OBSERVATIONS OF CREEP IN ALUMINUM AT ELEVATED TEMPERATURES

By

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ABSTRACT

The creep behavior of coarse grained high purity, 2 S, and 3 S aluminum was studied at 400° F, 700° F, and 1100° F over a stress range of 50 – 1200 psi. A high temperature microscope was utilized to study the steps in the creep process, permitting creep measurements over short distances in the various portions of the grains and across grain boundaries. Indentation produced by a sharp needle permitted a marking technique for reference purposes. The orientations of the grains were indicated by the use of the etch pit technique. Extensive metallographic work, frequently supplemented by X-ray work, was utilized to study the process of grain boundary sliding and grain boundary migration. Similarly the mechanisms of deformation in the grains were followed, the deformation being typified by the presence of slip bands, kinking bands, deformation bands, folds and sub-grains. The stepwise nature of the process of grain boundary sliding and migration was studied in detail as a function of temperature, stress, grain size and purity. The creep behavior across grain boundaries variously situated and for different conditions is noted and discussed. The formation of kinking bands, deformation bands, folds, slip bands, and sub-grains, particularly the latter two, is discussed. Intercrystalline failure is considered in the light of the purity of the metal.

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I. INTRODUCTION

Creep studies and measurements in most instances are based on a relatively gross gage length. Even in some recent theoretical studies on the mechanism of creep, changes were followed by means of X-rays and metallography without regard for the behavior of the individual grains and grain boundaries[1,2,3]. Analytical studies of creep have frequently indicated that more than one competing process is operative and that the ultimate creep performance depends on the effects of temperature, time and strain rate as well as grain size[4,5,6]. The grains and grain boundaries are known to respond differently to these variables but the extent of the response is still little known, especially in polycrystalline materials where the relative orientations of grains and grain boundaries are important.

This thesis presents some experimental observations of the behavior of the grain boundaries and their effect on the deformation in the grains. In addition to high purity aluminum, 2 S and 3 S aluminum were also investigated in order to determine the effect of purity on the deformation along grain boundaries and in the grains.

II. EXPERIMENTAL PROCEDURE

A small vertical nichrome wound furnace was built to permit direct loading on the test specimen. This furnace is capable of operating up to about 1300° F with reasonable temperature distribution. A ground rectangular quartz window, 1.25 inches by 0.5 inches, is imbedded in the external stainless steel tube to permit observations of the creep process with a microscope. An argon atmosphere was provided to prevent oxidation. The quartz window caused a temperature gradient between the back and front surfaces of the specimen of 15° F at 900° F and 700° F, and 20° F at 1100° F. The temperature gradient could be decreased to 3° F along the length of the specimen by adjusting shunts across the furnace windings.

An ordinary metallurgical microscope with a working distance of 14 mm. was rebuilt so that it could be moved in three mutual perpendicular directions. Visual examination could be made with either a 30 X eyepiece or a 15 X filar eyepiece. Using an objective of focal length 24.3 mm and a numerical aperture 0.20 a total magnification of 120 X or 240 X could be obtained.

High purity aluminum rated at 99.995 percent and 2 S and 3 S aluminum were supplied by the Aluminum Company of America as 5/8 inch rod. The spectrographic analysis of these three grades of aluminum are shown in Table I. The rods were first machined to 1/4 inch diameter, after which the gage portion was further machined to 3/16 inch diameter. The ends were 1/4 inch diameter

TABLE I

Spectrographic Analysis of the Three Grades of Aluminum

Grade	Impurities, Percent								
	Cu	Fe	Si	Mn	Mg	Zn	Cr	Ti	Na,Ca
High purity	0.0017	0.002	0.001	-	0.0002	-	-	-	0.0006
2 S	0.10	0.46	0.12	0.01	-	0.01	-	0.03	-
3 S	0.09	0.36	0.16	1.15	-	0.01	0.01	0.01	-

and threaded to fit available specimen holders. Two opposite flat surfaces of the gage portion were obtained by milling, giving final dimensions of the gage portion of 3/4 inch x 3/16 inch x 3/32 inch.

A. Preparation of Specimens of High Purity Aluminum

Machined specimens were first electro-polished in Jacquet's solution[7]. Reference marks were produced by pressing a thin sewing needle into the surface of the specimen. The specimen was then annealed at 900° F for 24 hours and at 1150° F for 12 hours. Typical macrostructures of specimens treated in this manner are shown in Figure 1. The range of the grain sizes was 1 to 5.5 mm., (see Figure 1), but most of the grains were of 2.5 mm. and went through the whole thickness of the specimen. The specimen was re-electro-polished and etched electrolytically to reveal the grain boundaries. In order to reveal the orientation differences of the grains, etch pits were produced on the surface of some specimens by etching in a mixture of hydrochloric and nitric acids (HCl, 45 percent; HNO_3, 10 percent; H_2O, 45 percent, by volume).

B. Preparation of Specimens of 2 S and 3 S Aluminum

Machined specimens were first annealed at 600° F for about 12 hours. The specimens were then stretched 3 to 8 percent. Thereafter, the procedure applied to 2 S and 3 S aluminum specimens was the same as for high purity aluminum. The specimens were marked with reference marks, electropolished, annealed at 900° F for 24 hours, and at 1150° F for 12 hours, and repolished. In general, the grains produced after the above treatments were about 3 to 10 times longer in the direction of the specimen axis than in the direction of the width of the specimen.

There were about 2 to 4 grains across the width of the specimen

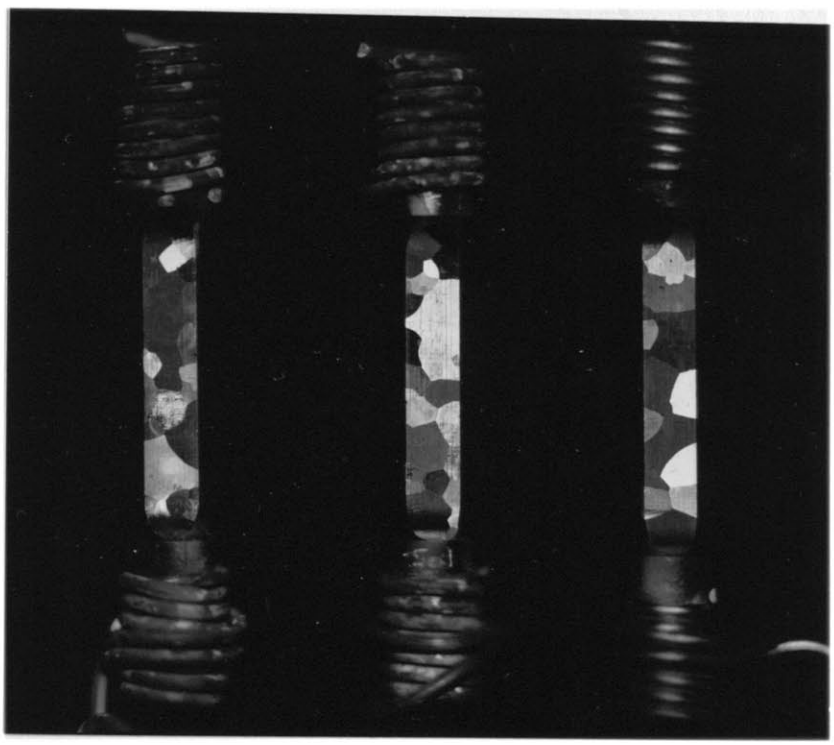


Figure 1. Macrostructures and dimensions of specimens of high purity aluminum. (Specimens of 2 S and 3 S aluminum are of the same shape and dimensions). Etched by Tucker's reagent. 1.8 X.

for 2 S aluminum, and about 10 to 20 grains for 3 S aluminum. The arrangement of the grains in the specimens was generally irregular, and therefore it was difficult to follow the course of the grain boundaries.

The specimens were very carefully put into the holders and furnace; yet even with this care, some fine slip lines were developed on the specimen surface. Later it was found out that, in addition to the etch pits, these slip lines were of great help in determining the orientation of grains and in evaluating the behavior of grain boundaries. The load needed for the low stress experiments was only 1.5 pounds (stress about 85 psi). In order not to disturb the specimen by loading when it was very weak at the creep temperature, direct load was applied at room temperature and the specimen was brought up to temperature under load. For stresses higher than 85 psi, water was siphoned into a container hung by the lower specimen holder when the specimen reached the test temperature.

The needle points produced in the surface of the specimen gave very bright and sharp points under the microscope and therefore served well as reference marks for creep measurement. Measurement could be made either by a filar eyepiece micrometer or by a dial gage. The accuracy of measurement depended on the size and the constancy of reference marks on the surface of the specimen.

With creep strain up to about 50 percent, no difficulty was encountered in measurement because the sharpness of the reference marks was maintained and their shapes did not change very much.

The size of the reference marks was about 0.01 mm. in diameter and the distance between two reference marks was about 0.6 - 0.7 mm.; thus the error of strain measurement should not be greater than 1.5 percent.

III. PRESENTATION OF RESULTS

A. Method of Presentation

In order to show the arrangement of grains, especially the direction and position of grain boundaries, traces of the grain boundaries on the four surfaces of the specimen were mapped out after macro-etching. Figures 2 through 6 show the structural development drawings of specimens P-3, P-8, P-6, P-10, and P-7, respectively. These figures will be referred to frequently in later discussions. The two flat surfaces (of which the front one faces the microscope) and the round edge surfaces of the specimen will be called the "plane surfaces" and "edge surfaces" respectively. Capital letters were used to distinguish individual grains. Arabic numbers beside the needle made indentations indicating the positions of reference marks. Small letters beside triangles indicate the approximate positions where back reflection Laue patterns were taken.

Photomicrographs were taken both during the actual creep process and after the specimen had been removed from the equipment. Most of the photomicrographs were taken with oblique light so that maximum detail could be shown in the same photomicrograph. All the photomicrographs will be so presented in the text that the direction of applied tension runs from top to bottom. Specimen numbers will be included in a bracket after the figure number. The grain letter will be indicated on the photomicrographs and schematic drawings in accordance with the development drawing of the specimen.

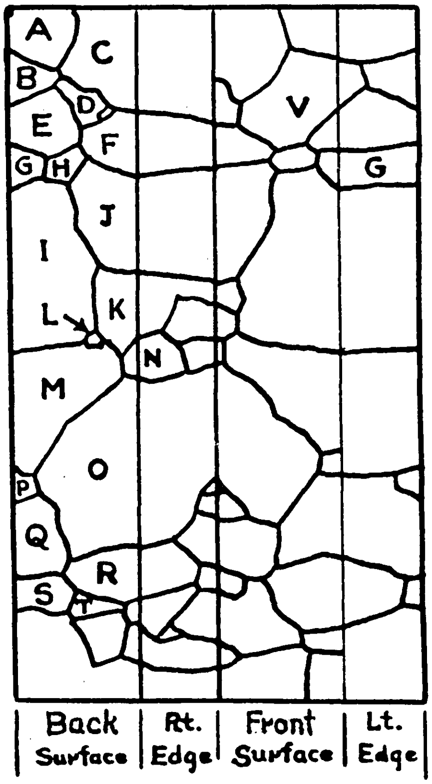


FIG. 2 SPECIMEN P-3

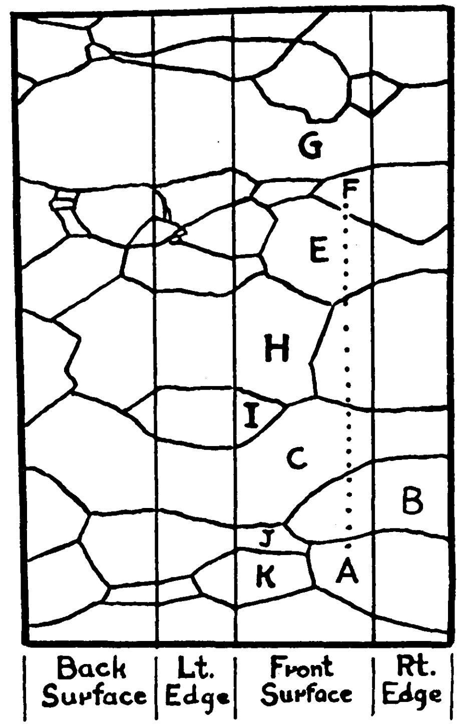


FIG. 4 SPECIMEN P-6

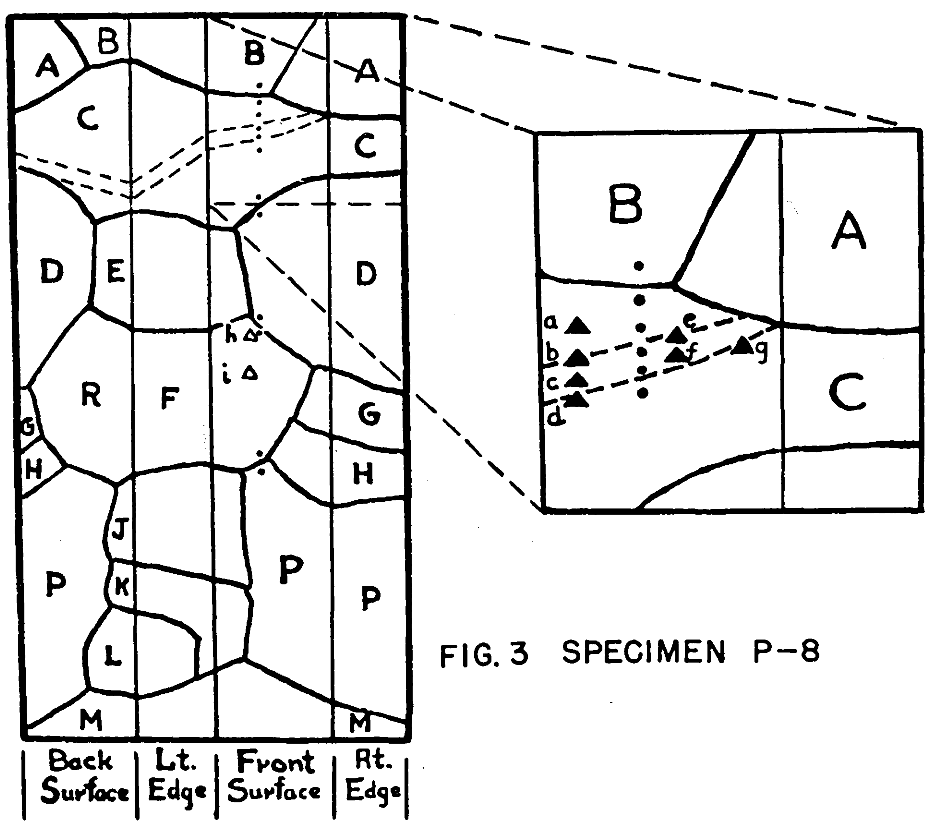


FIG. 3 SPECIMEN P-8

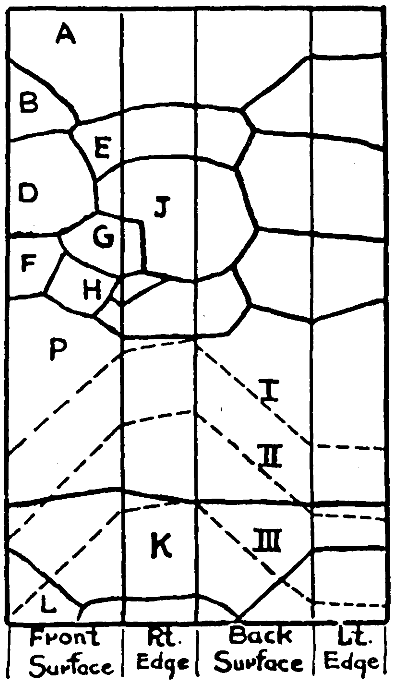


FIG. 5 SPECIMEN P-10

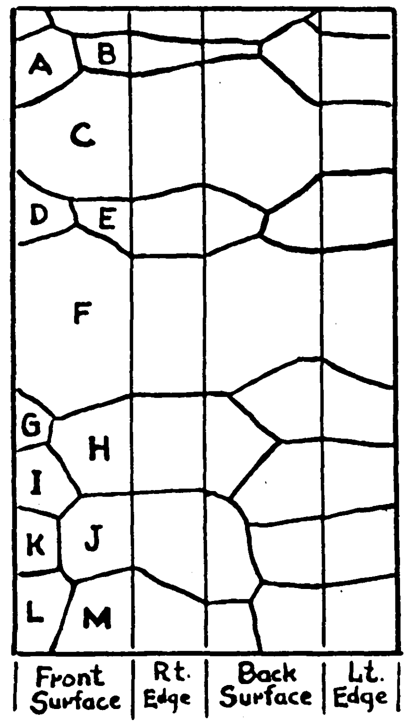


FIG. 6 SPECIMEN P-7

FIGS. 2 — 6 STRUCTURAL DEVELOPMENT DRAWINGS OF SPECIMEN P-3, P-8, P-6, P-10, P-7.

----- INDICATES THE COURSE OF SLIP BANDS

● " REFERENCE MARKS

▲ " THE POSITION WHERE BACK REFLECTION LAUE PATTERNS WERE TAKEN

For easy reference, Table II is prepared to list the test conditions and the status of the specimens when the test was completed.

TABLE II

Condition of Specimens at Time of Observation in Creep Studies

Spec. No.*	Load psi	Temp. °F	Total Elong. %	Duration of test hours	Condition of specimen when test stopped
P-7	1200	400	19.5	18	ruptured
P-11	400-750	400	12.5	475	not ruptured
P-3	85	700	2.5	1.5	not ruptured
P-8	85	700	7.7	81	not ruptured
P-1	100	700	28.5	234	ruptured
P-6	200	700	64.5	21.3	ruptured
P-9	65	1100	28	7	not ruptured
P-10	50	1100	33	30	ruptured
2 S-11	800	700	35.6	24.5	ruptured
2 S-7	300	900	-	20	not ruptured
2 S-6	450	900	12.3	30	ruptured
2 S-16	250	1100	8	12	ruptured
2 S-12	200-300	1100	5.875	41	ruptured
3 S	200-300	1100	19	79.5	ruptured

* P stands for high purity aluminum

2 S stands for 2 S aluminum

3 S stands for 3 S aluminum

B. Grain Boundary Sliding and Migration

Evidence of grain boundary sliding and migration: It can be seen from Figure 7 A (P-3) that the grain boundary between grains M and N underwent successively alternating sliding and migration. In order to provide a distinction from crystalline slip, the term "sliding" is introduced to refer to the process by which two grains slide with respect to each other along their common grain boundary surface. The term "boundary migration" is used in the sense that the grain boundary surface moves in some direction which does not lie in the grain boundary surface. The positions of the initial and final grain boundaries can be judged by the appearance of etch pits and are also indicated by letters I and F, representing the initial and final grain boundary, respectively. Since etch pits were put in before the creep test, the initial grain boundary should be the boundary between the regions of the two differently shaped etch pits corresponding to the two grains. The position of the final boundary was established by repolishing and etching the specimen after the creep test, whereupon the initial boundary was removed and the final boundary remained (see Figure 7 B). Evidence of the successively alternating and cooperative nature of boundary sliding and migration can also be obtained by observing the course of the slip lines of grain N which were produced during handling in putting the specimen into the furnace. The original straight slip lines are now sharply displaced in the field between the initial and final grain boundaries due to grain boundary sliding and migration. It is necessary to point out that the slip lines of

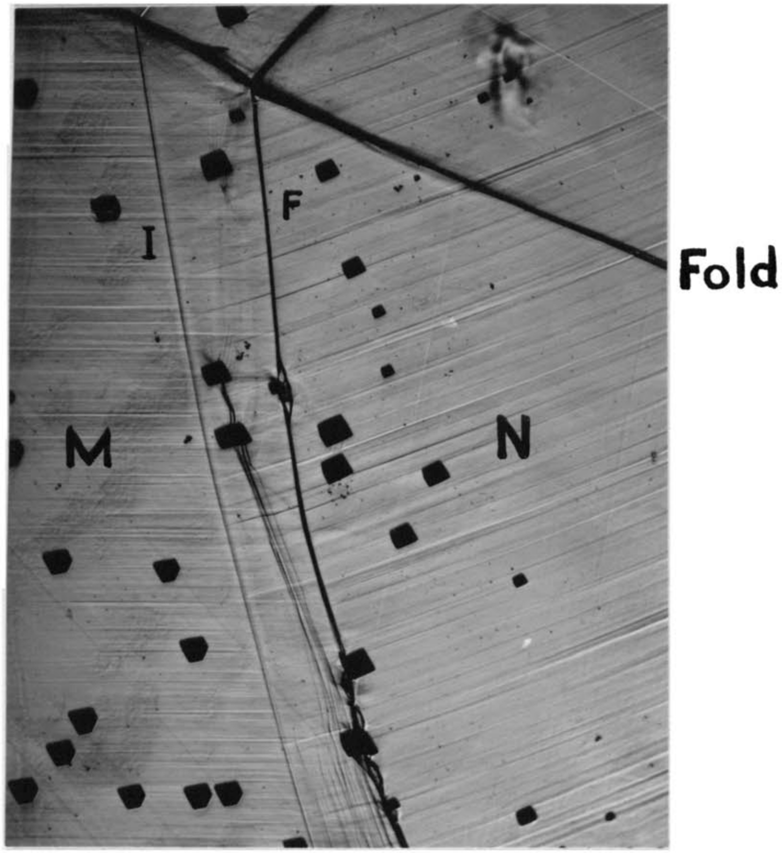


Figure 7 A.

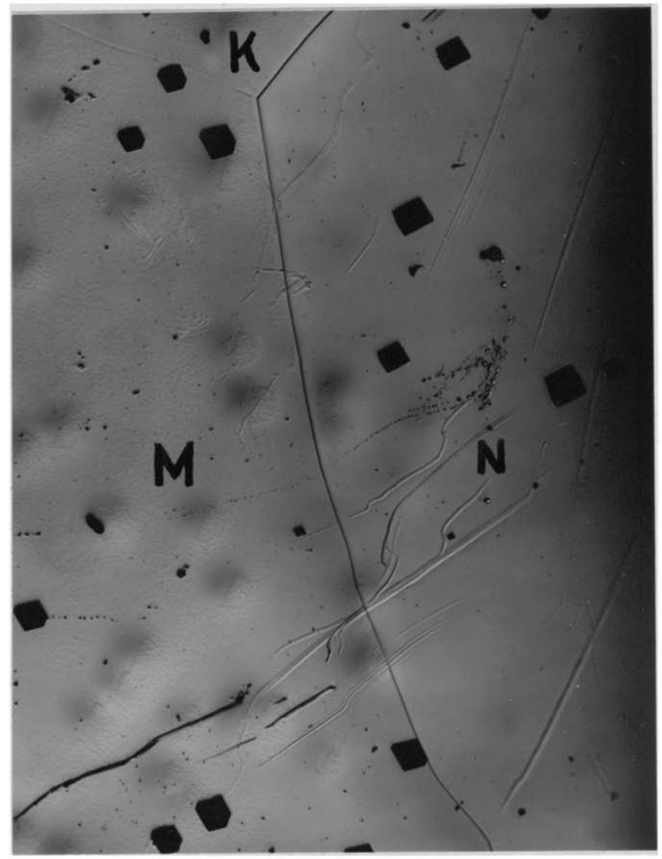


Figure 7 B.

Figure 7 A. (Specimen P-3). Deformed surface showing the steps of grain boundary migration and sliding during creep. Etch pits put in before test. I and F indicate the initial and final grain boundaries respectively. 150 X.

Figure 7 B. (Specimen P-3). Same field as Figure 2 A, repolished and etched. Initial grain boundary and fold (or band) disappeared. Etch pits put in after test. Crater-like regions are etch pit locations seen in Figure 2 A. 150 X.

grain M are propagated continuously across the migrated zone without being bent. The fact that they were not bent indicates that these slip lines must have been developed at the conclusion of boundary migration, probably by handling of the specimen after test.

The stepwise nature of grain boundary sliding and migration can be more clearly seen from Figure 8 (taken from the back surface of specimen P-3) which is a 500 X magnification of the triple point of grains M, O, and P from Figure 9. The boundary of grains M and O is inclined at about 45° to the direction of applied tension and at about 15° to the side of the specimen (see Figure 2). It was, therefore, in a favorable position to slide and did so. As a result of this sliding, an offset was produced along the boundaries of grains M and P, and O and P. As can be seen from Figure 9A, the extension of the initial grain boundaries of grains M and P and of grains O and P (marked as I in Figure 9 A) intersected within the dark zone between grains M and O, as indicated by dotted arrow. But, as is evident from Figures 8 and 9 B, the initial and final boundaries constitute the left and right borders of the black zone respectively. Furthermore, the initial boundary of grains M and P could not be curved through the black band because the specimen was fully annealed before the test. The offset so produced is indicated in the schematic drawing of Figure 9 c, in which solid arrows indicate the directions of boundary migration. As a result of this initial grain boundary sliding, it is expected that a condition of strain due to the disturbance of the atomic equilibrium configuration around the triple point must exist at least temporarily in that

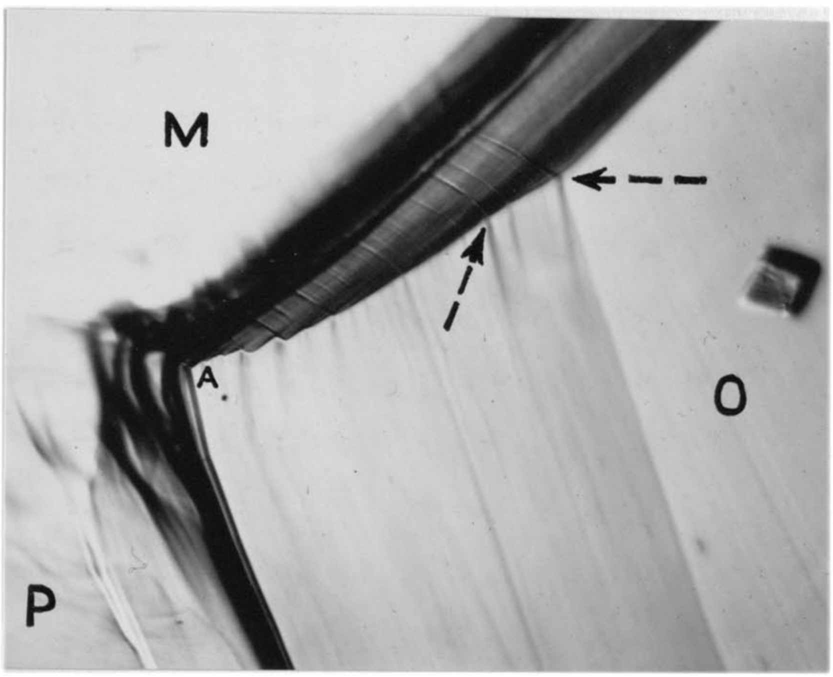
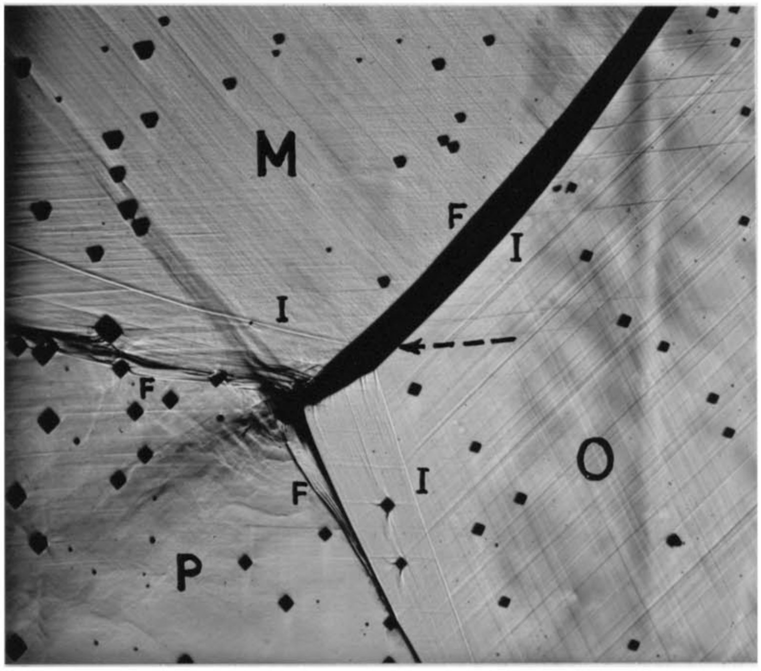


Figure 8. (Specimen P-3), back surface. The step-wise nature of boundary sliding and migration at the triple point of grains M, O, and P. Dotted arrows indicate the area swept by migrating boundary of grains O and P.



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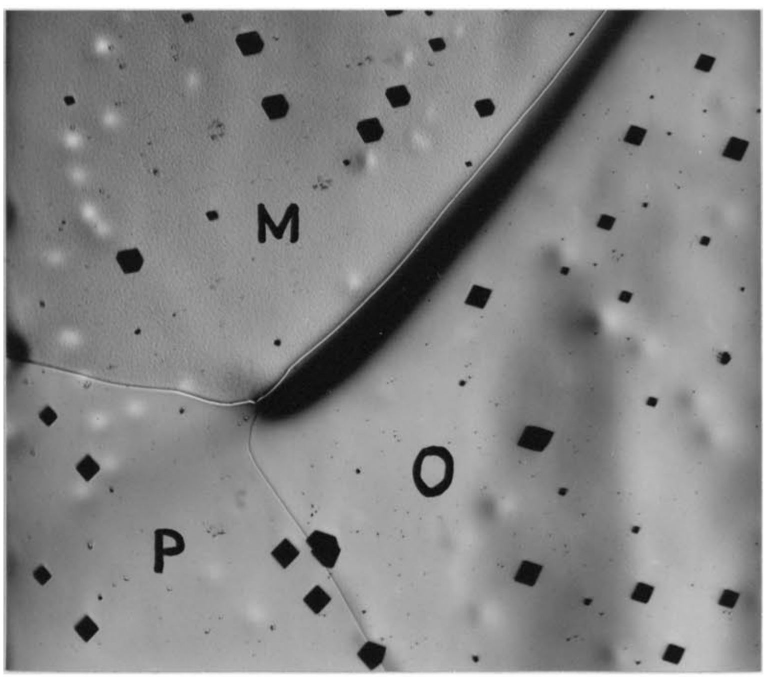


B

Figure 9 A. (Specimen P - 3). Sub-grain formation by bending around the triple point in the lower left grain. 100 X.

Figure 9 B. (Specimen P - 3). Same field as Figure 9 A repolished and etched. Sub-grain boundaries only very faintly revealed around the triple point in the lower left grain. 100 X.

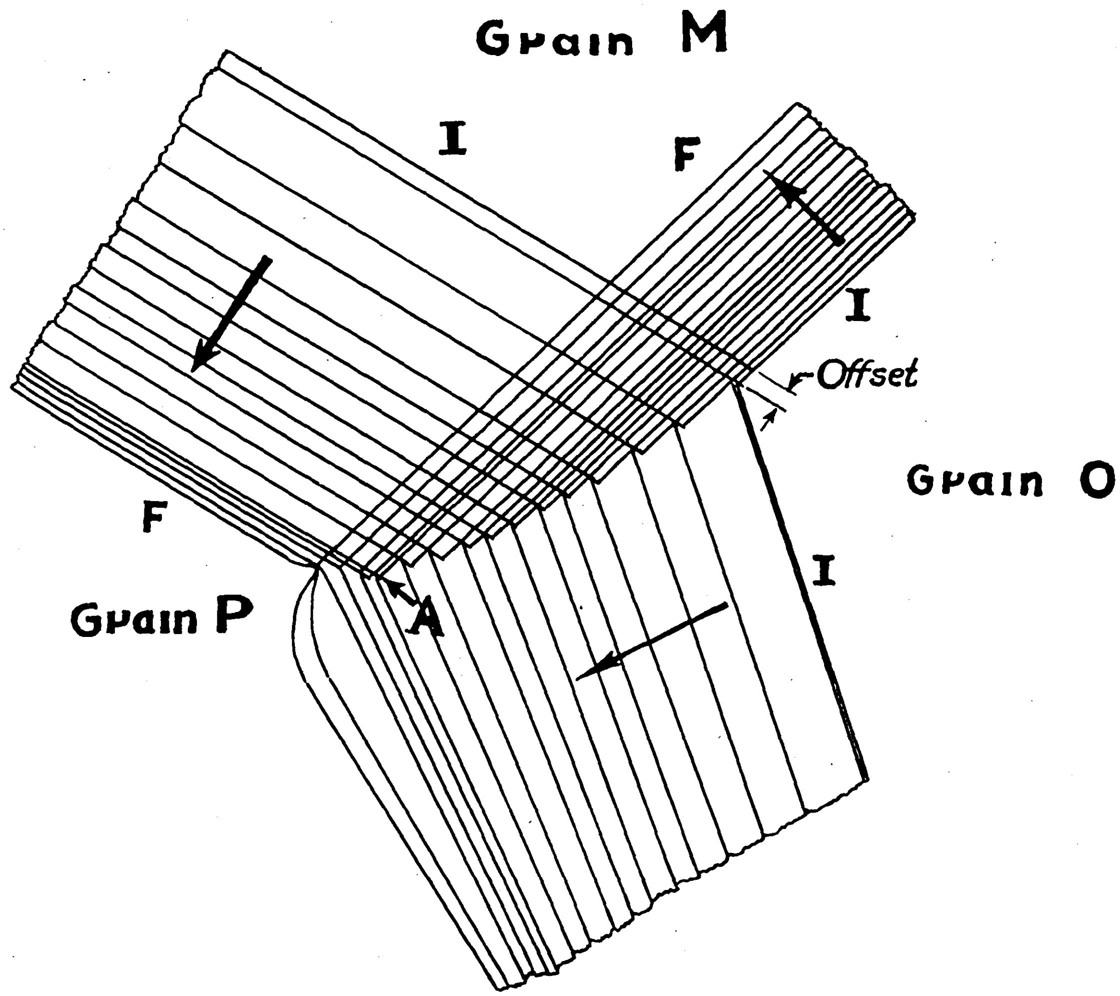


FIG. 9C SCHEMATIC DRAWING OF THE TRIPLE POINT OF FIG. 8. SOLID ARROWS INDICATE THE DIRECTION OF GRAIN BOUNDARY MIGRATION.

region. In order to maintain the triple point, the boundary of grains M and P migrated to the lower left by a diffusion process similar to a strain annealing process. Apparently after this cycle of grain boundary sliding and migration, the boundary of grains M and O was in a difficult position for further sliding. Thus, the boundary of grains M and P took over the sliding process. It can be seen from Figure 2 that this M-P boundary, with one end at the free edge of the specimen, could slide more easily than if it were bounded by two triple points at its ends. Sliding along the boundary of grains M and P caused the offset between the boundary of grains M and O and the boundary of grains O and P. A new triple point was formed by the migration of the boundary of grains P and O to the lower left direction. The narrow black band marked by the broken arrows in Figure 8 and swept by the boundary of grains P and O indicates this process nicely. This stepwise and cyclic process of boundary sliding and migration went on until point A (Figure 9 c) was reached. That this process was by no means uniform is evidenced by the unequal steps shown in Figure 8.

After point A was reached, heavy sliding along the boundary between grains M and O tended to cause fold formation (this will be discussed more fully later) in grain P, but the grain boundary between grains P and Q blocked the formation of the latter. Non-uniform deformation in grain P around the triple point resulted in heavy curvature of the boundaries of grains P and O and of grains M and P around the triple point, as noted in Figure 9 B.

C. Direction of Boundary Sliding and Migration

The direction of grain boundary sliding is governed primarily by the direction of shear stress acting on the boundary surface (but also in part by the orientation of the grains forming the boundary). This fact is clearly shown in Figure 10 in which a scratch line, originally straight, is displaced across the slid boundary.

Further, in specimen P-3 (Figure 2) a grain, G, bounded by a free edge of the test specimen, goes through the whole thickness of the specimen. This grain slid as a whole in the direction of the thickness of the specimen. As a result of this the back surface was depressed and the front side extruded, as illustrated in Figure 13. This could be readily observed visually. The direction of boundary migration is indicated by arrows in Figure 11 A and 12 A which are photomicrographs of the front and back surfaces of grain G, respectively. According to the geometry of this grain, it is natural that it should slide in the way observed in Figures 11 and 12. It is necessary to point out that the direction of boundary migration is generally toward the side which is depressed as shown by the solid arrows of Figure13, i. e., away from grain G in Figure 11 and toward grain G in Figure 12.

That the rate of grain boundary migration is non-uniform can be seen clearly in Figure 14 (specimen P-8). A schematic drawing of Figure 14 is shown in Figure 15 to show the sequence of steps in the migration of this boundary as observed during the actual test. It should be noted that the migrating boundary tends to

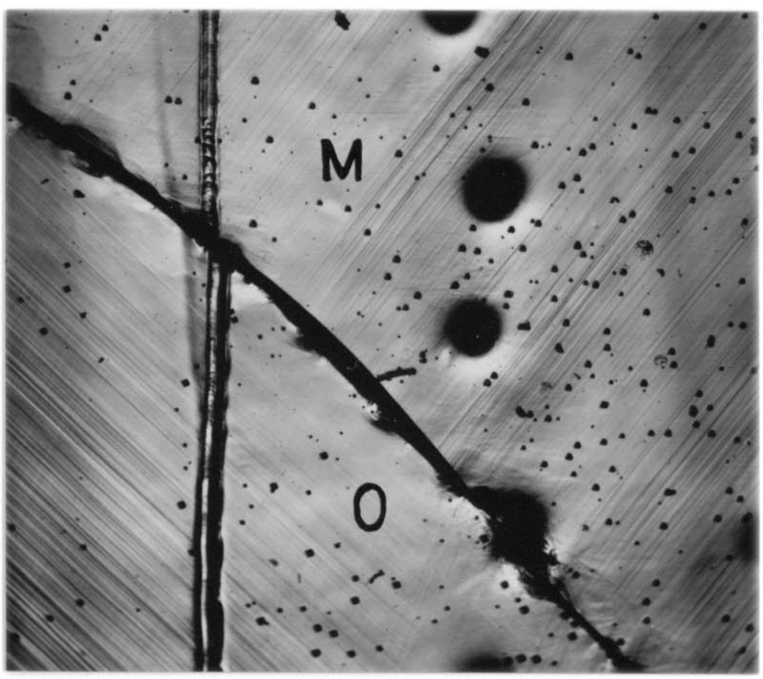


Figure 10. (Specimen P-3, front surface). Evidence and direction of boundary sliding by observing the offset of the scratched line and needle mark. 50 X.

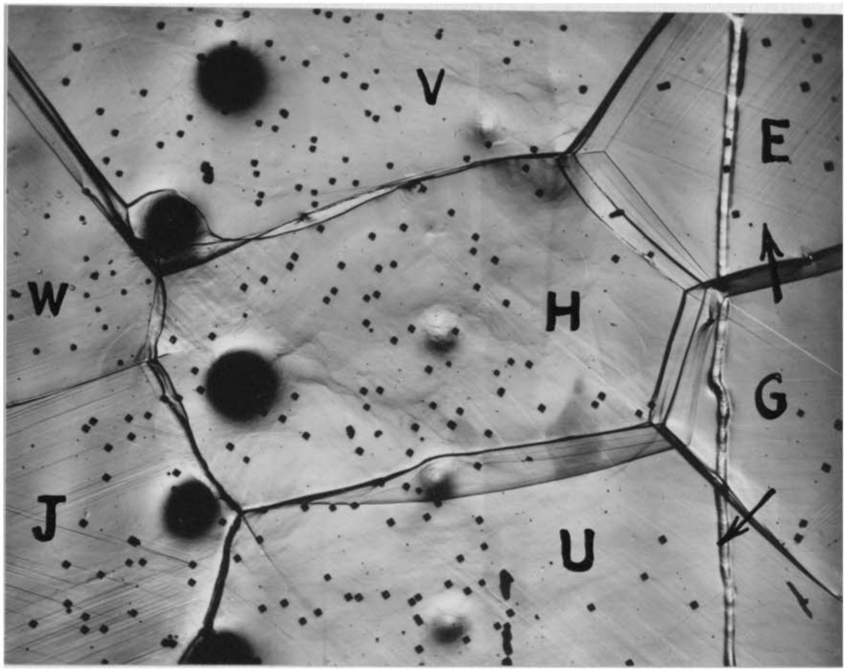


Figure 11 A. (Specimen P - 3, front surface). The enclosed grain was non-uniformly deformed; Grain G slid as a whole. 50 X.

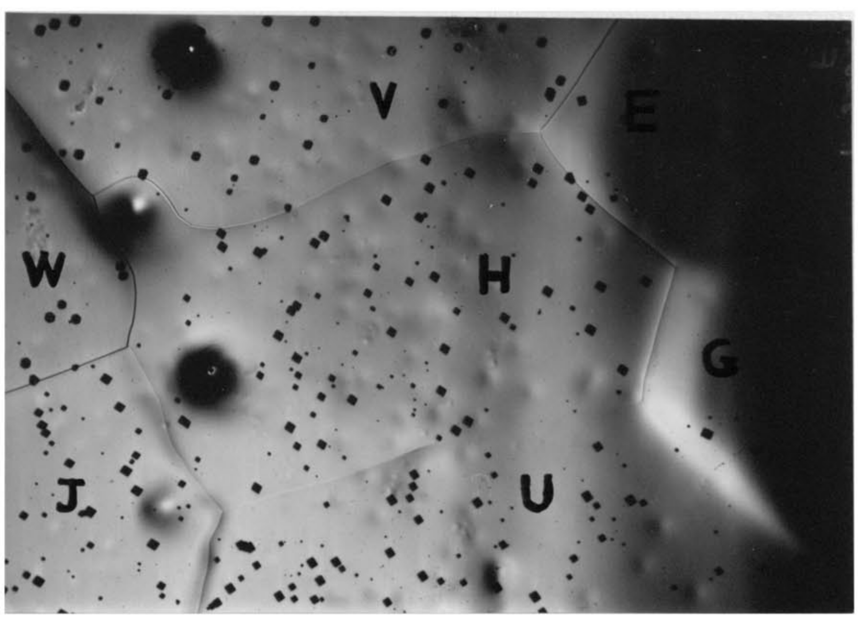


Figure 11 B. (Specimen P - s, front side). Same field as in Figure 5 A, repolished and etched.

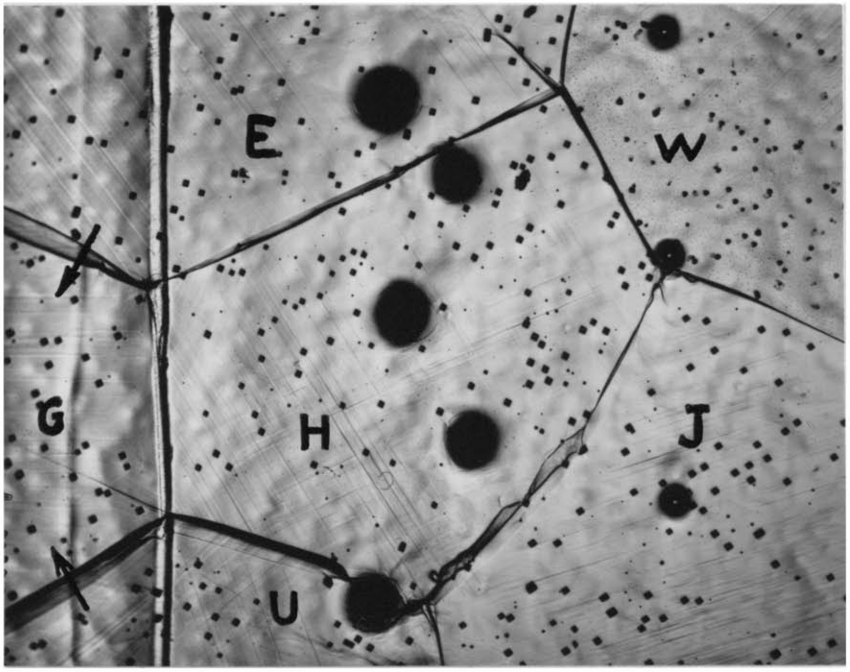


Figure 12 A. (Specimen P - 3, back surface). Grain G slid as a whole. 50 X.

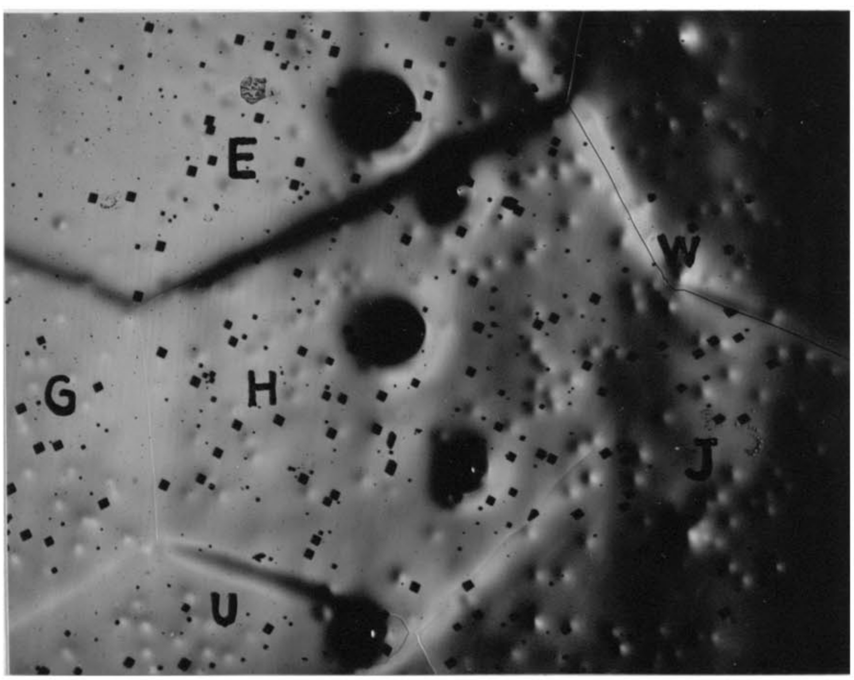


Figure 12 B. (Specimen P - 3, back surface). Same field as in Figure 12 A. Repolished and etched. 50 X.

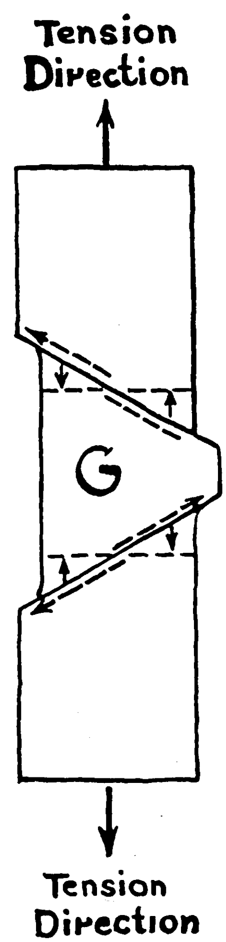


Figure 13. Schematic Drawing of the direction of boundary sliding and migration in specimen P - 3, Grain G. Broken Arrow indicates the direction of boundary sliding. Solid arrow indicates the direction of boundary migration. Dotted lines indicate a more stable position of the migrated grain boundaries.

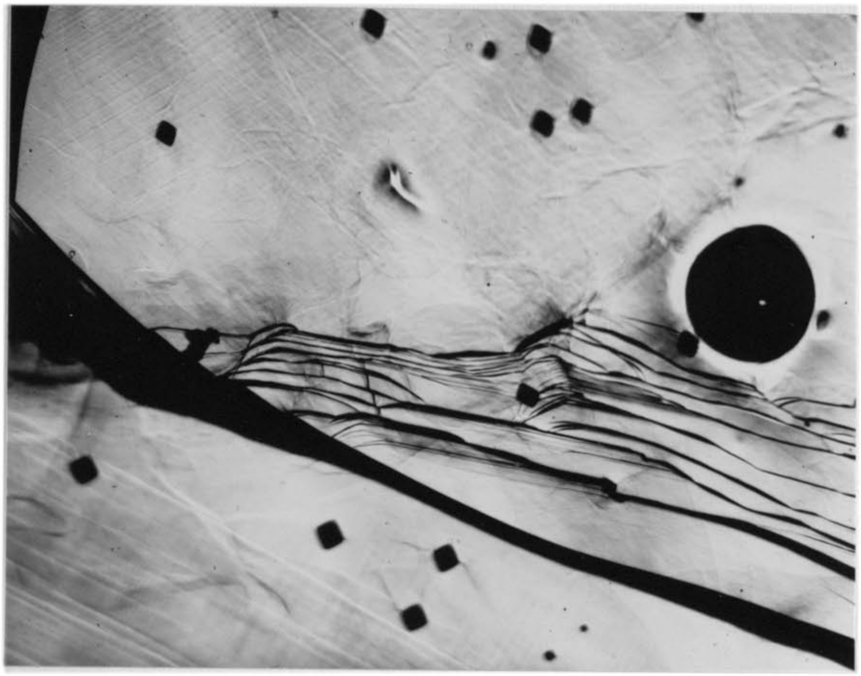


Figure 14. (Specimen P - 8, front surface). Uneven nature of grain boundary sliding and migration. 100 X.

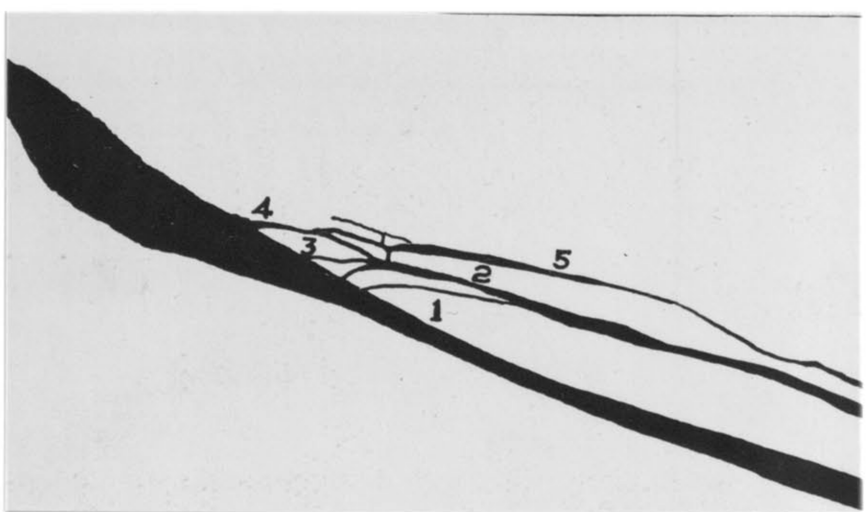


Figure 15. Schematic drawing of Figure 14 shows the uneven steps of boundary migration. The numbers indicate the consecutive steps of the grain boundary migration.

approach a position which is perpendicular to the specimen axis. Figure 16 is another example of this case. This position of the grain boundary is the one which has the smallest grain boundary area.

Figure 17 shows a grain boundary of grains A and B of specimen P-3. From Figure 2 it can be seen that grain B did not go all the way through the thickness of the specimen. The original double curved boundary tended to straighten out. It appears that the inflection point of this migrating boundary divides this boundary into two regions in which it migrated in opposite directions as shown by the arrows in Figure 17.

Figure 18 (specimen P-8) shows an interesting case in which grain E was apparently under compression since sliding had taken place along the boundaries of grains C and E and grains E and R. Furthermore, because of the blocking effect of the triple point of grains C, D, and E and of grains D, E, and R on the sliding of the boundaries of grains C and D and grains D and R, the grain boundary of grains D and E is subject to bending. The compression side of the bending is in grain E and the tension side is in grain D. It is probably due to this stress system and the locking effect of the two triple points that the upper part of the boundary of grains D and E bulges into grain E during progressive boundary migration. This kind of boundary migration typifies one in which the strain energy plays the dominant role.

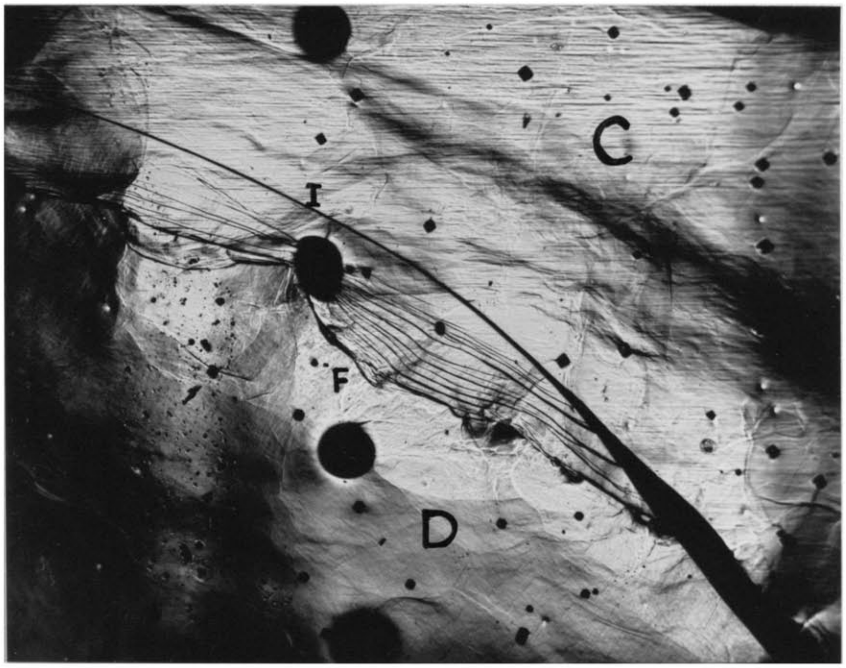


Figure 16. (Specimen P - 8, back surface). The grain boundary tended to rotate to a position perpendicular to the specimen axis. Note also the attracting effect of the needle mark on the migrating boundary. 50 X.

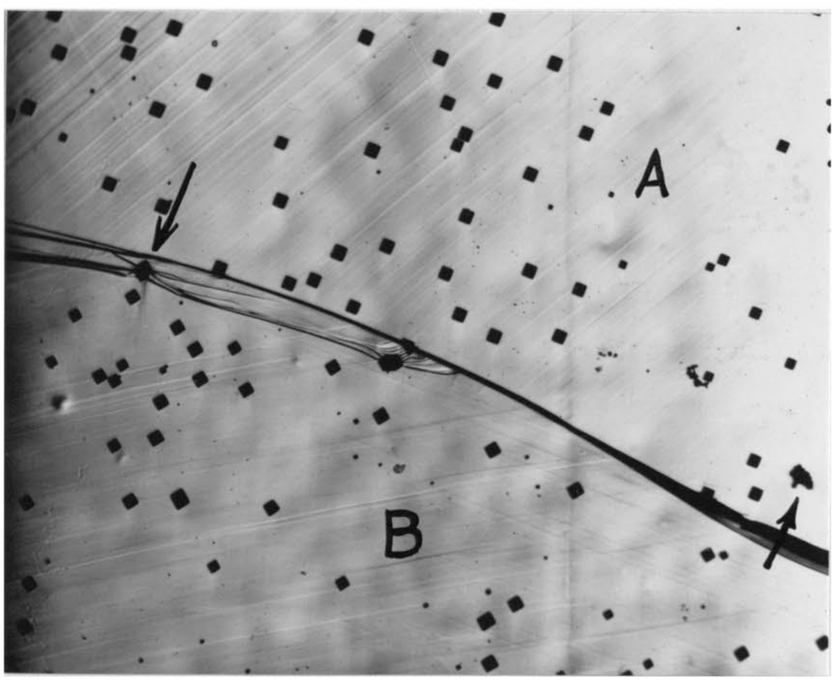
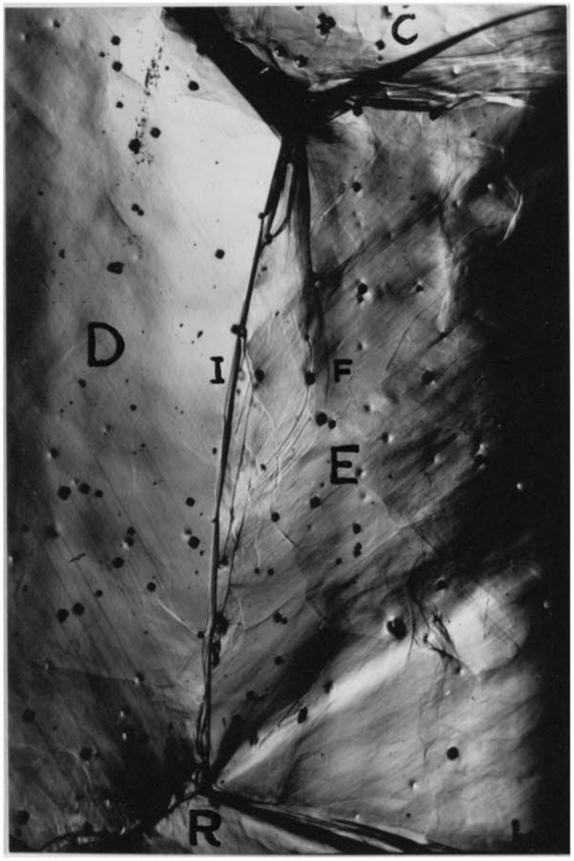


Figure 17. (Specimen P - 8, back surface). The boundary migrated in opposite directions, as shown by arrows. Note the attracting effect of the etch pit below the arrow at the left on the migrating grain boundary.



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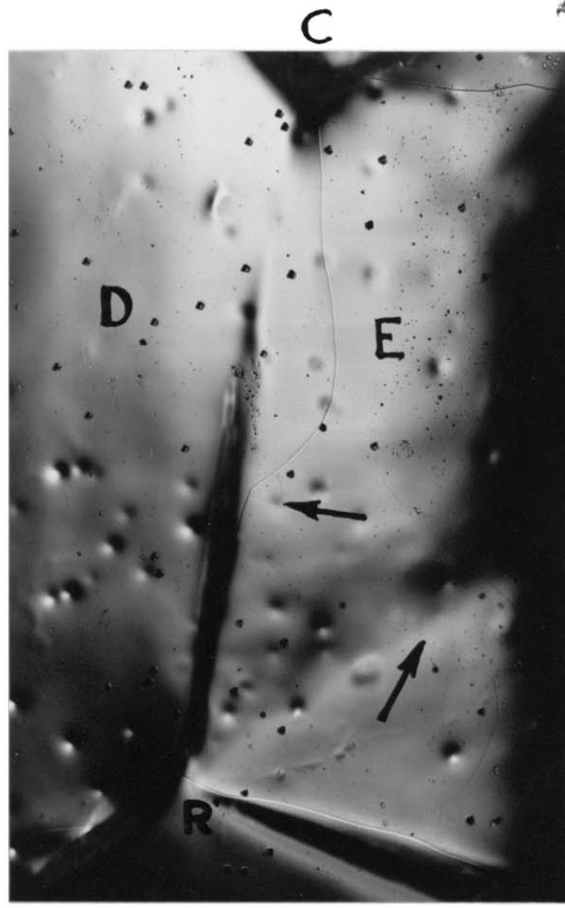


B

Figure 18 A. (Specimen P - 8, back surface). Uneven nature of grain boundary migration as a result of non-even distribution of stresses in grain E. 50 X.

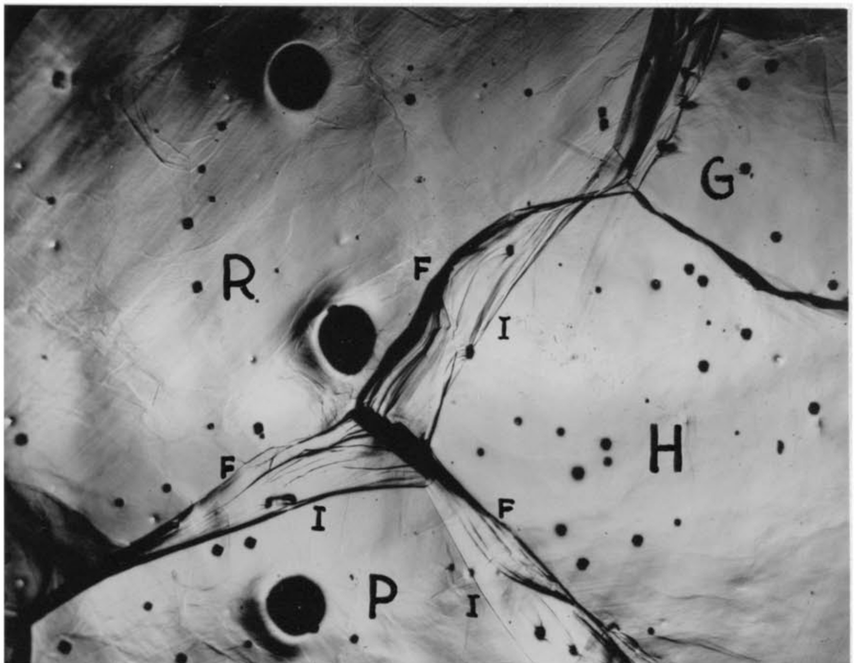
Figure 18 B. (Specimen P - 8, back surface). Same field as Figure 18 A. Repolished and etched. Sub-grain boundaries as shown by the arrows coincide well with the sharp bent marks on the deformed surface.

D. Effect of Grain Size on Boundary Sliding and Migration

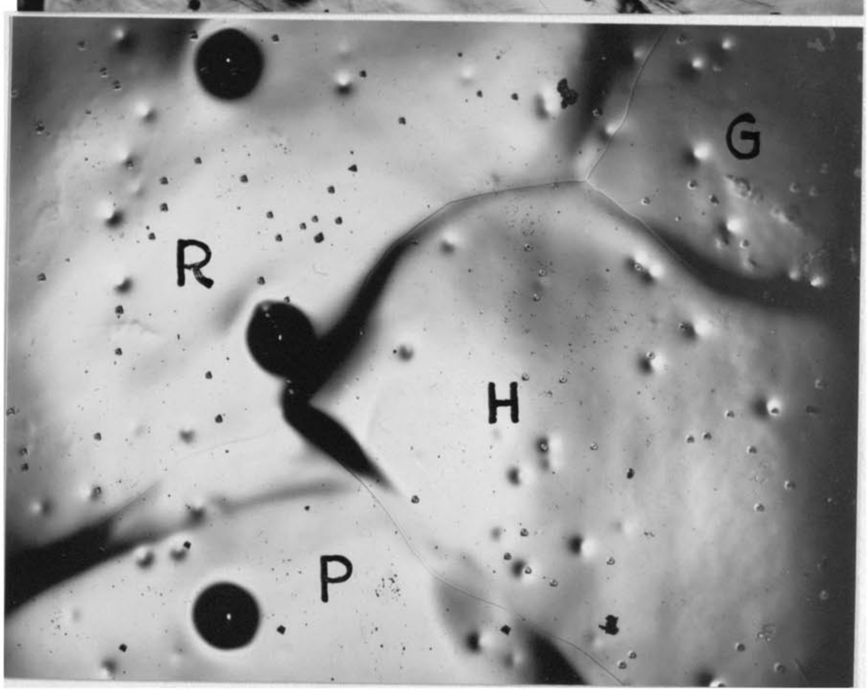
Figure 19 A shows a case where four grains go across the width of the specimen and all of them go through the thickness of the specimen. It is interesting to note the cooperative nature of grain boundary sliding and migration as evidenced by the appearance of the two triple points. Since the grain boundary of grains P and H is in a favorable situation (45° angle and plain boundary surface) to slide, the boundaries of grains P and R and of grains H and R migrated a large distance to meet this situation at the meighborhood of the triple point of grain P, R, and H. A clearer view of the migration process of the lower triple points of Figure 19 A can be obtained in Figure 19 C.

Figure 20 is a fine example showing the large extent of boundary migration when four grains go completely across the width of the specimen. I and F indicate the initial and final positions of the grain boundaries as before. It appears that the direction of the movement of a triple point is determined primarily by the particular boundary which is sliding at that instant. The observation that the path of movement of the triple point is zig-zag is not unexpected. It should be emplasized that the specimen from which Figure 20 was obtained was subjected to creep for 234 hours at 700° F and therefore more time was allowed for this extensive boundary migration to take place.

Figure 21 A shows an interesting case of how a small surface grain was consumed by three large neighboring grains. This is evident by a study of Figure 21 B, which shows the specimen repolished



A



B

Figure 19 A. (Specimen P-8). Deformed surface showing boundary sliding and migration around the triple points. Thickness of the shadows produced by grain boundary sliding suggests the relative amount of sliding. Note the sub-grain formation in the top left field. 50 X.

Figure 19 B. (Specimen P-8). Same field as Figure 19 A repolished and etched. Initial grain boundary disappeared; final grain boundary remained. Crater-like regions are etch pit locations seen in Figure 19 A. 50 X.

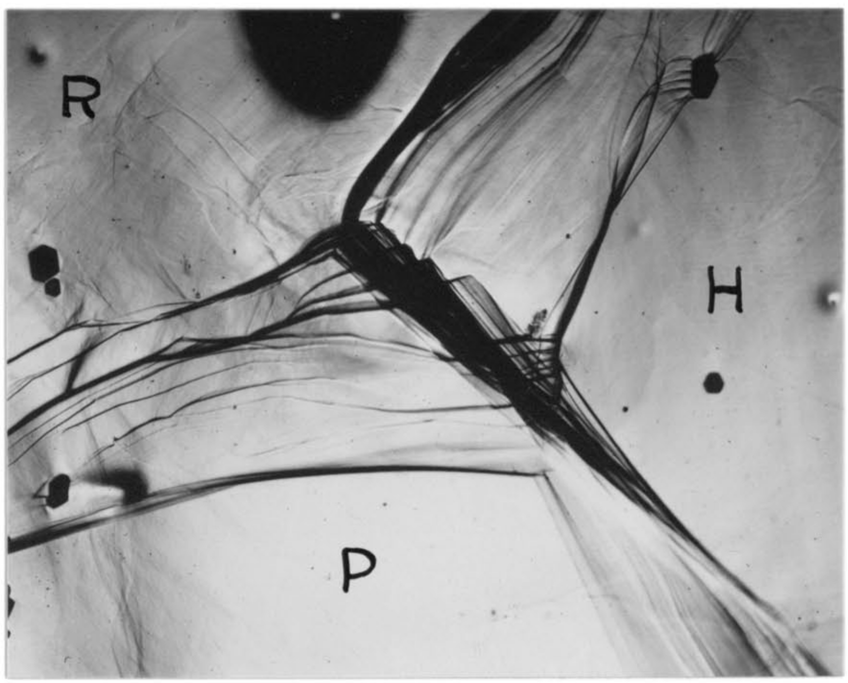


Figure 19 C. (Specimen P - 8). Higher magnification of the lower triple point of Figure 19 A. 150 X.

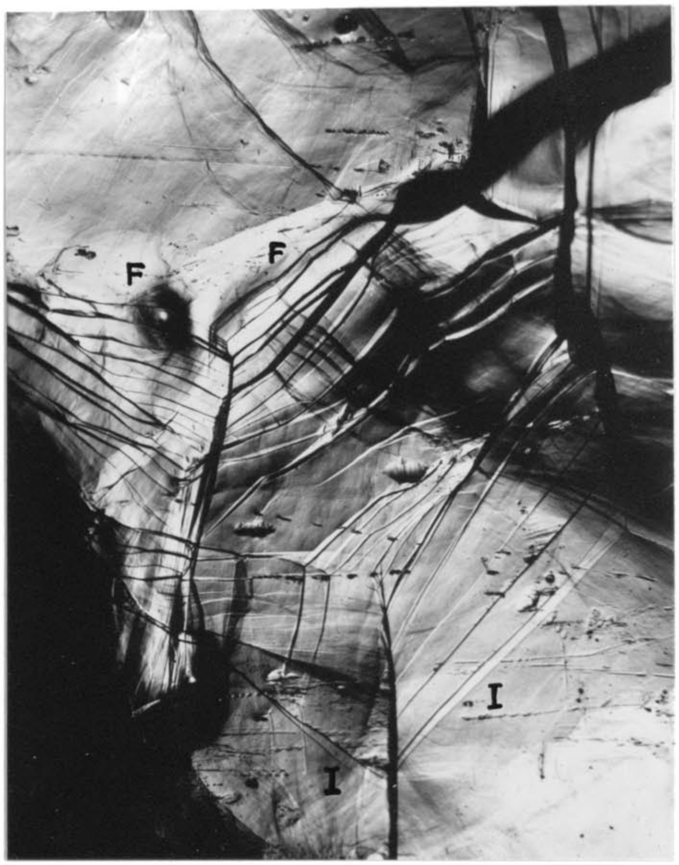


Figure 20. (Specimen P-1). Extensive boundary migration for a case where four grains of comparable size go across the width of the specimen. The zigzag path of the triple point is clearly shown. 50 X.

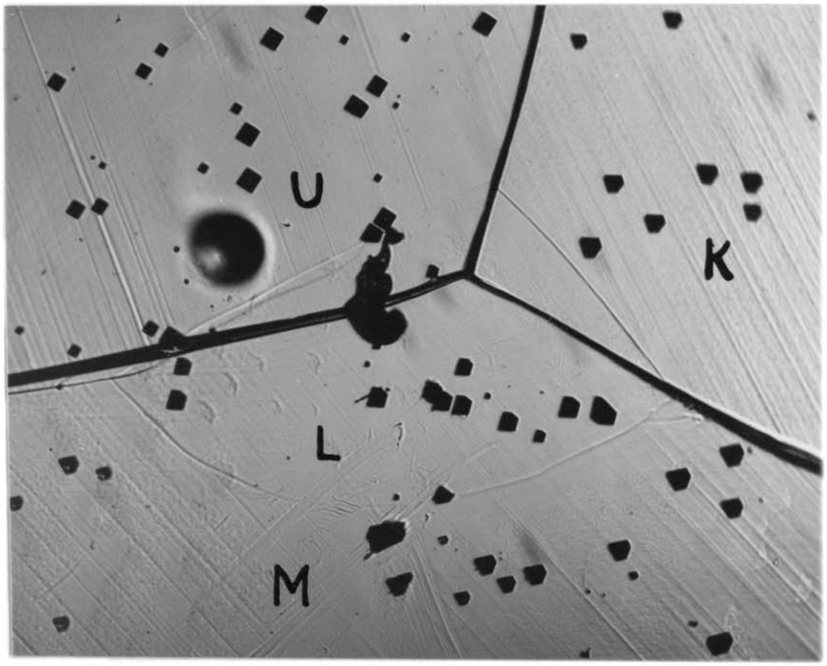


Figure 21 A. (Specimen P - 3, front surface). Deformed surface. Note how a small surface grain L was eaten up. 150 X.

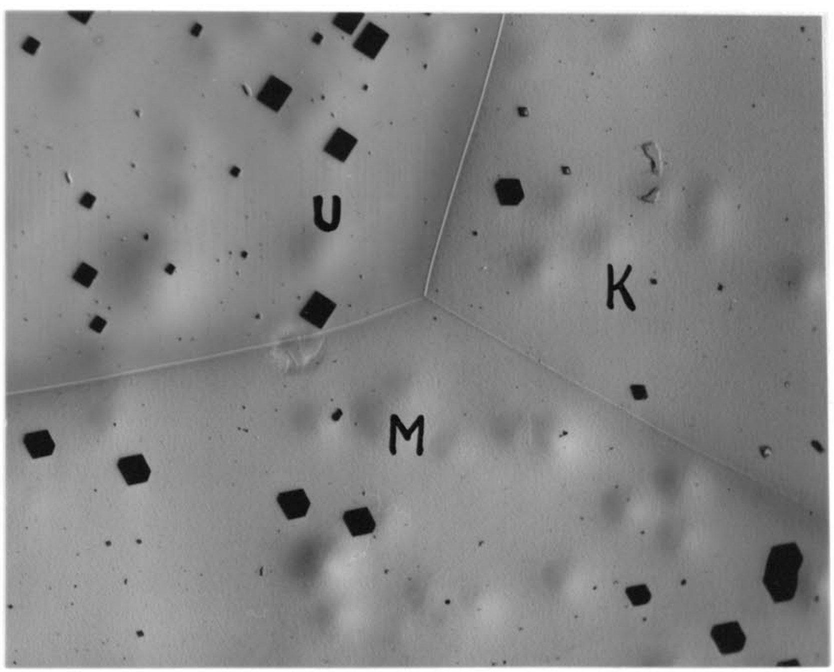


Figure 21 B. (Specimen P - 3, front surface). Same field as Figure 21 A. Repolished and etched. 150 X.

and etched. It can be seen from Figure 2 that after the disappearance of the small grain, the grain boundaries of the three large grains went through the thickness of the specimen.

E. Effect of Temperature on Boundary Sliding and Migration

As the temperature increases, the atomic mobility increases, and so does the rate of boundary migration. Figures 22 and 23 were taken from the same field during the creep process of specimen P-9 which was subjected to creep at 1100° F under a stress of about 65 psi. The rate of boundary migration was of the order of 1×10^{-5} cm/sec. at the beginning of the test. It is interesting to note how the boundary bounded by the two triple points rotated about 90° (Figure 23).

Figure 24 is a schematic drawing showing the steps of this rotation process, solid arrows indicating the direction of the migration of the triple points. The two triple points must come close to each other, or even meet at point O, before they can migrate in opposite directions. The final position lies at about 90° to the old grain boundary connecting the two old triple points.

Figure 25 through 29 show a similar case for specimen P-10 and were obtained at consecutive times during the test, 0.5, 3.25, 9.1, 18.5, and 30.4 hours after reaching the testing temperature, 1100° F.

The specimen fractured after 30.4 hours. The initial positions of the grain boundaries shown in Figure 5 were established before the test by macro-etching and are also indicated by the letter I in Figure 25. The triple point marked (1) in Figure 25 migrated through a zig-zag path in a stepwise manner toward the lower left direction while the triple point marked (2) migrated toward the

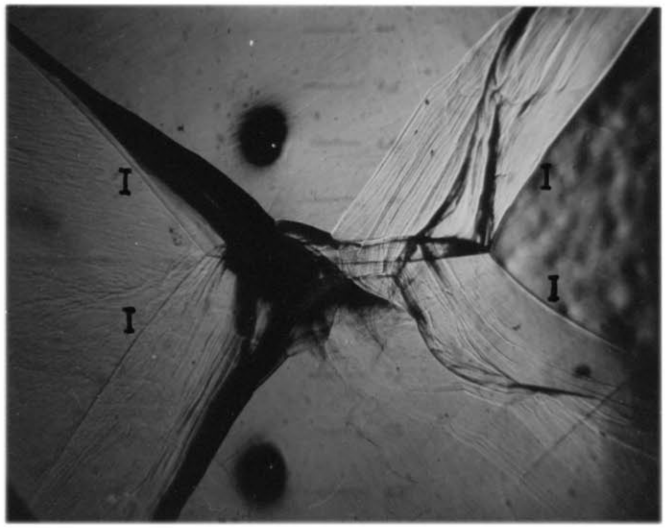


Figure 22. (Specimen P - 9). One hour after reaching 1100° F. Two triple points met in the center of the field and then separated in the top-bottom direction. 60 X.

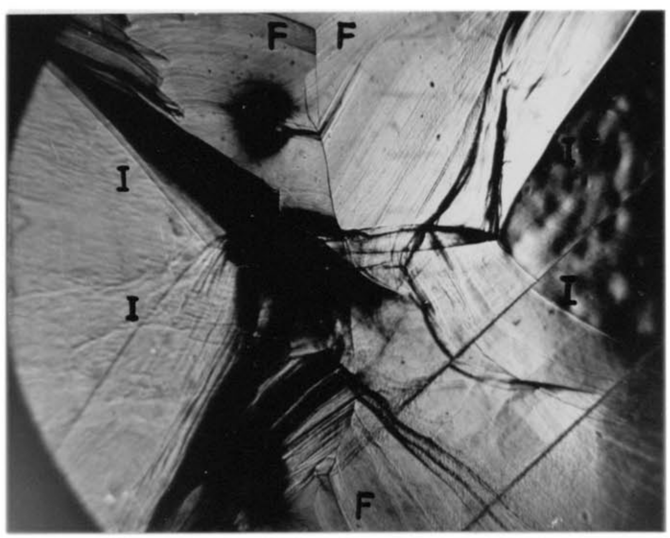


Figure 23. (Specimen P - 9). Same field as Figure 22. One hour and 35 minutes after reaching 1100° F. The grain boundary joining the two original triple points rotated 90 °. 60 X.

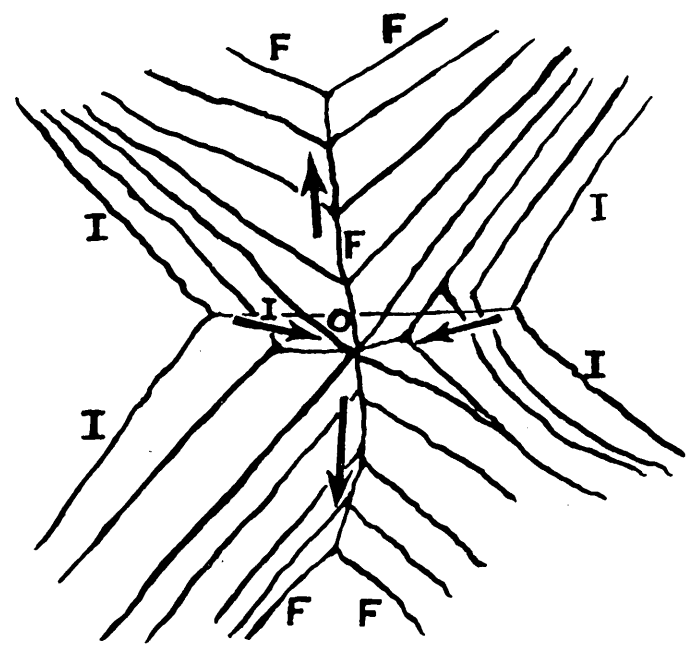


Figure 24. Schematic drawing of Figures 22 and 23 showing the steps whereby the grain boundary connecting two triple points rotated about 90°. I and F indicate the initial and final positions of grain boundaries and arrows indicate the directions of the migration of the triple points.

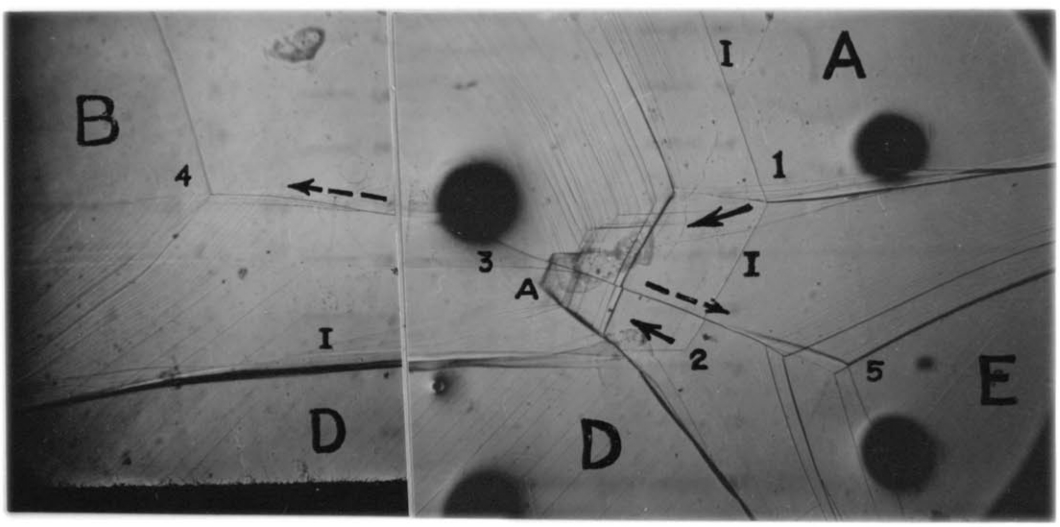


Figure 25. (Specimen P-10). One-half hour after reaching 1100°F. Note the initial grain boundary connecting two triple points, 1 and 2, rotated about 90°; now lying about 68° to the specimen axis. 60 X.
Solid arrows indicate the directions of the migration of the two triple points, 1 and 2, after which they met in the neighborhood of point 3. Dotted arrows indicate the directions of separation of the newly formed triple points to positions 4 and 5. 60 X.

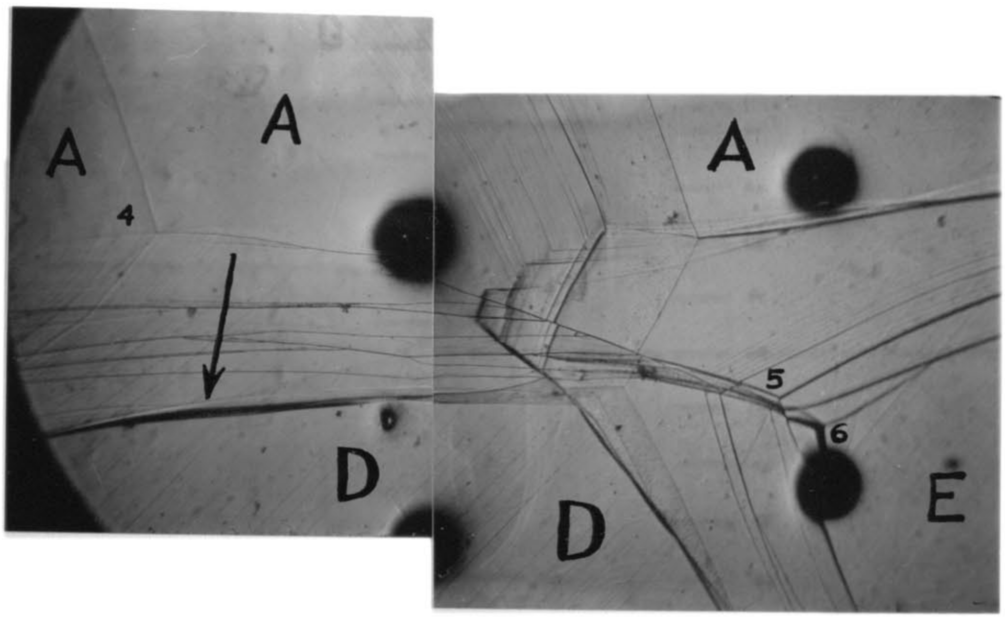


Figure 26. (Specimen P - 10). Same field as Figure 25. 3.25 hours after reaching 1100° F. The triple point 4 disappeared at the left edge of the specimen. The triple point 5 migrates to position 6. The new grain boundary tended to rotate perpendicular to the specimen axis as indicated by the solid arrow. 60 X.

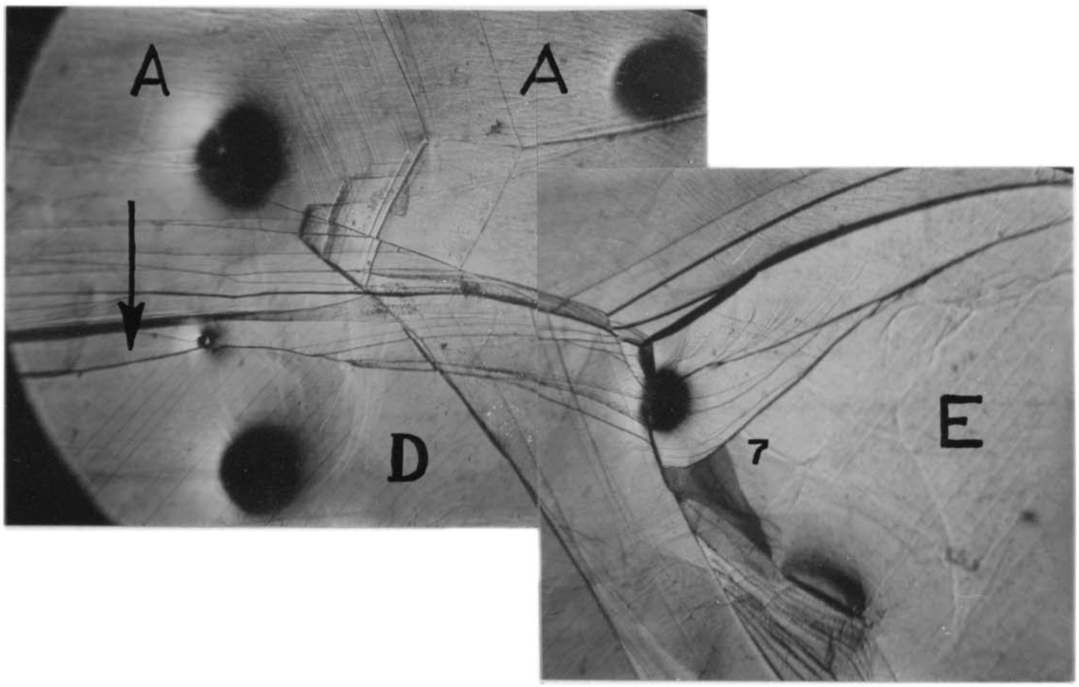


Figure 27. (Specimen P - 10). Same field as Figure 25. 9.1 hours after reaching 1100° F. The triple point 5 in Figure 25 continued to migrate toward the right edge of the specimen. The new boundary continued to rotate perpendicular to the specimen axis as indicated by the solid arrow. 60 X.

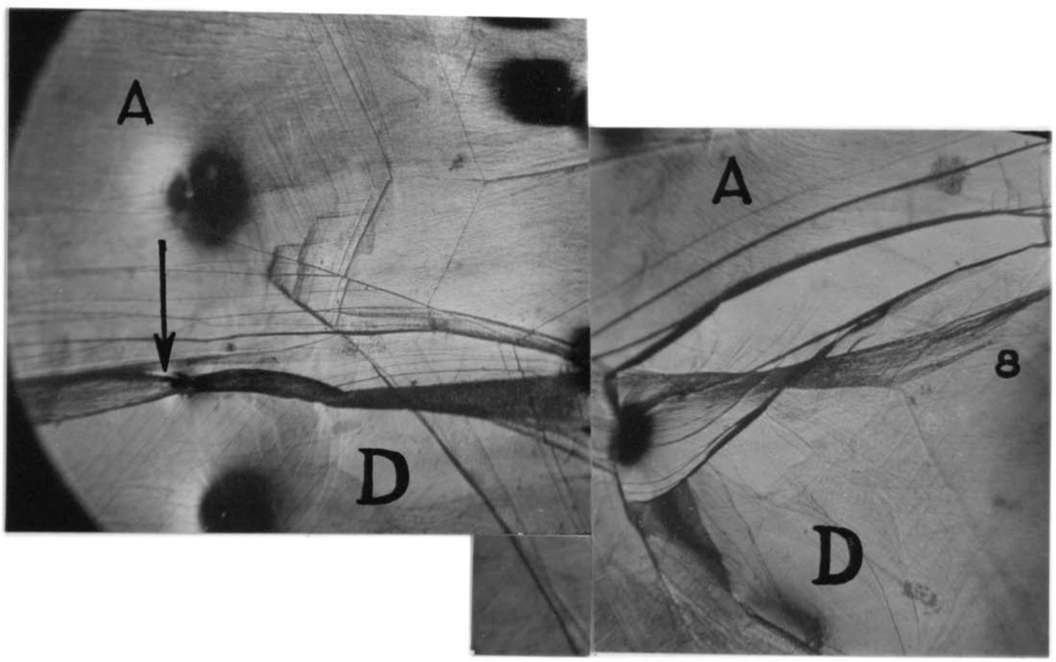
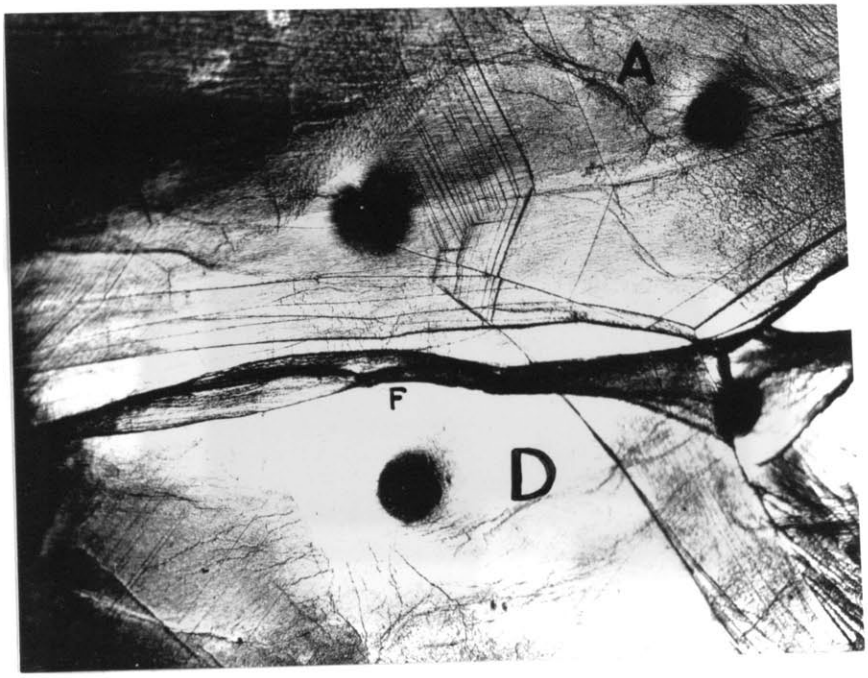


Figure 28. (Specimen P-10). Same field as Figure 25. 18.5 hours after reaching 1100° F. The triple point 5 in Figure 5 migrated to point 8. The migration of the new grain boundary indicated by the arrow almost stopped after reaching a position nearly perpendicular to the specimen axis. 60 X.



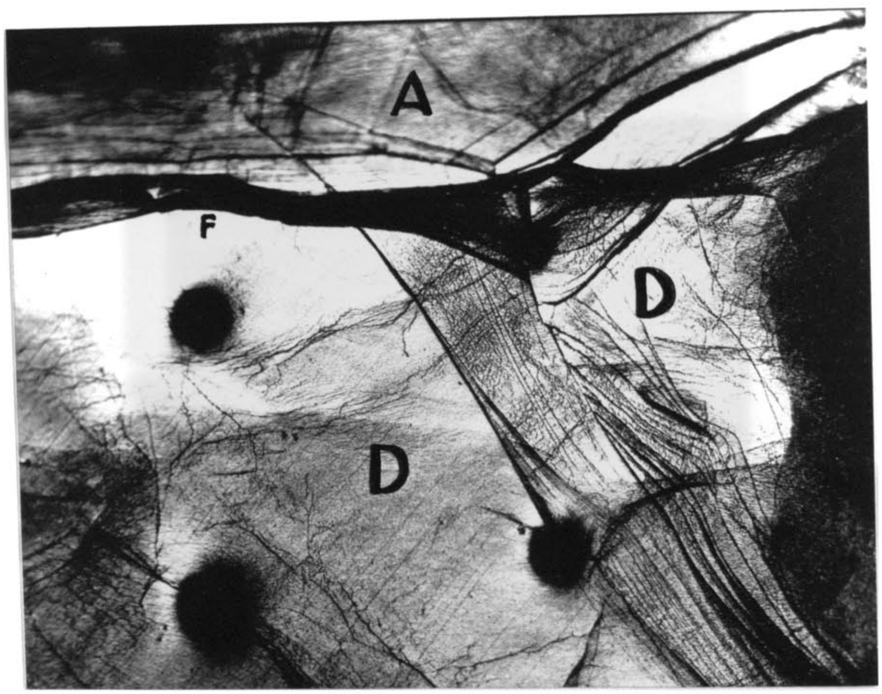


Figure 29. (Specimen P-10). Same field as Figure 25. 30.4 hours after reaching 1100° F. Specimen broke at this stage. The triple point 5 in Figure 25 almost disappeared at the right edge of the specimen. No appreciable migration of the new grain boundary indicated by the arrow. 50 X.

upper left direction in a stepwise manner, as indicated by the solid arrows in Figure 25. They came very close to each other, at point A (Figure 25). After point A, the appearance of the trails of the grain boundaries indicated that the two triple points must have separated very rapidly, one to the upper left (marked (4) in Figure 25) and the other to the lower right (marked (5) in Figure 25) as indicated by the dotted arrows. It is suggested that the two migrating triple points met at a common position marked (3) in Figure 25 during the creep process. This is a plausible suggestion based on the more exact picture shown in Figure 22. Once the two triple points occupy a common position, a maximum instability must exist from a grain boundary configuration viewpoint[8]. Due to the magnitude of this instability, two new triple points resulted. These new triple points migrated at a very rapid rate to new positions 4 and 5, indicated by the dashed arrows, leaving no trace of grain boundaries as is usual in a slower process of migration.

The left triple point (4) (Figure 25) disappeared at the left free edge of the specimen half an hour after reaching the creep testing temperature, 1100° F. While the right triple point marked (5) (Figure 25) continued to migrate toward the right edge of the specimen to a position (6), Figure 26, (taken 3.25 hours after reaching 1100° F). The grain boundary connecting the two new triple points now becomes one bounded by one triple point (6), Figure 26, and the left free edge of the specimen, and also is the boundary of grains A and D. The latter had migrated in steps in approaching a position almost perpendicular to the axis of the specimen, as shown by the arrow in Figure 26. Evidences of sliding of this boundary during consecutive steps of migration can be obtained by observing the offset of traces of the old grain boundaries (Figure 25). Figure 27, taken 9.1 hours after reaching 1100° F, indicate that the boundary of grains A and D swung to a position even still closer to 90° to the axis of the specimen and the new triple point, (6) in Figure 26, of grains A, E, and D moved closer to the right free edge of the specimen, as shown by (7) in Figure 27. At the end of the test, the grain boundary of grains A and D had migrated to a position almost perpendicular to the axis of the specimen while the new triple point of grain A, E, and D almost disappeared at the right free edge of the specimen, (Figure 29 A and Figure 29 B).

It is to be expected that as the grain boundary migrates to a position which is nearly perpendicular to the axis of the specimen further boundary migration would be small. Figures 30 and 31 show that that is the case. During the first 30 minutes at 1100° F under a stress of 65 psi, the boundary of grains D and F had moved a distance of about 0.05 cm. Figure 31, taken 2.5 hours after reaching 1100° F, shows that no appreciable further migration had taken place during a two-hour period.

No clear steps of boundary migration were found for specimens which were subjected to creep at 400° F for 475 hours. However, some evidence of grain boundary sliding was observed (Figures 73 A-F).

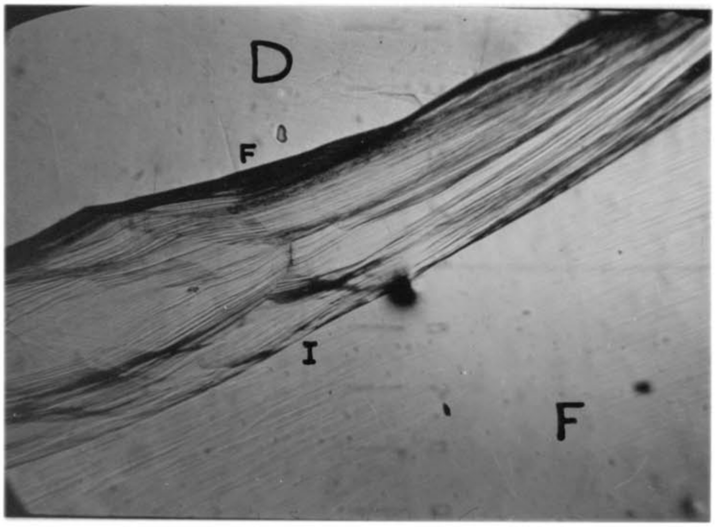


Figure 30. (Specimen P-9). 30 minutes after reaching 1100° F. Boundary migrated about .05 cm. during this period. 60 X.

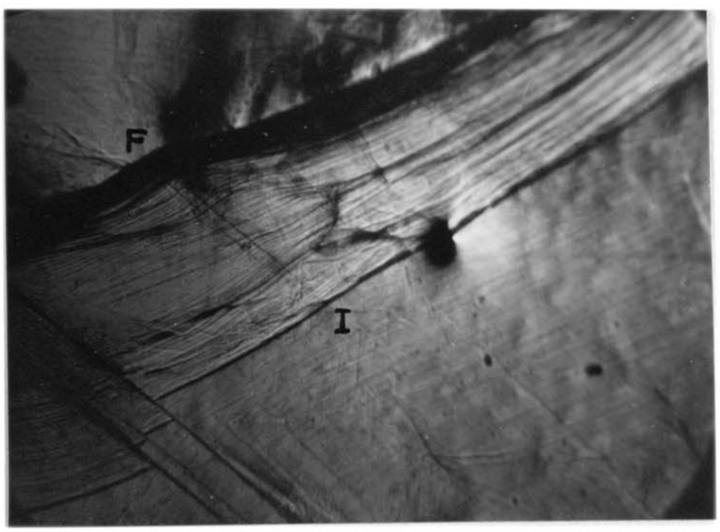


Figure 31. (Specimen P-9). 2 hours and 30 minutes after reaching 1100° F. Negligible migration during this period. 60 X.

F. Effect of Surface Irregularity on Boundary Migration

From all the photomicrographs presented above it can be seen that the course of grain boundary migration was affected to an important extent by the existance of etch pits. Figure 17 shows that the part of the grain boundary migrating in the direction of the arrow near an etch pit moves ahead of the rest of the grain boundary on the approaching fore-side of the etch pit. The balance of the grain boundary finally catches up with that portion already at the etch pit, but while the unimpeded grain boundary then proceeds to move on, the grain boundary at the etch pit lags behind in the migration process and appears reluctant to leave the etch pit vicinity. Needle marks exert a similar effect as shown by the grain boundary migration pattern in Figure 16. This would suggest that the grain boundary seeks a lower surface energy position; in the above case, this is offered by the etch pits or needle marks. It should be pointed out that this effect persisted quite deep into the thickness of the specimen since the same effect was still noted after removing the pits by electropolishing off a surface layer of above 0.02 mm.

It is interesting to examine closely the deformed surface and repolished and etched surface. The crater-like spots in Figure 7 B and 9 B are the old etch pits of Figure 7 A and 9 A which were put in prior to testing. Note that they do not coincide with the new etch pits after repolishing and etching. This statement also applied to other photomicrographs of repolished surfaces (Figures 11 B, 12B, 18 B, 19 B).

G. Appearance of Grain Boundary After Boundary Sliding and Migration

Figure 32 shows that an originally smooth grain boundary became angular after boundary sliding and migration. In fact, every sharply bent segment along this new wavy grain boundary appears to be due to the formation of sub-grains along this boundary as shown in Figure 33 A. (A higher magnification of the same boundary in Figure 32 and Figure 33 B is included to show the structure more clearly.) It should be emphasized that whenever a migrating boundary encounters a scratch, etch pit, inclusion or needle mark, it may become angular, however, it may also become sharply irregular even if there are no such disturbing physical barriers in its way (see Figure 14). This phenomenon occurs more frequently and prominently in the case of heavily slid boundaries. This effect could also be caused by second phase particles and various precipitates.

H. Slip Band Formation

Figure 34 A shows that two parallel slip bands which developed along the same slip system and overshot a considerable distance into the region where the other slip band was operative. Obviously high stress concentration in the overlapped region was relieved by cross slip[9] though oxide cracks slightly masked their appearance. Figure 34 B shows an extension of the area in Figure 34 A and was taken close to the left edge of specimen P-10. When these photomicrographs were taken the grain boundary of grains K and L had migrated below the reference mark (3) of Figure 34 B, as indicated by an arrow. Note also the steps left across the traces of the old grain boundary of

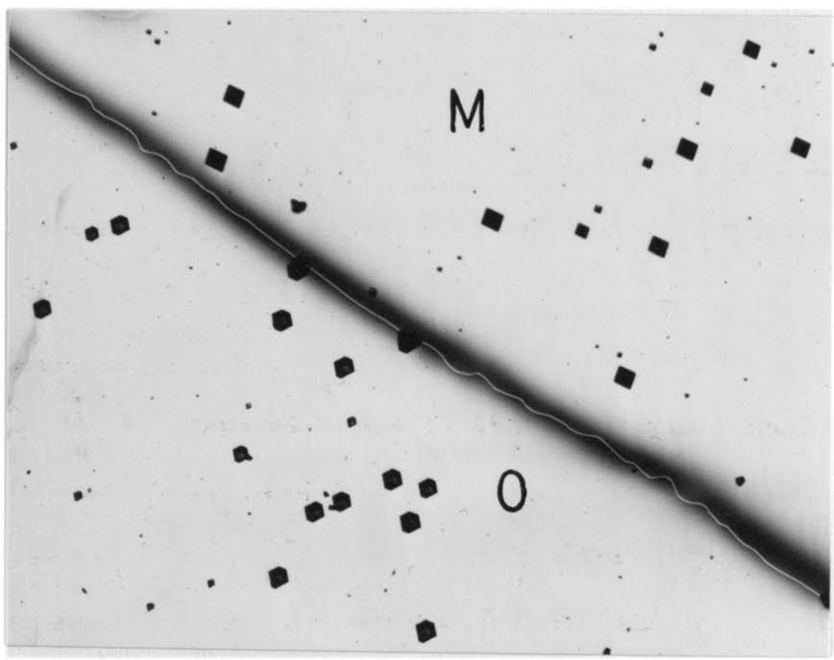
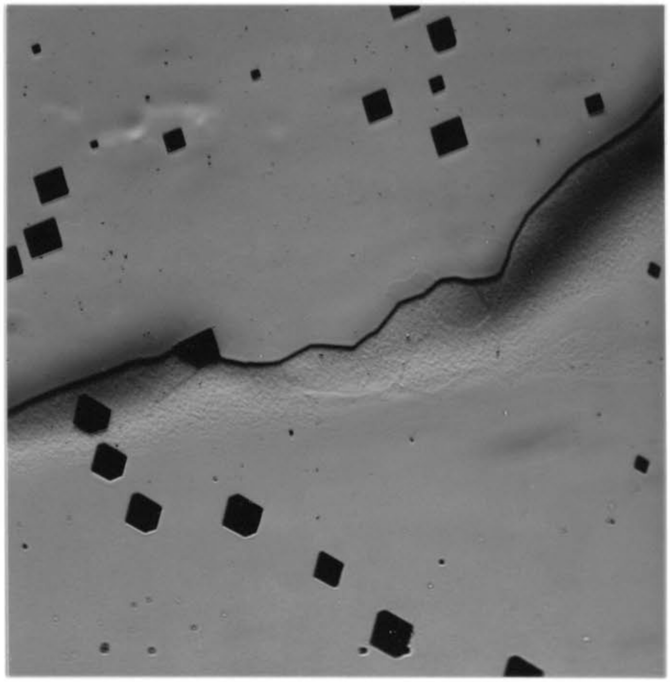
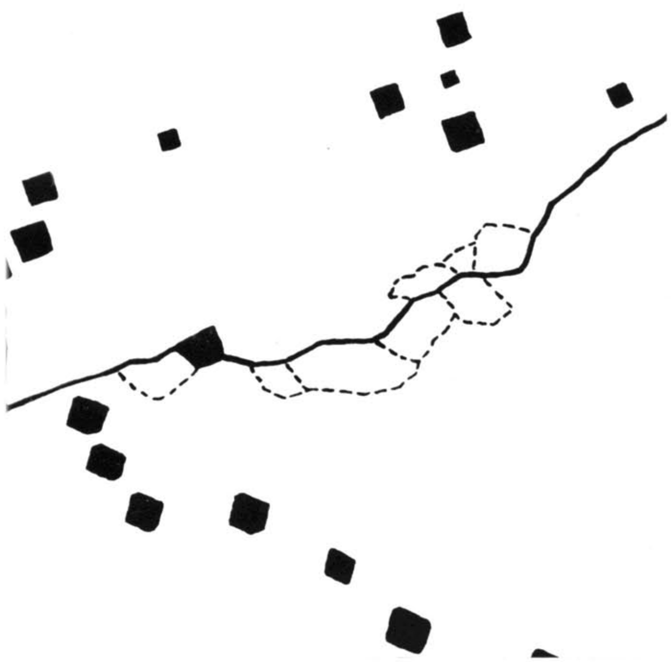


Figure 32. (Specimen P-3). Repolished and etched. Angular nature of grain boundary after boundary sliding and migration. 50 X.



A



B

Figure 33. (Specimen P-3). Appearance of boundary, originally straight, after creep test.
A. Photomicrograph at 150 X showing angular nature of sub-grain structure.
B. Sketch to show more clearly the structure of the grain boundary and sub-grains.

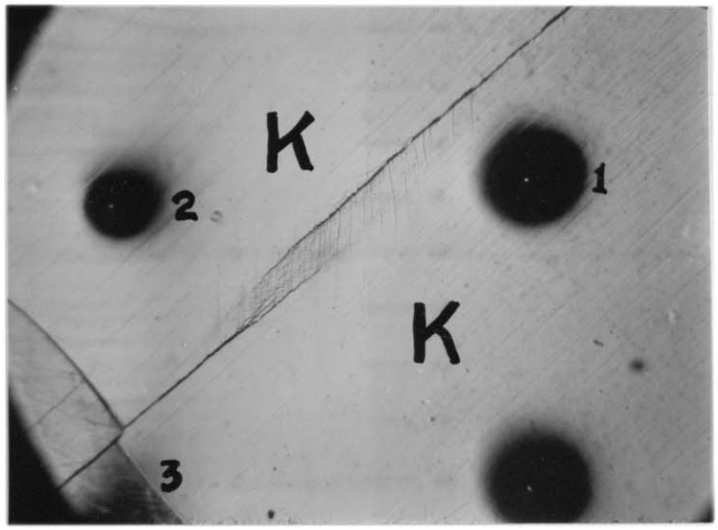


Figure 34 A (Specimen P-10, front side). 3.5 hours after reaching 1100° F. Two slip bands overshot each other and cross slip occurred between. 60 X.

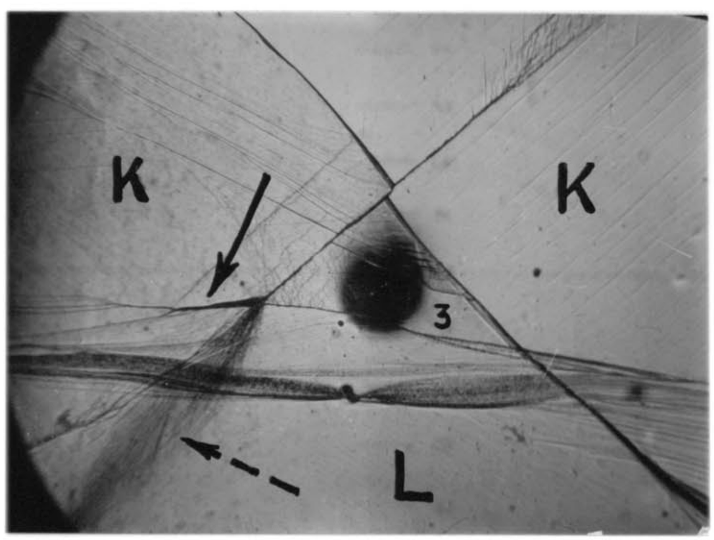


Figure 34 B (Specimen P-10, front side). 3.5 hours after reaching 1100° F. A continuation of Figure 34 A. Note the displacment of the old grain boundary caused by slip and the irregular deformation lines in grain L. 60 X.

grains K and L in Figure 34 B as a result of slip. Figure 35 is a photograph taken about 9.5 hours after reaching 1100° F for the same P-10 specimen. The two slip bands had merged into a wide band the width of which corresponds to the spacing of the original two slip bands. Individual bands within the wide band appear to cross each other in a complex manner to adjust for non-uniform deformation. It should be noted from Figures 34 - 36 that irregular deformation lines were produced in grain L, indicated by the dotted arrow in Figure 34 B, as a result of the development of the slip band in grain K. The fact that the general direction of the irregular deformation lines in grain L follows the direction of the slip band in grain K indicates that deformation tends to be continuous across the grain boundary. The course of the slip band is marked by the dotted line III in Figure 5 and also shown as the heavy solid line III on an enlarged scale in Figure 37. In fact, examination of the fractured specimen showed that the offset produced at the left edge of the specimen in grain L was similar to the one produced at the right edge of the specimen in grain K by the slip band. In order to accomodate the deformation produced by this slip band, an irregular deformation pattern was produced at the right edge of grain K in the later stage of the test as marked by an arrow in Figure 36.

Figure 38 shows two component creep curves across the same slip band as shown in Figures 34 - 36, and are composed of three cycles of decreasing and increasing creep rates. The term "component

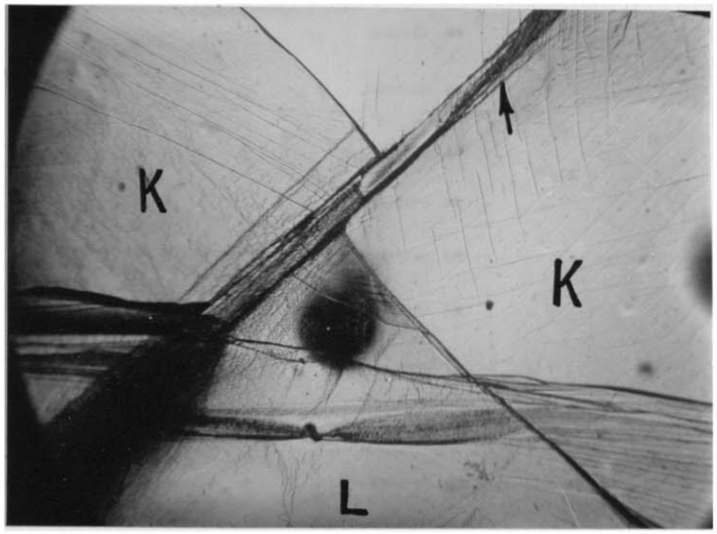


Figure 35 (Specimen P-10, front side). 9.5 hours after reaching 1100° F. Same field as Figure 34 B. Two original narrow slip bands merged into one wide slip band at this stage. Note also the steps left across the traces of the old grain boundary by the two original bands. 60 X.

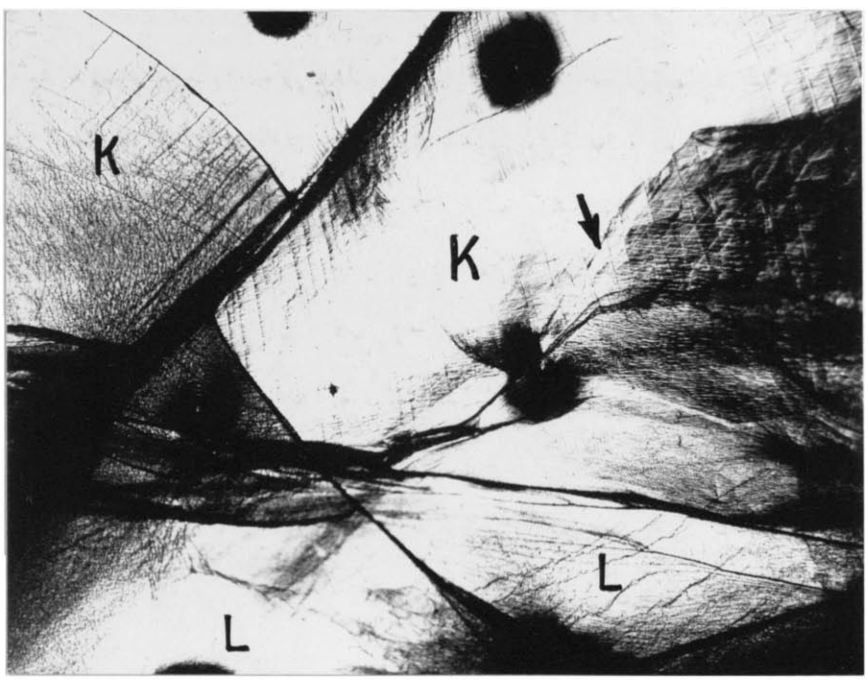


Figure 36 (Specimen P-10, front side). 30.4 hours after reaching 1100°F, fractured. The wide band bent at this step and non-homogenous deformation occurred close to the right edge of the specimen. 50 X.

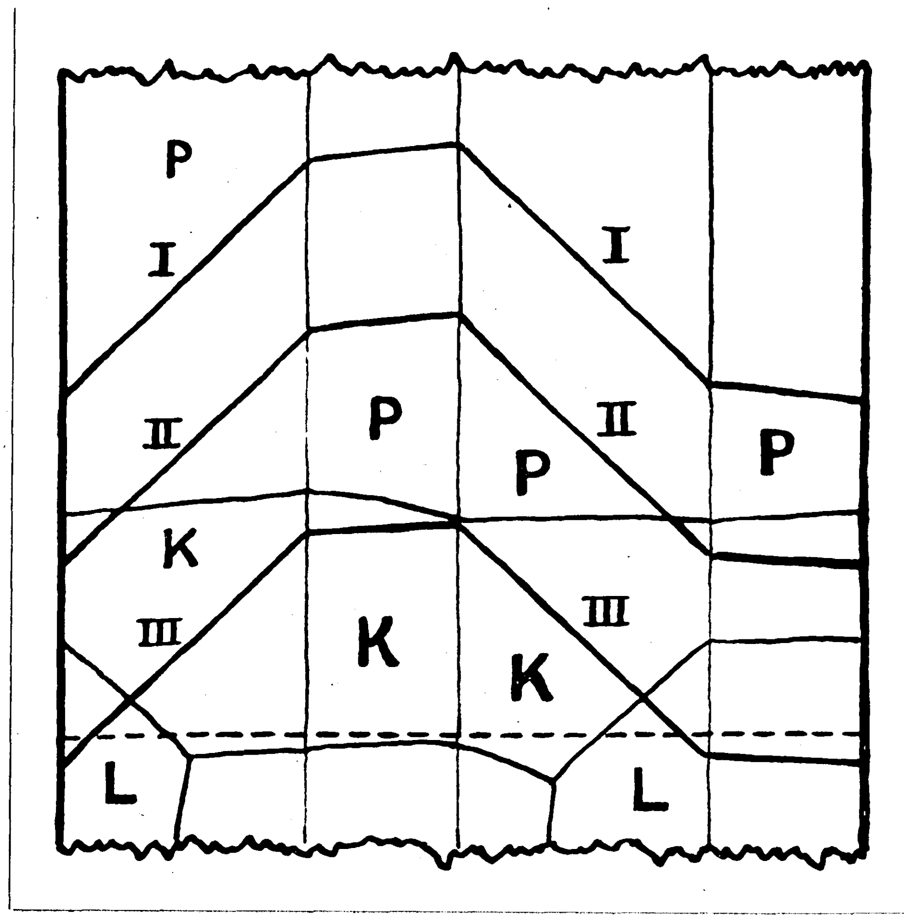


Figure 37. An enlargement of the lower part of Figure 5.
—— indicates the course of initial grain boundaries.
- - - indicates the courses of the grain boundary between K and L after migration.
━━━ indicates the courses of the slip bands.

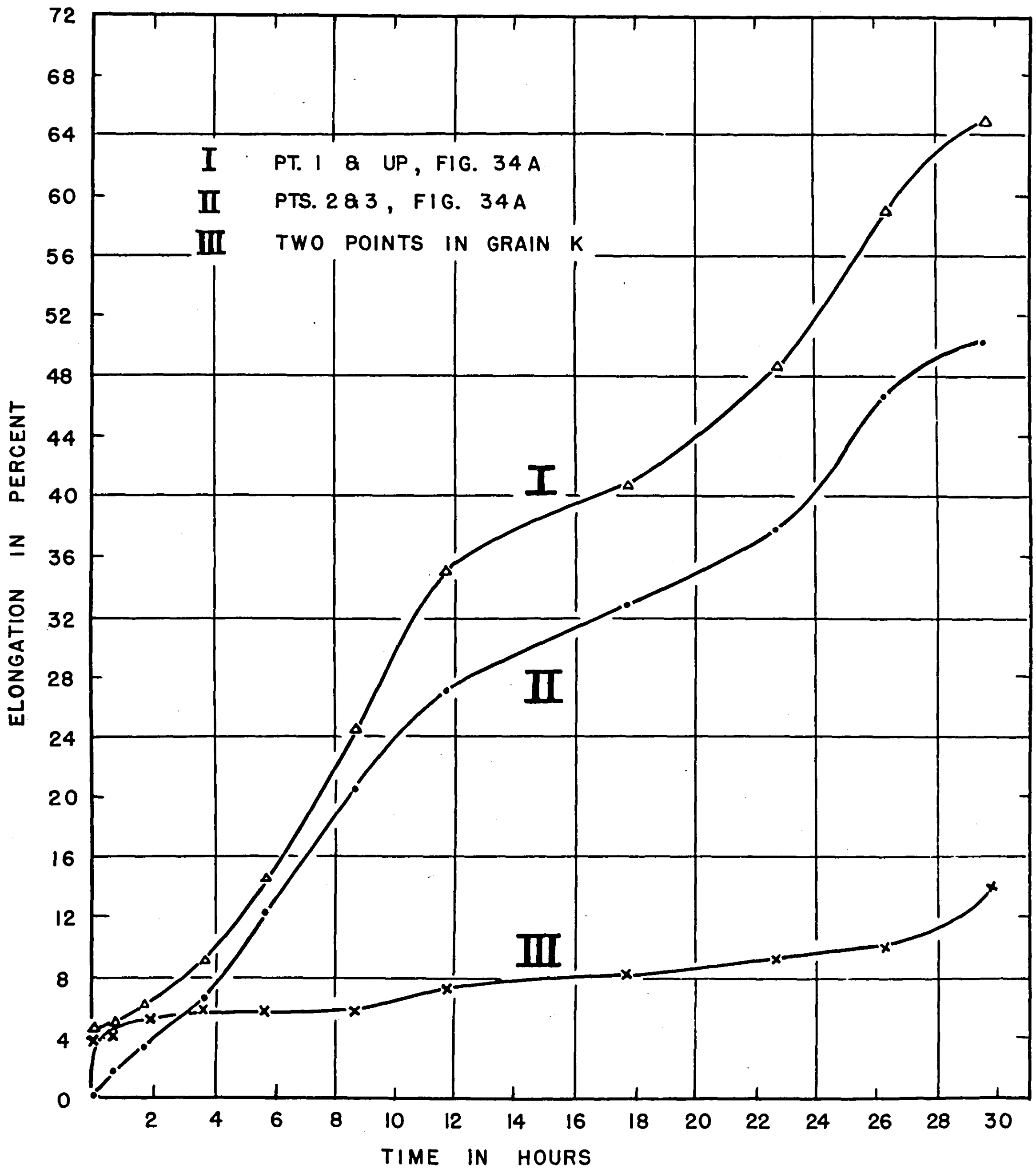


FIG. 38 COMPONENT CREEP CURVES IN GRAIN K
OF SPECIMEN P—10 (FIG. 37)

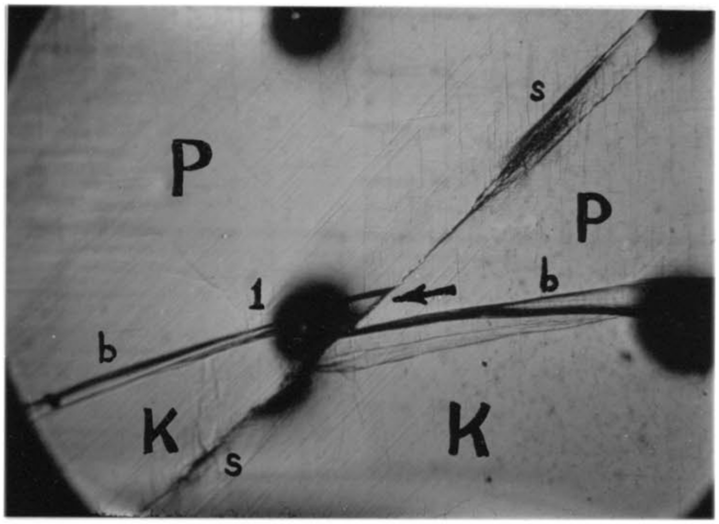
creep curve" is used here to indicate the creep curve obtained by measuring the change in distance between two reference marks in the direction of the applied tension. Curve I of Figure 38 was obtained between reference mark (1) in Figure 34 A and another reference mark immediately above the slip band. Creep curve II of Figure 38 was obtained between reference marks (2) and (3) in Figure 34 A. The course of this slip band is also indicated by the heavy solid line III in Figure 37. The region across which the curve I of Figure 38 was obtained was farther from, and therefore less influenced by, the boundary of grains L and K. Curve II, on the other hand, was obtained across the region which was very close to the boundary of grains L and K. Consequently, the elongation values shown in curve I are greater than those for corresponding times shown in curve II.

This observation implies that bending of the slip planes, or cross slip along different slip systems, or both, must occur in view of the unequal amount of slip in different parts of the same band. For the sake of comparison, a component creep curve across a slip band incompletely developed in grain K, owing to the blocking effect of the boundary of grains P and K, is also included in Figure 38, curve III.

Curve III has the same general characteristics as curves I and II, i.e., cycles of decreasing and increasing creep rates which, however, are much smaller in magnitude.

Figure 39 A shows that a slip band not only met the boundary between grains P and K but passed through and segmented this boundary into two parts. This boundary became continuous again only after a period of time was allowed for boundary migration to take place. This gave rise to a grain boundary bent at the slip band, as indicated by the dotted arrow in Figure 40 A.

At this stage it is necessary to refer to Figure 42 in which curves I and II were obtained across the same slip band shown in Figures 39 - 41. Curve I was obtained close to the left edge of the specimen and also close to the boundary between grains P and K while curve II was obtained close to the right edge of the specimen. For comparison, a creep curve III over the whole gage length of specimen P-10 is also included in Figure 42. Curve II shows that an initial period of about six hours results in a small strain of about one percent. This seems to indicate that a period of time is required to build up enough shear stress in the proper direction by thermal agitation to overcome the blocking effect of the boundary of grains K and L and whatever blocking effects may exist along the path of this would be slip bands[10]. As soon as these blocking effects were overcome, the strain increased very rapidly to about 20 percent elongation, but at a gradually decreasing creep rate. The behavior of the component creep curves during the period of the first 9.5 hours, agree well with Figures 39 A and 39 B, these figures being adjacent photomicrographs across the width of the specimen.



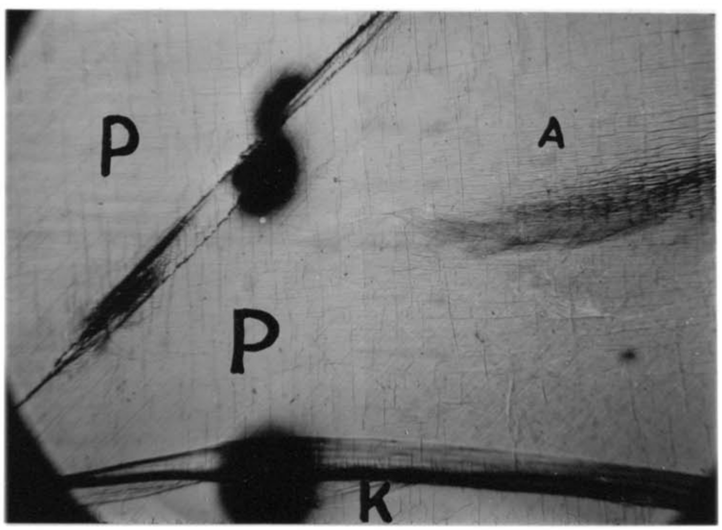
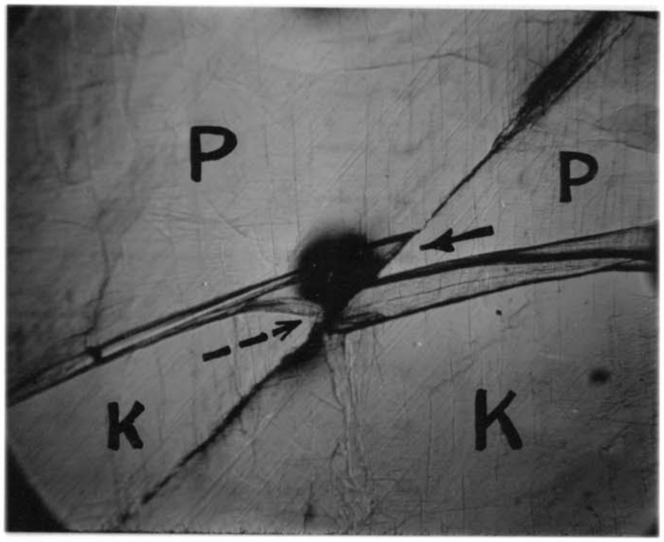


Figure 39. (Specimen P-10). 9.5 hours after reaching 1100° F. Figure 39 B is a continuation of Figure 39 A toward the right edge of the specimen. 60 X.

A. A slip band marked S passed through the grain boundary marked b and segmented this boundary into two parts. The offset so produced is indicated by an arrow.

B. Another slip band developed at the right edge of the specimen marked A.



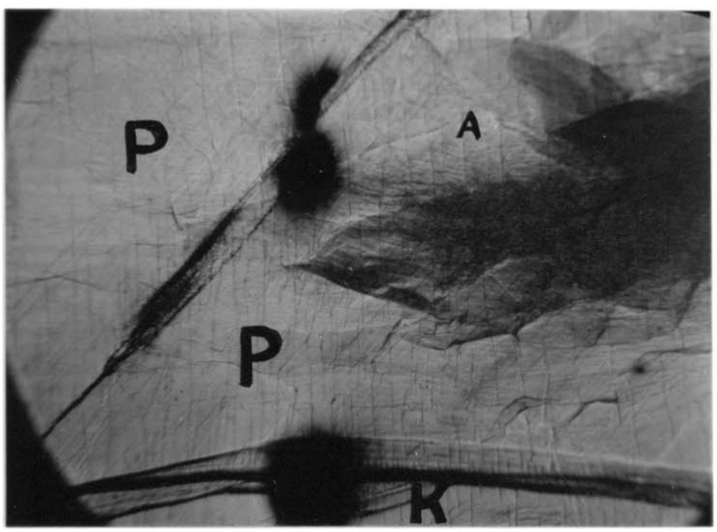


Figure 40. (Specimen P-10). 18.2 hours after reaching 1100° F. Figure 40 B is a continuation of Figure 40 A toward the right edge of the specimen. 60 X.

A. The displaced grain boundary of Figure 39 A becomes a continuous boundary bent at the slip band. No appreciable change in the amount of offset is shown by the arrow, as compared with Figure 39 A.

B. The fairly regular slip band indicated by A in Figure 39 A developed into a complicated deformation pattern as indicated by A in Figure 40 B.

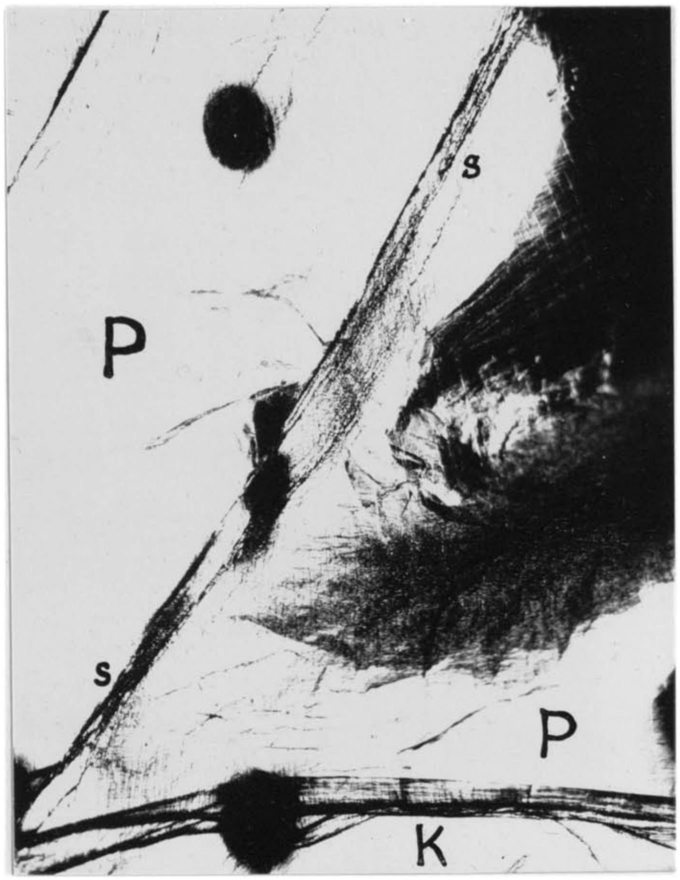


Figure 41. (Specimen P-10). 30.4 hours after reaching 1100° F. Same field as Figures 39 and 40. The band S was bent. Non-homogeneous deformation occurred in the region between the slip band and the grain boundary. 50 X.

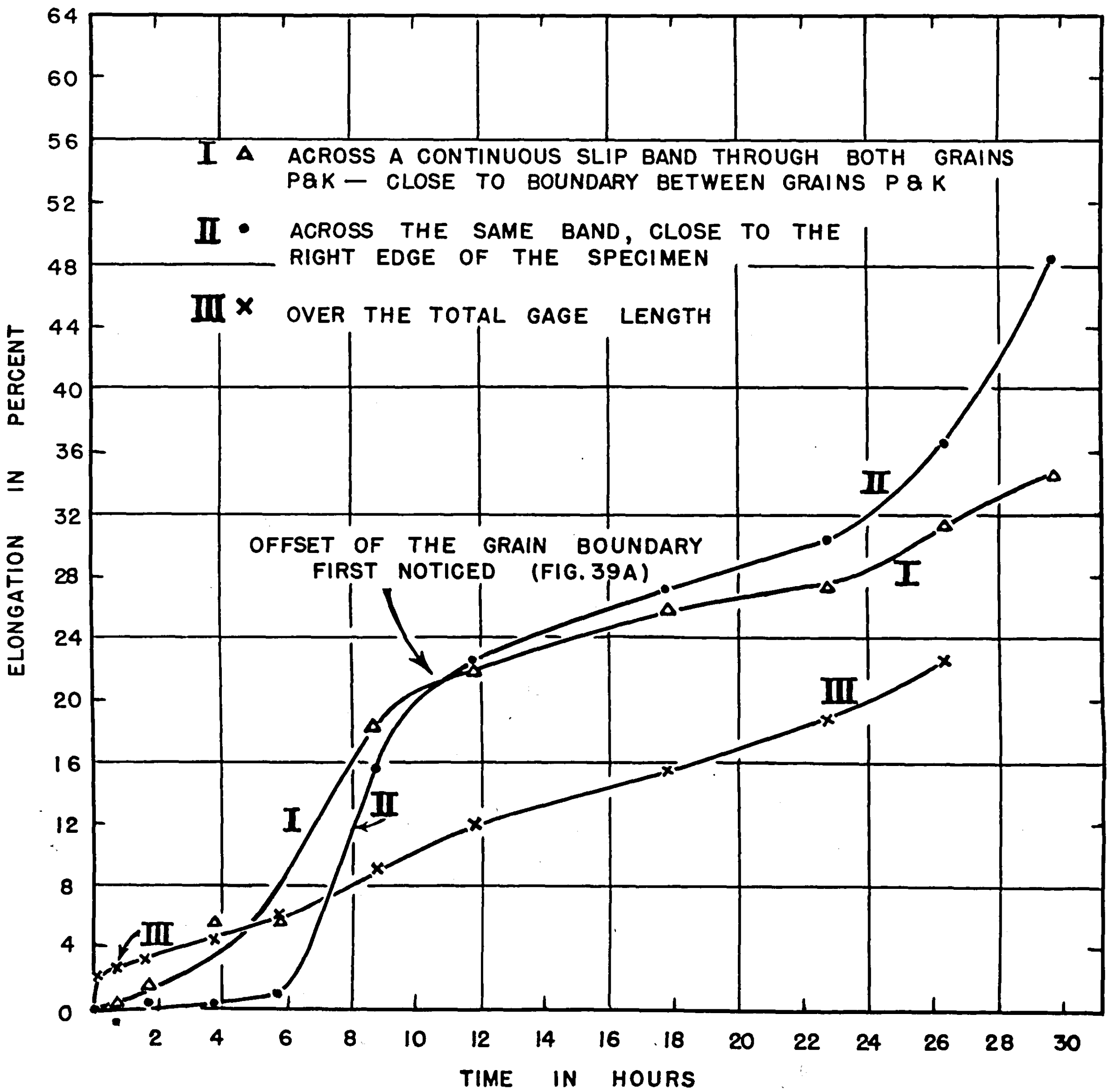


FIG. 42 CREEP CURVE OF THE TOTAL GAGE LENGTH AND TWO COMPONENT CREEP CURVES ACROSS THE SAME SLIP BAND THROUGH BOTH GRAINS P&K OF SPECIMEN P-10 (FIG. 37)

Figures 39 A and B were taken as soon as the slip band was developed. It can be noticed that the offset created by the slip band across the boundary between grains P and K (indicated by slid arrows in Figures 39 A and 40 A) shows practically no increase in magnitude in the period from the 9.5 to 18.21 hours of creep. This observation explains very well the flat portion of curves I and II in Figure 42. This result seems to show that the stress energy (or stress) built up along the slip band by thermal agitation is only enough to cause the two following changes. First, two new surfaces are produced along the slip band at the free edges of the specimen as indicated by 1 and 2, in the schematic drawing of Figure 39 A. Secondly, a certain amount of new surface area is formed between grains P and K along the slip band as shown by the arrow in Figure 39 A and designated as (3) in Figure 43 A. A period of boundary migration is required to change the appearance of that portion of the boundary designated as (3) in Figure 43 A, and shown in Figure 39 A, to that as shown in Figure 40 A and also shown as (4) in Figure 43 B. During this period the internal stresses created along the slip band must also have been reduced. It should be noted from Figure 39 B and 40 B that during the period from 9.5 to 18.2 hours, deformation occurred in the triangular region between the slip band and the boundary of grains K and P close to the right edge of the specimen. The deformation in this region started with fairly regular slip patterns (Figure 39 B), but ended up with a very complicated deformation pattern (Figure 40 B). Examination of the fractured specimen revealed that deformation at the right free edge of this

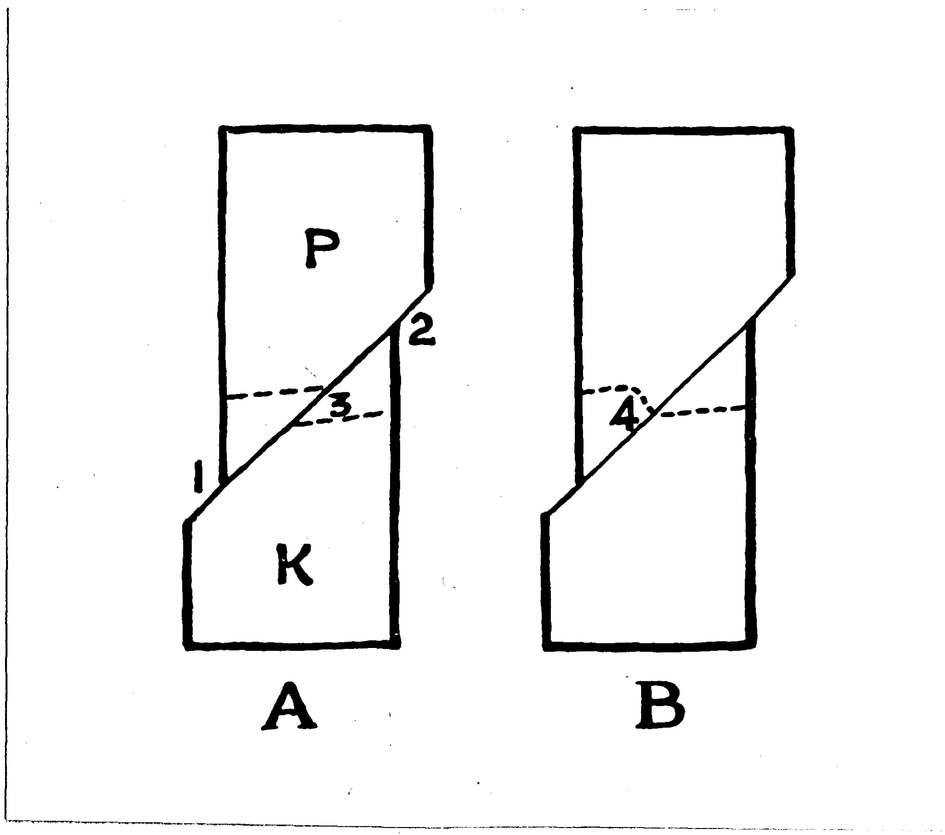


Figure 43 A and B.

A. Schematic drawing of Figure 39 A. Two new surfaces at the free edges of the specimen indicated by 1 and 2 and another new surface between grains P and K marked 3 were produced as a result of the slip band 1 3 2.

B. Schematic drawing of Figure 40 A. The displaced grain boundary becomes continuous again but bent at the slip band as indicated by 4.

\- - - indicates the grain boundary.

grain showed fairly regular traces of slip along the same slip system as the fully developed slip band in grain P. This observation implies that the material tends to deform by slip whenever and wherever it can. The complicated deformation pattern is the result of the restricting effects of the grain boundary between grains P and K, and the already developed slip band.

The final period of continuous increasing creep rate in Figure 42 is probably due to the fact that at this stage of creep the cross sectional area of the specimen was considerably reduced and the elongation of the other parts of the specimen permitted the rotation and bending of the slip band (132 in Figure 43 A) as evidenced in Figure 41.

It should be noted in Figure 42 that the curve I is above the curve II up to the 11th hour of creep after which the former is below the latter.

It can be seen from Figure 39 A that in the very early stages of creep the grain boundary of grains K and P migrated almost below the upper reference mark (1) in Figure 39 A (used for obtaining curve I in Figure 42), before the slip band S shown in Figure 39 A developed. The higher elongation values of curve I obtained in the early stage of creep may, therefore, be associated with the boundary sliding. Since curve II was obtained over a region far from the blocking effect of the boundary between grains K and P, the curve II is above the curve I in the later stage of creep as is the case in Figure 38.

It is interesting to examine further, the question of why the slip band shown in region A (Figure 39 B and 40 B) between slip bands II and III (Figure 37) did not develop fully. When this band started to develop, the boundary between K and L had already migrated to a position almost perpendicular to the specimen axis as shown by the dotted line in Figure 37. The boundary between grains K and L was therefore no longer in the path of this band. Consequently, on developing, this band would encounter only the same grain boundary (i.e., between P and K) as the slip band II did.

The only difference between these two bands is that their paths cover different areas in grain P and K though the area covered across the whole specimen is the same in both cases. It is reasonable to assume that the resistance of the grain boundary to be segmented is the same in both cases. Therefore, the explanation of this phenomenon must be sought in the different critical shear stresses of these two grains P and K.

Figures 44 through 47 show a series of photomicrographs indicating the development of a slip band in grain P (specimen P-10) to the time of fracture. The course of the slip band is shown by the line I in Figure 37. This grain started to deform by slip with a fairly uniform spacing, as shown in the region A in Figure 44. But as creep continued deformation tended to concentrate on a band of about 0.2 mm. width, as shown in the center of Figure 44, because this band did not encounter any grain boundaries.

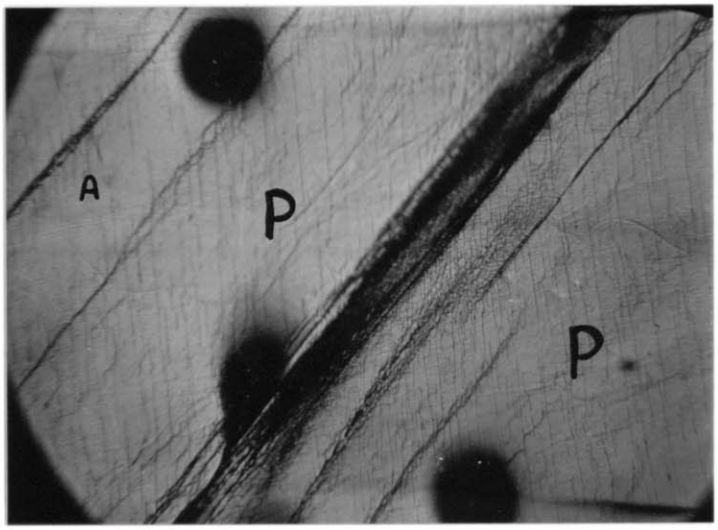


Figure 44. (Specimen P-10, front surface). 18.5 hours after reaching 1100° F. The slip band spacing was fairly regular in the early stages of the test as shown in the upper left field, A. Slip concentrated to the wide band in later stages of the test. 60 X.

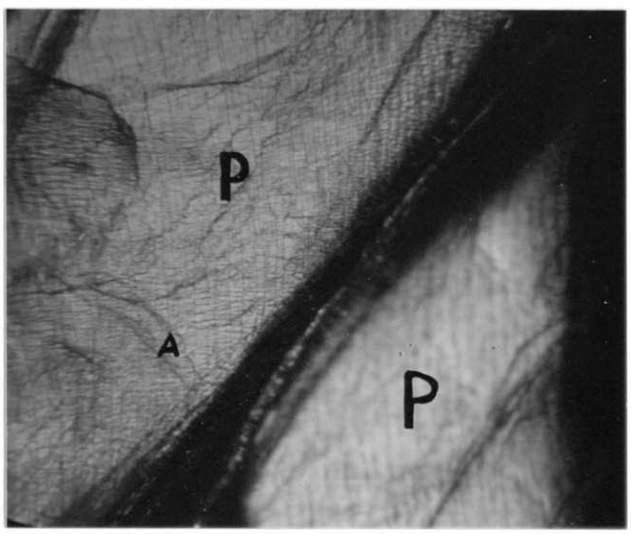


Figure 45. (Specimen P-10, front surface). Same band as Figure 44. 29.6 hours after reaching 1100° F. Non-homogeneous deformation occurred at the region, A, close to the left edge of the specimen and the heavily slipped band. 60 X.

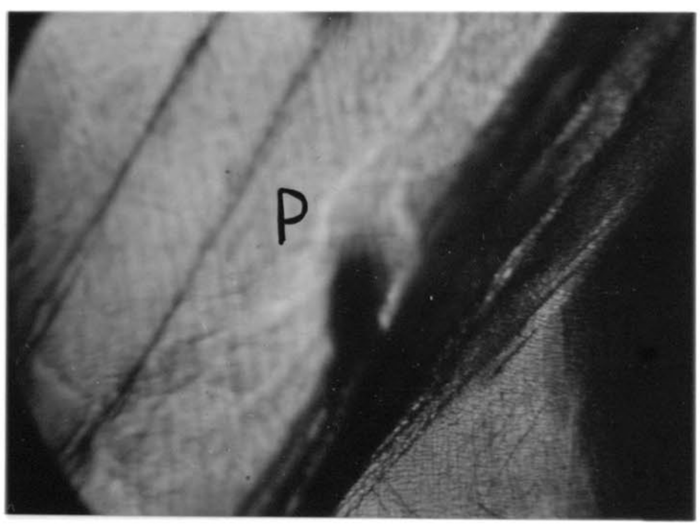


Figure 46. (Specimen P-10, front surface). Same band as Figure 44 and 45. 29.6 hours after reaching 1100° F. Note the steps left at the right edge of the specimen, as a result of shift of the operating slip bands from the upper to the lower bands. The amount of rotation of the slip band developed in the early stage of creep is greater than that developed in the later stage. 60 X.

Figure 47. (Specimen P-10). 30.4 hours after reaching 1100° F, fractured. Non-homogeneous deformation occurred at the region close to the right edge of the specimen and the heavily slipped band. 50 X.

With further deformation, this wide slip band exhibited an interesting slip and rotation process. Figure 46 clearly shows this process of rotation of the slip bands as creep continued. Schematic drawings are also presented in Figure 48 A and B to show the stages of this process. As a result of slip two new surfaces 1 and 2 are formed and are joined by a portion (3) of the same slip band (Figure 48 A). It may be seen from Figure 48 A that region (3) is not as free to rotate as (1) and (2) since the latter are free surfaces. As a result of this situation, rotation of the portion (3) tended to lag behind the rotation of (1) and (2). Since rotation of the slip band during slip was restricted by the blocking effects of the grain boundaries, bending of the slip band must occur. In view of the fact that slip along the bent band is limited, further slip must occur on adjacent planes which are not bent as shown in b, Figure 48 B. This may explain why the elongation in the early part of curve I, Figure 49 (obtained across this band) is rather small. With a decrease in cross sectional area with respect to the load applied, a continuously increasing creep rate (later portion of curve I) is obtained over this band.

This specimen was eventually fractured by a process of slip along successive slip bands. By examining the fractured surfaces, it was revealed that the steps produced along the successive slip bands during the early stage of the development, are small. In other words, the number of the slip bands participating in this process is large. The reverse is true during the later stages of development.

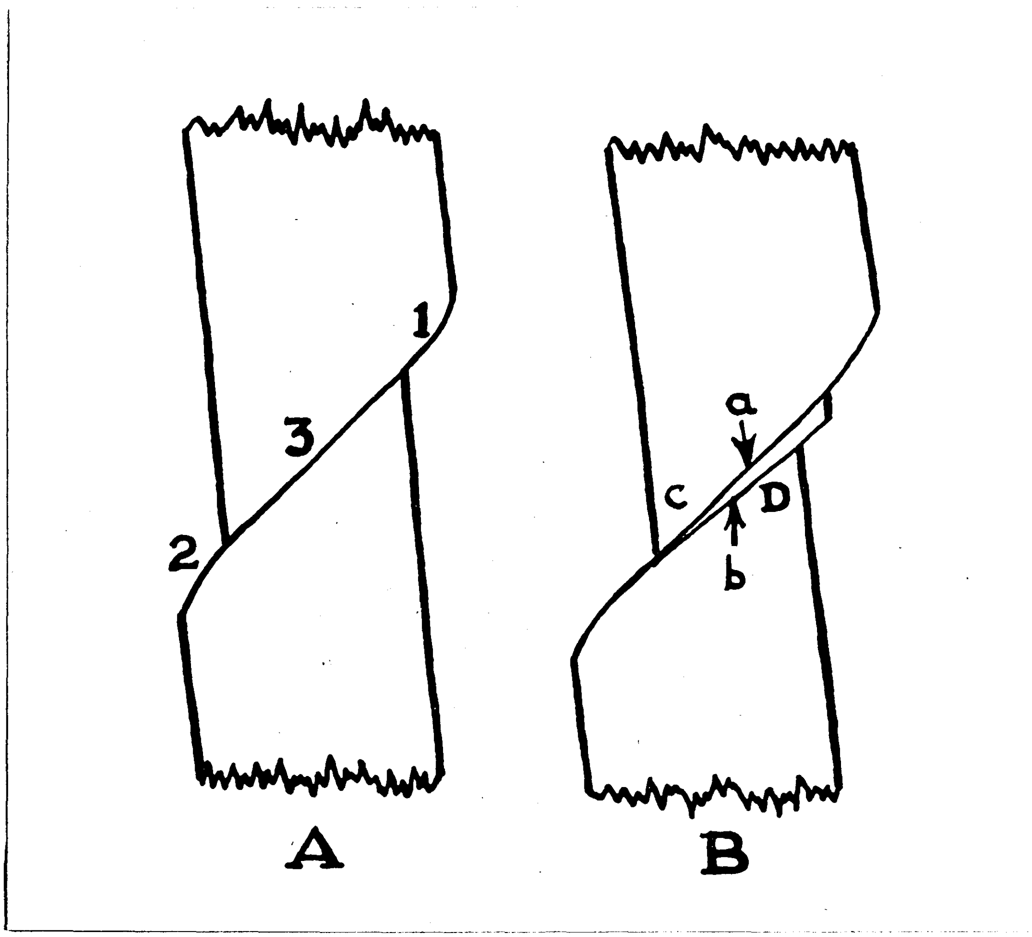


Figure 48. Schematic drawing of Figure 46.

A. Rotation of portion 3 lags behind the rotation of portions 1 and 2.

B. Slip band **b** operated after slip band a became bent and rotated away from favorable direction for slip. C and D indicate the regions of non-uniform deformation.

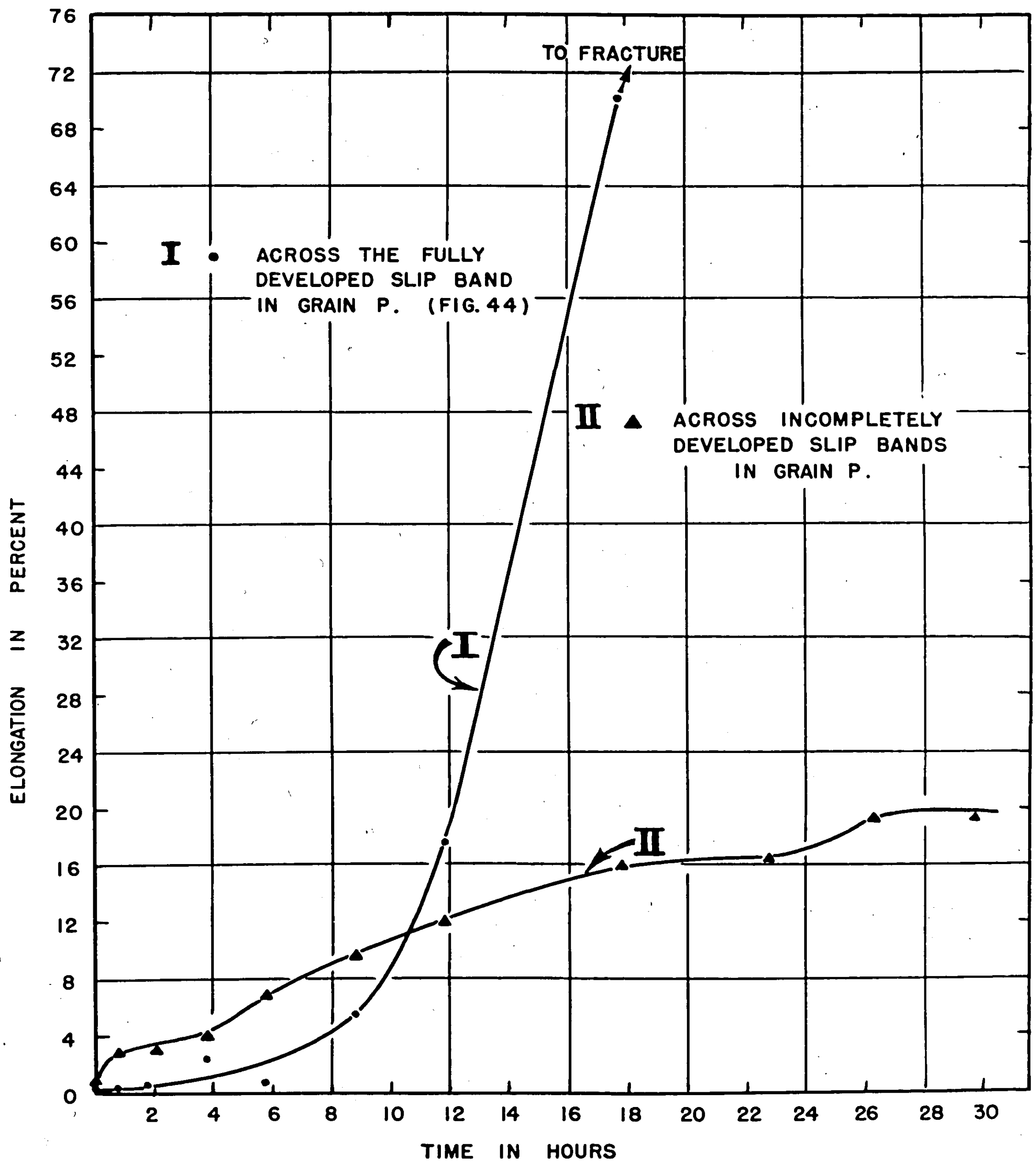


FIG. 49 COMPONENT CREEP CURVE ACROSS SLIP BANDS IN GRAIN P OF SPECIMEN P-5 (FIG. 5)

As the portion (3) of the slip band lags behind in rotation in comparison with the rotation of portions (1) and (2), bending of the heavily slipped slip band would occur. This bending would give rise to compression on the left tip of the upper part of the crystal and on the right tip of the lower part of the crystal. These two regions are designated by C and D in Figure 48 B. Figures 45 and 47 show how these two regions of the same grain had been non-uniformly deformed as a result of this compressive stress. This non-uniformly deformed region exhibits fairly regular traces by slip at the free edge of the specimen. These traces by slip indicates that they are formed along a slip system being different from the slip band, I, Figure 37. That the slip bands in the regions C and D, Figure 48 B, cannot fully developed is believed to be due to the blocking effect of the heavy slip band, I, Figure 37.

Two dotted lines in grain C of Figure 3 indicate the courses of the two bands developed during the creep test. The upper band was fully developed all the way across the cross section of the specimen except at the very right edge of the specimen where it encountered the boundary of grains A and C. However, the lower band was not fully developed because it encountered the boundary of grain C and D at a considerable distance inside the width of the specimen on the back surface of the specimen. Figures 50 and 51 show the course of these two bands on the front side of the specimen; the former was taken close to the left edge, while the latter was taken close to the right edge of the specimen. Since they are far from the

Figure 50. (Specimen P-8, front surface). Effect of grain boundary on the development of slip bands. The upper band was fully developed, the lower band not. The arrows indicate the regions where cross slip has taken place. 50 X.

Figure 51. (Specimen P-8, front surface). A continuation of Figure 50 to the right. The upper band encountered the grain boundary at the very right edge of the specimen, the lower band encountered a grain boundary at the back surface of the specimen, incompletely developed. 50 X.

influence of the grain boundaries, close to the left edge of the specimen (Figure 50) their course of development seems to follow a specific direction. But when the lower band approaches the boundary of grains C and D on the back surface of the specimen, its course of development deviated away from the grain boundary as shown in Figure 52 (taken on the back surface close to the right edge of the specimen). The course of the lower band on the front surface of the specimen was also so affected that it merged with the upper band at the very right edge of the specimen (Figure 51).

By examining the deformation marks, left on the specimen surface, it can be concluded that slip had taken place on different slip systems within the band, as indicated by the arrows in Figures 50, 51, and 52. However, these two bands left regular slip steps at the left edge of the specimen where they were not influenced by grain boundaries. Back reflection Laue photographs (Figures 53 B and 53 C) taken at different positions on the front surface of the specimen, which are indicated by small triangles in Figure 3, show that the material situated on the upper band and between the upper and lower band close to the left edge of the specimen were very slightly distorted while the material in the lower band was broken down into three sub-grain spots (Figure 53 D) with a spread of about 2°; apparently, as a result of the incompletely developed nature of the lower band. Patterns taken farther along the width of the specimen show that the material situated both in the upper band and between these bands

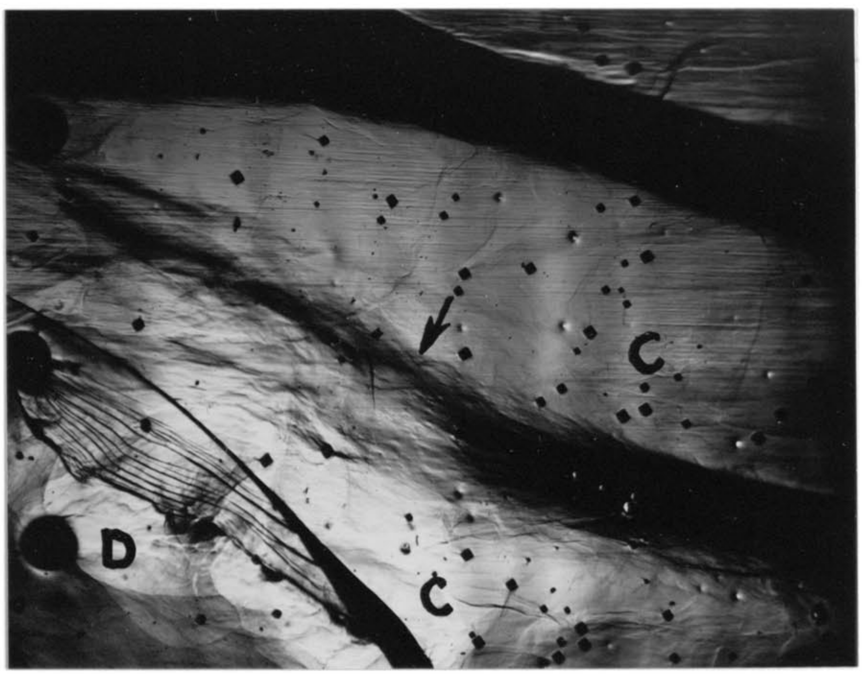
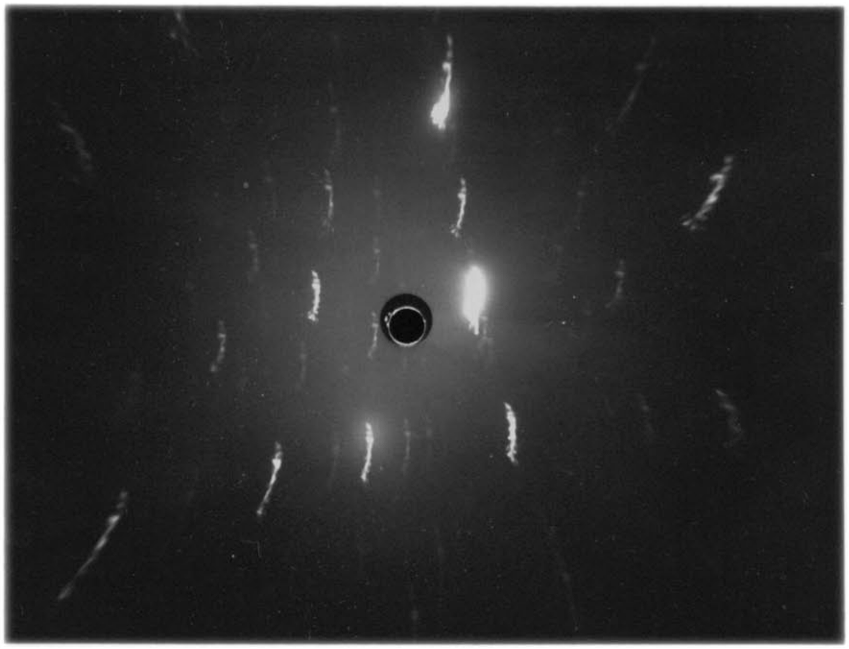


Figure 52. (Specimen P-8, back side). Effect of the grain boundary on the development of slip bands. The same two bands are shown in Figures 50 and 51. The lower band deviated on approaching the grain boundary. 50 X.

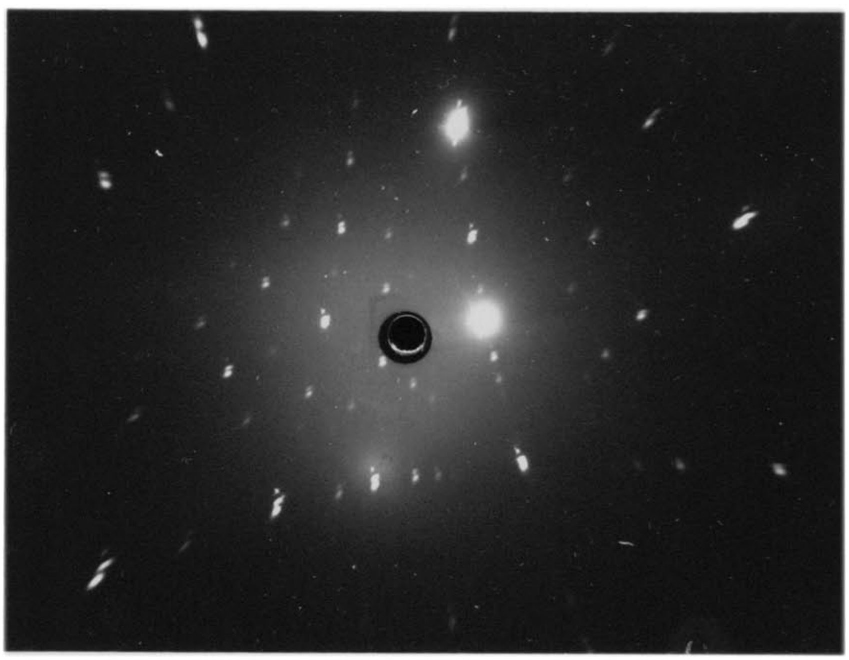


a

b

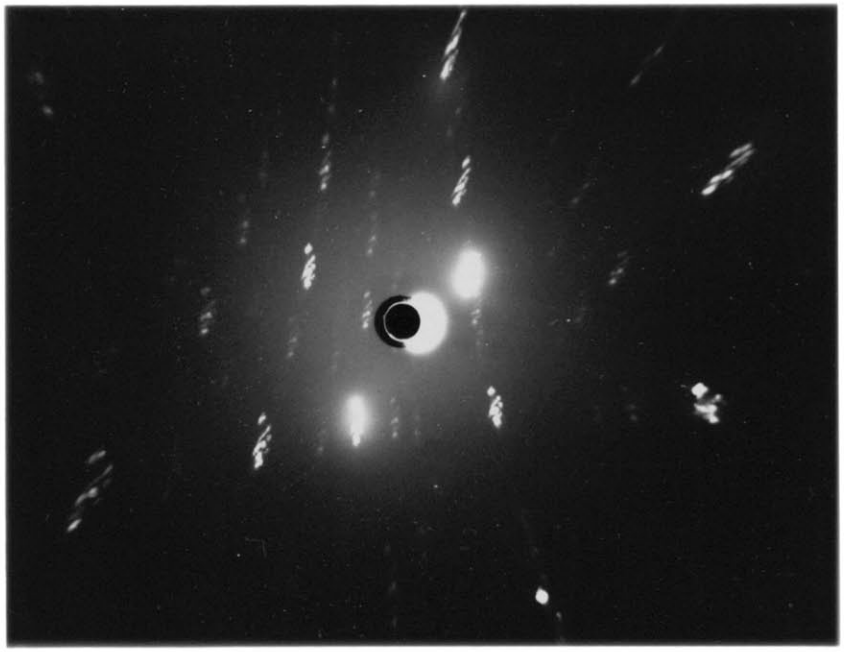
Figure 53 a - g. (Specimen P-8, front surface). Back reflection Laue patterns taken at locations corresponding to the triangles in Figure 3.

c

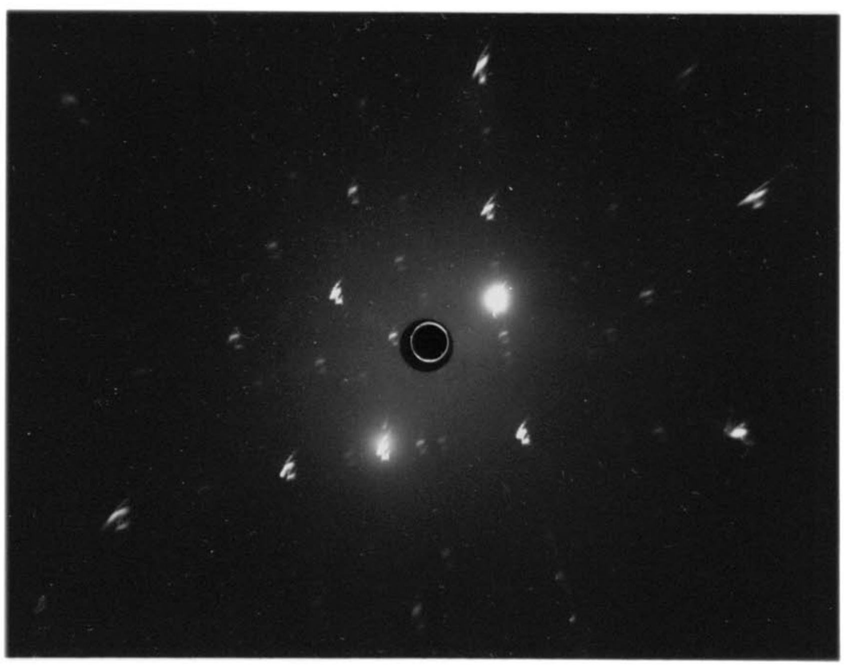


d

Figure 53.

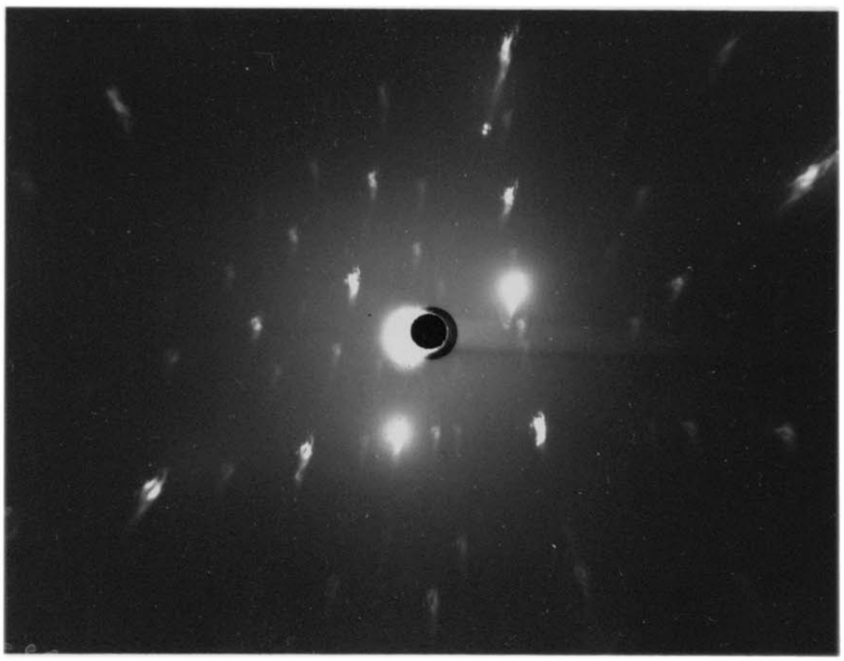


e



f

Figure 53.



g

Figure 53.

were broken down into sub-grain spots; the former (Figure 53 E) with a spread of about 5° while the latter (Figure 53 F) had a spread of about 2°. It should be remembered that the farther the upper band goes along the width of the specimen, the closer it gets to the boundaries of grains A and C, and C and D, on the front and back surface, respectively. Patterns taken on the front surface of the specimen close to the right edge (Figure 53 G) where the lower band deviates its course, show not only sub-grain spots but also streaks with a total spread of about 5°. This may mean that the regions where different slip systems had been operative were too small to be resolved distinctly by the presently employed X-ray technique. It should be pointed out that the material between the upper band and the boundaries of grains A and C and of grains B and C was more severely broken down into sub-grains with a spread of about 6° than the material within the band and between the bands; it was even true at the left edge of the specimen (Figure 53 A). This fact is apparently due to non-homogeneous deformation which has to take place in order to accommodate the deformation taking place in the upper band.

Figure 54 shows three component creep curves which were obtained by measuring between two reference marks, [illegible], across the bands and between the bands. The region across the upper band, Curve I, deformed initially with decreasing creep rate up to about 4 percent elongation and then deformed at a very small constant creep rate until the band became observable. The deformation

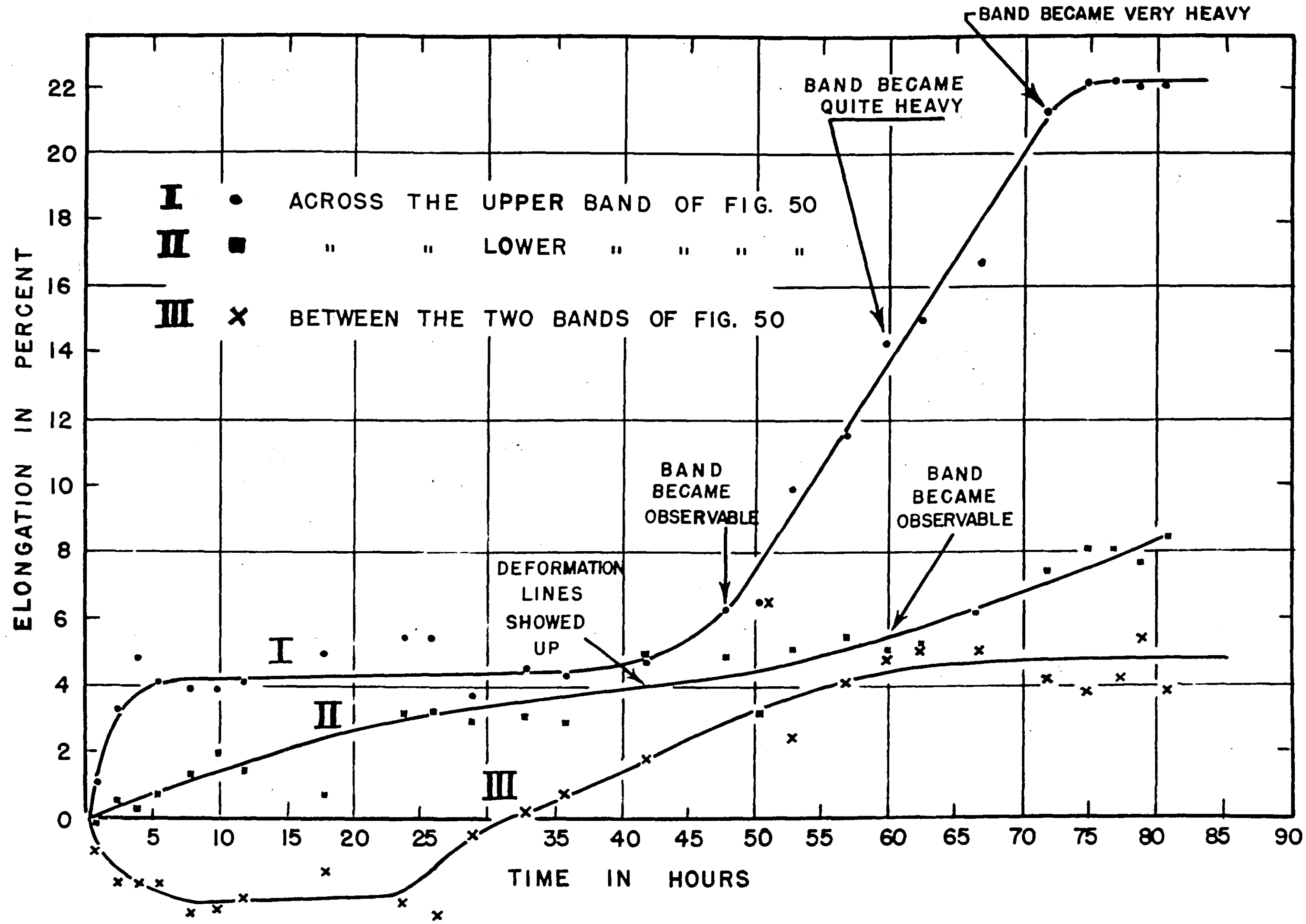


FIG. 54 COMPONENT CREEP CURVES ACROSS THE SLIP BANDS AND BETWEEN TWO BANDS OF SPECIMAN P—8 (FIG. 3)

characteristics of this initial period may be a result of the blocking effect of the boundary of grains A and C at the very right edge of the specimen (Figure 51). The period, characteristic of very small creep rate, is very probably the one during which enough stress is being built up to overcome this blocking effect by deviating the course of development of the upper slip band at the very right edge of the specimen. As soon as this stress is built up, deformation proceeds rapidly as shown by the next stage of curve I, until bending of the slip planes and strain hardening is large enough that further deformation becomes difficult. The same general behavior occurs in the region of the lower band, Curve II, but at different times. It is interesting to note that the region between the bands, Curve III, shows an apparent initial contraction which may be a result of the tilting of this block because slip along these two bands was not only different in magnitude, but also occurred at different times. It should be emphasized that although the amount of elongation produced by the lower band is small it gives rise to rather severe non-homogeneous deformation in the grains (Figures 52 and 53 G).

Figure 55 illustrates another example of the effect of a grain boundary between grains J and P (specimen P-8). It can be seen from Figure 3 that the grain J occupies the whole thickness of the specimen and is bounded by three grain boundaries. Examination of the specimen after removal showed that the offset produced by slip at the free edge of the grain was much larger than the offset in the neighborhood of the grain boundary. This is also evidenced by the fact that fine

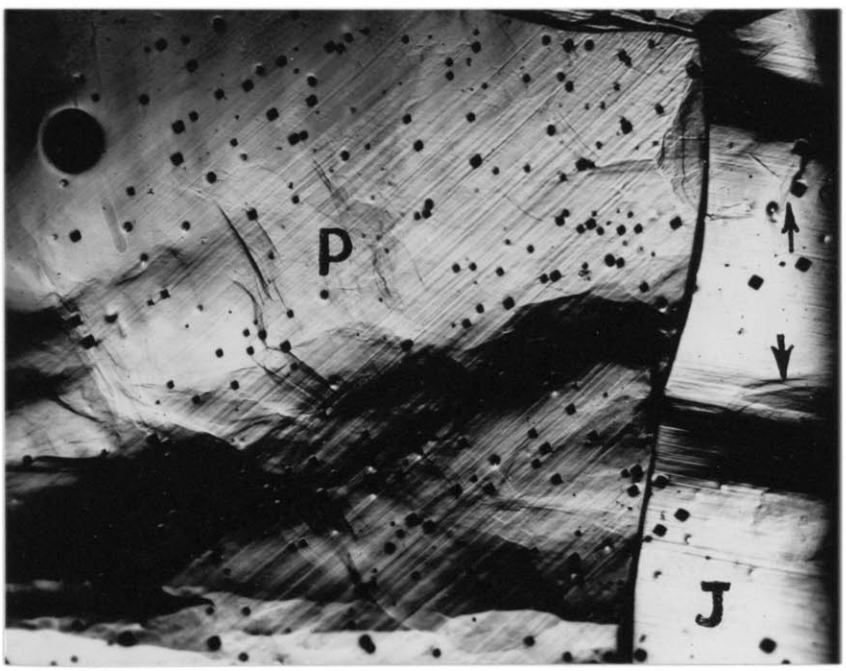


Figure 55 A. (Specimen P-8, back surface). The amount of slip close to the right edge of the specimen along the slip bands of the right grain is much larger than that close to the grain boundary. Note also the sub-grain formation in the left grain. 50 X.

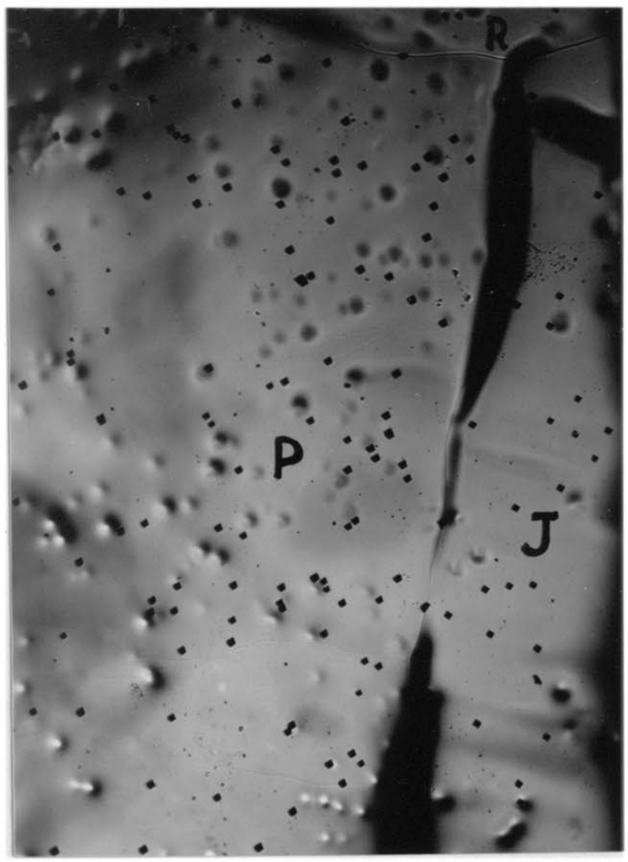


Figure 55 B. (Specimen P-8, back surface). Same field as Figure 55 A, repolished and electro-etched. Sharply bent boundary remained, slip bands and deformation lines disappeared. 50 X.

traces of slip lines can be seen in the neighborhood of the grain boundary but not at the free edge of the band, Figure 55 A. That the amount of slip is different on the same slip plane in the slip direction means that either bending of the slip plane or rotation of the slip plane about an axis perpendicular to the slip plane, or both, may occur. It is believed that as a result of this non-homogeneity of slip in the band, non-homogeneous deformation occurs both in the band and in the material near the band, as indicated by arrows in Figure 55 A.

Figure 56 A shows a fold in grain K of the same specimen P-8, the formation of which is closely associated with the sliding along the boundary of grains P and J. The development of folds associated with boundary sliding will be presented more fully in a later section. Note that the pre-existing slip lines, produced during handling of the specimen, are rather sharply bent at the borders of the fold. Figure 57 shows a back reflection Laue photogram taken in this region. It turns out that the major axis of rotation of the sub-grain spots is perpendicular to the plane which is delineated by the traces of this fold. It is believed that this fold is formed as a result of slip followed by rotation of the slip plane about an axis whose major direction is perpendicular to the slip plane. Rotation of the slip planes about an axis perpendicular to the slip plane has been observed with the electron microscope.[11] The fact (Figure 56 B) that the slip lines produced by small amounts of cold work can go through the fold with only slight deviation in directions

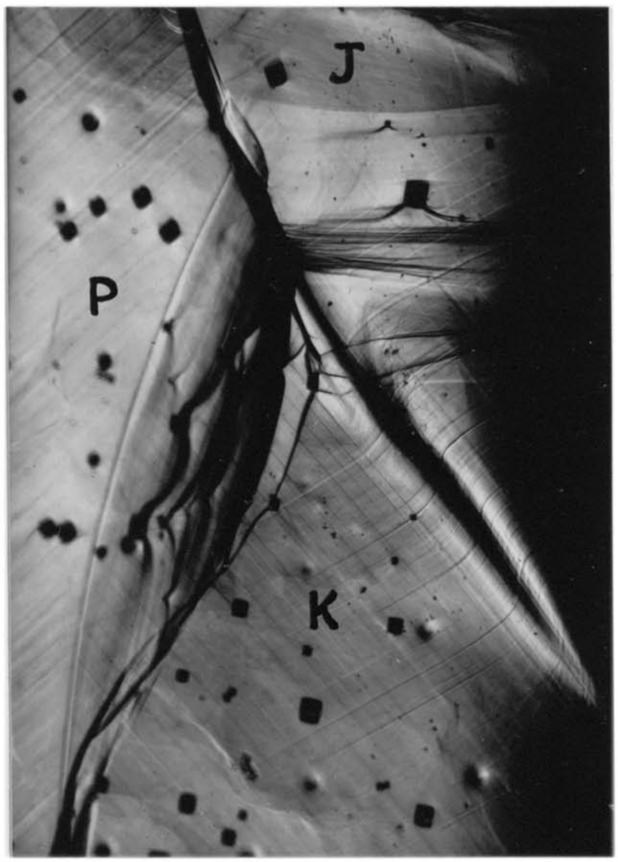


Figure 56 A. (Specimen P-8, back surface). Fold, produced by sliding of the opposite grain boundary, caused pre-existing slip lines to be rather sharply bent through this band. Note also the offset of the other two grain boundaries produced by sliding along the third boundary. 100 X.

Figure 56 B. (Specimen P-8, back surface). Same field as Figure 56 A. Slip lines produced by small amount of deformation at room temperature go through this band, without appreciable change in direction. 100 X.

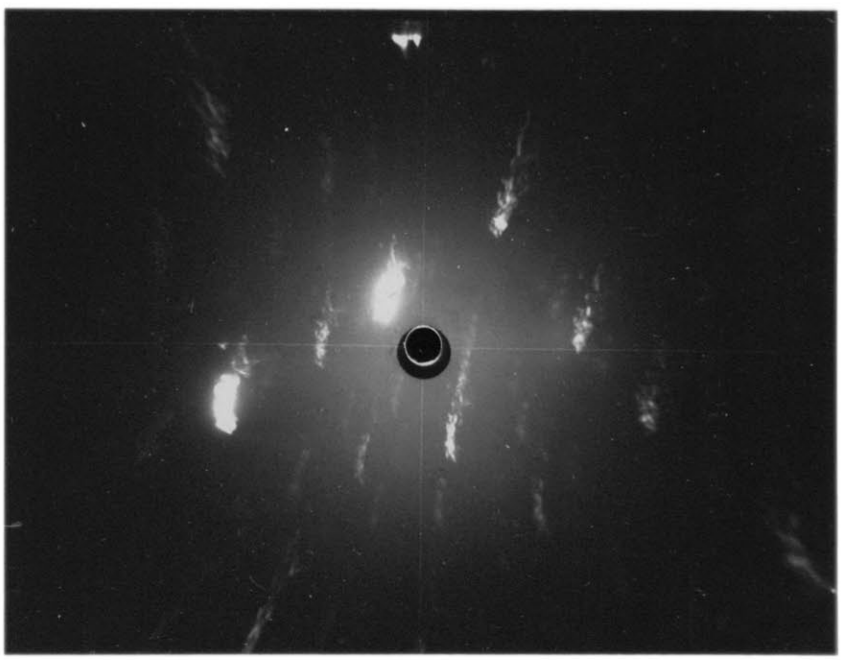


Figure 57. (Specimen P-8, back surface). Back reflection Laue photograph taken on the fold of Figure 56 A.

may be explained in considering the new slip lines, produced along the same slip system as the previous bent ones, but through different planes of the slipped and rotated lamellae from the previously bent ones (see Figure 58).

I. Kinking and Deformation Band Formation

Kinking bands were observed only after a considerable amount of slip had taken place along the slip planes. Figures 59 A through 59 G show the sequence of development of the kinking bands, (specimen P-11, tested at 400° F). A kinking band made its first appearance only 66 hours after loading with a stress of 400 psi when the grain E had elongated about 3.5 percent by slip. As time went on, other kinking bands developed in grain E and tended to extend into grain F without regard to the grain boundary. More new bands tend to develop within the already developed wide bands. The borders of this kind of band seem to follow a general direction, but they are definitely not developed along a crystallographic plane. The nature of this kinking band can be clearly seen in Figures 60 and 61. Note that the pre-developed slip bands are bent around the rather sharp borders of the kinking bands.

Deformation bands may occur either in the very early stage of creep (Figure 62 A) or after considerable slip had taken place (Figure 80 B). The term "deformation band" is used here to represent a band without a sharply defined border, in which slip has taken place along one or more than one slip systems being different from the slip system outside the band.

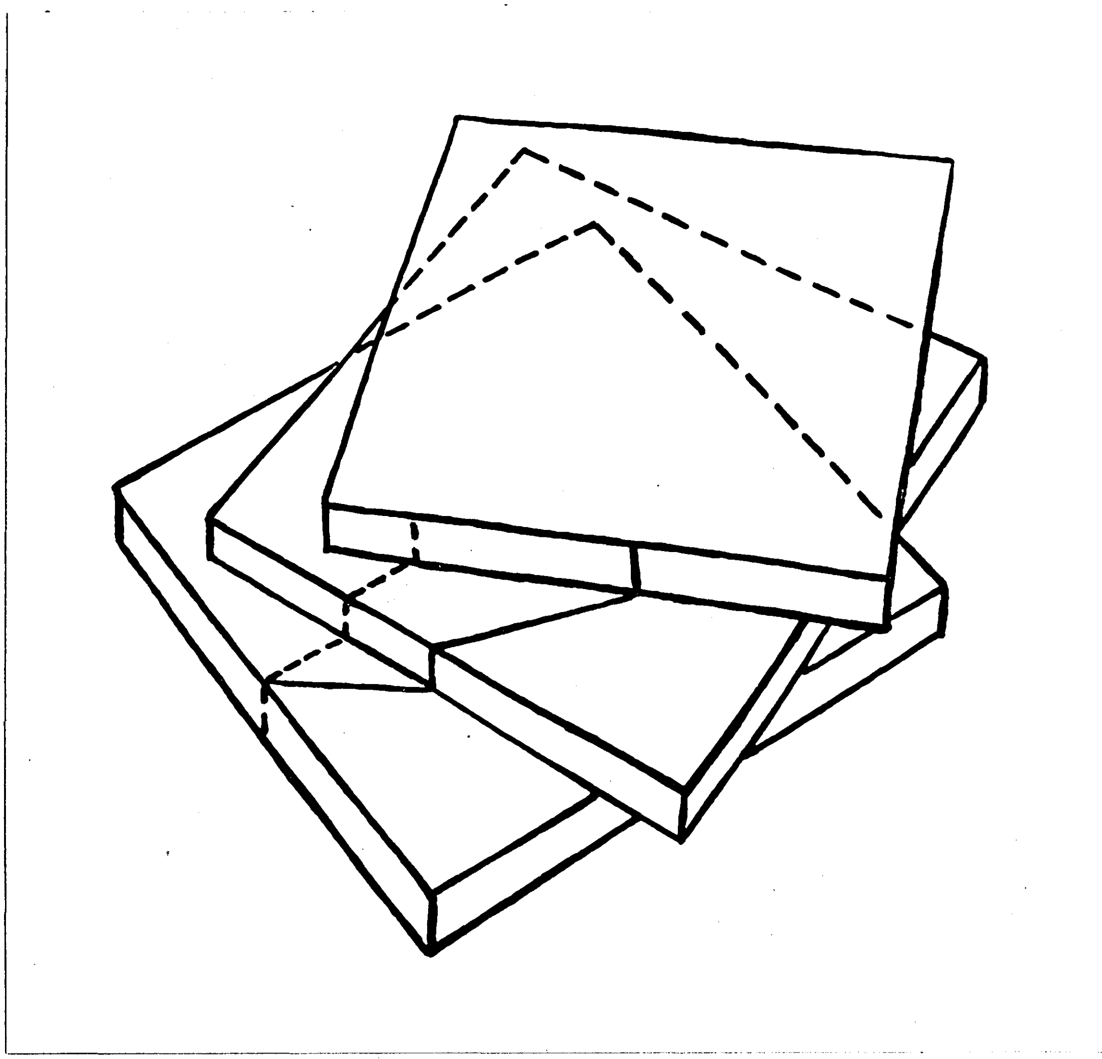
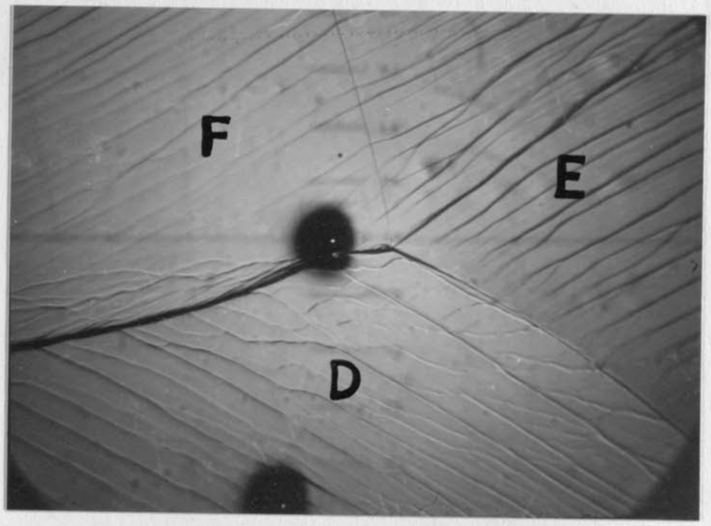
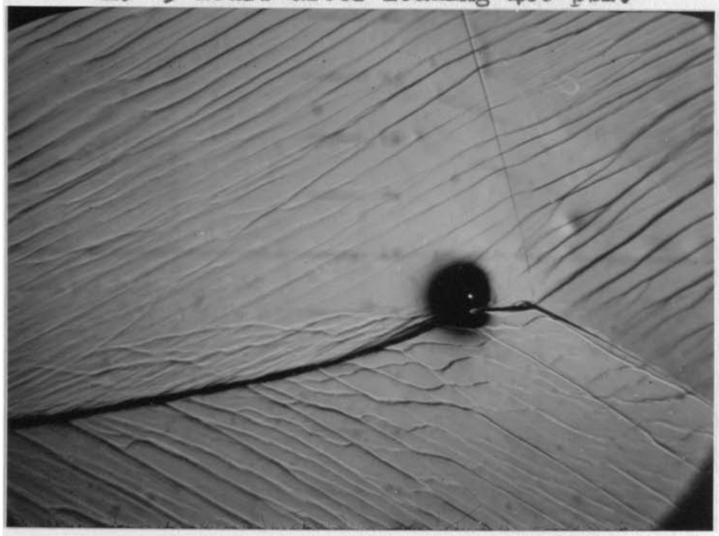


Figure 58. Schematic drawing of Figure 56 B shows slip followed by rotation of slip planes in the fold. Cross slip line (dotted) produced by deformation at room temperature after fold formation appears to go through the fold less deviated than cross slip line (solid line) produced prior to fold formation.



A. 3 hours after loading 400 psi.



B. 8.5 hours after loading 400 psi.

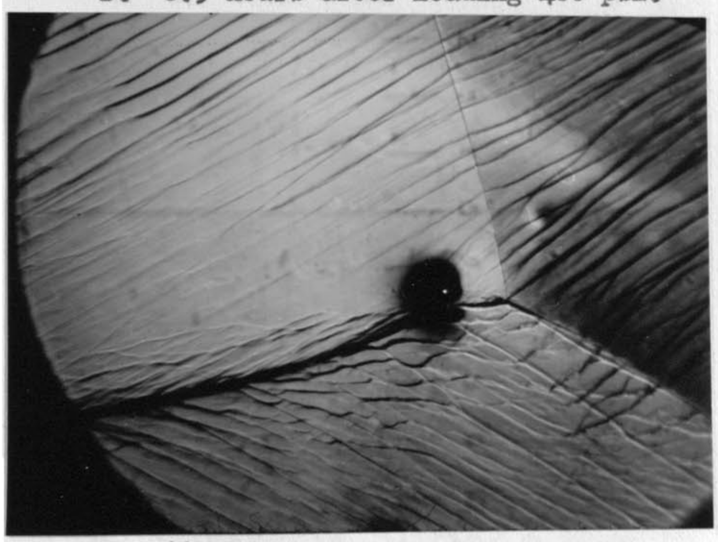
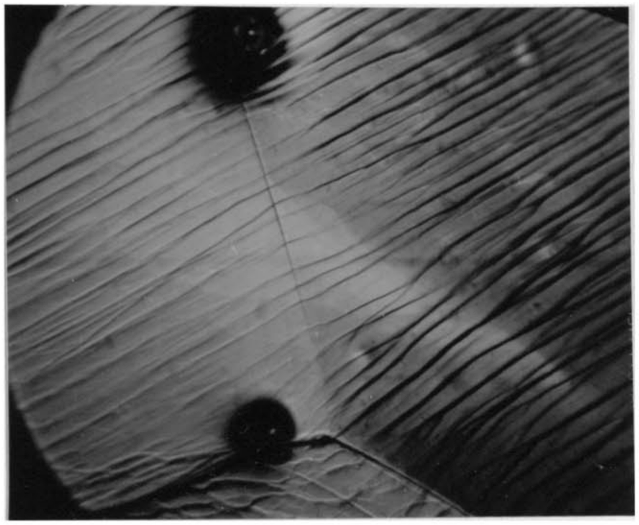


Figure 59. C. 66 hours after loading 400 psi.



D. 74 hours after loading 400 psi.
11 hours after loading 550 psi.

E. 162 hours after loading 400 psi.
99 hours after loading 550 psi.

Figure 59. F. 192 hours after loading 400 psi.
129 hours after loading 550 psi.

G. 385 hours after loading 400 psi.
322 hours after loading 550 psi.
220 hours after loading 750 psi.

Figure 59. A - G. (Specimen P-11). Sequence of development of kinking bands. Kinking bands developed in the upper right grain in the early stage tended to propagate into the upper left grain in a later stage. Note also the continuous nature of the slip bands across the grain boundary of the upper two grains. 60 X.

grain A

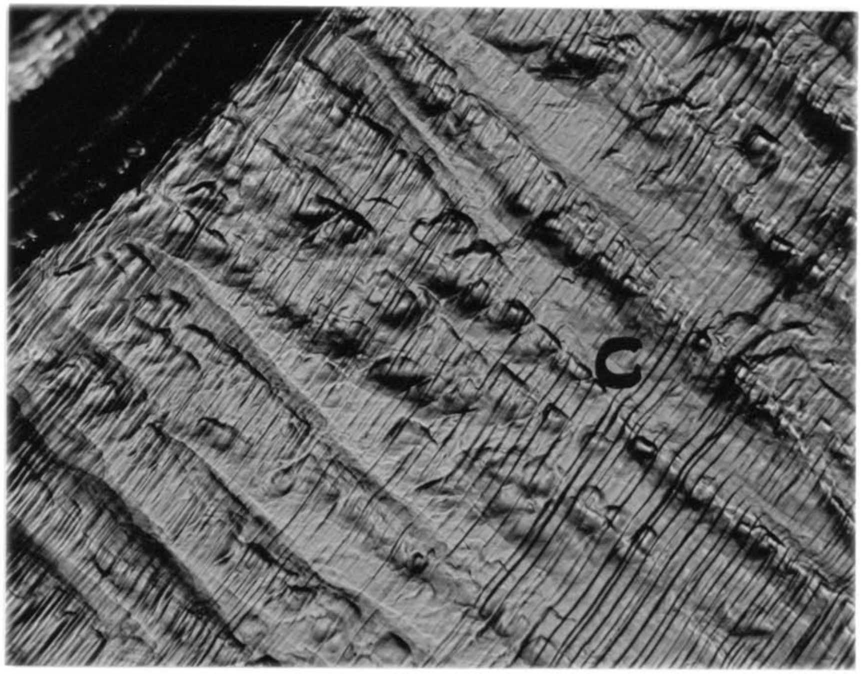


Figure 60. (Specimen P-7, front surface). Appearance of kinking bands close to the grain boundary at the upper left corner. 100 X.

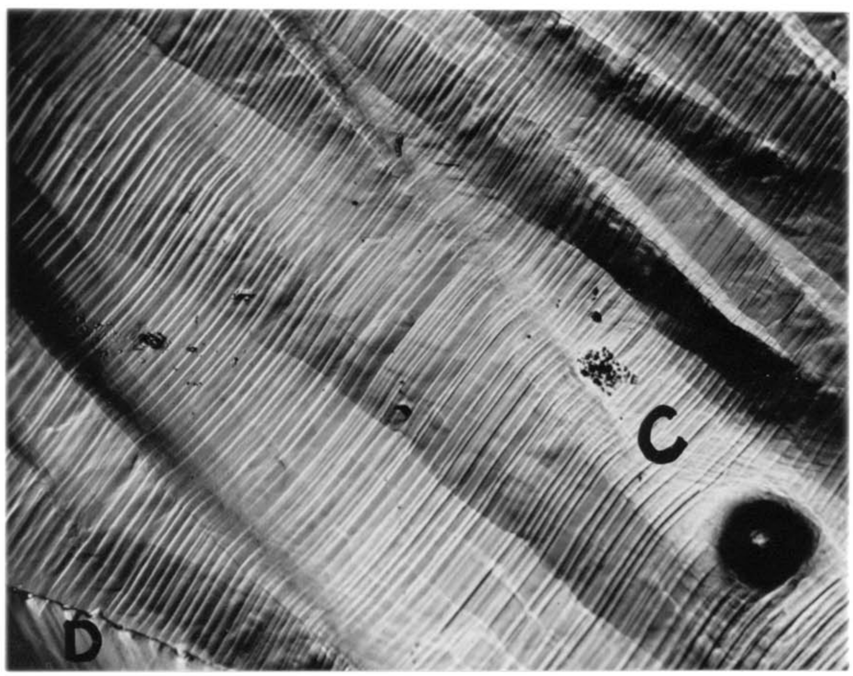
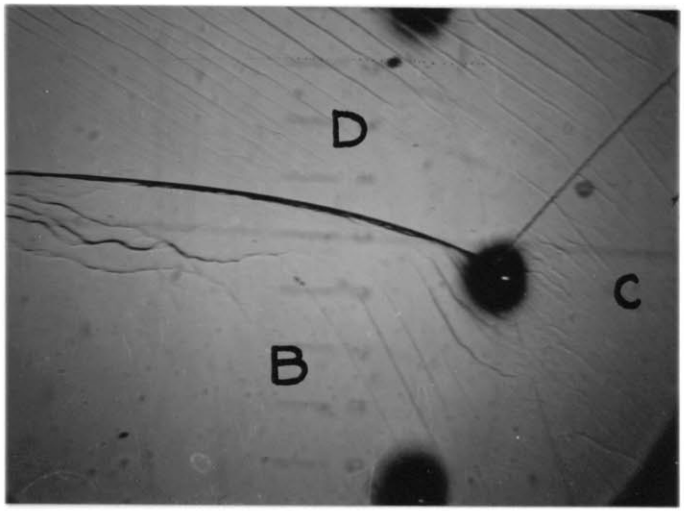
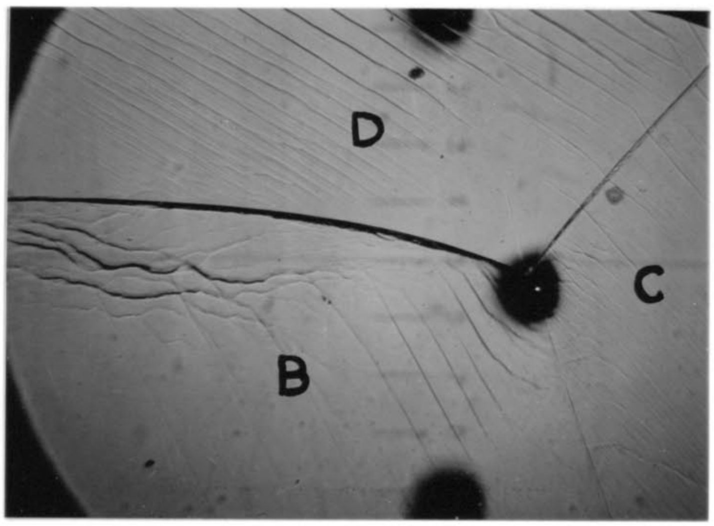


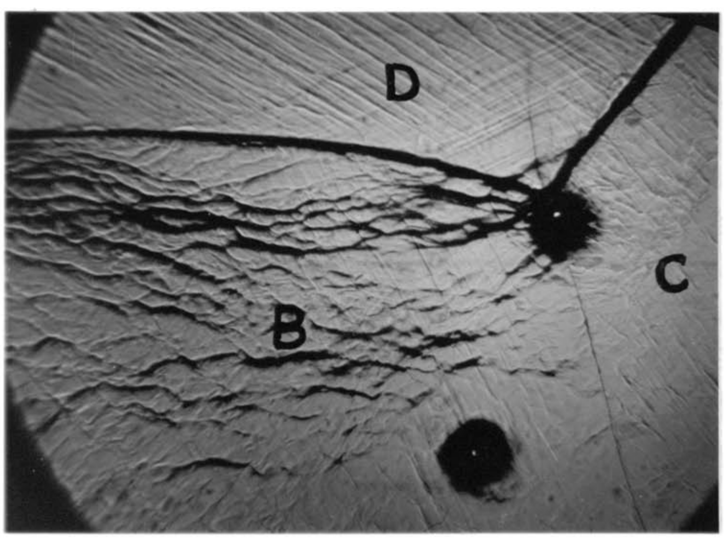
Figure 61. (Specimen P-7, front surface). Appearance of kinking bands close to the specimen edge, same grain as Figure 60. 100 X.



A. 3 hours after loading 400 psi.



B. 66 hours after loading 400 psi.



C. 192 hours after loading 400 psi.

129 hours after loading 550 psi.

27 hours after loading 750 psi.

Figure 62. A - C. (Specimen P-11). Development of deformation bands in the lower left grain. 60 X.

It should be pointed out that the term"slip band" used in the last section resembles the deformation band in some respects, for example, cross slip may occur in the band. The main feature, characteristic of the slip band, is that slip occurs along one single slip system. This feature is primarily maintained with regard to the slip bands of the above discussion, hence the term "slip band" was used.

The points from which deformation bands start are either at the free edge of the specimen (Figure 63) or at that part of the grain boundary exhibiting sharp shenges in radius of curvature as a result of non-uniform boundary sliding and migration (Figure 64). As shown in Figure 67 A, the deformation band just started to develop at the grain boundary as indicated by the arrow. All these places are the places of high stress concentration.

J. Fold Formation

The term "fold formation" is used to indicate that kind of deformation band, the formation of which is closely associated with the sliding of a grain boundary lying directly opposite to the band. The fairly straight black band which appears to be an extension of the grain boundary between grains K and M is an example of a fold (Figure 7 A). In view of its curved nature and the fact that its traces do not correspond to the traces of slip lines produced during handling, it can be concluded that it is not developed along a particular crystallographic plane. The curved nature of the fine traces left on the deformed surface along its path, indicated by an arrow in Figure 65 A, suggests that slip has occurred along different slip systems. During creep the width of the fold has been observed to increase with sliding and migration of the opposite grain boundary

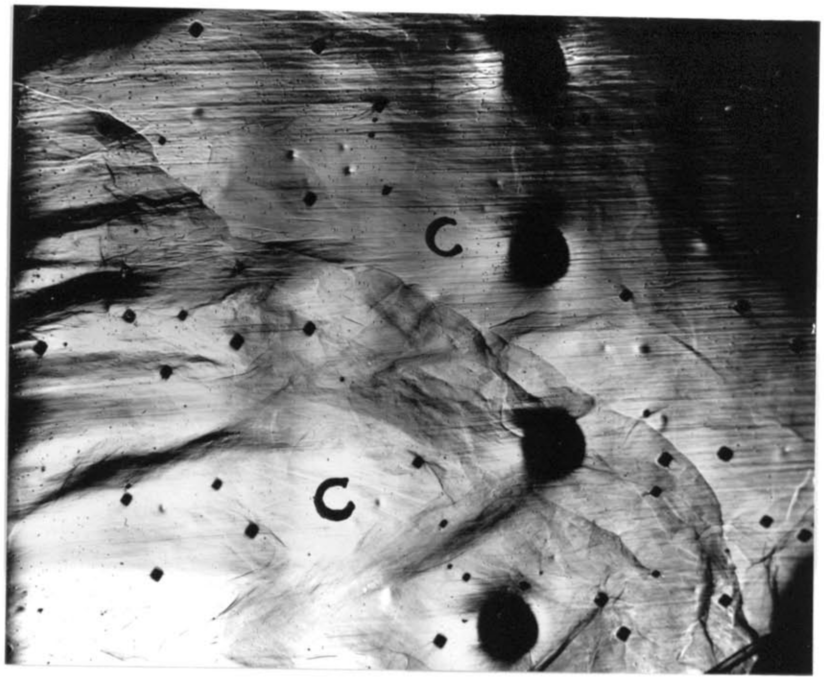
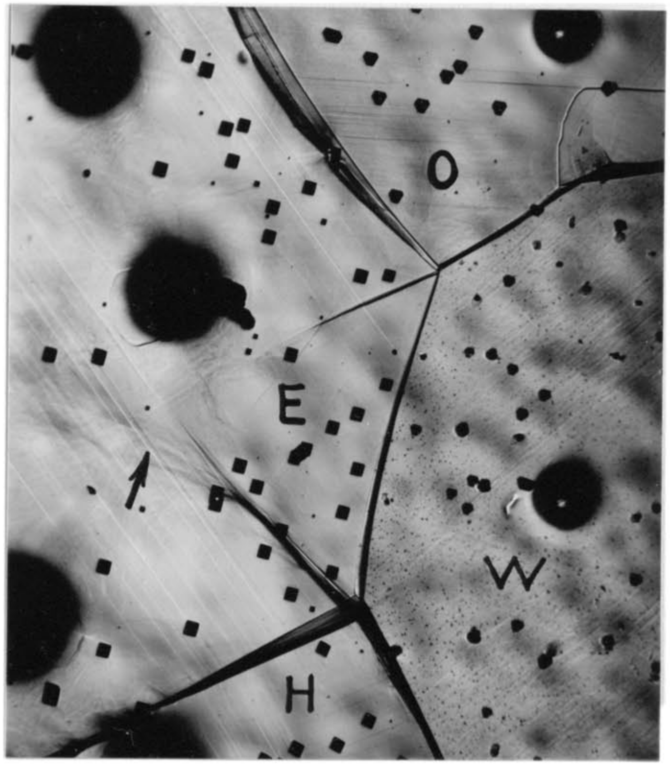
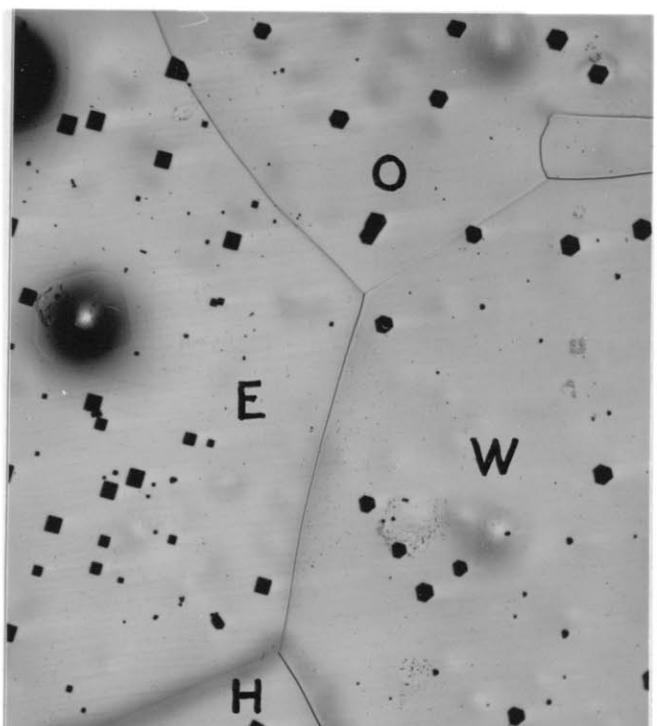


Figure 63. (Specimen P-8, front surface). Deformation bands started from the free edge of the specimen. Note also the sharp sub-grain boundary formed by bending. 50 X.

Figure 64. (Specimen P-6). Deformation bands started from that part of grain boundary where sharp curvature occurred as a result of uneven boundary sliding and migration. 100 X.



A



B

Figure 65 A. (Specimen P-3). Fold formation in the top center grain caused by sliding of the opposite grain boundary. 100 X.

65 B. (Specimen P-3). Same field as Figure 65 A, repolished and etched. Folds cannot be revealed by repolishing and etching. 100 X.

(Figure 65 A). This fact suggests that sliding of the opposite boundary is the primary cause of fold formation. Figure 56 is also an example of a fold. None of the folds can be revealed again by electro-etching after repolishing, (Figures 7 B and 65 B). Slip lines reproduced by small amounts of deformation at room temperature were shown to go through the position of the fold without appreciable bending. Both these facts suggest that disorientation across the fold is rather continuous and not appreciable.

By examining the traces of several folds of this sort left on the free surfaces of the specimen, it revealed that their plane of formation makes an angle of about 40 - 50° to the direction of applied tension. This is also the direction favorable for slip.

This observation and the X-ray results regarding the rotation of the crystal lattice across the fold (Figure 57) seem to indicate that the direction of deformation is governed by the amount of shearing force acting in, and the magnitude of the blocking effects (for example, grain boundaries and inclusions) existing along this particular direction. The deformation in this direction may be contributed by slip along several slip systems. It is believed that the above statement may apply also to the formation of deformation bands. The full development of folds depends on:

1. Whether there is grain boundary in its path
2. Whether the size of the grain in which the fold may form is small enough in comparison with the length of the opposite grain boundary along which sliding may take place.

This fact is schematically shown in Figure 66 A and B. It is assumed in Figure 66 A and Figure 66 B that grains 1, 2, and 3 occupy the whole thickness of the specimen and have plain grain boundaries. The geometrical arrangements of grains 1, 2, and 3 are the same in Figure 66 A and B, but the grains are assumed to occupy different volumes across the width of the specimen. Grain 3 in Figure 66 B is assumed to occupy a much larger volume than that in Figure 66 A. The boundary between gfains 1 and 2 is deliberately set into a favorable position for sliding. As shown in the discussion of boundary sliding and migration, during a creep test at 700° F and above, grain boundaries in a favorable position for sliding deform before deformation in the grains can take place. This means that the yield strength of the grain boundaries is weaker than that of the grains for creep tests at 700° F and above. Suppose the yield strength of the grain boundary between grains 1 and 2 and the strength of grain 3 is Y_B and Y_G respectively, and let the area of the boundary between grains 1 and 2 and the area of the extension of it in grain 3 be A_1 and A_2 respectively, for Figure 66 A and similarly A_1' and A_2' for Figure 66 B. Assume F is the shearing force across the whole cross section of the specimen in the direction of the grain boundary between grains 1 and 2. The condition that a fold can develop fully in grain 3 is

$$F \geqq Y_B A_1 + Y_G A_2.$$

Since $Y_G > Y_B$ and $A_2' >> A_2$, the term $Y_G A_2'$ becomes more important and makes $Y_B A_1' + Y_G A_2' > F$. Consequently the shearing force sufficient

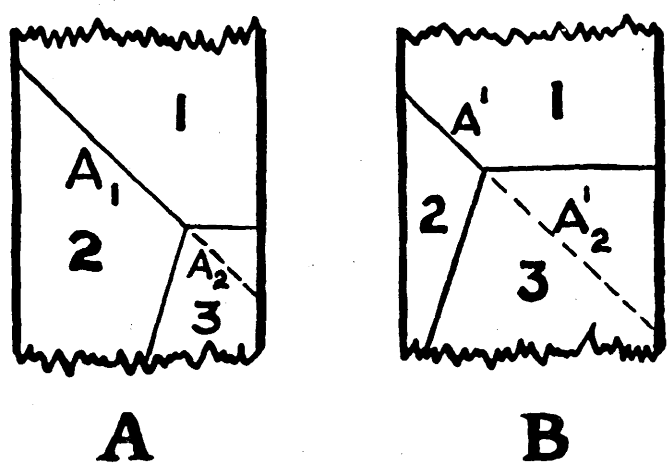


Figure 66. A schematic drawing shows that fold can form in grain 3 in Figure 66 A, but can not in grain 3 in Figure 66 B.

for fold formation in the case of Figure 66 A, may not be enough in the case of Figure 66 B, grain 3 in Figure 66 A being smaller than grain 3 in Figure 66 B.

Figure 65 A shows a case of two developed folds due to boundary blocking effects (see also grain E in Figure 2). The folds tended to stop and split into several finer bands as they entered into the grain.

The rumpled appearance of small grain P (Figure 9 A) is believed to result from the fact that sliding along the boundary of the grains M and O cannot develop a fold owing to the blocking effect of boundary between grains P and Q (see also grain P in Figure 2).

Folds are not observed when the specimen is subject to creep at 1100° F even if the arrangement of the grains is favorable for their development. Because of the high mobility at this temperature, boundary migration occurs before appreciable sliding can take place. This means that the stress built up at the triple point by boundary sliding is not large enough to cause a fold. The grain boundaries migrate so fast from the very beginning of the test that in a short time only a few grains with boundary surfaces practically perpendicular to the direction of applied tension are left in the specimen. Consequently, further sliding along the grain boundaries becomes very improbable.

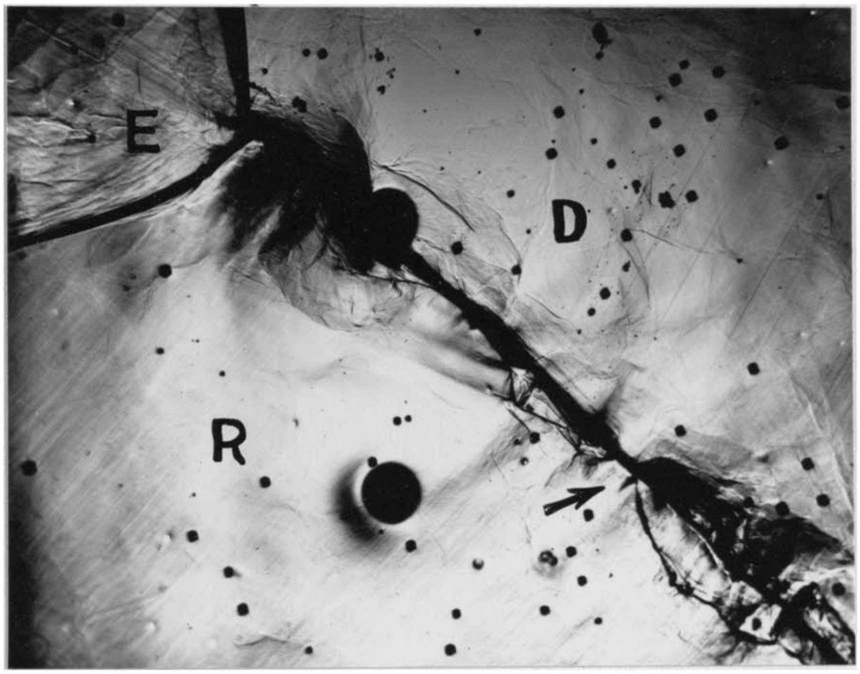
Folds were observed only in two cases at 400° F. In both cases the grain boundaries lying opposite to them were very favorably oriented for sliding. Since only small amounts of boundary sliding were observed at this temperature, boundary sliding might still play an important role in fold formation. The possibility that stress concentration created around the triple point owing to non-homogeneous deformation of the grains may cause fold formation cannot be ruled out.

K. Sub-Grain Formation

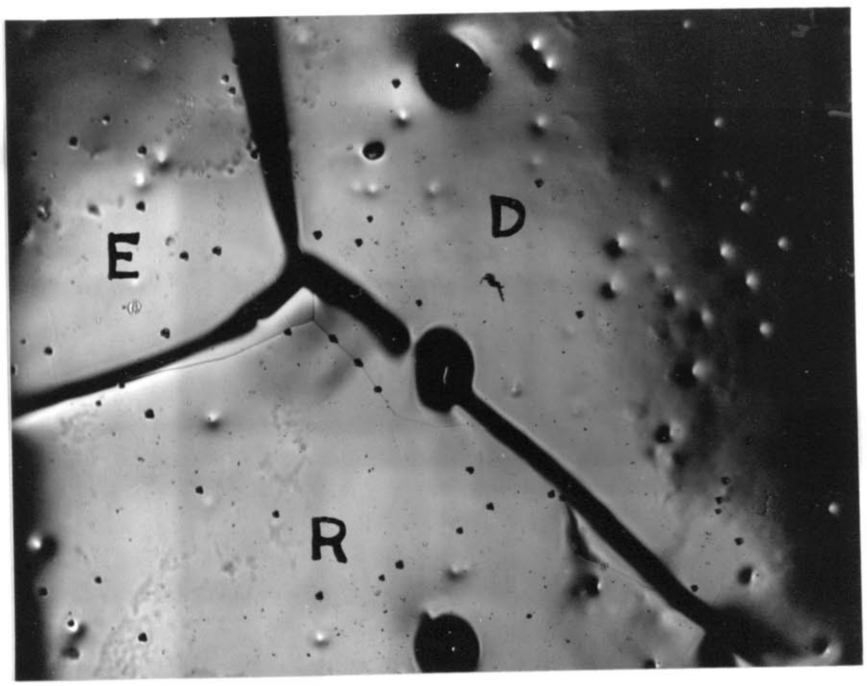
Grain boundary sliding may cause a ridge and valley appearance on the deformed surface if fold formation is prevented (grain P, Figure 9 A). These areas were made up of a series of such valleys and ridges which propagated into the grain away from the boundary as the amount of boundary sliding increased. The same type of deformation shown in Figure 67 A took place not only around the corner of the triple point but also along both sides of the heavily slid boundary. The sharpest ridges or valleys could be revealed by electro-etching after repolishing, as shown in Figure 67 B. The ridges or valleys of Figure 67 A correspond completely with the faint lines of Figure 67 B.

It should be pointed out that a tail of deformation band (indicated by the arrow of Figure 67 A) at the turning point of the migrating grain boundary (Figure 67 A). This tail eventually developed to a deformation band when enough deformation was allowed (Figure 64).

It is interesting to note from Figure 67 A that the effect of boundary sliding was by no means restricted to a thin layer of several atomic distances thick (the thickness of a grain boundary as suggested by Ke[12] on the basis of internal friction measurement),but extended to



A



B

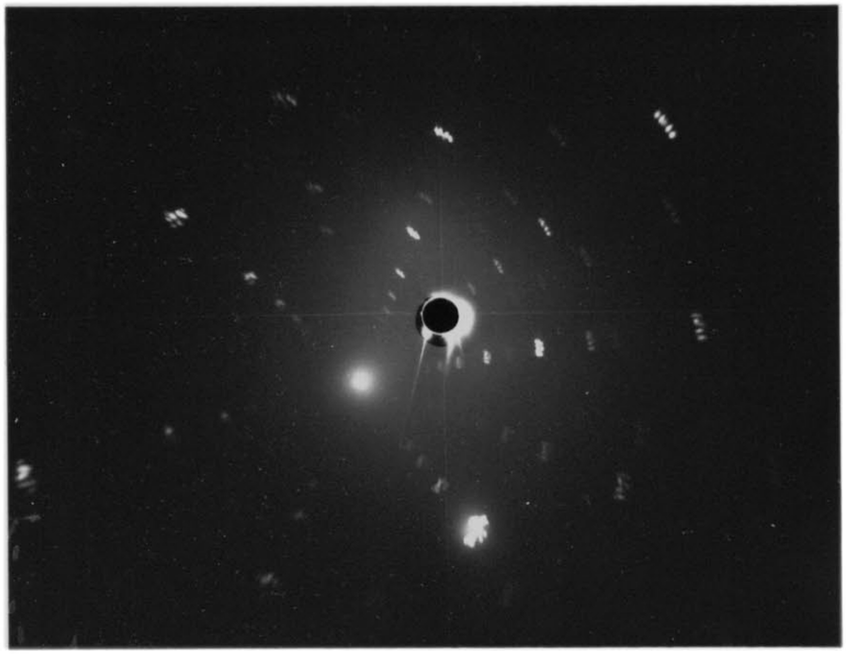
Figure 67 A. (Specimen P-8). Sub-grain formation by bending around the triple point and along the heavily slid grain boundary. 50 X.

67 B. (Specimen P-8). Same field as Figure 67 A, repolished and etched. Final grain boundary becomes angular; sub-grains revealed by electro-etching after repolishing. 50 X.

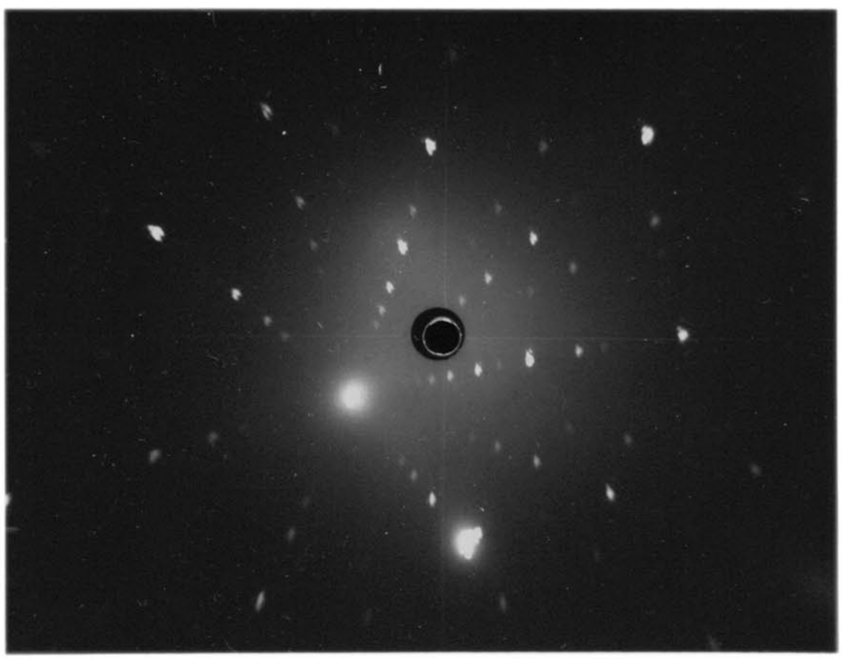
a rather large volume along both sides of the heavily slid boundary, in this case the affected area was about 0.2 mm. wide to each side of the slid boundary.

It has been shown that the effect of etching on a grain boundary depends on the orientation difference of the two neighboring grains[13]. This fact suggests that the orientation difference across the sharp ridges or valleys of Figure 67 were quite small but large enough to give electro-etching effects. The etching effect persisted even after a surface layer of 0.02 mm. thick was removed by electro-polishing. Accordingly, this effect was more than a mere surface phenomenon. That the orientation difference across this kind of boundary is quite small is further proved by the fact that the slip lines produced by a small amount of deformation at room temperature can go through this kind of boundary. One may properly call this type of boundary a sub-grain boundary, and the grains bounded by them sub-grains.

These faint lines revealed by electro-etching after repolishing are also proved to be sub-grain boundaries by studying the back reflection Laue patterns (taken at the positions indicated by triangles h and i in grain R, Figure 3) shown in Figures 68 A and B. The degree of breakdown of Laue spots decreases the farther away they are taken from the slid boundary. This is readily evident from the diffraction patterns and agrees with the metallographic evidence. The sub-grain size appearing in Figure 67 B is 0.07 to 0.2 mm. The X-ray beam with a diameter of 1 mm. can cover 5 - 14 sub-grains. In view of the fact that sub-grains of identical orientation will register



A



B

Figure 68. (Specimen P-8, front surface). Back reflection Laue photograms taken at positions indicated by Δ in the enlargement of grain R of Figure 3.
A. at Δh
B. at Δi

on the same spot, the number of the breakdown spots of a single Laue spot agree fairly well with the size of the sub-grains appearing on the photomicrographs.

It should be noted that a Laue spot breaks down into several sub-grain spots with negligible background between them, particularly the one taken very close to the slid boundary. This phenomenon is in sharp contrast to the Laue patterns taken in folds and deformation bands; the latter two showing streaks and a fairly strong background between strong breakdown spots (Figure 57). At times, due to this strong background, it is not possible to distinguish the separate spots. This fact may imply that the sub-grain boundary is produced by sharp bending of the lattice which may or may not be accompanied with bending glide. The sharp bending nature of the sub-grain boundaries can be even more clearly seen in Figure 69 (the center grain is bounded all around it by other grains).

It is necessary at this stage to distinguish the sub-grains with sharp boundaries as shown above from the sub-grains without sharp boundaries. The latter cannot be revealed by electro-etching after repolishing. The sub-grains of the second kind are observed both in the interior of the grain and in the neighborhood of the grain boundaries at 700° F and 1100° F. As shown in the upper left grain of Figure 19 A, the sub-grain boundaries are delineated by narrow bands. They could not be revealed again by electro-etching after repolishing as shown in Figure 19 B. This kind of boundary is more frequently observed at 400° F around the triple point and in the

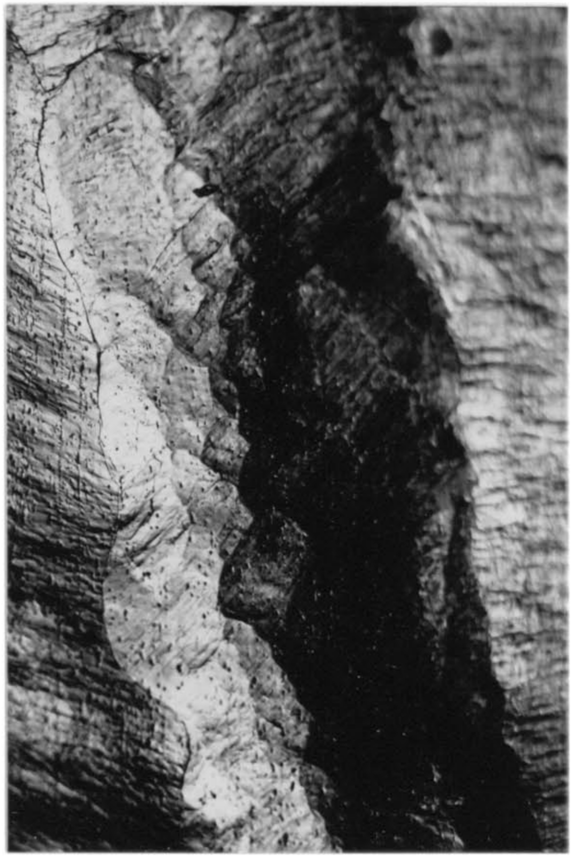


Figure 69. (Specimen 2 S – 7). Sub-grain formation by bending of the lattice. 100 X.

neighborhood of the grain boundaries than in the interior of the grain. As shown in Figure 70 (specimen P-11), it appears that the sub-grain boundaries around the triple point in grain J are delineated by bands in which slip had taken place on more than one slip system. They are very probably imcompletely developed deformation bands. This kind of sub-grain is shown even more clearly in grain D of Figure 71.

It should be mentioned that when the conditions are such that extensive boundary migration is permissible boundary sliding accompanied by sub-grain formation is less than otherwise (compare Figure 19 A and B, C, with 67 A). The conditions for extensive boundary migration are summarized as follows:

1. High temperature
2. More than three grains of comparable size across the width of the specimen.

Figure 72 A through F shows how part of the specimen P-11 deformed at 400° F. In the early stage of creep the specimen deformed by slip. Three slip systems had operated in grain G but they tried to avoid the grain boundary between grain G and E. In the later stages of creep kinking bands developed in grain E tended to extend into grain G and caused non-homogeneous deformation in grain G, particularly in the region slip had been restricted to develop. Sub-grains were gradually developed in this region, and as may be seen from the photographs were formed after slip bands had developed in the early stage of creep. At the time of fracture this region was broken down into many sub-grains randomly distributed (Figure 72 E

Figure 70. (Specimen P-11). Sub-grain formation by deformation bands. 100 X.

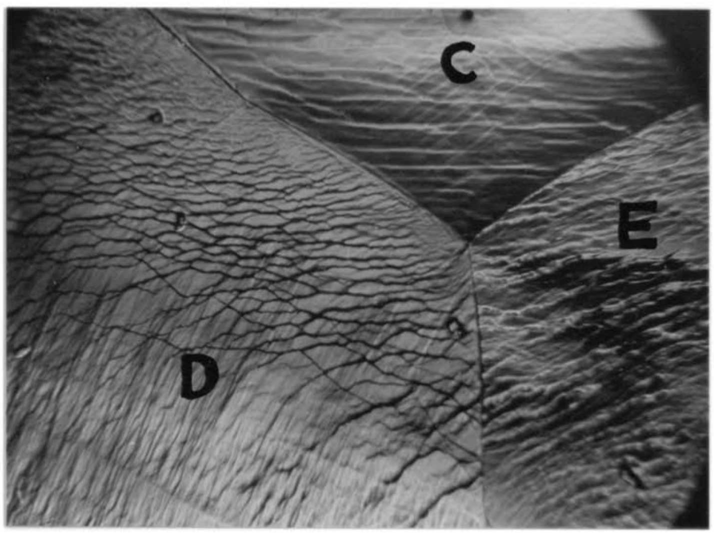
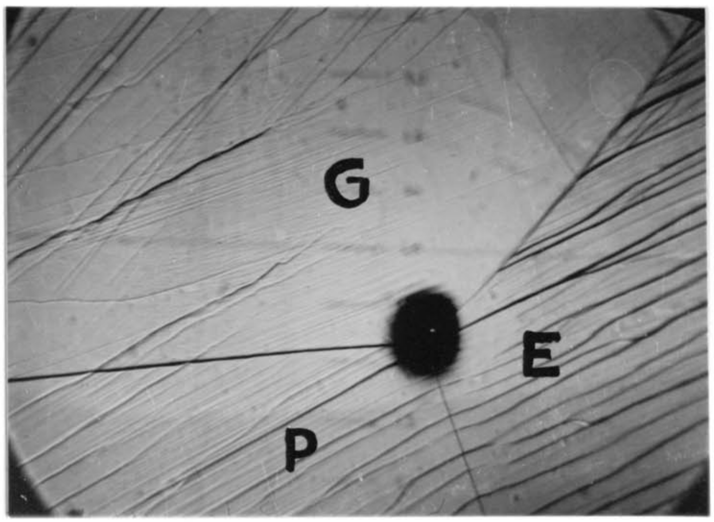
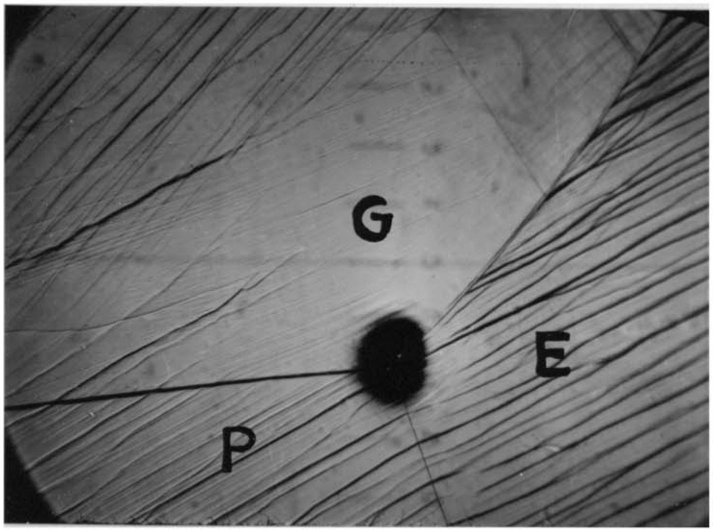


Figure 71. (Specimen P-7). 1 hour after loading 1200° F.
Sub-grain formation by deformation bands.

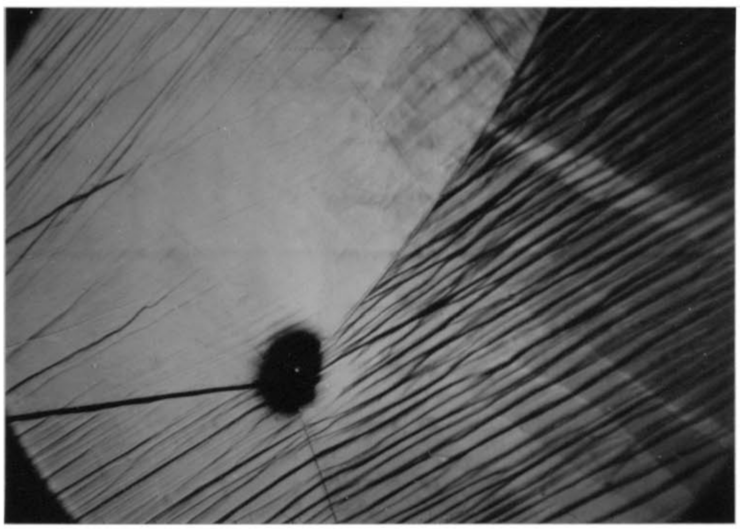


A.

Figure 72. A - F. (Specimen P-11). 3.5 hours after loading, 400 psi. Sequence of photomicrographs show the development of kinking bands, (grain E) and sub-grains (grain G). Note also the continuous nature of slip bands across the grain boundary of the lower grains E and P and the evidence of sliding along the boundary between E and P. 60 X.



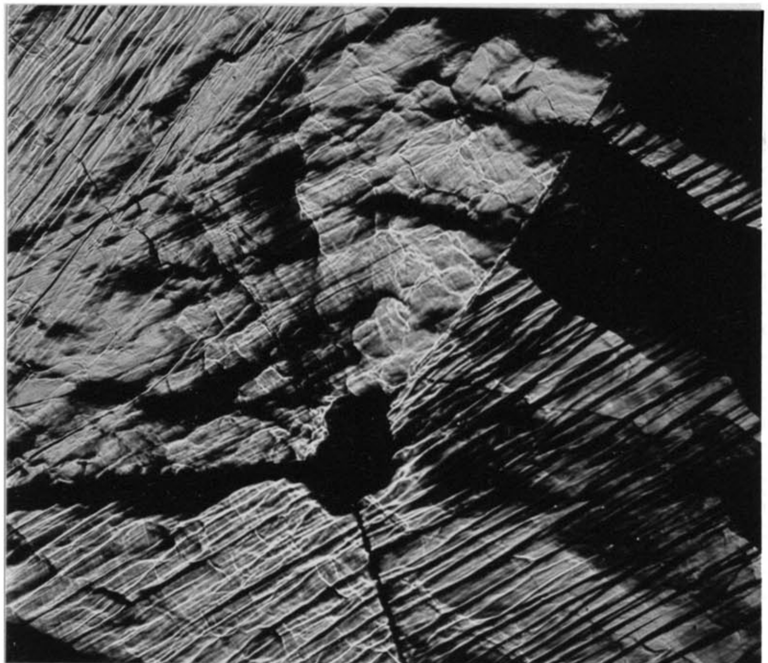
B.
8.5 hours after
loading, 400 psi.



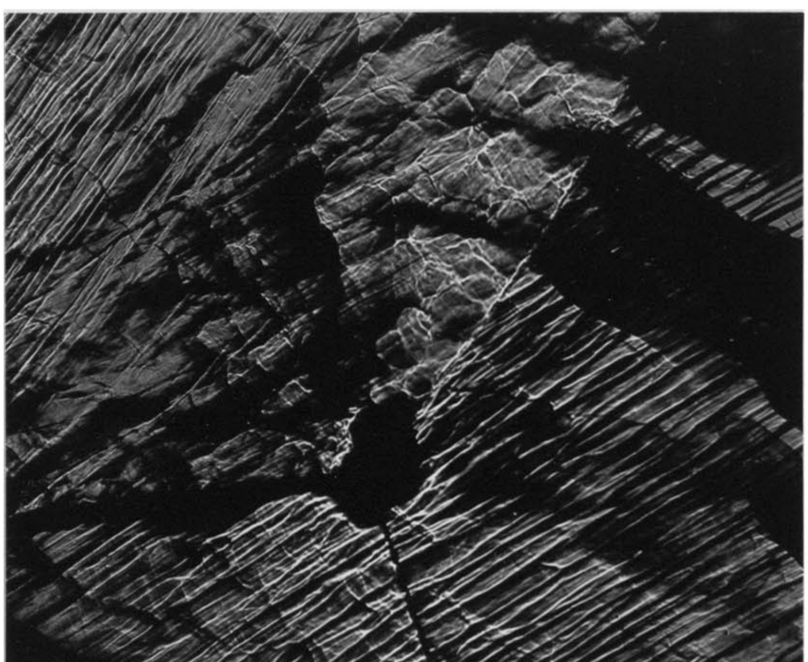
C.
74 hours after
loading, 400 psi.
11 hours after
loading, 550 psi.

D.
385 hours after
loading, 400 psi.
322 hours after
loading, 550 psi.
220 hours after
loading, 750 psi.

Figure 72.



E.
After rupture, oblique illumination. 50 X.



F.
After rupture, oblique illumination. Sub-grain boundaries are shown more clearly than Figure 72-E, by setting the microscope a little "out of focus". 50 X.

Figure 72.

and F). Laue patterns taken in this region (grain G) substantiate this observation (Figure 73). In this case the size of sub-grains are 0.03 - 0.12 mm. A Laue pattern, (Figure 74), taken in grain E in which kinking bands were superimposed on the earlier developed slip bands, shows that the direction of breakdown is not random and the intensity of individual breakdown spots varies quite a lot. In photomicrographs (for example, Figure 75) if the kinking band boundaries are considered as the sub-grain boundaries, then the number of sub-grains visible in the photomicrographs agree well with the number of sub-grains that are indicated from the Laue patterns. Note that the kinking band boundaries run in a general direction but their spacing varies quite a lot.

The same type of results were observed in specimen P-7 which was subjected to creep at the same temperature as specimen P-11 but at a much higher stress - 1200 psi. It can be seen from Figure 60 (taken close to the grain boundary between grain A and C) that sub-grain boundaries follow the general direction of kinking bands but not the direction of slip bands. The photomicrograph (Figure 61) taken away from the grain boundary of grains A and C, shows that sub-grains had not fully developed and only kinking bands are seen. In comparison with the X-ray patterns obtained from specimen P-11, (Figures 73 and 74), those obtained from specimen P-7, (Figures 76 a and b) show that the number of the sub-grain spots is much larger in the latter than in the former. Therefore the sub-grains

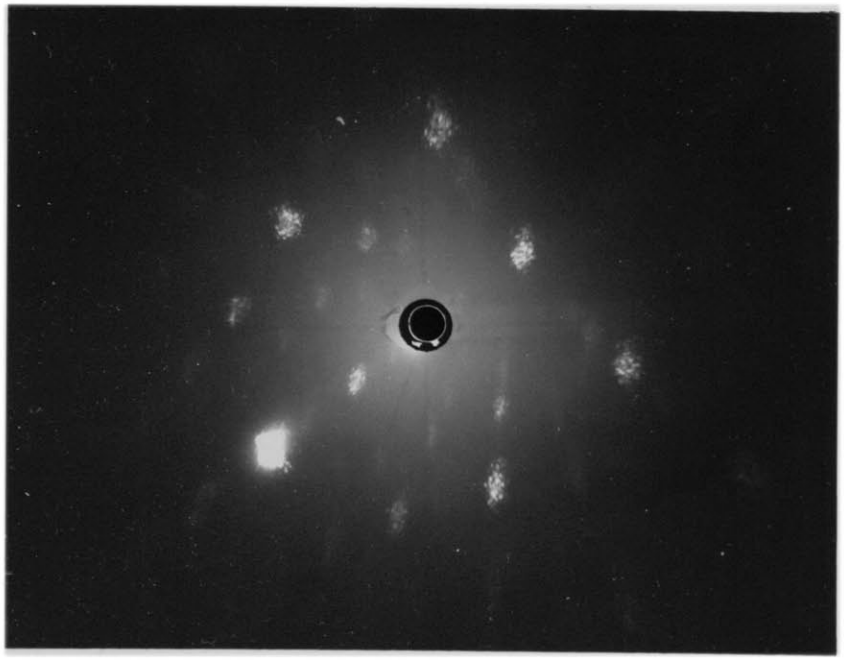


Figure 73. (Specimen P-11). Back reflection Laue photograph, taken in grain G close to the boundary between grain G and E, shows random breakdown of sub-grain spots.

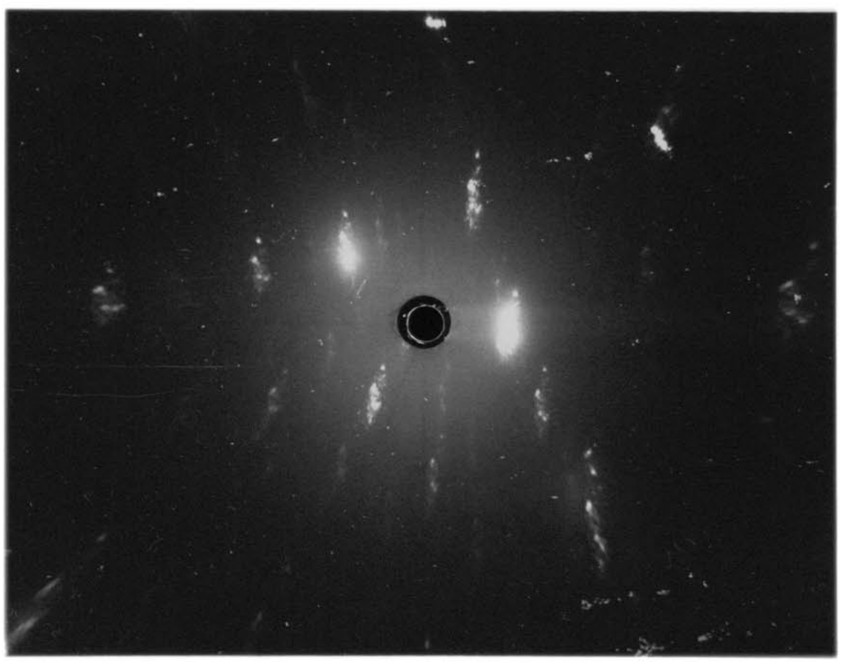


Figure 74. (Specimen P-11). Back reflection Laue photograph taken in grain E (Figure 72 F) shows breakdown spots with varying intensity.

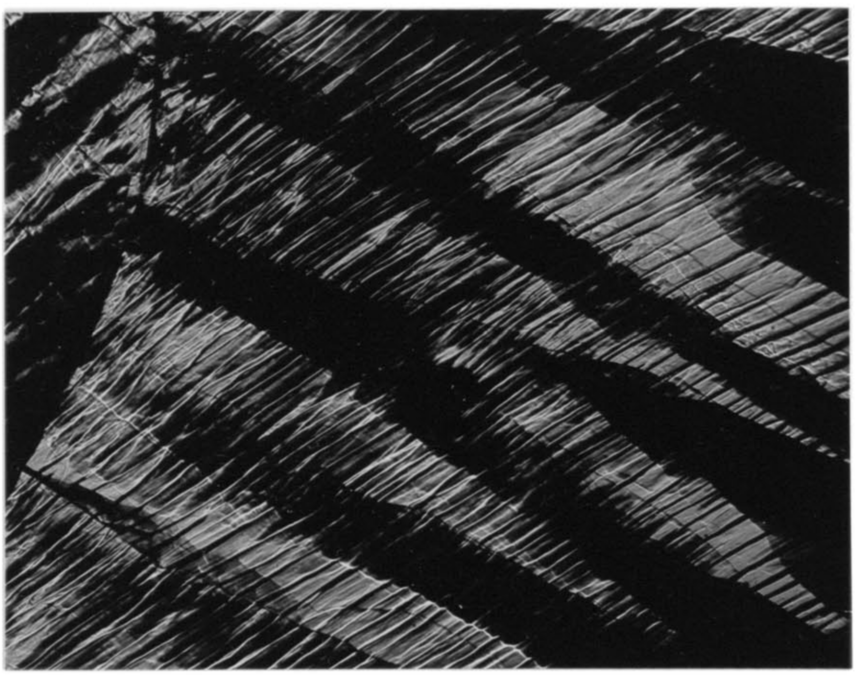
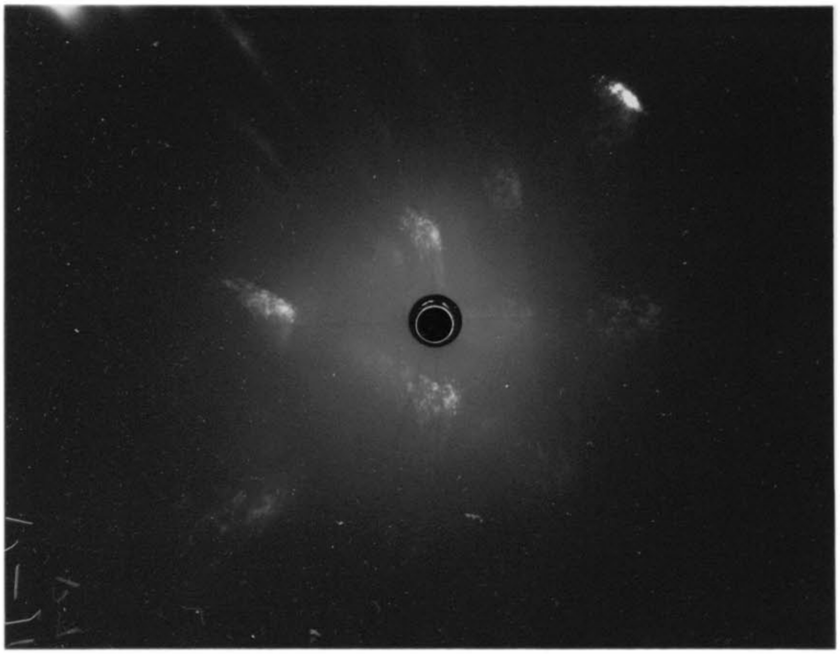
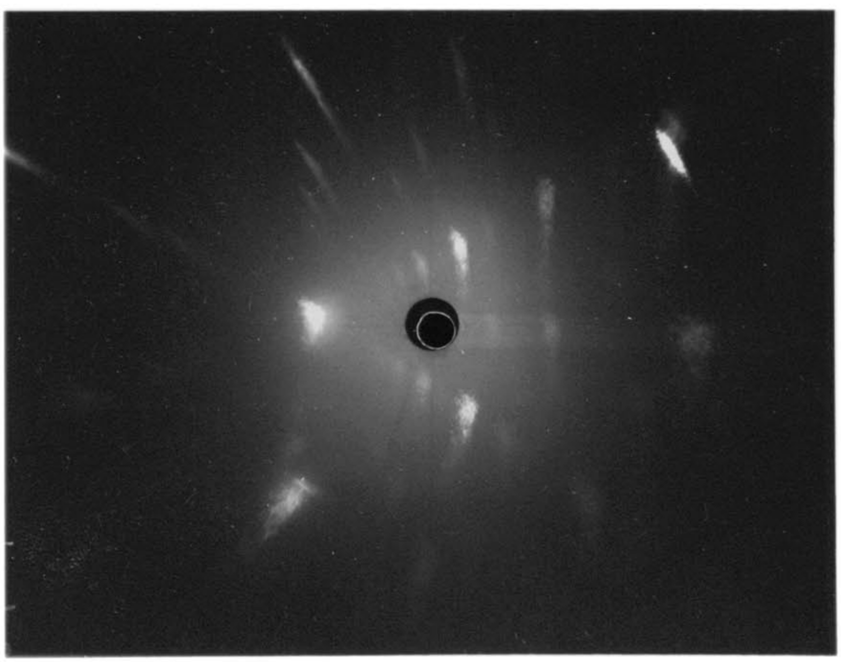


Figure 75. (Specimen P-11). Kinking bands superimposed in previously developed slip bands. 50 X.



A



B

Figure 76 A and B. (Specimen P-7). Back reflection Laue photographs.
A. Taken close to the grain boundary of grain A and C.
B. Taken close to the right edge of the specimen (Figure 61).

in P-11 are much larger than those in P-7. Comparing Figure 76 A (taken close to the grain boundary) with Figure 76 B (taken away from the grain boundary) it is revealed that the brokendown sub-grains spots of the former are more discreet than those of the latter. However, the background among breakdown spots is quite intense in both cases.

The size of sub-grains counted in the present investigation agrees with the results of Servi, Norton and Grant[14].

In discussing the formation of slip bands, reference has been made to the material between the bands being deformed by sub-grain formation when rotation and full development of the slip bands was restricted (Figure 50). This phenomenon is clearly shown in Figure 77. This result gives a good metallographic proof to the results of Servi, Norton and Grant that the size of sub-grains counted by X-ray methods is one to four times smaller than the average slip band spacing.

L. <u>Effect of Impurities on the Mode of Deformation</u>

Boundary sliding and migration was observed in 2 S aluminum for creep testing temperatures, 900° F and 1100° F. This is shown in Figure 78 A and B for 900° F. The extent of grain boundary migration was observed to be a little greater at 1100° F than that at 900° F for 2 S aluminum. However, the extent of boundary migration for 2 S aluminum was much smaller even at 900° F than in the case of high purity aluminum at 700° F (compare Figure 78 A with Figures 19 and 20). It has been observed in high purity aluminum that the grain boundary usually tended to reduce its surface area during migration. It may be seen from Figure 78 A that 2 S aluminum behaves in a similar

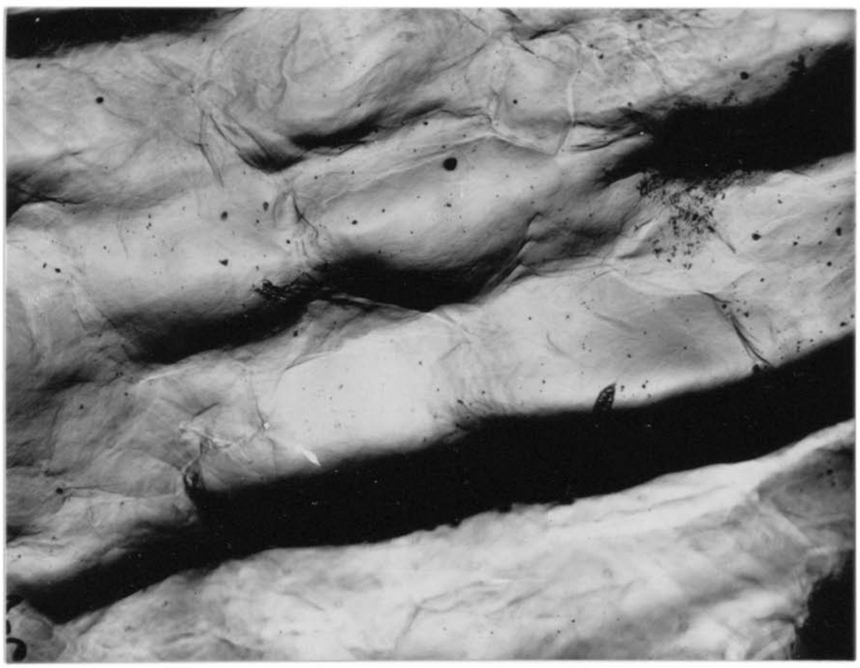


Figure 77. (Specimen P-6). The irregular nature of slip bands and the formation of sub-grains between the bands. 100 X.

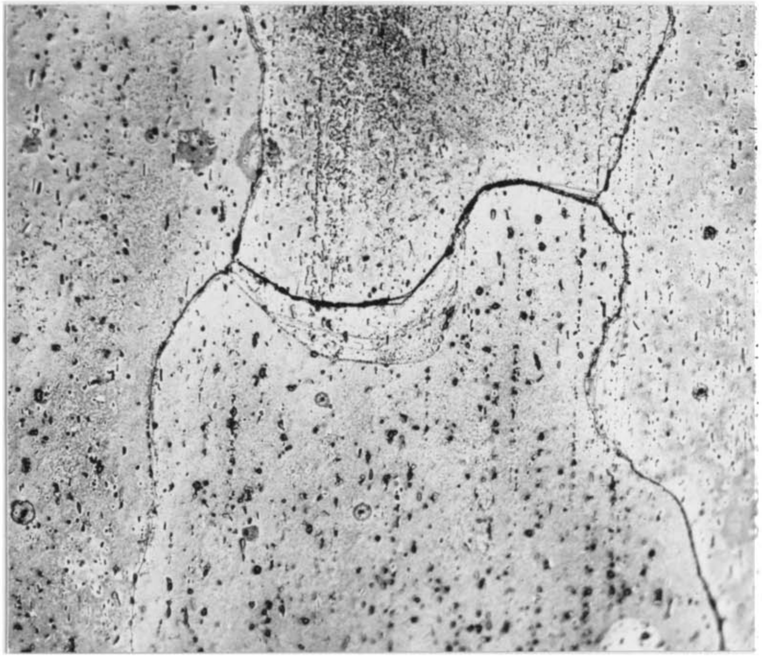


Figure 78 A. (Specimen 2 S - 7). Grain boundary migration in 2 S aluminum at 900° F. 150 X.

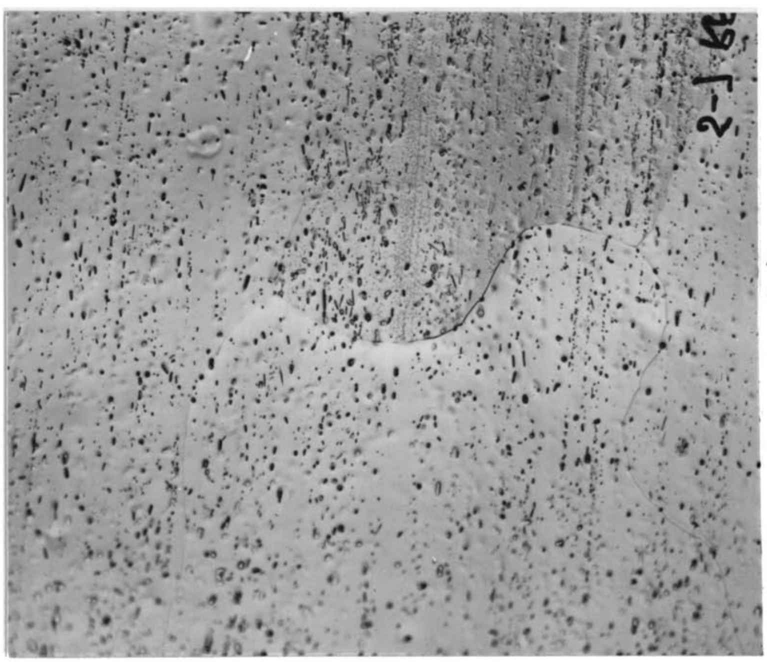


Figure 78 B. (Specimen 2 S - 7). Same field as Figure 78 A, repolished and etched. 150 X.

manner. The course of grain boundary migration is more irregular than what has been presented above for high purity aluminum. A 3 S aluminum specimen subjected to creep at 1100° F for 79.5 hours showed almost no evidence of boundary migration although the usual signs of boundary sliding were observed.

Figure 79 was taken after rupture from specimen 2 S – 6, tested at 900° F; Figure 80 A and B were taken at consecutive times during the test of specimen P-11 at 400° F. It can be seen by comparing Figure 79 with Figure 80 A and B that the deformation in 2 S aluminum was less concentrated in a certain region and tended to spread over the whole grain as opposed to the behavior of high purity aluminum. In other words, for practically the same amount of plastic strain the band spacing is much less for 2 S aluminum than for high purity aluminum. These results indicate that the impurities impede the annealing effect considerably so that deformation cannot continue on the pre-developed bands. This phenomenon corresponds to the effect of impurities on recrystallization temperature, in general, impurities increase the recrystallization temperature of the mother metal.

No folds of types shown in Figure 56 A and 65 A for high purity aluminum, were observed in 2 S aluminum at 900° and in both 2 S and 3 S aluminum at 1100° F, although concentrated deformation around a sharply curved boundary was observed (as shown by the solid arrow 2 in Figure 79). This result is expected because the irregular nature of grain boundary and the impurity contents of 2 S and 3 S aluminum would not give rise to appreciable boundary sliding.

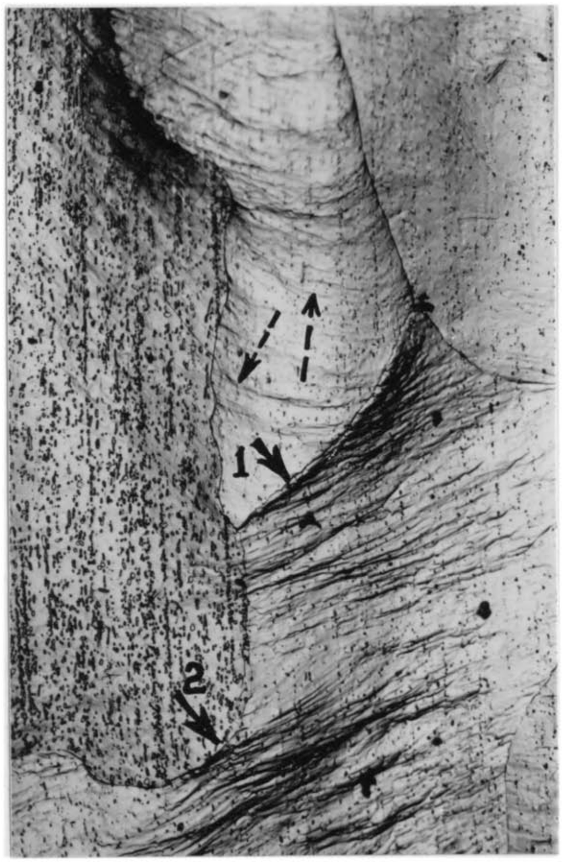
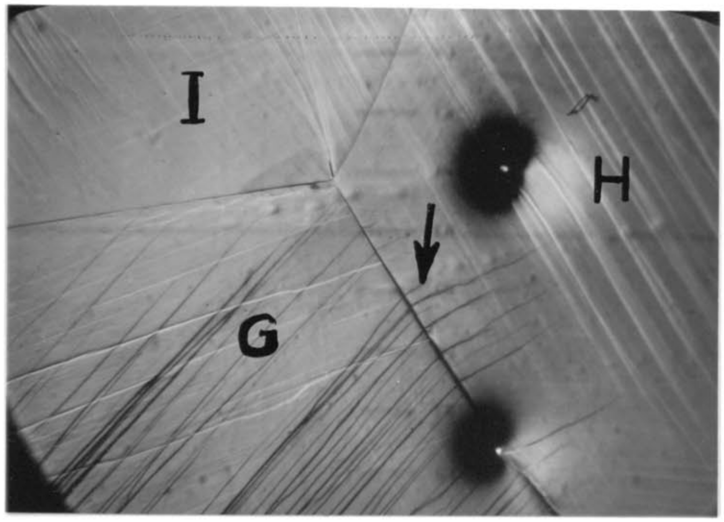
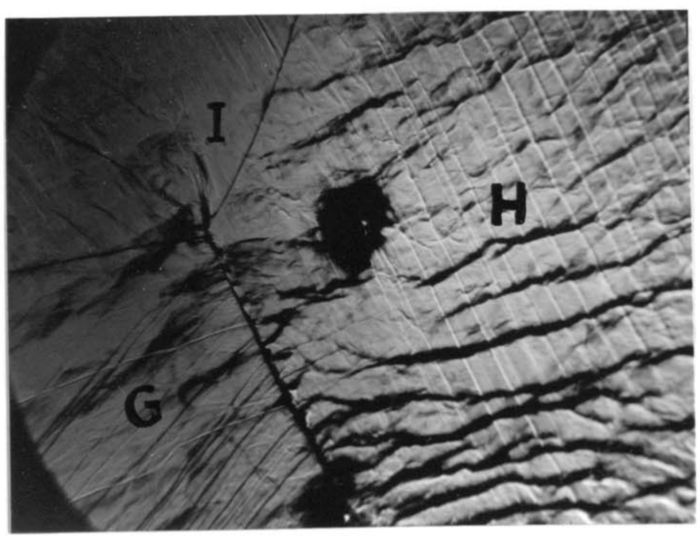


Figure 79. (Specimen 2 S - 6). Deformation tended to concentrate a little distance away from the grain boundary (solid arrow 1) and also to concentrate at the region where grain boundary changes its curvature (solid arrow 2). Note also the deformation lines in the shape of an arch (dotted arrows). 100 X.



A



B

Figure 80. (Specimen P-11).
A. Slip bands of the grain G caused partially developed slip bands in the grain H. 60 X.
B. Deformation bands developed in grain H in a later stage of creep. 60 X.

Sub-grains formed in 2 S aluminum usually exhibited sharp borders, showing a clear sign of being formed by bending (Figure 69). Sub-grain formation was also observed in 3 S aluminum as shown in Figure 81 which was repolished and etched after deformation in order to show the sub-grains formed in the neighborhood of the original grain boundary. It should be remembered that the grains were invariably lens-shaped with the longest dimension parallel to the axis of the specimen.

M. Effects of Grain Boundary on the Modes of Deformation in Their Neighborhood

A summary is presented below to show the effects of grain boundaries on the different forms of deformation in their vicinity.

1. When the orientations of the grains across the grain boundary are very close to each other, slip bands may go continuously through the grain boundary (see grain E and P in Figures 59 and grains P and K in Figure 39).

2. When the orientations of the grains across the grain boundary are not too different, slip bands developed in one grain can cause partially developed slip bands in the other grain (Figure 80 A, as indicated by the arrow).

3. Slip bands, with wide band spacing in the grain tend to split into finer bands on approaching the grain boundary (indicated by the arrow in Figure 82). This is apparently the result of limited amounts of slip which the grain boundary can accommodate in any one zone.

Figure 81. (Specimen 3 S -1). Repolished and etched. Sub-grain formation in 3 S aluminum. 150 X.

Figure 82. (Specimen P-11). Slip bands tended to split into finer bands on approaching the grain boundary. 60 X.

Figure 83. (Specimen P-7). A triangular region of less deformation is formed in the right grain around the triple point. 60 X.

4. Slip tends to concentrate a little distance away from the grain boundary (indicated by the solid arrow 1 in Figure 79); slip along two or more than two slip systems may occur there.

5. When a small grain bounded by the other grains deforms by slip, the slip lines, starting from the grain boundaries of the small grain join each other in the interior of the grain in the shape of an arch as if slip takes place along one slip system and bends during continuous deformation (indicated by dotted arrows in the upper center grain of Figure 79).

6. A triangular region around a triple point deforms much less than the rest of the grain (Figure 83 as indicated by the arrow). More than one slip system was operative in this region and the kinking bands stopped at the bordef of this region.

7. Sub-grains may form around the triple point as shown in Figure 70 (grain J) as indicated by the arrow. This region deforms much less and the slip bands developed in the other part of this grain tend to stop at this region.

8. Sub-grains may form along both sides of the heavily slid grain boundary, particularly around the triple point (Figure 67 A).

9. Folds in the grains form as a result of sliding of the grain boundary which lies opposite to that grain (Figure 56 A and Figure 65 A).

N. Inhomogeneity in Deformation and Component Creep Curves

The term "component creep curve" was defined above to describe a creep curve obtained by measuring the change in distance between two closely spaced reference marks on the specimen surface. Accordingly, a component creep curve of a grain boundary indicates the deformation not only by boundary sliding alone but also the deformation in the grains associated with boundary sliding. The original distance between two reference marks was about 0.6 – 0.7 mm. The error of measurement reported as precentage elongation was of the order of ± 1.5 percent. A curve was drawn through all the experimental points. In view of the stepwise nature of boundary sliding and migration and the blocking effect of a grain boundary on the development of slip bands, a smooth creep curve does not imply that deformation really takes place smoothly.

Figures 84 and 85 show four grain boundary component creep curves for the deformation of specimen P-8. One typical component creep curve of the grain being deformed by sub-grain formation and one creep curve over the whole gauge length are also included for comparison.

Referring back to Figure 3 (specimen P-8), it can be seen that the grain boundary of grains B and C makes about 75° to the direction of applied tension. The behavior of the creep curve of this grain boundary (Curve I, Figure 85) is in accord to the geometrical condition of this grain boundary.

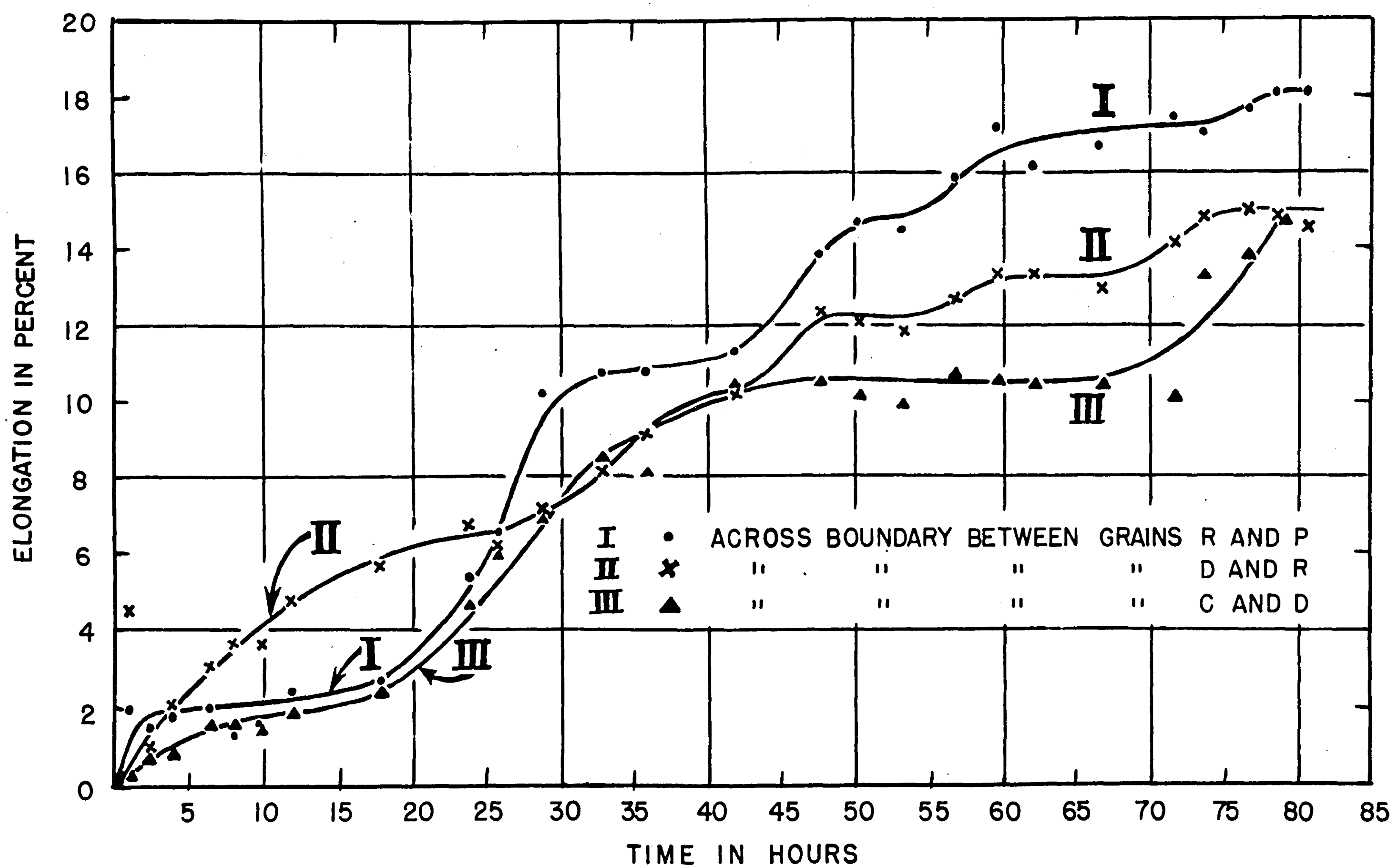


FIG. 84 COMPONENT CREEP CURVES ACROSS GRAIN BOUNDARIES OF SPECIMENS P-8 (FIG. 3)

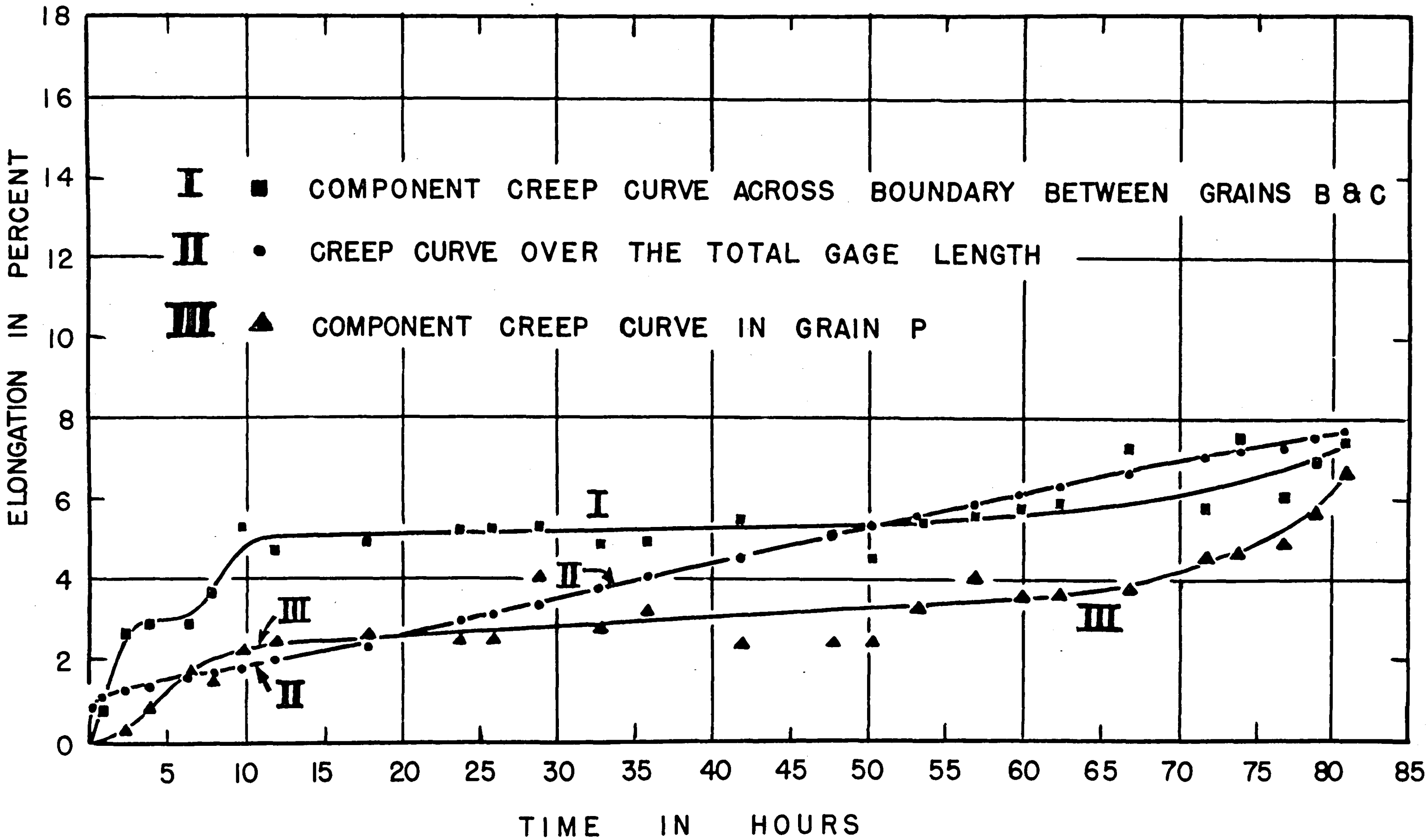


FIG. 85 CREEP CURVES OF SPECIMEN P — 8 (FIG. 3)

Though the grain boundary between grains C and D is fairly favorably oriented for sliding, it was initially curved close to the right edge of the specimen (Figure 16). After an initial period of about 15 hours during which this grain boundary was straightened out by boundary migration and the blocking effect at the triple point of grains C, D, and E was overcome, deformation increased (Curve III, Figure 84) quite rapidly until enough strain hardening set in to cause the creep rate to decrease. A period of recovery was required before another period of increasing creep rate set in again.

The same observations can be applied to the behavior of creep curves (Curve I, Figure 84) of the grain boundary between grains R and P except that the jump in elongation from 15 to 30 hours was associated with the formation of a fold in grain J as a result of sliding along the boundary between grains R and P as shown in Figure 86.

Since the grain boundary between grains D and R with two small grains E and G (Figure 3) at its ends offering relatively small resistance, was favorably oriented for sliding, it continued to slide from the very beginning showing a fairly continuous increase in elongation (Curve II, Figure 84). It is interesting to note that the period of a cycle of decreasing and increasing creep rate is smaller for the creep curve of this boundary than the other grain boundaries. The explanation of this behavior must be sought in the extensive sub-grain formation along both sides of this grain boundary and consequently in the angular nature of this grain boundary

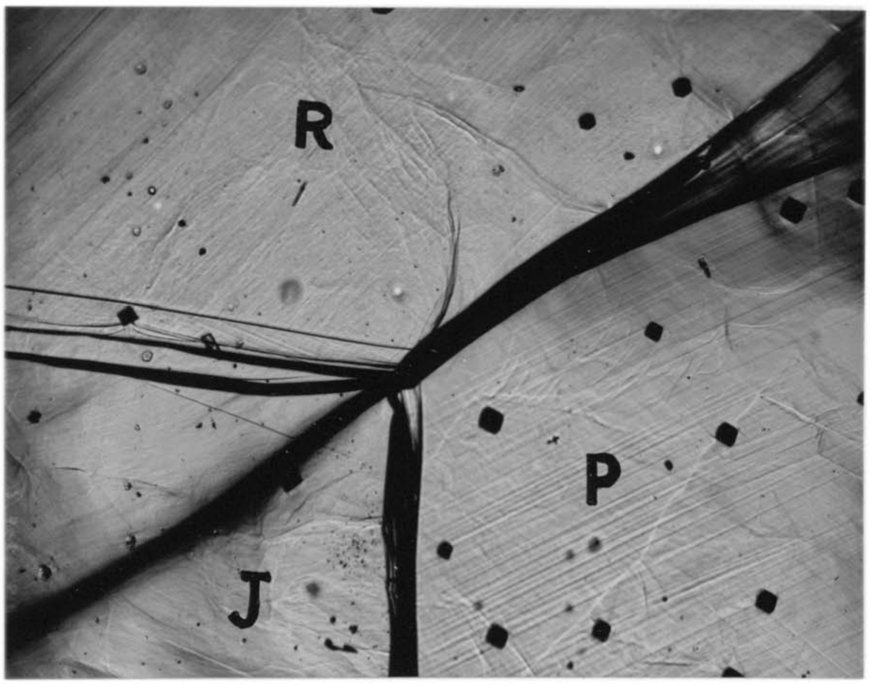


Figure 86. (Specimen P-8) front surface. Fold formation in the grain J caused by sliding of the opposite boundary. 100 X.

after a period of boundary migration (Figure 67). It is obvious that the more angular the grain boundary becomes the shorter the distance the grain boundary can slide, and therefore, the more frequently are grain boundary migration and recovery needed for further boundary sliding.

Boundary sliding will inevitably upset the original equilibrium arrangement of atoms on both sides of the slid boundary and particularly around a triple point of three grain boundaries. In other words, a high energy region is created by grain boundary sliding. This energy may either appear as stored elastic energy or may generate new surfaces through sub-grain formation. The grain boundary cannot continue to slide as a result of these disturbing effects, and boundary migration sets in in order to decrease the overall energy. Another possible phenomenon which might occur in conjunction with boundary migration is also deserving of consideration, namely, recovery through movement of dislocations to form polygons[9]. During the process of grain boundary migration, small deformation may take place by readjustment of atoms[15]. When the grain boundary migrates to a more stable position, grain boundary sliding may set in again. Boundary migration depends on the mobility of atoms and therefore is a time dependent process. The appearance of a creep curve obtained between two reference marks including a boundary, which has undergone successive sliding and migration, shows just what may be expected; namely, a repeated process of "active boundary sliding period" and "boundary migration period". The period during which active boundary sliding takes place is characterized by the fact that the creep rate increases to rather high values before the period of boundary migration takes place, resulting in a decreasing creep rate.

Curve III, Figure 85, shows a component creep curve of the material within grain P (Figure 3). The region between the two reference points, used in determining this curve, was deformed mainly by sub-grain formation. Curve III shows that after an initial period of increasing creep rate, which is characteristic of a very small creep strain, the elongation increased quite rapidly until a second period of decreasing creep rate sets in again. Curve II (Figure 42) was obtained across a slip band, the formation of which was affected by a grain boundary between grains P and K (Figure 37). It may be seen that the behavior of these two curves is very similar. This fact may be explained by considering the sub-grain boundaries as having the same general behavior as the boundary-affected slip bands. This observation seems to be substantiated by the fact that most of the sub-grain boundaries are made of narrow bands, in which slip has taken place along two or more slip systems.

The degree of disorder produced along sub-grain boundaries would be expected to be larger than that along slip bands, for the same amount of deformation. This would lead to smaller total creep strain for each cycle of increasing creep rates followed by decreasing creep rates in the case of deformation by sub-grain formation. This observation is in accord with the experimental results. Other component creep curves of regions deformed mainly by sub-grain formation exhibit similar behaviors to the creep curve III, Figure 85, described above.

Since this specimen is so coarse grained (P-8), the creep curve II, Figure 85, over the total gauge length is surprisingly smooth. In view of the fact that different regions of the specimen behave differently during creep, a smooth creep curve obtained over a rather long gauge length has its meaning only in a statistical sense.

In order to show the effect of an increase of stress on the behavior of component creep curves and on the mode of deformation, specimen P-6 was subjected to creep at 700° F and 200 psi until rupture. Typical component creep curves across the grain boundary and in the grains are shown in Figure 87 and 88, respectively. The initial period of rather rapid increase of creep strain across a grain boundary is in accord with optical observations during creep that boundary sliding and migration were primarily responsible for the deformation in this period. It has been pointed out previously that heavy boundary sliding caused the grain boundary to become angular after boundary migration. Consequently, the angular nature of the grain boundary would be expected to make further sliding difficult and therefore would result in a decreasing creep rate. In the meantime, this angular nature of the grain boundary would initiate slip bands at those places of the grain boundary where sharp changes of curvature have taken place. The initiation of slip bands at the angular grain boundary results in creep in the grain with a continuous increase of creep rate; and,

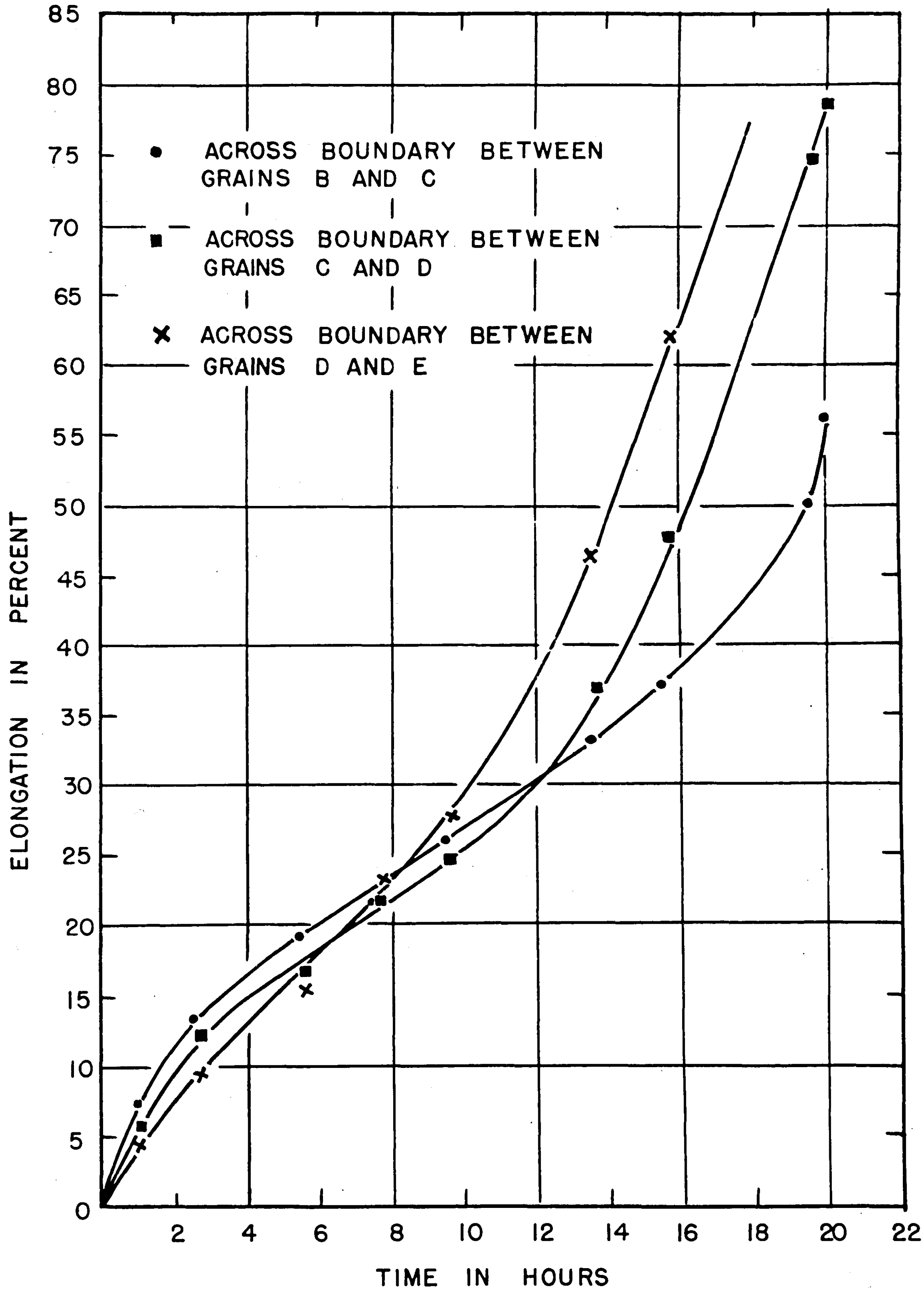


FIG. 87 COMPONENT CREEP CURVES ACROSS GRAIN BOUNDARIES OF SPECIMEN P-6 (FIG.4)

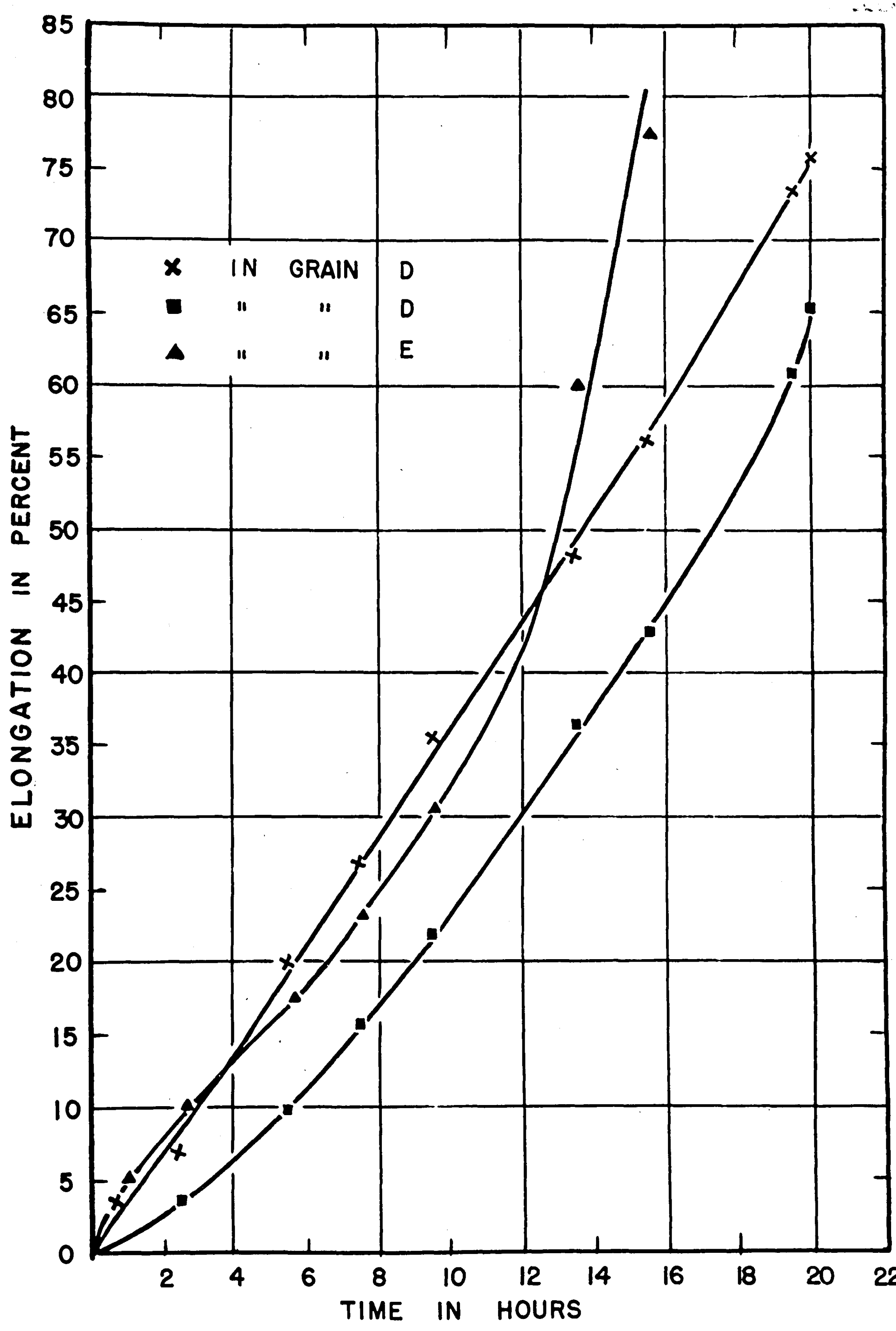


FIG. 88 COMPONENT CREEP CURVES IN THE GRAINS OF SPECIMEN P - 6 (FIG. 4)

in fact, the creep strain in the grains does overtake that across the grain boundary in the later stages of creep.

Accordingly, the contribution of the creep strain coming from the grain material along both sides of the heavily slid grain boundary would increase in the later stages of creep.

It is interesting to note that the stepwise nature of component creep curves at high stresses (Figures 87 and 88) is less distinctly shown in the case of those at low stresses (Figure 84 and 85). The infrequency of taking measurements relative to the rupture life of this specimen may be one of the reasons. It may well imply that the resistance, offered by the disordered material created along the sub-grain boundaries and slip bands, would be relatively ineffective in the case of high stress.

The point mentioned above that the amount of deformation in the grains overtakes that across the grain boundary in the later stages of cfeep is again illustrated in Figure 89 in which the percentage of elongation is plotted against the distance along the gage length of the specimen at four different times during creep. The elongation of the middle point between two reference marks in the grain is considered as the elongation between these two reference marks. The elongation at the point of a grain boundary is arbitrarily considered as the elongation between two reference marks across the grain boundary. That the elongation across the boundary between grains E and R (Specimen P-6) is relatively small in comparison with the other three equally favorably oriented boundaries

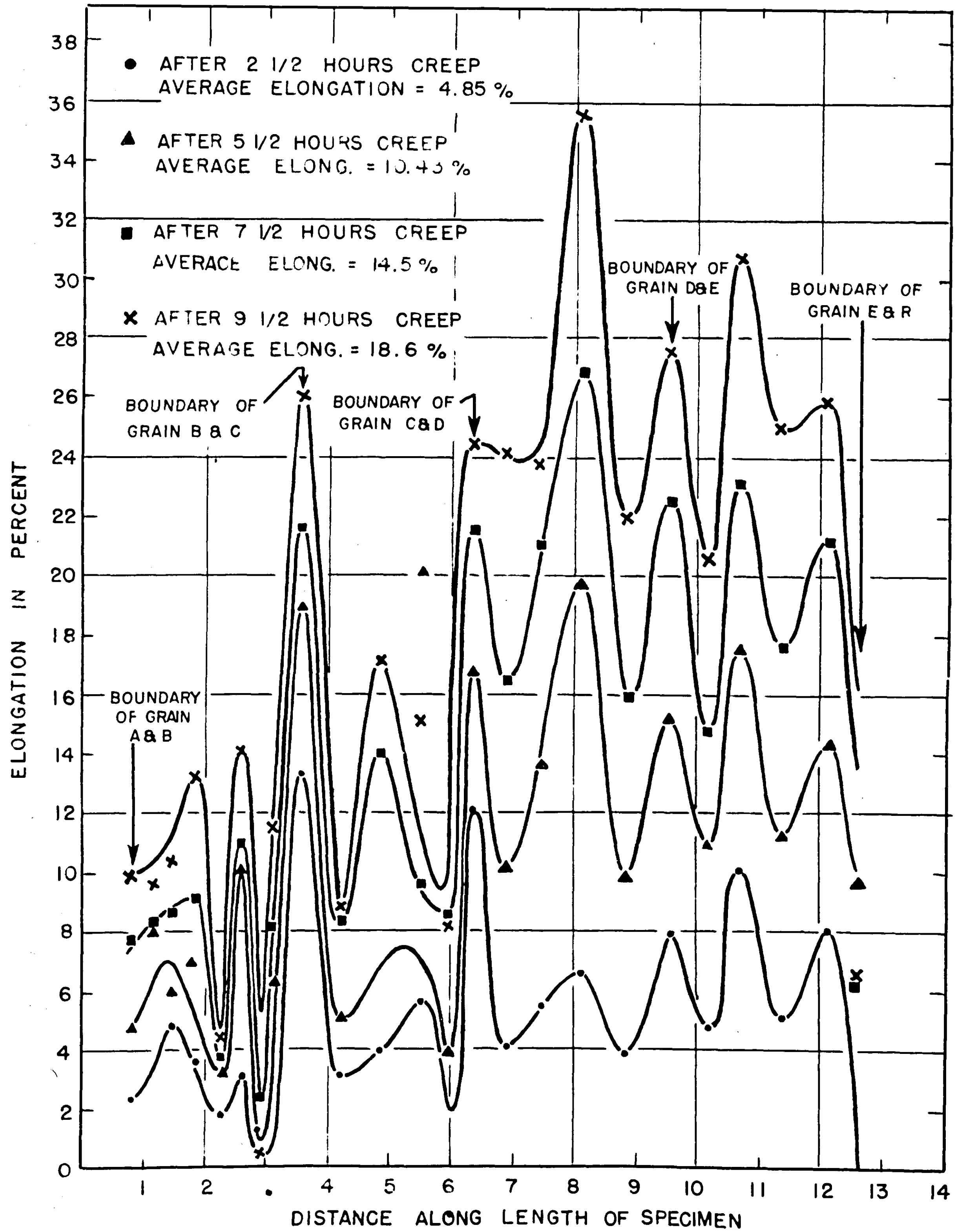


FIG. 89 DEFORMATION AS A FUNCTION OF TIME AND DISTANCE ALONG THE LENGTH OF THE SPECIMEN P-6 (FIG. 4)

may be a result of a slight orientation difference across this grain boundary, in addition to the complicated arrangement of grains in that part of the specimen.

Another point of interest is that the elongation in that part of the grain which is close to the grain boundary favorably oriented for sliding is usually less than either the deformation close to the center of the grain, or the deformation across the grain boundary. The explanation of this fact must be sought in the severely non-homogeneous nature of deformation along both sides of the heavily slid and migrated boundary. Though, as mentioned above, slip bands may be initiated at this region, yet, the amount of slip is restricted by grain boundaries in this region. Furthermore, the fact that the grain surface in this region may be tilted either upward or downward with respect to the grain boundary would give rise to an apparent elongation of less magnitude than it should be if measured parallel to the tilted surface.

As pointed out in the presentation of the section on grain boundary sliding and migration, when the specimen is subjected to creep at 1100° F, the grain boundary migrates very fast to a position almost perpendicular to the axis of the specimen during the early stage of the test in a stepwise manner, each step of migration being accompanied with quite small amounts of sliding. Figure 90 shows two component creep curves obtained on specimen P-10. The upper curve represents a component creep curve obtained across the old boundary between grains A and B, Figure 5. It shows that an elongation of

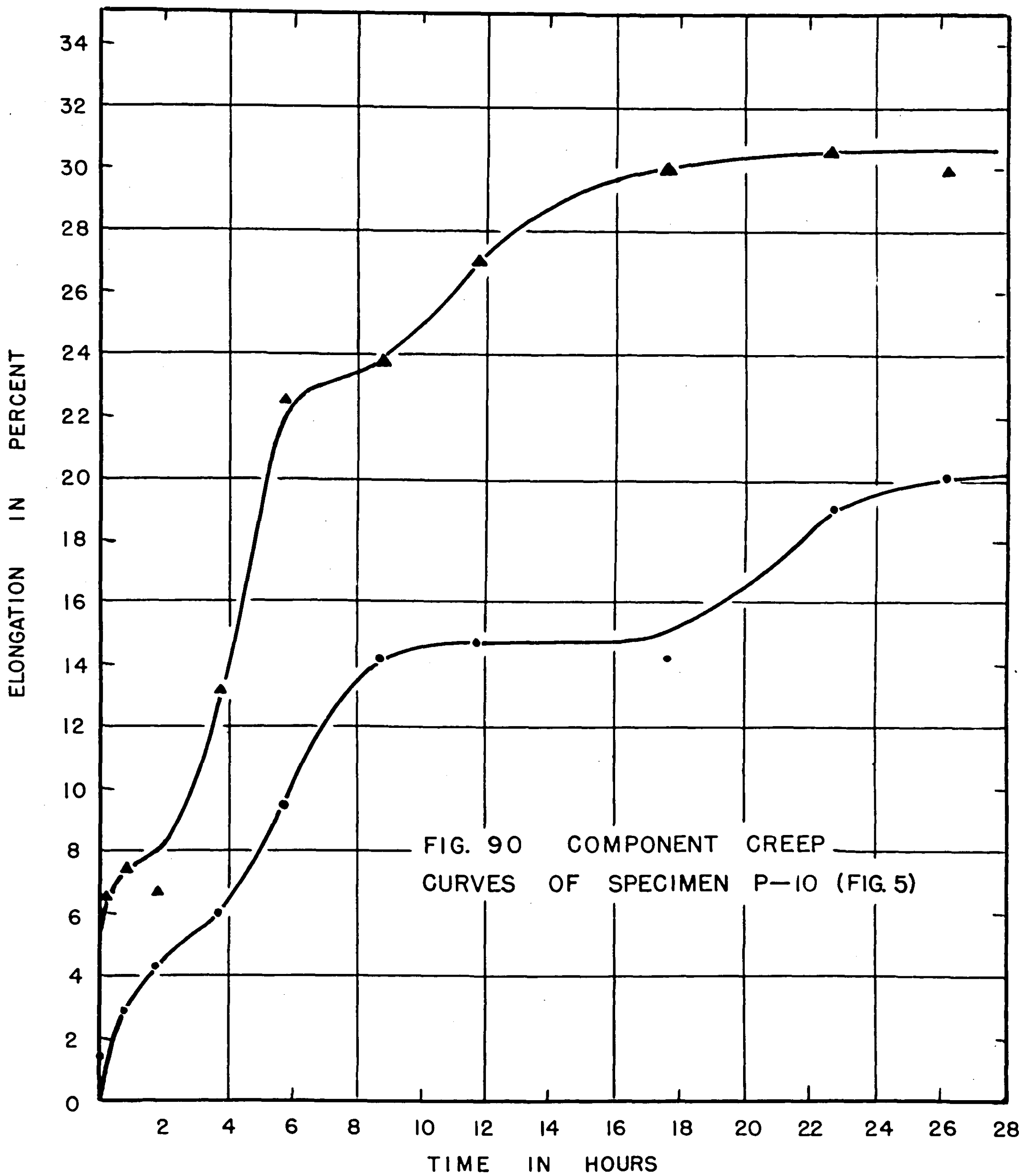


FIG. 90 COMPONENT CREEP CURVES OF SPECIMEN P—10 (FIG. 5)

about 7 percent occurs in the first one-half hour period. During this time the grain boundary between grain A and B slid and migrated out of the field covered by these two reference marks. In comparison with the upper curve obtained in grain A, the lower curve shows a rather small amount of creep in the first half hour period. Thereafter, both curves represent the deformation in the grains by slip and sub-grain formation. Since the lower one was close to the enlarged end of the specimen its amount of deformation was comparatively small.

It has been shown that the deformation at 400° F is characterized by slip band formation in the early stages of creep and by slip and sub-grain formation in the later stages of creep. The behavior of the grain boundary at 400° F can be divided into two types. When the orientation difference across the first type of grain boundary is such that deformation in one grain can be transmitted to the other grain, the second type of grain boundary is such that the deformation in both grains is restricted. The former case is shown in Figure 91, Curve II.

Curve II shows the elongation across the boundary between grains A and C, Figure 6. The elongation value shown in Curve II lies in between those shown for Curves I and III. Curves I and III represent the creep of regions in grains A and C respectively, in the immediate vicinity of the boundary between grains A and C.

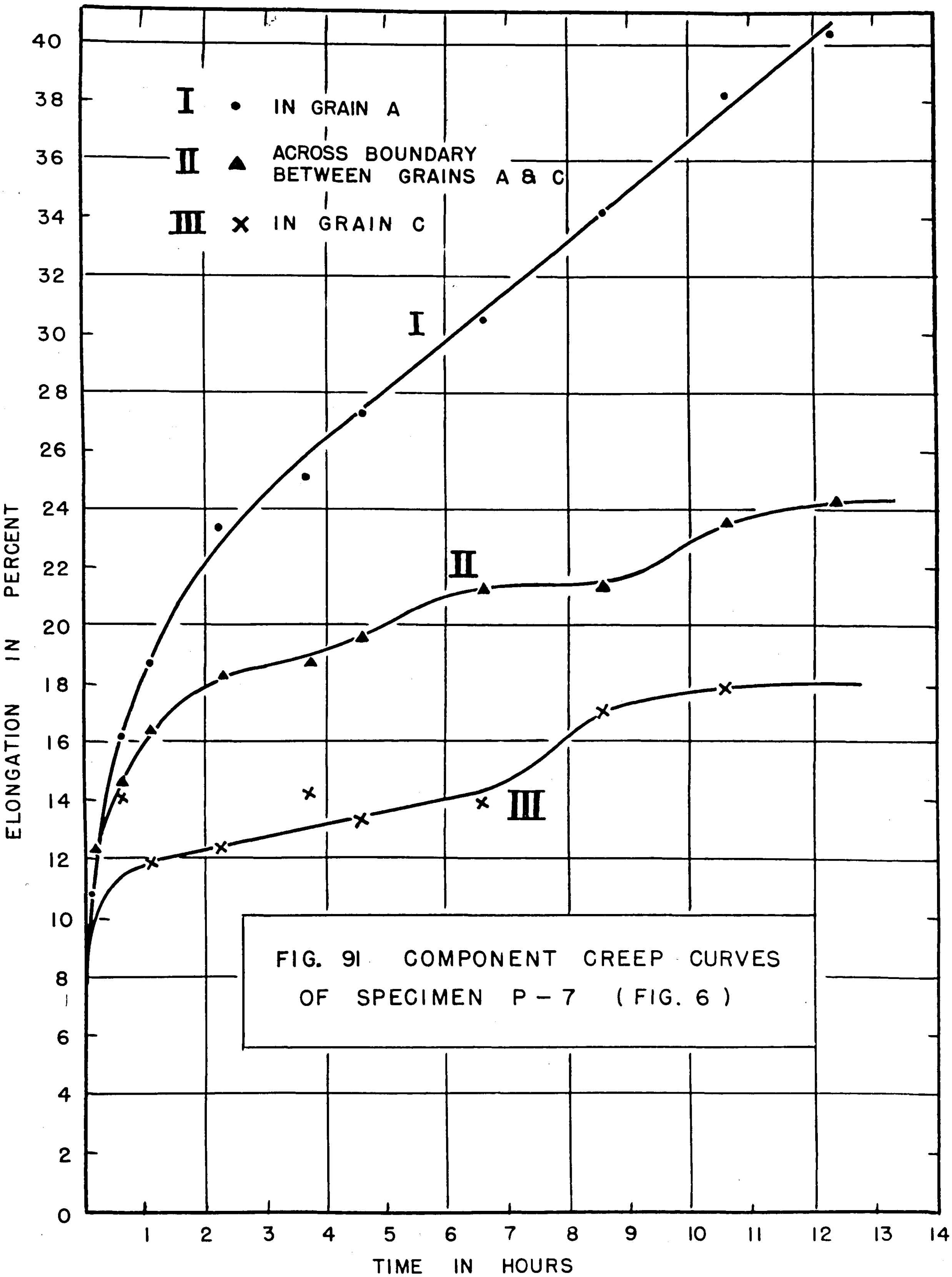


FIG. 91 COMPONENT CREEP CURVES OF SPECIMEN P – 7 (FIG. 6)

The latter case is shown in Figure 92. The extent of deformation across the grain boundary is smaller than that in both grains. These results agree with those of Boas and Hargreaves[16]. A creep curve over the whole gauge length is also shown in Figure 92 for specimen P-7.

Figure 93 is prepared by plotting elongation against the distance along the axis of the specimen at two different times, in order to show the effect of time on the deformation across grain boundaries and in the grains. It can be seen that the deformation in the grains follows the same general trend at two different times. The peaks and valleys correspond to whether heavily slipped bands are included between those two reference marks. However, deformation across a grain boundary becomes more important in later stages of creep, such as boundaries between grain D and F and between grain H and I, Figure 6. This corresponds well to the optical observation that at 400° F boundary sliding occurs in later stages of creep (Figure 72 d).

The two types of deformation across the grain boundaries mentioned in conjunction with Figure 89 are also shown in Figure 93.

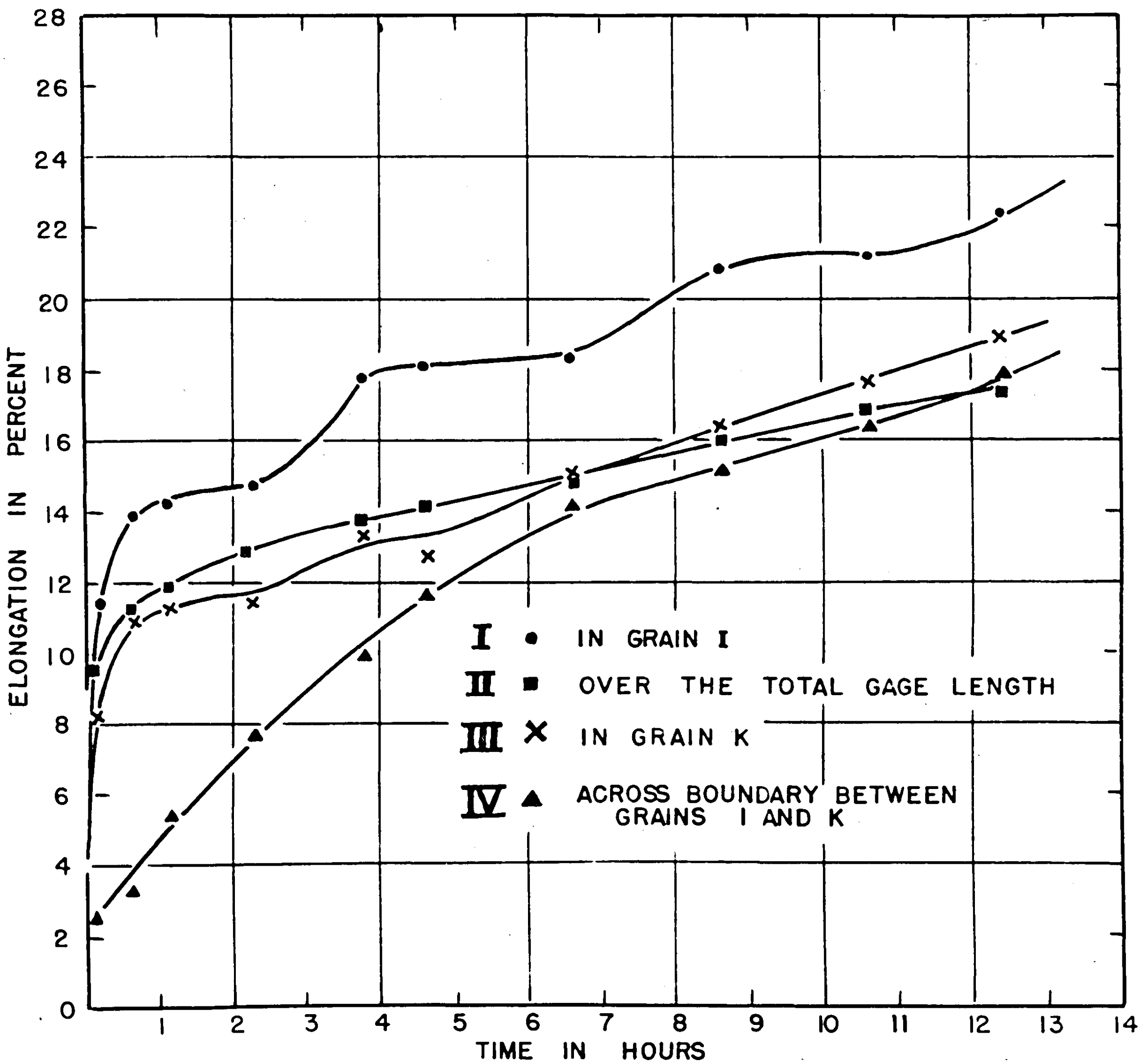


FIG. 92 COMPONENT CREEP CURVES AND CREEP CURVE OVER TOTAL GAGE LENGTH OF SPECIMEN P-7 (FIG. 6)

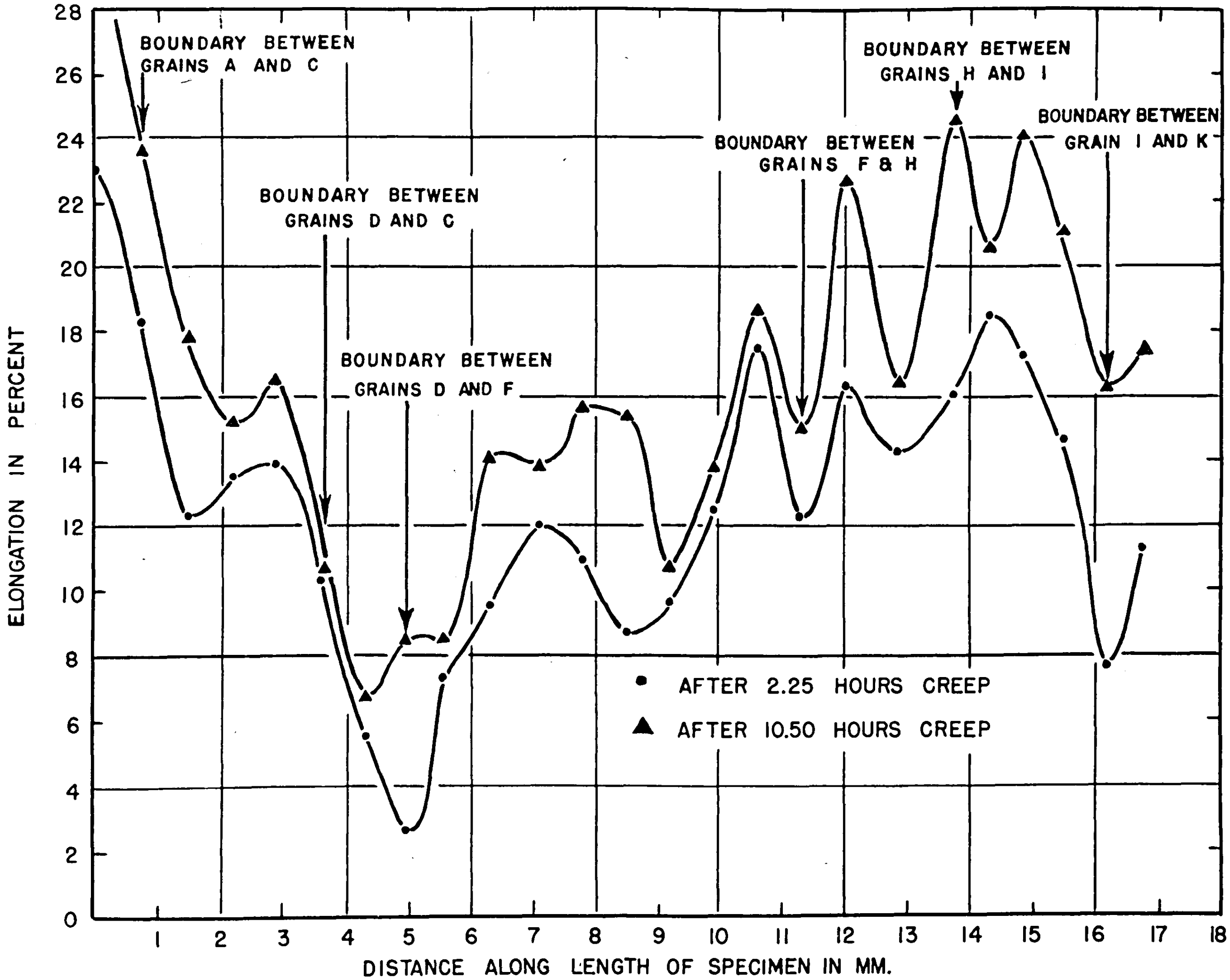


FIG. 93 DEFORMATION AS A FUNCTION OF TIME AND THE DISTANCE ALONG THE LENGTH OF THE SPECIMEN IN SPECIMEN P — 7 (FIG. 6)

IV. DISCUSSION

A. Sub-Grain Formation

The mechanism of sub-grain formation during creep is still in a state of uncertainty at the present time. The argument rests on whether sub-grain formation is a mechanism of deformation in itself[1,2,17] or the end product of non-homogeneous deformation[14,18,19]. The results presented above clearly show that sub-grain formation arises from the fact that normal slip cannot be fully developed due to restricting effects of the grain boundaries. The prominence of sub-grain formation around the triple point and along heavily slid grain boundaries further substantiates this case.

Two types of sub-grains were observed. The first type is characterized by the fact that its boundaries show clear bending nature and can be revealed by electro-etching after repolishing (Figures 55 A grain P, 60, 67 and 69). The second type seems to be formed because of slip on different slip systems in neighboring regions (Figure 19 c). The regions delineating the sub-grains of the second type has the appearance of the deformation band but their boundaries are not sharp and cannot be revealed by electro-etching after repolishing. The X-ray technique used in the present investigation is not able to locate and identify these two different types of sub-grain boundaries. The limited results from X-ray work seem to show that the broken-up sub-grain spots are more discreet for the first type than for the second type. It should be pointed out that even in the first type slip along different slip systems usually takes place in different sub-grains.

Chen and Mathewson[21] have shown that kinking bands, or cross slip and duplex slip, may result in single crystals of aluminum under tensile deformation. The occurrence of either of these mechanisms, they suggested, depends on the orientation of the crystal with respect to the tensile axis. In polycrystalline aluminum, the different grains have a range of orientations. Blocking effects of the grain boundaries enhance the operation of these two mechanisms. Therefore, it is reasonable to expect that during deformation, some of the grains in a polycrystalline aluminum specimen would exhibit kinking band formation, whereas others would deform by cross slip and duplex slip. It has been suggested above that one of the two types of sub-grains is formed from kinking bands whereas the other is formed as a result of cross slip and duplex slip in neighboring regions in the same grain. Cross slip and duplex slip, however, are produced by different slip systems being operative in neighboring regions because of the blocking effects arising from restrictions imposed on grain boundaries by adjacent grains. Consequently, it is expected that both types of sub-grains will be found in different grains of the same polycrystalline specimen.

A grain in a polycrystalline material cannot deform along only one slip system for large deformations because rotation of the slip plane is limited by the grain boundaries. As shown by Taylor[22], slip along five slip systems is needed for one grain to deform, by the same relative amount in different directions, as the whole specimen. There is no question about the operation of cross slip and duplex slip in a polycrystalline material. The question is why kinking band formation sometimes occurs in preference to cross slip and duplex slip. As shown by Chen and Mathewson[21], the orientation of the material in the kinking band lags behind during rotation compared to the orientation of the material outside the band. This results implies that for the same amount of deformation the amount of rotation of the whole crystal is less in the case of a grain containing kinking bands than in the case of a grain which has deformed by continuous slip along one single slip system. In other words, the kinking band performs the function of duplex slip as far as the restriction of the rotation of slip planes of a single slip system is concerned. The results of the present investigaion indicate that kinking bands occur after a considerable amount of slip along one slip system has taken place (Figure 59 A – G and Figure 72 A – F). It is conceivable that the disorganized material created both along and between the pre-developed slip bands would resist another slip system from passing through. Kinking bands occur because intersection or movement through the pre-developed slip bands is not required. Polycrystalline material is particularly apt to kinking band formation because less displacement across the grain boundary is required for kinking band formation. As shown in Figure 59 a – g, kinking bands can propagate from one grain to another grain almost without regard to the grain boundary.

In view of the complicated stress pattern along a grain boundary and around a triple point, the evidence that the size of sub-grains may vary by a factor of 3 or 4 times even in the same grain is not unexpected. However, the size of sub-grains tends to increase as the stress decreases and temperature increases. On the basis of dependence of annealing effects on critical internal stress[24], the dependence of slip band spacing on temperature is ably explained by Brown[23]. Servi and Grant's results[25] showing that slip band spacing in creep tests depends more strongly on stress than on temperature is not surprising if it is remembered that Patterson and Orowan's results[24] with aluminum show annealing effects even at room temperature. The result of the present investigation and the results of Servi, Norton and Grant[14] that the size of sub-grains is not very strongly dependent on temperature is not unexpected. Brown's explanation of the dependence of slip band spacing on temperature may apply to the temperature dependence of the size of sub-grains of the second type in the formation of which, slip is believed to play an important role. The internal stresses created in the vicinity of the sub-grain boundaries of the second type would be expected to be much higher than those in the vicinity of the slip bands formed in single crystals. This region would be prone to concentrated deformation because of its high annealing rate. Observations during creep tests seem to show that the boundaries of the second type become heavier as deformation continues. There is an indication, however, that the amount of bending along sub-grain boundaries of the first type is limited. Further deformation in this first type tends to occur inside the pre-formed sub-grains, and the newly formed sub-grain boundaries seldom go through the pre-formed ones.

B. Comparison of Boundary Sliding and Slip Band Formation

Both slip along slip planes and sliding along grain boundaries have been shown to exhibit cyclic behavior in elongation-time creep plots. The results further show that as soon as slip is initiated in a band, this slip causes an avalanche of slips along slip planes in a wide band. The deformation may amount to 20 percent due to the avalanche of slips until a period of decreasing creep rate sets in. When there is less restriction in the rotation of the slip planes by grain boundaries, slip can continue on the slip band until fracture occurs along that slip band. In view of this result, it can be concluded that the period of decreasing creep rate must be caused by restrictions on the rotation of the slip planes, and the restriction on the amount of displacement the slip band can produce across the grain boundary (Figures 39 A, 40 A, and 41). The latter is particularly important in polycrystalline material because new grain boundary surfaces are created between the two grains through which the slip band goes. Although the case shown in Figure 39 A, of a slip band going across a grain boundary, may be a special one because of similarity in orientation of the two grains, every grain boundary must have a capacity to accommodate a certain amount of slip. In discussing the slip band spacing in polycrystalline material, one factor, namely the capacity of a grain boundary to accommodate slip, must be taken into consideration, although the influence decreases as the distance from the grain boundary increases (Figure 82).

Grain boundary sliding results in the creation of new surfaces and a disturbance in the form of an increase in strain energy in the neighborhood of the slid boundary. Boundary migration, which follows this process of boundary sliding, reduces both the increase in surface area and the surplus strain energy. The component creep curves (Figures 38 and 42) over a region containing a boundary affected slip band show that a large increase in elongation occurs soon after the first formation of slip bands. This fact can be interpreted by supposing that an annealing effect must have taken place very rapidly. Creep curves (Figure 84) obtained across a grain boundary undergoing sliding and migration also show a similar increase in elongation soon after migration. However, the increase is of a much greater magnitude in the case of the boundary affected slip bands than it is in the case of the migrating grain boundaries. It may, therefore, be implied that the annealing effects following slip are much faster than the overall recovery caused by boundary migration.

The results, obtained by King, Cahn and Chalmers from bi-crystals of tin subjected to creep, show that the creep strain increases rapidly in the early stage of the test and reaches a rather steady value in the later stage of the test[26]. This result may correspond to one cycle of the component creep curves across grain boundaries in the present investigation. Consequently, the cyclic behavior of the component creep curves across a grain boundary is a general one. It may result from the fact that larger distances of atomic movement are required because a larger orientation difference is involved in the latter case than in the former. Boundary sliding occurs also in an avalanche manner, but it is very frequently intervened by boundary migration and the distance it can slide in every step is much smaller than that in the case of slip. It is conceivable that polycrystalline material differs from bicrystal material only in the extent of sliding in each step and the frequency of boundary migration, but not in the nature of sliding process.

The amount of boundary sliding in each step may be limited by the disturbance created along the slid grain boundary. A grain boundary apparently smooth, is not expected to be smooth on an atomic scale at 500 magnification. It has been shown that after a cycle of boundary sliding and migration, the grain boundary becomes angular even on an macro scle. The amount of sliding in each step, after the grain boundary becomes angular, would be still smaller. In the case of polycrystalline material, the amount of sliding in every step would be smaller than in the case of bicrystals because energy has to be supplied to create new surfaces at each end of the slid boundary, in addition to the new surface created at the free edge of the material.

The process of boundary migration is a strongly time and temperature dependent process. Thus, it is not surprising to find that the grain boundary sliding process is a highly strain-rate dependent process while slip is not much so. The results of Hanson and Wheeler[27] shows that boundary broadening occurs in the later stage of creep in commercial aluminum. This led Orowan[15], on the basis of empirical formula obtained by Andrade[28], to propose a mechanism of boundary sliding and to account for the mechanism of secondary creep of the conventional creep curve. The results of the present investigation for high purity aluminum tested at 400° F agree with the results of Hanson and Wheeler, namely, boundary sliding becomes important in later stages of creep. For creep tests at 700° F, boundary sliding and migration operate from the very beginning of the test and indeed account for the bulk of the deformation in the early stages of creep (Figures 84 and 85). At very high temperatures (1100° F for high purity aluminum) the deformation in the grains becomes important again because the grain boundary migrates very fast to a position to make further sliding difficult, leaving deformation possible only in the grains. Consequently, any theory proposed to describe the behavior of grain boundaries during creep over a wide range of temperatures must consider the cooperative process of boundary sliding and migration.

In view of the similar shape of component creep curves across a grain boundary and across a boundary affected slip band the subdivision of conventional creep curves into transient creep and steady state (or quasi-viscous) creep seems incompatible. It is no wonder that the value of K of the so-called K flow[28] does not follow the Newtonian law of viscous flow, i.e., it is not proportional to the applied stress. As far as creep performance is concerned, grain boundary sliding and grain boundary-affected slip have some similarity. Whether these two processes differ only in degree or fundamentally is still unknown.

C. Intercrystalline Failure

It has been shown that grain boundary migration is very structure sensitive. It has also been shown that boundary migration is a kind of recovery process which makes further boundary sliding possible. In impure or alloyed materials, grain boundary migration becomes less significant, restricting sliding to a fixed boundary. Sliding along the same boundary would eventually give rise to cracks as a result of interlocking effects at the triple point. This is why commercial alloys subject to high temperature creep show intercrystalline fracture whereas no sign of intercrystalline failure has been observed for high purity aluminum in the present investigations.

V. SUMMARY AND CONCLUSIONS

From studies of very coarse grained high purity, 2 S, and 3 S aluminum at 400°, 700°, and 1100° F, the following conclusions can be drawn.

1. The types of deformation observed are as follows: slip bands, kinking bands, deformation bands, fold formation, sub-grain formation, and boundary sliding and migration.

2. Boundary sliding and migration have been shown to take place in a stepwise and successive manner. The zig-zag nature of triple point movement, (i.e., movement of the point where three grains meet), is considered to be a necessity of these highly co-operative processes, because the direction of boundary migration depends primarily on the direction in which the grain boundary slides in the previous sliding step. Component creep curves across grain boundaries which undergo successive sliding and migration show several cycles of "active boundary sliding" and "boundary migration". This behavior is supported by optical observations during the actual test. The driving force for boundary migration is a combination of strain energy and surface energy. The latter is important at 1100° F. Boundary sliding may cause fold formation in the grains. Boundary migration is also shown to be very structure sensitive. Boundary sliding was observed in the later stage of creep at 400° F.

3. Boundary sliding and migration and the deformation associated with them are very important mechanisms of deformation at 700° F. They may account for most of deformation in early stages of creep. The deformation in the grain may overtake that of boundary sliding

and migration in the later stages of creep. At 1100° F, boundary sliding and migration are important only in the very early stage because the grain boundary migrates very fast to a position to make further sliding difficult.

4. In high purity aluminum, slip tends to concentrate on certain pre-developed slip bands and wide slip band spacing results. Slip bands are fully developed if rotation is permitted and no grain boundaries are in their path. A component creep curve across a fully developed band shows continuously increasing creep rates until fracture occurs on this band, while a similar curve across a boundary-affected band shows continuous increasing creep rate followed by a decreasing creep rate. The latter behavior is explained by the restricting effect of grain boundary on further unimpeded slip.

5. Folds in the grains are considered to be formed by a combination of deformation bands and kinking bands. The formation of folds in the grains is caused by sliding of the opposite grain boundaries. The full development of folds has been shown to depend on

a. whether there are grain boundaries in its path
b. the orientation of the sliding boundary with respect to the applied stress
c. the relative areas of the grain and its opposite sliding boundary in the direction of boundary sliding
d. the relative orientation difference of the neighboring grains across the sliding boundary.

Sub-grains are shown to be the result of grain boundary restricting effects on the normal development of slip bands. The formation of sub-grains is often prominent along the heavily slid grain boundaries and around the triple points.

6. Two types of sub-grains were observed. One type is believed to be associated with kinking band formation and the other, deformation band formation. The size of sub-grains measured metallographically may vary by 3 or 4 times. The size of sub-grains tends to increase as the temperature increases and stress decreases.

7. Boundary migration and sliding were observed in 2 S aluminum at 900° F and 1100° F. As a result of impurity content, the amount of boundary migration is much smaller and its course more irregular than is observed in high purity aluminum. Normal boundary sliding (no evidence of boundary migration) was observed in 3 S at 1100° F. The slip band spacing is smaller in 2 S aluminum than in high purity aluminum. Fold formation was not observed in 2 S and 3 S aluminum. This is interpreted as due to the small extent of boundary sliding. Sub-grain formation was observed both in 2 S and 3 S aluminum.

VI. SUGGESTIONS FOR FUTURE RESEARCH

1. By running creep tests on single crystals of high purity and 2 S aluminum, the following questions may be quantitatively answered as regards the relationship between structural changes and elongation-time behavior as a function of purity.

a. What is the extent of annealing effects in a slip band as a function of stress and temperature?

b. Under what conditions will kinking bands form at elevated temperatures? Whether the formation of kinking bands makes the deformation easier or difficult? To what extent does its formation influence the elongation-time behavior?

c. What is the nature of sub-grain boundaries? Are they weak or strong regions?

d. The rupture life and minimum creep rate obtained on 2 S aluminum can serve as a more fundamental proof that the occurrence of a break on a log-stess log-rupture life (or log minimum creep rate) plot is associated with the behavior of the grain boundary.

2. By using high purity and solid solution type (Al, Mg alloys are preferable because more data are available) aluminum which contains 3 - 6 grains with predetermined orientations, the information on the following processes can be obtained as a function of temperature, stress, strain rate, purity and orientation differences.

a. The rate, the amount, and the direction of boundary migration.

b. The rate and the amount of boundary sliding.

c. The relationship between structural changes and elongation-time behavior in different grains.

It is hoped that after the information from the above proposed research program is secured, a formula and a theory, based on the mechanism of deformation, can be established to describe the creep behavior of single phase alloys in a statistical manner. Further knowledge about grain boundary can also be obtained.

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