to sufficient size to be able to interact directly with one another; each group acting at large distances as if it were a single dislocation of Burgers vector nb where n is the number of dislocations in the group and b is the Burgers vector of each individual dislocation. The main hardening effect might then be due to the longer range interactions of large groups of dislocations in neighboring slip lines. On this basis the large strengthening effect of a $.05^{o}$ boundary and the small increase in strengthening effect for boundaries above 1^{o} seems reasonable.

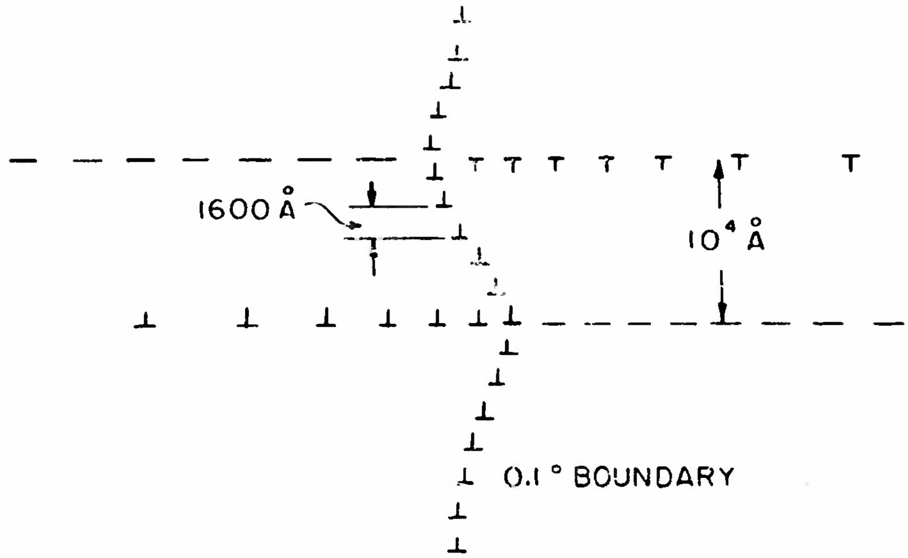

Fig. 32. Suggested mechanism whereby a dislocation boundary could nucleate large dislocation groups which would be unable to pass one another because of their mutual repulsion.

Little or no strengthening effect was observed when a specimen was tested immediately after introduction of a boundary without an intermediate anneal. Plastic flow began at extremely small stresses and the linear part of the stress strain curve was only reached after a quite considerable strain. The most likely structure for a 1° boundary formed by plastic flow at liquid nitrogen temperature is shown in Fig. 33. It is analogous to 1000 parallel .001 degree boundaries. The outer dislocations of each horizontal group would be only weakly held and could act as a source of plastic flow at low stresses. It should be emphasized that in these experiments the total strain was usually limited to .006 and even for those tests where the linear part of the curve was approached gradually the pre-yield-stress

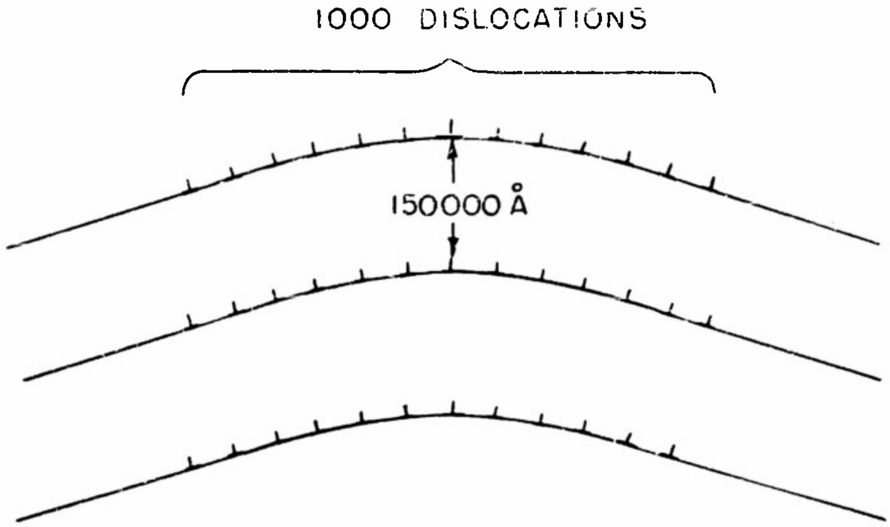

Fig. 33. Suggested structure of a 1° boundary immediately after formation at liquid nitrogen temperature.

plastic flow was confined to the range of strain below about .002. This strain corresponds to a motion of one dislocation across a set of planes separated by 1000 Å or by 100 dislocations on each of a set of planes 10^5 Å apart. Therefore a considerable part of the total strain might have been due to motion of dislocations already present.

Screw Dislocation Arrays

The exact distribution of screw dislocations introduced into the slip planes by twisting about the c-axis was less clearly defined than in the case of the edge arrays. In the hexagonal structure it is most likely a crossed grid of screw dislocations forming a trigonal network in each of the active slip planes. The distribution of rotational slip among the possible parallel slip planes was also unknown. However, assuming that the spacing of active planes was about the same as for unidirectional slip, (about 10^5 Å) then the spacing of screw dislocation lines per slip plane for a twist of $.1^{\circ}$ per mm would be about 50 Å.

The contribution of such an array to strength of a crystal is probably through an increase in the frictional term discussed by Mott(30). The rapid rise of yield stress for small twists followed by a smaller increment for each additional increment in angle of twist may mean that the average spacing of active slip planes during twisting does not change rapidly with increasing twist; the spacing of screw dislocations within each active slip plane is merely decreased. In this way the period of the internal stress field in the material between the active planes of rotational slip would increase with angle of twist but the magnitude might remain essentially constant. The considerable pre-yield stress plastic flow observed with this type of array may be due to motion of some segments of the screw dislocation arrays.

Summary and Conclusions

Three types of dislocation array were introduced into zinc crystal specimens and the effect of each on the stress strain curve was investigated. For simple edge dislocation boundaries lying at right angles to the active slip direction a quantitative study of yield stress as a function of boundary angle was made. The presence of a pair of .05° simple edge dislocation boundaries across the gage section of a specimen caused a 25% increase in the yield stress. Further increases in boundary angle were accompanied by smaller increments in strengthening effect. A saturation value of about 50% increase in yield stress appeared to be approached for boundary angles above one degree. For a given boundary angle the strengthening effect was determined by the temperature at which the specimen containing a boundary had been annealed. A pair of one degree boundaries introduced by plastic bending at liquid nitrogen temper-

ature produced no strengthening at all if the crystal had been heated only to room temperature, very little strengthening if heated to 300° C, but a large increase in yield stress if heated to 400° C. More complex arrays of pure tilt boundaries produced by a conical indentation passing through the gage section of the specimen showed qualitatively similar results.

An array of screw dislocations was introduced into the (0001) planes by twisting about the c-axis. Quantitative measurements of strengthening effect as a function of angle of twist gave results similar to those for simple edge dislocation boundaries; the first small increment of twist angle produced the largest increment in yield stress. A somewhat smaller dependence on annealing temperature was observed for this type of array.

Two further qualitative observations were common to all the types of dislocation array studied: (1) no change in the slope of the linear part of the stress strain curve was observed, (2) in general the yield was not as sharp as in macroscopically perfect crystals; appreciable plastic strain accompanied by rapid strain hardening occurred before the linear part of the curve began.

The following conclusions can be drawn from the results.

(1) The yield strength of a metal crystal is determined not only by the submicroscopic and microscopic distribution of dislocations but also by concentrations of dislocations on a macroscopic scale forming isolated small angle boundaries.

(2) Rate of strain hardening at larger strains is not affected either by isolated edge dislocation boundaries or by a more uniformly distributed array of screw dislocations in the slip planes. It therefore seems probable that the small linear rate of hardening of hexagonal crystals is due to a process occurring on a submicroscopic scale.

(3) The detailed structure of a small angle boundary, as determined by prior strain and thermal history, determines its effectiveness as a barrier to slip.

(4) The relatively large strengthening effect of a pair of $.05^{\circ}$ boundaries makes it seem improbable that the effect can be due solely to interactions between the boundary dislocations themselves. A possible interpretation is that the boundary acts as a nucleus for the formation of a more effective barrier.

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