SUBSTRUCTURE AND RECRYSTALLIZATION OF DEFORMED CRYSTALS OF HIGH-PURITY SILICON-IRON

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I. INTRODUCTION

Recent advances in the techniques of transmission electron microscopy have made it possible to directly observe the dislocation substructures of metals.

Many studies have been undertaken to relate the mechanical behavior of metals to the substructure during plastic deformation. Some investigators have followed the changes in the substructure and mechanical properties during annealing treatments. As a result of these studies, various mechanisms for recrystallization have been proposed based on observations of the modification of the substructure when the deformed metals are heated.

It is not the purpose of the present paper to examine the several proposed mechanisms for recrystallization but, rather, to describe in some detail the derivation and modification of substructure in a deformed and annealed single crystal of silicon iron. It will be shown that the substructure, as evolved during deformation, directly prescribes the state and orientation of nuclei for primary recrystallization. This is not to say that the presently described mechanism for recrystallization holds for all metals and all crystal orientations, however. Additional effort will be required to determine whether there is a single mechanism for recrystallization or whether the mechanism is dependent upon the initial orientation of the crystal being deformed and the type and extent of deformation. This aspect of the situation will be briefly explored in context with the proposed mechanisms for recrystallization.

II. EXPERIMENTAL PROCEDURE

The single crystals used in this study all had a (100) [001] orientation with respect to the rolling plane and rolling direction. The crystals contained 3% silicon and were prepared as follows. High-purity iron and silicon were melted and cast, in vacuo, into slab ingots. The ingots were hot-and cold-rolled to sheet, 0.012" thick. Portions of the sheet were annealed in vacuum, hydrogen, or argon at 1200°C to effect growth of grains with (100)

planes parallel to the sheet surface by means of the surface energy driving force (1-4). Crystals with (100) planes within 1 degree of the plane of the sheet were removed from the sheet and rolled, at room temperature, parallel to the [001] direction to reductions of 10% to 90% of thickness.

Samples of the rolled crystals to be used for transmission electron microscopy were thinned by electropolishing successively smaller areas of the sample until a small hole formed (5). The thinnest parts of the sample, adjacent to the hole, were removed and placed in the microscope. Other portions of the rolled crystals were annealed in a salt bath at temperatures from 535°C to 700°C. These were thinned after annealing. The majority of the transmission microscopy was performed on a Phillips electron microscope type EM-100B at 100 KV.

III. EXPERIMENTAL RESULTS

a. Substructure of Rolled Crystals

Figure 1 shows the dislocation substructure and the beginning of cell formation in a crystal rolled to 10% reduction. The planar orientation, as determined by selected area diffraction, is (100) and a <011> direction of the crystal is parallel to the <011> trace superimposed on the figure. It appears that the dense tangles and incipient cell walls consist of short segments of edge dislocations parallel or nearly parallel to the <112> directions (short traces in Fig. 1). There are also some longer segments of screw dislocations (areas marked a) parallel to the <011> trace. The bent dislocations in areas b and c are probably screws that are cross-slipping on to other {110} planes. The configuration of cross-slipping screw dislocations has been described by Swann and Nutting (6).

Rolling to 20% reduction leads to the formation of distinct cells, 0.2-0.3 microns in diameter as shown in Fig. 2. These cells are a factor of 5 smaller than the cells found in iron deformed a comparable amount (7)*.

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* It is possible that the addition of silicon to iron results in a decrease in cell size as is the case for the addition of aluminum to copper (8).

The cell walls contain a higher density of dislocations than was the case for the crystal rolled to 10%. The disorientation across the cell walls is less than 1 degree. Surveys of the crystal with the diffraction beam showed no differences of orientation greater than 1 degree within the crystal in agreement with X-ray diffraction data.

When rolled to 50% reduction, the cells are found to be elongated and the cell walls are aligned parallel to the rolling direction. The elongated cells can be seen in Fig. 3. The cell elongation is roughly a factor of 2 and appears to occur not so much by the enlargement of the cells in the rolling direction as by the breaking of cross tangles or walls to combine two cells into one. Several examples of cross-wall breaking may be seen in the figure. The width of the cells remains constant at 0.2-0.3 microns.

While the same elongated cell structure exists everywhere within the sample, in certain regions of the crystal substantial reorientations are found. The diffraction pattern in Fig. 3 illustrates the nature of the reorientation which may amount to as much as 25 degrees. The diffraction pattern, which shows a rotation of 10°, encompassed approximately 15 cells. Since the (100) plane is parallel to the plane of the sample, the reorientation may be characterized as a rotation about an axis normal to the (100) or rolling plane. The average angle of disorientation per cell wall is 0.6-0.7°.

The regions containing the reorientations we have called "transition bands". The transition bands alternate with regions wherein there is no orientation shift and we have classified these latter regions as "deformation bands". As far as can be determined, the substructure in the transition bands is identical to the substructure in the deformation bands. This situation changes, however, with increasing deformation.

Figure 4 shows the structure of a transition band in a crystal rolled to 60% reduction. The further elongation of the cells into sub-bands is apparent as is the narrowing and sharpening of the walls between the sub-bands. However, as seen in Fig. 5, the cell structure within the deformation bands has altered considerably in that the average cell diameter has been reduced to 0.1-0.2 microns and in many areas the cells have been filled with dislocations. The diffraction pattern, obtained from this deformation band, shows no significant orientation shifts.

Elimination of cells in the deformation bands is virtually

complete at 70% reduction and the width of the transition bands is considerably reduced as shown in Fig. 6a. This figure shows a transition band and the orientations of the deformation bands on either side of it (Figs. 6b and 6c) in a crystal rolled to 70%. The transition band now consists of clearly defined sub-bands, still only 0.2-0.3 microns wide, running parallel to the rolling direction. Diffraction patterns show the deformation bands to have (100) planes parallel to the plane of the sample and a change in orientation of the [001] direction of about 40 degrees across the transition band. This rotation occurs as a series of small, discrete orientation changes associated with the individual low-angle boundaries of the transition band. Since there are 18-20 sub-bands within the transition band, the average angle of disorientation of the low-angle boundaries is approximately 2 degrees. The [001] direction of the center sub-band is parallel to the direction of the transition band and hence, parallel to the rolling direction. The structure of the low-angle boundaries is shown in Fig. 7 at high magnification. The structure of these boundaries varies considerably from place to place as does the density of dislocations within the sub-bands. Further examples of the structures of the low-angle boundaries will be given later.

When rolled to 90% reduction, the transition bands become even narrower although the width of the sub-bands remains constant. The average angle of disorientation of the low-angle boundaries has increased to 3-4 degrees. Figure 8a shows a typical example of a transition band in a crystal rolled to 90% reduction. The deformation bands have (100) planes parallel to the sample (diffraction patterns, Figs. 8b and c)and there is a change in orientation of the [001] direction across the transition band of 15 degrees. This relatively small change in orientation across the transition band is frequently observed for crystals rolled to 90% reduction. In many cases, in the more heavily rolled crystals, the transition bands are split and the [001] direction of the center sub-band does not always coincide with the rolling direction. In some cases, where the transition bands are closely spaced, the deformation band between them contains an elongated cell structure similar to that observed in crystals rolled to 50% reduction. This effect is not understood.

Repeated diffraction surveys of all the deformed crystals show that the majority of the crystal is comprised of regions with the (100) planar orientation but other orientations are occasionally found that have their origin in the formation of mechanical twins during rolling. Figure 9a shows a transition band and deformation bands in a crystal rolled to 70% reduction. The diffraction

patterns (Figs. 9b and c) show that the deformation bands have (111) planes parallel to the rolling plane. The change in orientation across the transition band is 21 degrees about an axis normal to the (111) plane.

Many twins have been observed after reductions of 10-20% and these should reorient during subsequent cold-rolling to place (111) planes nearly parallel to the rolling plane. Diffraction patterns of deformed twins observed in the electron microscope show these to have (111) planes in the rolling plane and a <011> direction parallel to a <011> direction of the matrix (9).

b. Textures of the Rolled Crystals

Up to about 50% reduction, the reorientation during rolling may be characterized as a spread of the initial single crystal in two directions about an axis normal to the rolling plane. At 50% reduction, this spread is approximately 15 degrees in both directions. Beyond about 50% reduction, the crystal begins to form two texture components. The components are shown in the pole figure of Fig. 10 which shows the (200) poles of a crystal after rolling to 70% reduction. The initial orientation of the single crystal is given by the open squares. The central portion of the pole figure was not obtained since the symmetry of the orientation made this unnecessary.

The two components, A and B, are related to the initial orientation by rotations of 25 degrees, both clockwise and counterclockwise, mainly about an axis normal to the rolling plane. The dashed lines arbitrarily delineate the orientations of the major components from the transition region (TR), the region of continuous spread from the initial single crystal to the orientations of the components.

Rolling to 90% reduction increases the rotation of the major components away from the rolling direction to about 35° while slightly increasing the spread away from the periphery of the pole figure. There is a decrease in the volume fraction of material in the transition region and an increase in the volume fraction contained within the major components.

The presence of the major components in the rolled crystal can be seen directly by proper etching of the rolled crystal. Figure 11 shows the macroetched surface of a (100) [001] crystal rolled to 70% reduction. The etchant reveals the cube faces; thus, the edges of the pits are parallel to <001> directions. A [001] direction in the region marked A lies at an angle of about 20

degrees counterclockwise to the rolling direction, whereas, in the region marked B, the [001] direction lies at an angle of about 12 degrees clockwise to the RD. The regions A and B, of single but different orientation, are the deformation bands. The narrow band between A and B is the transition region of Fig. 10 across which the change in orientation between adjacent deformation bands is accomplished. As has already been shown in the examination of the transition bands, the change in orientation between the deformation bands is gradual and spread over a distance of about 5 microns in crystals rolled to 50% to as little as 1 micron in crystals rolled 90%.

c. Substructure of Annealed Crystals

The changes that occur in the substructure upon annealing crystals that have been rolled to reductions of 50% or greater may be generalized as follows: a) a decrease in the width of the transition bands (particularly in the case of crystals rolled to 50%), b) a sharpening of the low-angle boundaries, c) nucleation of recrystallization grains within the transition bands, and e) polygonization to sub-grains within the deformation bands.

Figure 12a shows a transition band in a crystal rolled to 50% reduction and annealed for 15 min. at 600°C. The elongated cell structure that comprised the entire substructure of the rolled crystal is no longer present; instead, the transition bands consist of numbers of clearly defined sub-bands, 0.2-0.3 microns wide, separated by sharply defined low-angle boundaries. The deformation bands are structureless, the cells having been replaced by a general distribution of dislocations.

While the total width of the transition bands has decreased for comparable changes in orientation of the [001] directions (compared to the width of the transition bands in the rolled crystal), and the width of the sub-bands remains constant, the average angle of disorientation of the low-angle boundaries has increased to 1.3 to 1.5 degrees.

The diffraction patterns in Figs. 12b and c show that the deformation bands, Band C and B, on either side of the transition band, have (100) planes parallel to the plane of the sample. The change in orientation of the [001] direction across the transition band is 28 degrees.

The nature of the rotation across the transition band is shown more clearly by referring to Fig. 13 which shows a transition

band in a crystal rolled to 70% reduction and annealed for 5 min. at 700°C. Diffraction patterns were obtained at points 1-5 and show that within the deformation bands (points 1 and 5) and the transition bands (points 2, 3, 4) (100) planes are parallel to the rolling plane. The change in orientation of the [001] direction occurs as step-wise rotations about the normal to the rolling plane, for a total rotation of 47 degrees. The [001] direction in the center of the transition band coincides both with the rolling direction and the direction of the transition band. The number of sub-bands in this transition band is 22; dividing by the total orientation change of 47 degrees gives an average angle of disorientation per low-angle boundary of 2 degrees. The nature of the rotations across the transition band suggests that the low-angle boundaries are tilt boundaries and should, therefore, consist of edge dislocations. The structure of the low-angle boundaries is shown more clearly in Fig. 14. This shows another area of the same transition band shown in Fig. 13, but at much higher magnification. The transition band is traversed by a very narrow twin (400 Angstroms wide) which was formed at the time the sample was being thinned or cut in preparation for electron microscopy. The twin changes direction where it crosses a low-angle boundary; the change in direction is a bend with the bend axis normal to the sample surface. There is no apparent twist at the low-angle boundary. Measurements of the twin's angular change at the low-angle boundaries range from 1 to 3 degrees with an average bend of 2 degrees.

If one counts the number of dislocations per cm length of boundary, then, from $Db = \theta$ (10) for a simple tilt boundary, values of 1 to 2 degrees are obtained. D is the number of dislocations per cm of boundary and b is the Burger's vector (2.5 angstroms). Thus, electron diffraction, the change in direction of the twin, and dislocation counts confirm the tilt nature of the low-angle boundaries.

As was pointed out earlier, the transition bands in crystals rolled to 90% reduction contain low-angle boundaries with angles of disorientation of 3-4 degrees. When annealed, the angle of disorientation remains constant as does the width of the sub-bands.

Figure 15a shows a typical transition band in a crystal rolled to 90% reduction and annealed for 30 sec. at 657°C. The deformation bands, B and D, have (100) planes in the plane of the sample (diffraction patterns 15b and d). The change in orientation of the [001] direction across the transition band is approximately 35 degrees.

d. Recrystallization

The most striking characteristic of recrystallization in the rolled crystals is that of nucleation; nuclei are observed to arise only within the transition bands. This is illustrated in Fig. 16 which shows the polished and etched surface of a crystal rolled to 90% reduction and annealed for 1 min. at 650°C.

The broad bands are deformation bands and the narrow bands are the transition bands. The recrystallization grains are either contained within or are contiguous to the transition bands. Recrystallization did not occur within the deformation bands; however, growth of new grains into the deformation bands occurred readily.

Recrystallization grains were easily identified in the electron microscope by 1) the general configuration of the grain within the transition band, 2) a very low density of dislocations, 3) a tendency to polish through at the boundary between the new grain and the transition band, and 4) the presence of Kikuchi lines in the diffraction pattern. Kikuchi lines indicate greater perfection of the new grain as compared to the sub-bands of the transition band (11).

One such new grain, approximately 1.5 microns wide, in a sample rolled to 70% and annealed for 5 min. at 700°C, is shown in Fig. 17a. Diffraction patterns, obtained at point 1 (within the transition band), at point 2, within the grain, and at point 3, within the deformation band to the right of the transition band, are given in Figs. 17b, c, and d, respectively. All three areas of the crystal have (100) planes parallel to the plane of the sample. Across the boundary between points 1 and 2 there is a change in orientation of the [001] direction of only 2 degrees. Across the boundary between the new grain (point 2) and the deformation band, however, there is a change of 10 degrees in the [001] direction. Since the planar orientation is the same, it is evident that the new grain originated as a part of the transition band. Furthermore, the disparity of the angles of disorientation across the boundaries between 1 and 2 and between 2 and 3 indicate that the point of origin of the new grain was adjacent to point 1. The grain increased in width and the boundary 2-3 increased its angle of disorientation as it migrated toward point 3.

The new grain, marked C, in Fig. 15a also has a (100) plane in the plane of the sample as do the deformation bands on either side of the transition band. It is obvious that grain C originated near the right hand side of the transition band since the difference in orientation of the [001] direction between C and D is only 3-4

degrees.

The recrystallization grain always has the same planar orientation as the transition band from which it originates. For instance, Fig. 18a shows a recrystallization grain in a crystal rolled to 90% reduction and annealed for 1 min. at 650°C. The new grain, C, has a (111) plane parallel to the plane of the sample (diffraction pattern, Fig. 18c), as do the deformation bands, B and D, on either side of the transition band (Figs. 18b and d). Regions and grains of this orientation are the result of the formation of mechanical twins during the early stages of rolling. The deformed twins recrystallize completely before recrystallization even begins in the matrix. At the points of intersection of the twins, grains of many different orientations are found.

It has been observed that nucleation occurs more often near the edges of the transition bands than in the center. That this is to be expected will be discussed later.

IV. DISCUSSION

a. Origin of Primary Recrystallization Nuclei

It is characteristic of the nucleus for primary recrystallization, in this particular system, to have the same orientation as the orientation of the particular sub-band which served as the point of origin of the nucleus.

This characteristic suggests that the nucleation event is the result of the enlargement of a portion of a sub-band by migration of the low-angle boundary separating the growing sub-band from adjacent sub-bands in the transition band. Figure 19 gives an example of low-angle boundary migration leading to formation of a recrystallization nucleus in a crystal rolled to 70% and annealed for 5 min. at 700°C. The low-angle boundary, which originally separated the sub-band marked X from the adjacent left-hand sub-band, has migrated to the point marked by the arrow 1. It has combined with the existing boundary to form another boundary of higher angle of disorientation. With this migration, a curvature has been imparted to the connecting boundaries (arrows 2) such that they must move in the direction of the arrows. Thus, X-block will widen and lengthen at the expense of neighboring sub-bands.

The angle of disorientation of the migrating boundary progressively increases as it crosses successive sub-bands; it

encounters the polygonized deformation band as a relatively high-angle grain boundary (see, for instance, Fig. 17). The arrows marked 3, near the bottom of Fig. 19, show a region where parts of a low-angle boundary have migrated across an adjacent sub-band in an irregular fashion.

The driving force for migration of the low-angle boundary derives from the difference in energy-density across the boundary (12). The sub-band boundaries within the transition band add to the driving force for growth of the nucleus. Thus, the recrystallization grains are generally long and narrow with points at one or both ends (see Fig. 16).

The sub-grain boundaries within the polygonized deformation bands also add to the driving force for growth once the nucleus boundary reaches the deformation band; there are cusps where the grain boundary intersects the sub-grain boundaries. These cusps cause a curvature to be imposed upon short segments of the grain boundary with the centers of curvature in the direction of migration of the boundary.

This description of the nucleation event is based on the supposition that the low-angle boundaries are mobile. As the photomicrographs and the diffraction patterns show, the low-angle boundaries are tilt boundaries and, as such, they should be mobile, according to Cottrell (13). Thus, it remains to determine the mechanism by which such low-angle tilt boundaries and the transition bands are formed during deformation and how these boundaries might be changed during the anneal. The following section considers a mechanism for formation of the transition bands.

b. Formation of Transition Bands

The formation of the transition bands appears to occur as a result of interactions of edge dislocations of different Burger's vectors approaching each other along the two operative <111> glide directions in each {110} glide plane.

In the early stages of deformation, the <111> glide directions are symmetrically disposed with respect to the rolling direction and {110} glide planes have the highest resolved shear stress. Thus, dislocations with $[\bar{1}11]$ and $[\bar{1}\bar{1}1]$ Burger's vectors, gliding in the (101) plane, for instance, approach each other, the leading edge components meeting to form a wall. This wall is shown schematically as the center wall in Fig. 20. The following screw components undergo cross glide on to $(10\bar{1})$ and other {110} glide

planes and meeting another screw dislocation of opposite sign, are annihilated. Left behind, then, in the original glide plane are equal numbers of edge dislocations with different Burger's vectors. This basically is the mechanism of formation of cells as described by Seeger (14) and examined by Swann and Nutting (15).

Thus, after a 10% reduction (see Fig. 1), the incipient cell walls and tangles appear to consist of edge dislocations parallel to several <112> directions. Also, the small number of screw dislocations (parallel to the <011> trace) and the evidence for abundant cross-glide appear to substantiate the annihilation of screw dislocations. The cell walls represent the early stages of formation of the transition bands.

As the amount of deformation is increased, the <111> glide directions begin to rotate away from the symmetrical glide position to a position where glide occurs more readily along one of the <111> directions than along the other. This rotation occurs in different directions in different regions of the crystal. That is, on the right-hand side of the cell wall, the rotation will be clockwise and on the left hand side, counterclockwise. Referring to Fig. 20, the succeeding dislocation walls will then contain more edge dislocations of one Burger's vector and fewer of the other Burger's vector. As the rotation increases, each succeeding wall will contain fewer and fewer dislocations of one type. Eventually, the low-angle boundaries near the edge of the transition band will contain only one family of edge dislocations. If the amount of deformation is sufficient to cause rotations of about 30 degrees (reductions beyond about 70%) the edge dislocations in the low-angle boundaries along the edge of the transition band will have Burger's vectors parallel to the rolling direction. In Fig. 20 these walls are adjacent to the deformation bands.

The dislocation structures of these outer low-angle boundaries can be clearly seen in Fig. 21 where the boundaries in the bottom left-hand corner of the photograph are adjacent to the deformation band. This sample was rolled to 70% reduction and annealed for 1 min. at 700°C.

Within the deformation bands, glide occurs along a single <111> direction and the cells are eventually filled with dislocations. Because of this single glide, the deformation bands retain a single orientation which gradually changes with increasing deformation beyond about 50% reduction to provide the major components of the texture. Since there are no significant variations of orientation within the deformation bands there is no nucleation

of recrystallization grains.

c. Effect of Annealing on Structure of Transition Bands

Annealing short of recrystallization results in some modifications of the structures of the transition bands.

In the case of the crystals rolled to 50% reduction, the transition bands become narrower and the low-angle boundaries straighten and become more sharply defined. Also, the angle of disorientation of the low-angle boundaries increases from 0.6-0.7 degrees (in the as-rolled crystal) to 1.5-1.7 degrees after the anneal. The total change in orientation across the transition band remains constant, however. There must be some combination of the low-angle boundaries during the anneal to produce the higher-angle boundaries.

In the case of the crystals rolled to 70% and 90% reduction, however, there is no significant change in the angle of disorientation of the low-angle boundaries during the anneal. In fact, the only change in the structure of the transition bands in the more heavily rolled crystals is the simplification of the structure of the low-angle boundaries. This simplification is probably due to the expulsion of whatever screw dislocations might be contained within the low-angle boundaries (16, 17) to convert them to pure tilt boundaries during the anneal.

Since the boundaries near the edge of the transition band are more likely to consist of a single family of edge dislocations, it is expected that nucleation would occur in the sub-bands adjacent to the deformation bands rather than within the center of the transition band. The present observations indicate this to be the case. However, the limited number of instances in which nucleation is far enough along to be recognizable yet not so far along that the new grain traverses the transition band prevents making a sweeping generalization.

d. Other Mechanisms

It is possible that the above proposed mechanism for recrystallization holds only for the (100) [001] orientation in a body centered cubic crystal or for other crystals under similar conditions of symmetrized glide. Hu (18), in a study of the structures of rolled (110) [001]-oriented crystals of silicon-iron, observed, upon annealing, rapid formation of sub-grains followed by a slow increase in sub-grain size prior to recrystallization. He postulated that sub-grains coalesced as a result of the disap-

pearance of some of the sub-grain boundaries, the coalescence being followed by rotation of the sub-grains. This same coalescence and rotation mechanism was applied by Hu in an independent study of the structures of rolled and annealed (100) [001]-oriented crystals of silicon-iron (19). In that study he observed a grain with the (113) orientation in a transition band with the (100) orientation and suggested that the grain was formed as a result of very large rotations of some of the sub-bands of the transition band.

In the present study, the recrystallization grains were always observed to have the same planar orientation as the transition band from which the grains originated, at least within the accuracy of the electron diffraction measurements.

Weissmann, et al (16), and Fujita (20), in studies of the substructures of cold-worked aluminum, observed that growth of sub-grains during an anneal appeared to proceed by a gradual disappearance of sub-boundaries, i.e., by coalescence of sub-grains. The sub-grains became surrounded by high-angle boundaries, presumably as a result of migration of dislocations from the sub-grain boundaries to some point outside the coalescing group of sub-grains.

Votava (21) observed a cell structure in cold-worked copper which polygonized to sub-grains upon annealing. However, the recrystallization nuclei appeared suddenly and simultaneously with polygonization. Baily (22) suggests that recrystallization in deformed silver results from the migration of already present grain boundaries but could not determine the source of the necessary high-angle boundaries in heavily worked silver.

It seems that additional study will be required to provide a detailed description of formation of high-angle boundaries when sub-grains coalesce. Particular attention must be paid to the presence and conditions of local curvature (23) at the points of nucleation in view of the body of evidence suggesting that nuclei are formed in regions where turbulent glide has occurred (24), i.e., where the lattice has become curved.

In the present study and in the study by Hu (19), the observations confirm the presence of the necessary regions of disorientation required for nucleation.

V. SUMMARY AND CONCLUSIONS

1. (100) [001]-oriented crystals of high-purity 3% silicon-iron were rolled, at room temperature, to reductions of 10-90% of

thickness. The structures of rolled and annealed crystals were observed by means of transmission electron microscopy.

2. Cells are formed at low reductions (10-20%). The cell walls consist mainly of edge dislocations parallel to <112> directions.

3. With heavier reductions (up to 50%), the cells elongate in the rolling direction. In certain regions of the crystal there are significant reorientations. The reorientation is characterized as a rotation about an axis normal to the (100) or rolling plane. These regions have been called "transition bands". The regions in which there are no reorientations are called "deformation bands".

4. At 60-70% reduction, the elongated cells in the transition bands become sub-bands separated by low-angle tilt boundaries which have angles of disorientation of about 2 degrees. The elongated cell structure in the deformation bands is replaced by a general distribution of dislocations.

5. The low-angle boundaries in the transition bands in the crystals rolled to 90% reduction have angles of disorientation of 3-4 degrees.

6. When annealed, the low-angle boundaries of the transition bands are converted to pure tilt boundaries. The deformation bands undergo polygonization to sub-grains.

7. Nucleation of recrystallization grains occurs only within the transition bands by a process of migration of low-angle boundaries across adjacent sub-bands. The driving force for migration derives from differences in dislocation density from sub-band to sub-band.

8. The recrystallization grains have the same orientation as the sub-band which served as the point of origin of the nucleus.

9. The majority of recrystallization grains have (100) planes parallel to the plane of the sample. Occasional recrystallization grains are observed to have orientations near (111). These are found in conjunction with (111)-oriented transition bands and deformation bands located within deformed twins.

10. The proposed mechanism for recrystallization in (100) [001]-oriented crystals of high-purity silicon-iron may not hold for crystals of other orientations or for metals with different deformation characteristics.

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REFERENCES

1. J.L. Walter and C.G. Dunn, Trans. Met. Soc. AIME, 215, 465 (1959).

2. J.L. Walter, Acta Met., 7, 424 (1959).

3. J.L. Walter and C.G. Dunn, Trans. Met. Soc. AIME, 218, 914 (1960).

4. J.L. Walter and C.G. Dunn, Acta Met., 8, 497 (1960).

5. V.A. Phillips and J.A. Hugo, J. Sci. Instr., 37, 216 (1960).

6. P.R. Swann and J. Nutting, J. of Inst. of Metals, 90, 133 (1961).

7. A.S. Keh and S. Weissmann, to be published in Proc. of 1961 Conf. on "The Impact of Trans. Electron Microscopy on Theories of Strength of Crystals", Interscience Pub., New York.

8. See Ref. 6, p. 134.

9. For additional information concerning twins in high-purity silicon-iron crystals see J.L. Walter and E.F. Koch, Acta Met., 10, 1059 (Nov. 1962).

10. W.T. Read, Dislocations in Crystals, McGraw-Hill Book Co., Inc., New York (1953) p. 158.

11. Z.G. Pinsker, Electron Diffraction, Butterworths Sci. Publ., London (1953) p. 217.

12. C.G. Dunn and K.T. Aust, Acta Met., 5, 368 (1957).

13. A.H. Cottrell, Dislocations and Plastic Flow in Crystals, Oxford Univ. Press, London (1953) p. 188.

14. A. Seeger, Dislocations and Mechanical Properties of Crystals, Wiley, New York and London (1956) p. 243.

15. See Ref. 6, p. 135.

16. S. Weissmann, T. Imura, and N. Hosokawa, to be published in the Proceedings of the Symposium on "Recovery and Recrystallization of Metals", AIME Annual Meeting, Feb. 1962.

17. J.C.M. Li, to be published.

18. H. Hu, Trans., Met. Soc. AIME, 224, 75 (1962).

19. H. Hu, Letter to Ed., Acta Met., 10, 1112 (Nov. 1962).

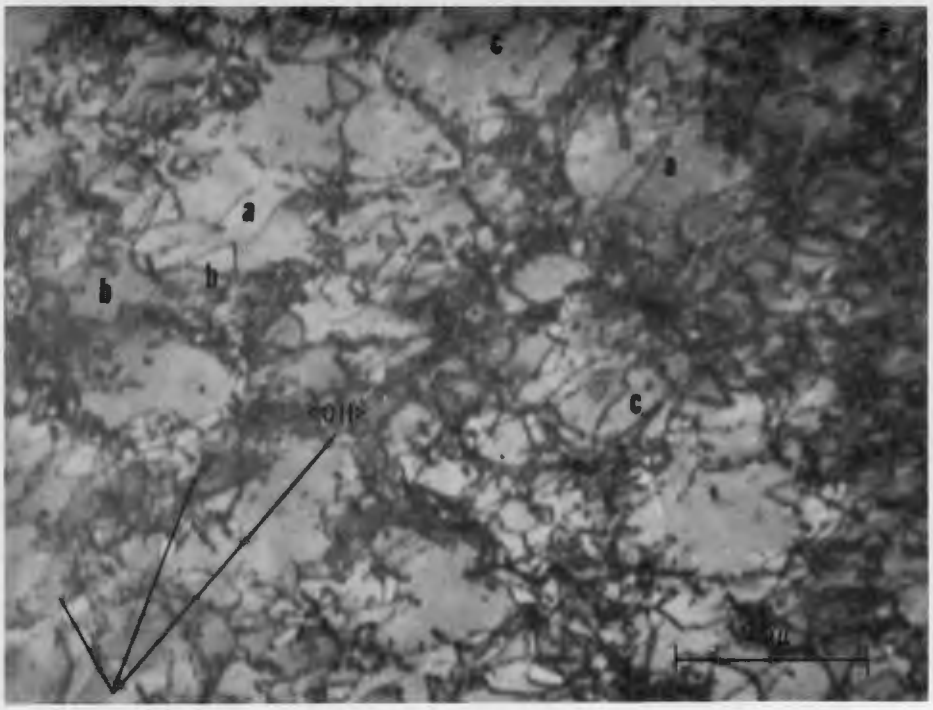
20. H. Fujita, J. Phys. Soc. Japan, 16, 397 (1961).

21. E. Votava, Acta Met., 9, 870 (1961).

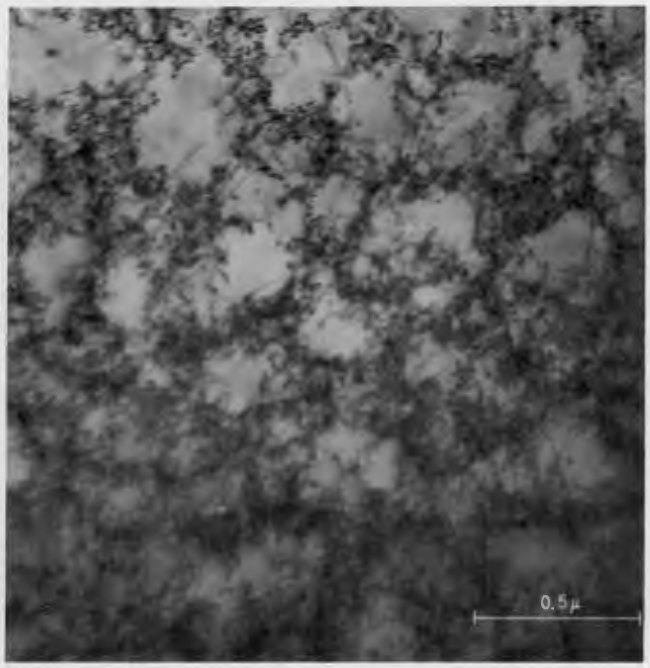
22. J.E. Bailey, Phil. Mag., 5, 833 (1960).

23. R.W. Cahn, Proc. Phys. Soc. A63, 323 (1950).

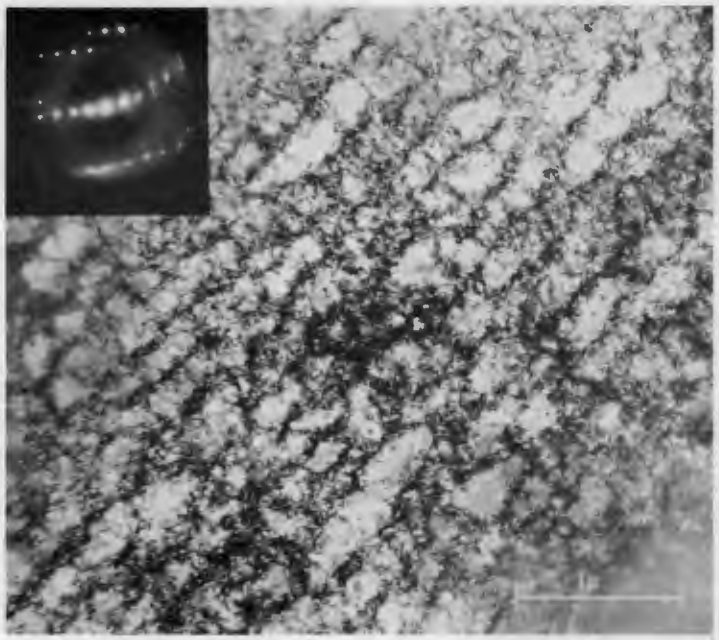
24. R.W.K. Honeycombe, J. Inst. Metals, 80, 45 (1951).



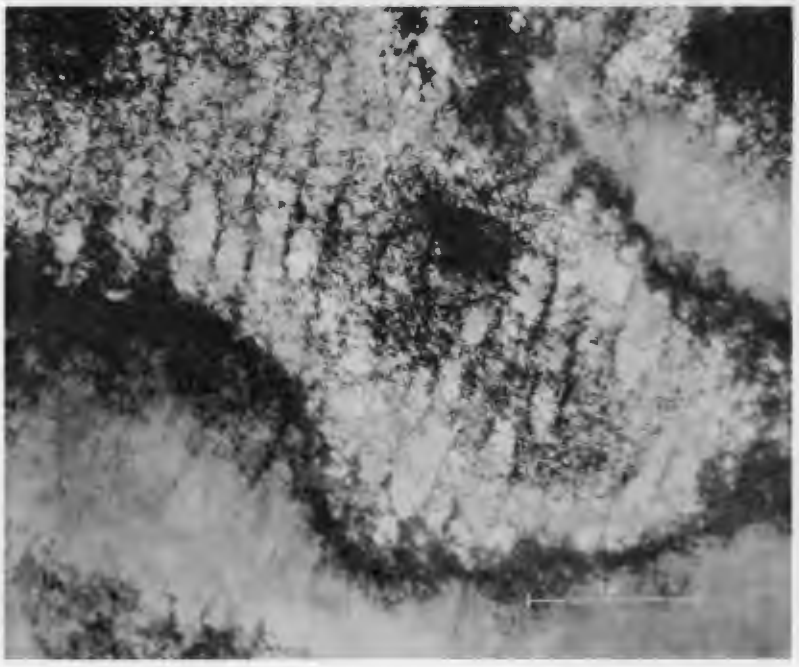
1. Incipient cells and dislocation tangles in crystal rolled to 10% reduction. X 110,000.



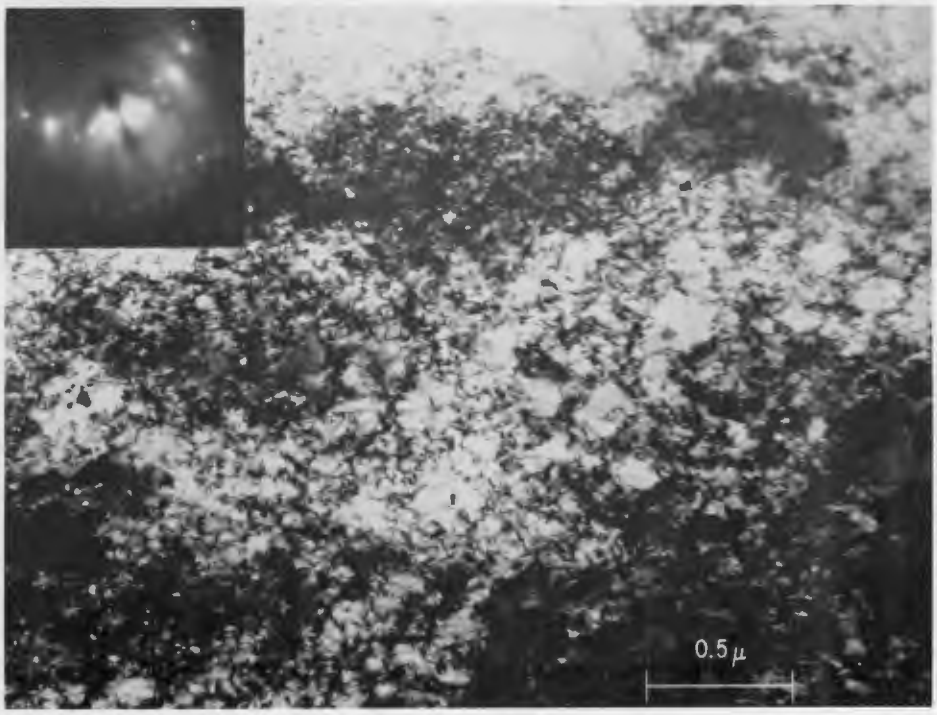
2. Cells in crystal rolled to 20% reduction. X 100,000.



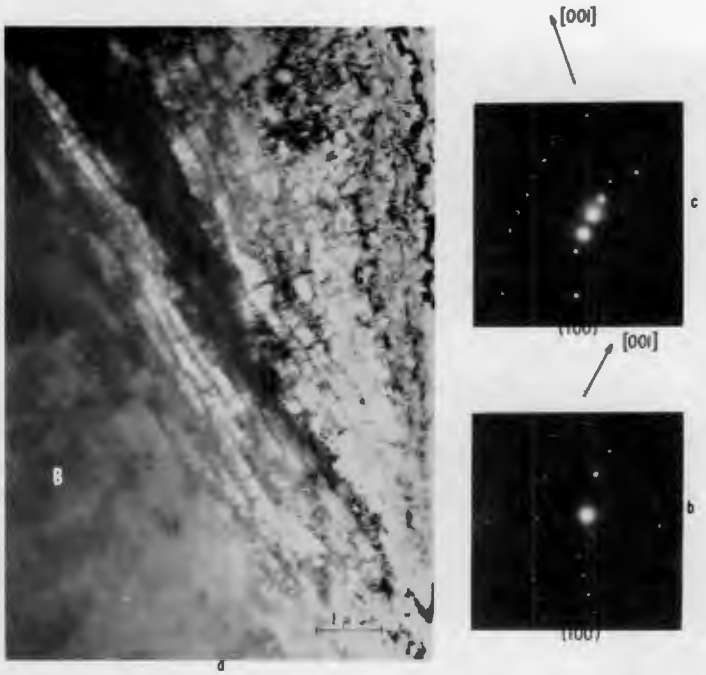
3. Elongated cells in transition band in crystal rolled to 50% reduction. Diffraction pattern shows nature of reorientation. X 48,000.



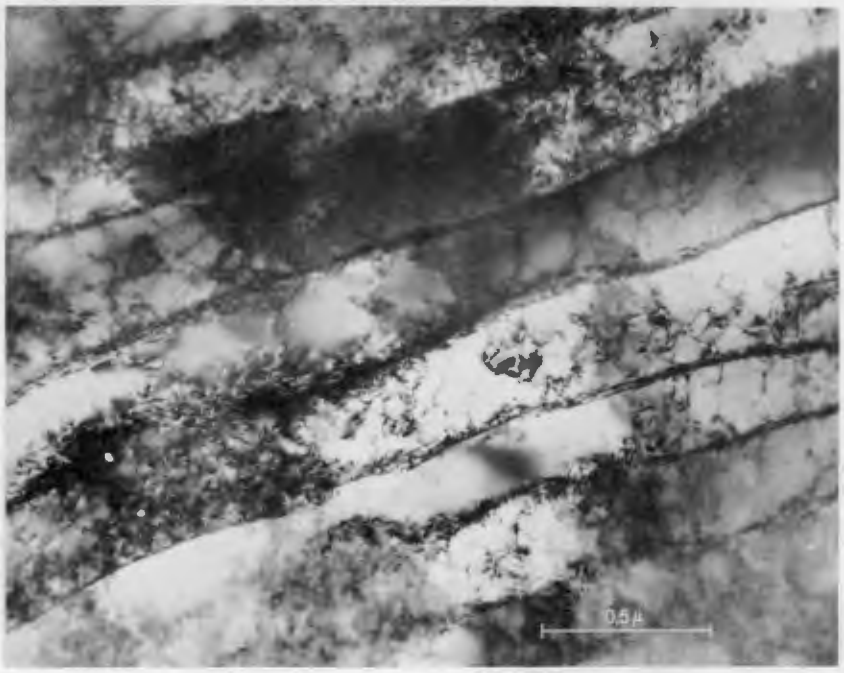
4. Sub-bands in transition band in crystal rolled to 60% reduction. X 50,000.



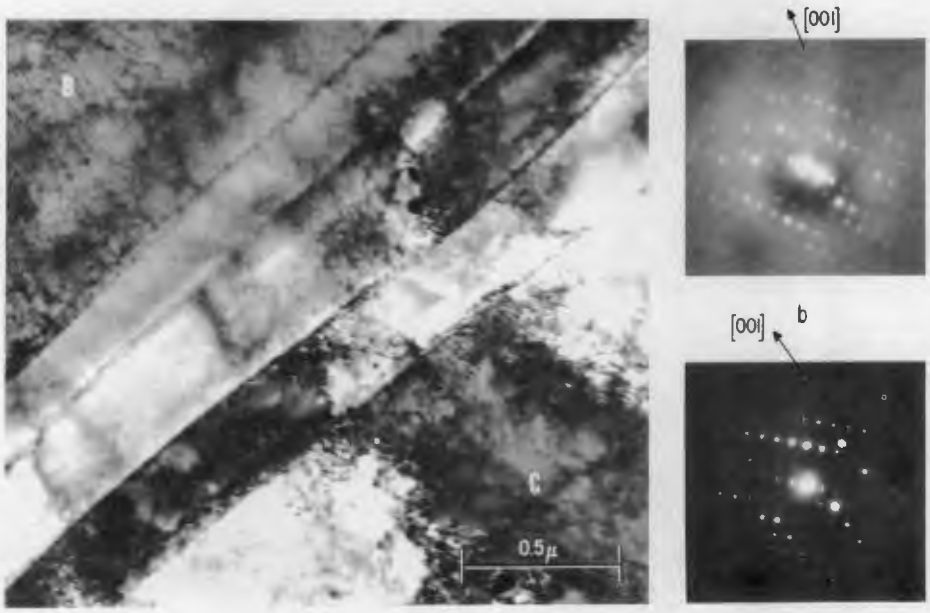
5. Dislocation structure and diffraction pattern of deformation band in crystal rolled to 60% reduction. X 84,000.



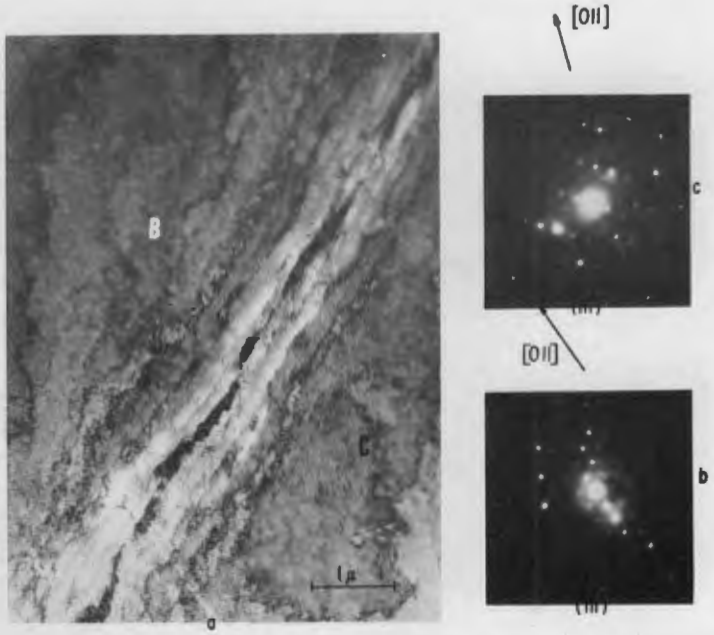
6. Transition band and diffraction patterns of deformation bands, B and C, in crystal rolled to 70% reduction. X 19,000.



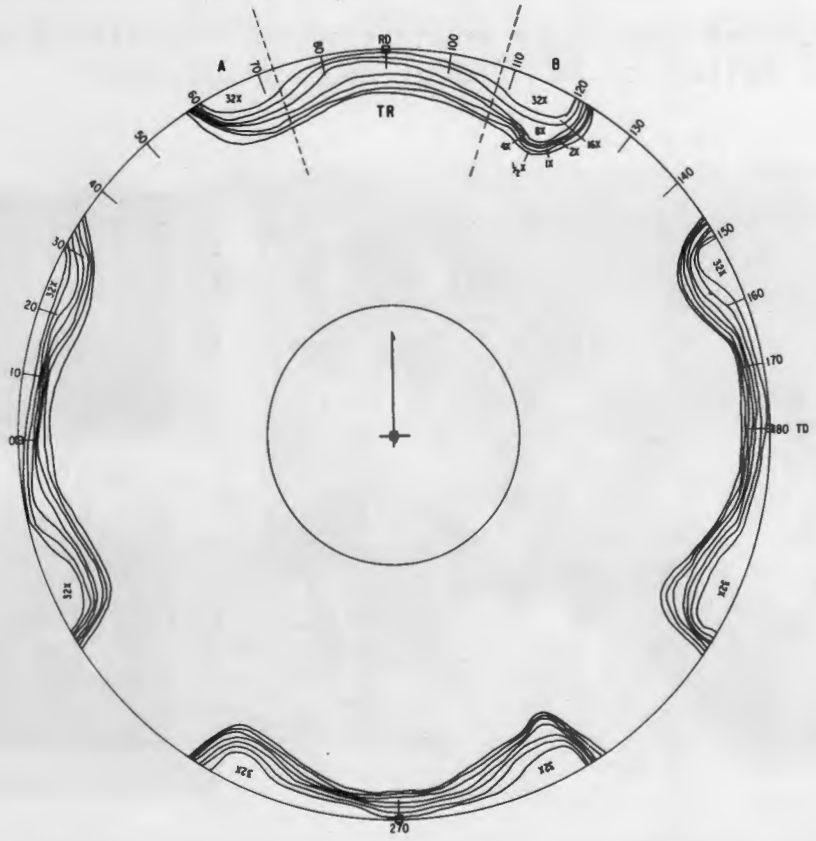
7. Structure of low-angle boundaries in transition band in crystal rolled to 70% reduction. X 100,000.



8. Transition band and diffraction patterns of deformation bands, B and C, in crystal rolled to 90% reduction. X 90,000.

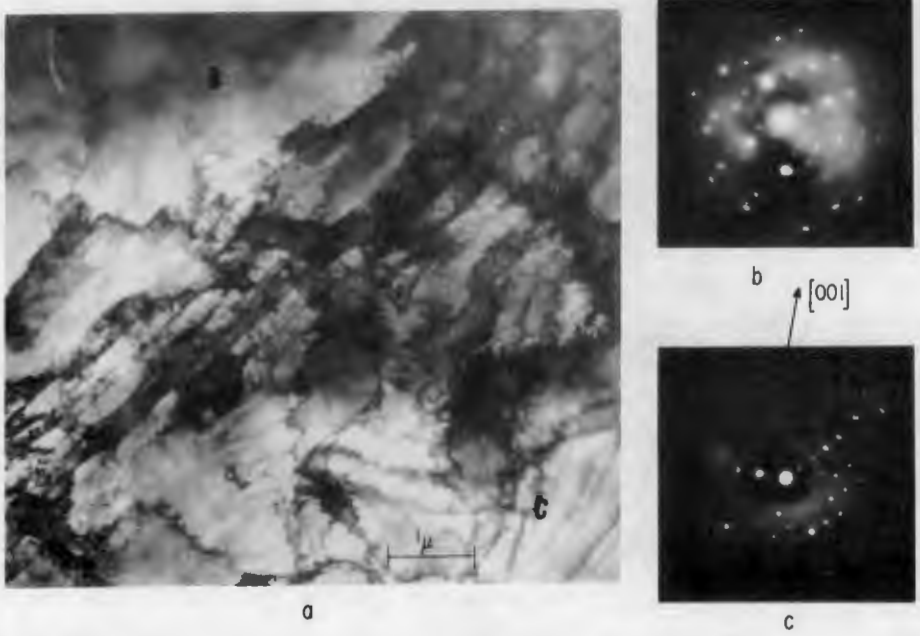


9. Transition band and (111)-oriented deformation bands B and C, (diffraction patterns b and c) in crystal rolled to 70% reduction. X 24,000.

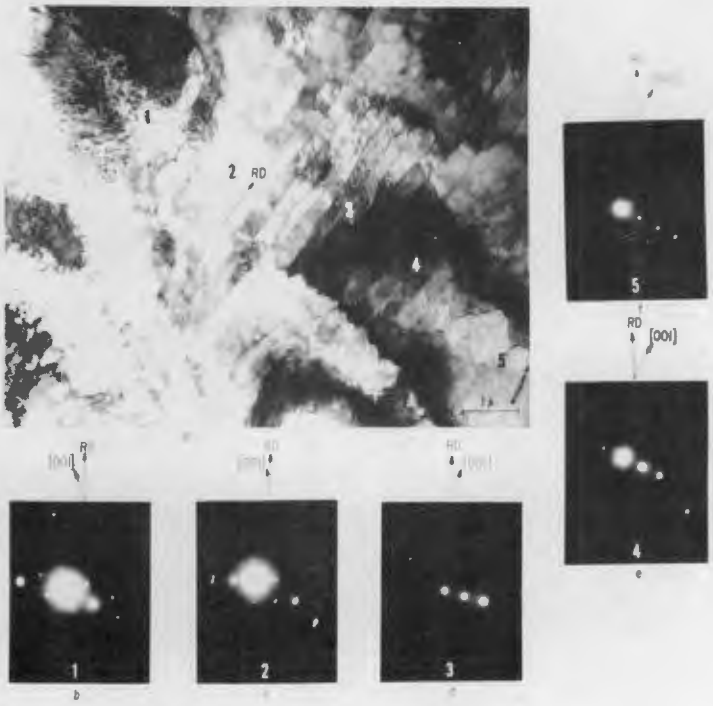


10. (200) pole figure of crystal rolled to 70% reduction. Open squares give initial orientation, (100) [001].

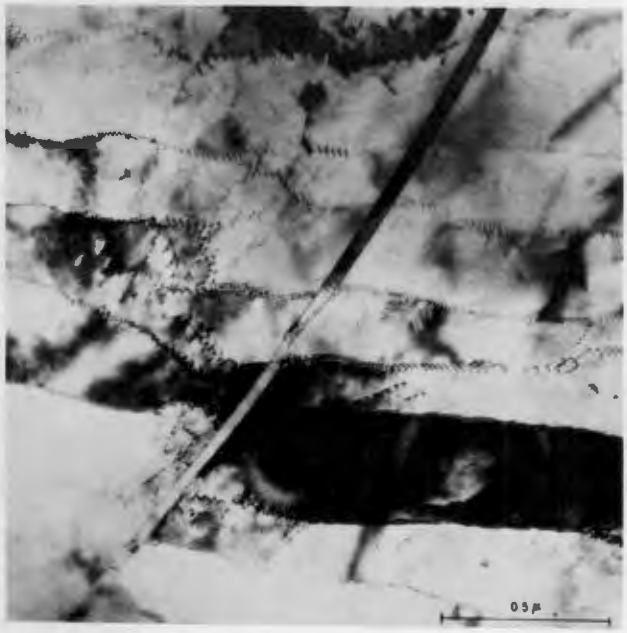
11. Macroetched surface of crystal rolled to 70% reduction. Shows variety of orientations. A and B are deformation bands. X 500.



12. Transition band and diffraction patterns of deformation bands, B and C, in crystal rolled to 50% and annealed 15 min. at 601°C. X 25,000.

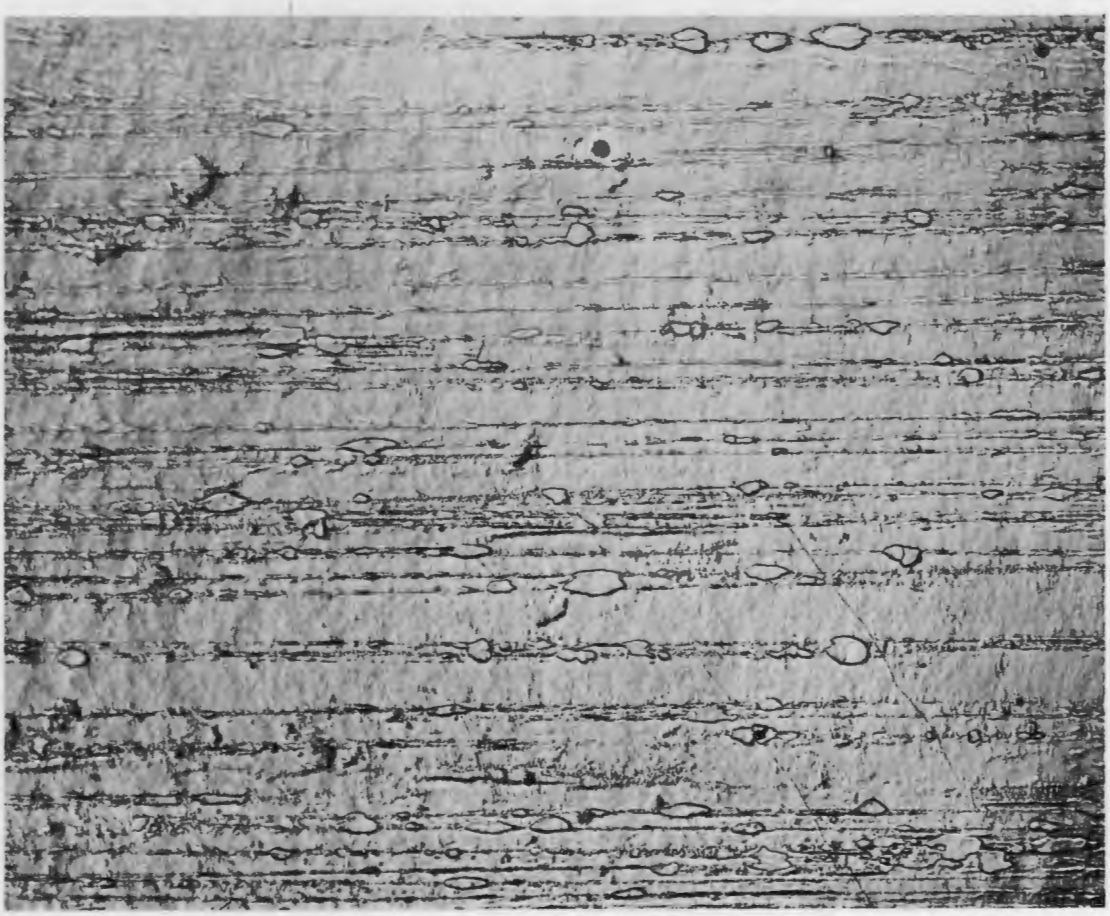


13. Transition band in crystal rolled to 70% reduction and annealed for 5 min. at 700°C. Diffraction patterns taken at points 1-5 to show change of orientation of [001] direction across transition band. X 17,000.

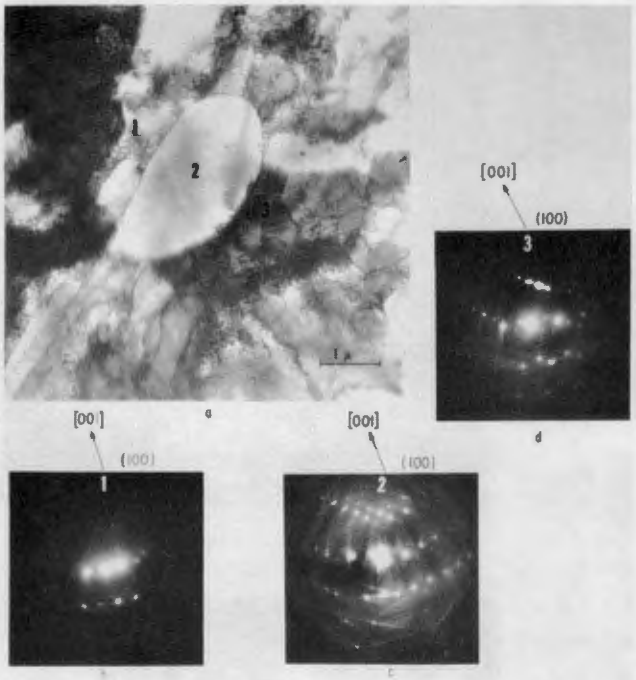


14. Structure of low-angle boundaries in transition band in crystal rolled to 70% and annealed for 5 min. at 700°C. Narrow band traversing transition band is twin. X 108,000.

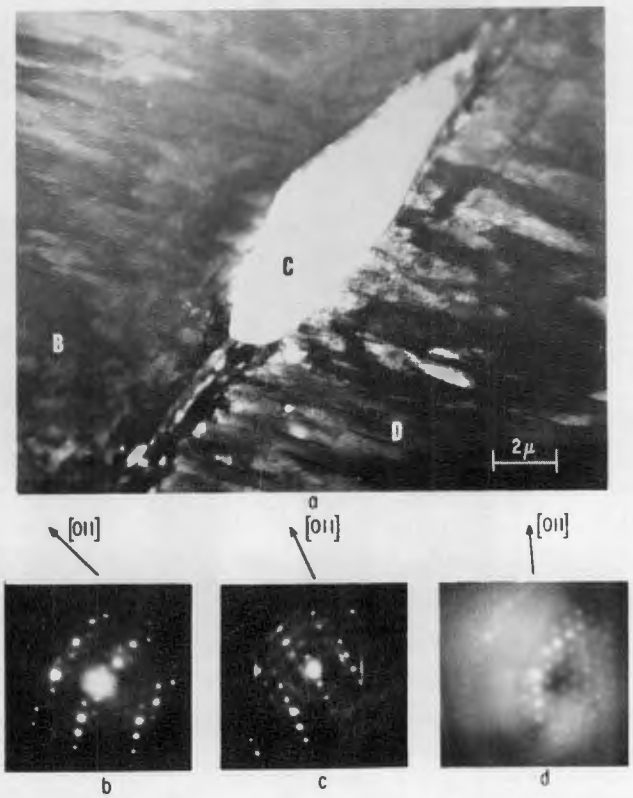
15. Transition band and recrystallization nucleus in crystal rolled to 90% and annealed 30 sec. at 657°C. Diffraction patterns b, c, d show (100) orientations of deformation bands, B and D, and nucleus, C.



16. Polished and etched surface of crystal rolled to 90% and annealed 1 min. at 650°C. Wide bands are deformation bands and narrow bands are transition bands. Nucleation occurs within transition bands. X 150.

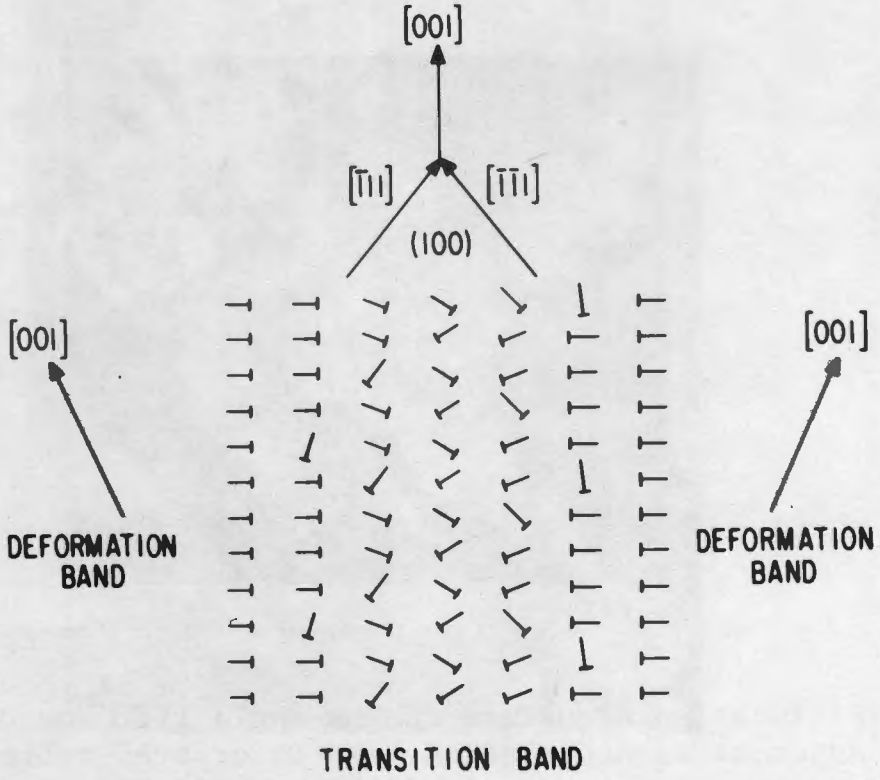


17. Transition band, recrystallization nucleus (2) and polygonized deformation bands in crystal rolled to 70% and annealed 5 min. at 700°C. Diffraction patterns show orientations at points 1-3. X 19,000.



18. (111)-oriented recrystallization grain and deformation bands in crystal rolled to 90% and annealed 1 min. at 650°C. Diffraction patterns b and d show orientation of deformation bands. X 10,000.

19. Origin of recrystallization nucleus within transition band in crystal rolled to 70% and annealed 5 min. at 700°C. X 53,000.



20. Schematic diagram of dislocation structure of low-angle boundaries of transition bands.

21. Dislocation structure of low-angle tilt boundaries adjacent to deformation band in crystal rolled to 70% and annealed 1 min. at 700°C. X 100,000.