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TECHNICAL NOTE 3556

GRAIN-BOUNDARY BEHAVIOR IN CREEP OF ALUMINUM BICRYSTALS

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SUMMARY

The gliding of one metal crystal with respect to another parallel to their mutual grain boundary has been studied in pure aluminum bicrystals during isothermal creep at temperatures ranging from 200° to 650° C under static stresses of 10 to 1,600 psi. The motion is spasmodic and begins with an induction period. Its direction is determined by that of the maximum resolved shearing stress in the plane of the grain boundary, but its rate depends as well upon the angle through which the active slip systems are bent as they cross the grain boundary, being the higher the greater the distortion at the grain boundary. The overall gliding rate is a linear function of the cube root of time and bears an Arrhenius relationship to the temperature, and its logarithm is proportional to the stress, over small ranges of stress. Shearing occurs within a zone of finite thickness, which becomes thicker with time, increasing temperature, and increasing stress. The metal within this zone is composed of subgrains that rotate back and forth about an axis which is sometimes the octahedral axis of the parent crystal. Rupture, which is never intergranular, is preceded by a sudden increase in the rate of uniform extension in one of the crystals. The mechanism of grain-boundary gliding is described as a coordinated alternation of slip and recovery in a chain of subgrains along the grain boundary. This concept is employed to account both for the rate of primary creep and for the transition from primary to steady-state creep.

INTRODUCTION

Recent efforts to develop alloys of superior elevated-temperature creep resistance have heightened interest in the function of grain boundaries in creep, both because coarse-grained materials are found to exhibit greater creep resistance at high temperature and because high-temperature creep rupture is usually intergranular. The present research was undertaken in an effort to gain a more complete understanding of the behavior of grain boundaries in creep in the hope that such knowledge might prove useful in guiding the continuing search for better alloys. This paper summarizes the results of 4 years of work, a portion of which was reported in reference 1.

The amorphous-metal hypothesis, through its assumption that metal grains are joined by a layer of viscous cement, was responsible for what was probably the first observation of intergranular displacement. Rosenhain and Humfrey (ref. 2), seeking experimental support for this hypothesis, observed that iron, mildly deformed at a high temperature, yields predominantly along the gamma grain boundaries. More or less concurrently, Andrade (ref. 3) employed the amorphous-metal concept to account for steady-state creep, which he associated with viscous flow upon grain boundaries and operated slip planes. When the tungsten lamp filament was under development, it was found that the principal cause of sagging is high-temperature creep by grain-boundary flow, or "offsetting," as the effect was then known. Clark and White (ref. 4) first pointed out that fine-grained metals are generally less resistant to creep under low stress and high temperature than coarse-grained metals.

Through subsequent years grain-boundary flow has been observed in several more metals and alloys including lead, tin, aluminum, magnesium, and alloys involving these metals (refs. 5 to 21), but only recently has there been any intensive inquiry into the nature of the phenomenon itself (refs. 7 to 20). Upon the basis of results published in 1952 and 1953, it is apparent that grain-boundary motion is heterogeneous in several respects. First, it is spasmodic, proceeding in a succession of surges. Grant and coworkers (refs. 8 to 13) were the first to report this, but they interpreted their observation as meaning that the possibility of flow upon any one grain boundary becomes exhausted and that motion is resumed only upon a fresh boundary, having been produced by the migration of the original boundary. In the earlier report upon the present work (ref. 1), spasmodic gliding upon what appeared to be stationary grain boundaries was recorded. Further studies, described below, seem to bear out this interpretation.

The heterogeneous nature of grain-boundary displacement is felt also in the distribution of plastic deformation in the metal adjacent to the grain boundary. Betteridge and Franklin (ref. 7) were early in pointing out that grain-boundary shear in polycrystals is necessarily accompanied by compensating distortions in those grains upon which the ends of the grain boundaries impinge. Grant and coworkers (refs. 8 to 13) and McLean (ref. 16) remarked upon a localized intensification of distortion of all crystals adjacent to sliding grain boundaries. Puttick and King (ref. 15) went farther in postulating the development of corrugations in the grain boundary in such a way as to deter motion along it. They offered, in evidence, pictures of separated tin crystals having wavy grain surfaces that were exposed by parting the bicrystal subsequent to creep testing by the intergranular penetration of mercury. A more detailed discussion of deformation along the grain boundary was included in the earlier report upon the present research (ref. 1), wherein it was indicated that the distortion consists in an intensification of the block rotation, which is occurring to a lesser degree elsewhere within the extending crystals. Further verification of this interpretation is now available.

Despite such evidence of the heterogeneous nature of grain-boundary displacement, it appears to have been widely assumed that the process occurs as a shear upon an interface between the crystals. One of the present authors is already on record (ref. 1) to the contrary, however, and new evidence will be presented here to show that the motion which is called grain-boundary shear is, in fact, distributed through a layer of metal of substantial thickness.

As of now, the present investigators alone have reported an intensive study of the relationships between crystal orientation and grain-boundary motion. In the early studies (ref. 1) it was found that there is no relationship between grain orientation and the direction of motion at the grain boundary; this direction is determined by the direction of the maximum shearing stress parallel to the grain boundary, a conclusion which has since been verified by Grant and coworkers (refs. 8 to 13). The rate of the grain-boundary displacement is, however, critically sensitive to grain-orientation relationships. The original statement of the present authors to the effect that the rate increases with the degree of orientation mismatching (ref. 1) can now be particularized in the statement that the rate increases with the bending and twisting of the slip elements as they operate across the grain boundary.

With the exception of the results of Ke (refs. 22 to 24) and contrary to the prediction of Andrade, all published evidence to date indicates that the gliding rate decreases with time. Essentially identical time-displacement patterns have been reported by Puttick and King (ref. 15), Grant and coworkers (refs. 8 to 13), McLean (ref. 17), and Findley and Cochardt (ref. 1). McLean comments to the effect that Ke's results can apply only to an extremely early stage and as such represent a special case.

Unique to the present investigation is the finding of an induction period preceding motion upon the grain boundary and the recognition of two kinds of rates of gliding, one associated with the individual surges and one with the cumulative movement over long time intervals. The instantaneous motion varies, in its magnitude, linearly with the temperature, while the cumulative displacement conforms to the Arrhenius relationship with an activation energy of approximately 10,000 calories per mole. These observations have already been reported (ref. 1).

The new findings presented in the present paper are the product of the detailed observation of grain-boundary displacement and concurrent crystalline flow in nearly 100 pure aluminum bicrystals tested in creep over almost the full ranges of stress and temperature within which grain-boundary displacement could be made to occur. About half of the bicrystals were tested to fracture, some for times approaching 1 year. Both before and after testing most of the samples were further subjected to metallographic and X-ray crystallographic examination. From this comprehensive study it is now possible to describe grain-boundary gliding in considerably greater deatil than has been possible heretofore.

The present investigation was conducted at the Carnegie Institute of Technology under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics. Many persons have contributed to the planning, conduct, and interpretation of this research. Among those who have made especially important contributions to the planning and interpretation are Drs. A. W. Cochardt and H. W. Paxton. Those who have participated at one time or another in various capacities in the conduct of the work include also Messrs. T. W. Eichelberger, R. C. Schell, L. T. Lloyd, A. Platt, J. Rydzak, F. Bourriaud, G. Hook, J. Hohman, C. Vogley, E. G. Haney, F. DeMarco, E. Kalinowski, R. Rummel, A. Nemy, and K. Semlak. Special thanks are due to members of the staff of the Metals Research Laboratory who have aided by their counsel and particularly to Dr. G. T. Horne and Rev. R. W. Turner. The metal used and its detailed analysis were furnished by the Aluminum Company of America. The special cold-top process employed in making the reference grids upon the bicrystals was made available and the necessary materials were supplied through the kind cooperation of Mr. D. L. Goffredo of the American Newspaper Publishers Association Laboratories of Easton, Pennsylvania.

EXPERIMENTAL MATERIAL

Pure aluminum (99.95 percent pure) was used exclusively in this research, partly because of the existing background of knowledge of the creep behavior of this metal, but mainly because of the advantages that it offered in the ease of specimen preparation and the convenience of the temperature range in which the effects to be observed were known to occur. Spectrographic analyses made upon specimens ready for testing showed only a minor deterioration of the purity of the metal. The major impurity was copper, about 0.005 percent, followed by iron, chromium, silicon, and magnesium, each about 0.001 percent, and sodium and calcium, each 0.0001 percent. No other element was found in a spectrographically detectable amount.

EXPERIMENTAL METHOD

Most of the studies referred to in this paper were made upon bicrystals which were in the form of bars approximately $1\frac{1}{2}$ inches long, 3/8 inch wide, and 1/8 inch thick having at midlength a nearly straight and flat grain boundary perpendicular to the broad faces and crossing the breadth of the bar at a predetermined angle, usually 45° to the longitudinal axis (see fig. 1). All but a very few of these bars were cut from coarse-grained rolled aluminum plate which had been prepared by strain-annealing. The few others were cut from cast bicrystals that had been made from the same metal by a thermal-gradient technique. After etching and electropolishing to remove all strained metal, a pattern of square reference marks, 133 to the inch, was lightly photoengraved upon one of the broad faces of each bar (see fig. 2). The electropolishing left a transparent oxide coating which could be largely removed with a phosphoric acid solvent but which was usually left in place as protection against rapid tarnishing at the temperature of creep testing because nearly all tests were run in air.

A plan of the measurements made during the creep testing of these bars is shown schematically in figure 3. Shear parallel to the grain boundary was measured by the lateral displacement A of the two portions of a reference square which straddled the grain boundary. Similar measurements were made simultaneously at three or more positions along the grain boundary and the results were averaged. At the same time, the extension of each grain $\left(B_1 \text{ and } B_2\right)$ close to the grain boundary was measured over an initial gage length of 0.32 centimeter each. "Total extension" C was measured across the grain boundary over a gage length initially 0.7 centimeter long.

Prior to testing, the orientation of each crystal was determined. The bar was then suspended between file-faced grips in a vertical tube furnace open to the atmosphere. At the beginning of the test the furnace was first adjusted to the desired temperature and an initial set of measurements was taken with a measuring telescope focused through an optical glass window in the furnace. Then a weight adjusted to the cross section of the individual sample was carefully suspended from the lower grip. Immediately another set of readings was taken, followed by readings at increasing time intervals until the frequency became once daily, which frequency was maintained throughout the balance of the test. A summary of the productive tests is shown in table I. Whenever possible, after testing, the specimen was reexamined metallographically and by X-ray diffraction.

PRECISION OF MEASUREMENTS

When the precision of the measuring telescopes used and the magnitude of the human error in locating the reference points from day to day are considered, it is probably safe to assume that the measurements of grain-boundary displacement are accurate within 0.005 millimeter. In comparing the results among all tests, differences in grain-boundary displacement exceeding 0.010 millimeter are believed to exceed the total of the experimental error from all sources. The same lineal error applied to the short gage lengths used in measuring the extension within each grain and the total elongation astride the grain boundary amounts to 0.5 percent for the former and 0.2 percent for the latter.

Aside from periods of actual control failure, the temperature variation did not exceed ±5° C. Although thermal gradients of as much as 5° C were found within the length of individual specimens, it is unlikely that the temperature variation along the length of any one grain boundary exceeded 1° C. Testing temperatures were read by means of an auxiliary thermocouple placed close to the specimens at the grain boundary. Errors in this reading resulting from calibration drift are thought not to have exceeded 3° C.

The orientations of the two grains of each bicrystal were usually determined prior to testing by the Laue back-reflection method using Greninger charts. This method is capable of an accuracy approaching 1° of arc. Higher precision was attained by goniometer readings of etch-pit orientations, but the use of this method was employed in only a few instances.

Loads were calculated upon the basis of the initial dimensions of the samples and were made up in shot that was carried in a tared bucket. No good means is available for assessing the error in this procedure, but it is unlikely that it exceeded 1 percent. The variation in the unit load resulting from changes in the dimensions of the samples at the grain boundary could not have much exceeded this figure because the necking of the samples, prior to fracture, never was observed to extend to the region of the grain boundary.

EXPERIMENTAL RESULTS

The experimental results, which are too voluminous for presentation in full, are here summarized in terms of the relationships that they exhibit. In order to make the presentation easier to follow, it is introduced by a brief statement of the grosser aspects of grain-boundary gliding.

Although the application of a small stress to an aluminum bicrystal held at an elevated temperature results in an immediate and continuing overall extension, the lateral displacement of one grain with respect to the other does not begin until the expiration of a more or less extended induction period. Then an apparent shear parallel to the grain boundary begins at its maximum velocity and decelerates until there is again no more perceptible motion over an extended period of time (see fig. 4). Later there is another surge of displacement of one grain with respect to the other, another rest period, and so on in a series of cycles. The magnitude and frequency of the cycles decrease somewhat with the passage of time and increase with rising temperature and stress. While deviations from this kind of behavior are common and will be described presently, a clearly defined cyclic grain-boundary displacement appears to be the basic pattern of the process.

At the lowest temperature at which the effect is observed, shear along the grain boundary has the outward appearance of being sharp. As the temperature rises, however, it seems less and less that there is an interface of shear and more and more as though the shear is occurring inhomogeneously within a grain-boundary layer of very substantial thickness. Below a certain temperature, which decreases with increasing stress, grain-boundary gliding fails to develop at all, while in the range of high temperature and stress the effect so merges into the gross general distortion of the grains themselves as to lose its individuality. A comparison of the present findings with the results of published studies relating to grain-boundary displacement in polycrystalline aluminum suggests that the effect is observable over somewhat broader ranges of temperature and stress in polycrystalline material.

Lateral motion at the grain boundary remains parallel to the reference surface so long as the grain boundary is perpendicular to the reference surface. This is true regardless of the crystallographic orientations present in the bicrystal. The rate of grain-boundary gliding, however, is highly sensitive to the orientation relationships, their initial influence persisting through the life of the creep test, even though the crystals themselves may suffer considerable elongation and apparent distortion. In none of the present studies did grain-boundary displacement proceed to failure along the grain boundary. Rupture, when it occurred, was always transcrystalline and never close to the grain boundary.

Induction Period

The induction period varies in its length over a broad range, depending upon the crystallographic orientation relationships, the temperature, and the stress. Short induction periods, amounting to as little as a matter of minutes, are associated with large orientation differences between the crystals, high temperature, and high stress. At the opposite end of the scale, periods as long as a month (in one case 5 months) preceding grain-boundary gliding are encountered. The onset of movement at the end of the induction period is always abrupt and seems in no way related to any external mechanical or thermal disturbance. Because of the vigor with which motion always proceeded at the end of the induction interval, it seems that the threshold below which there is no motion is in effect the temperature and load limit below which the induction period becomes longer than the patience of the experimenter.

In a series of studies conducted later than those discussed in the present paper it was found that the induction period can be observed only if the measurements are made in a direction other than that of the applied stress. If measurements equivalent to those at points A in figure 3 are made vertically instead of horizontally, an uninterrupted extension is found, its rate being somewhat larger than that measured over the lengths B_1, B_2, and C.

Cyclic Motion

Cyclic motion in many cases became indistinct after the first one or two surges. Two rather closely associated circumstances were identified as causes. One of these related to the fact that motion was not always equal or concurrent at all positions along the grain boundary, so that the averaging of displacement readings taken simultaneously at three positions tended to smear out cycles which were locally distinct (see fig. 5). Such irregularities were more noticeable in samples tested at the highest temperature and stresses. In the most extreme case, a bicrystal under test at 800 psi exhibited extensive displacement along half of its grain boundary while no displacement at all had occurred along the other half.

The other cause of the obscurement of cyclic motion is associated with the broadening of the zone within which the grain-boundary motion occurs and with the fine-scale inhomogeneity of the movements within this zone. When the active zone so increases as to engulf the reference points, the readings become erratic, often to such a degree as to conceal all but the coarsest features of the grain-boundary displacement (see fig. 6). Under these circumstances it is common for the measurements to report fictitious negative displacement from time to time. This occurs because the reference points are being rotated back and forth (almost as though tossed about upon a choppy sea) by the large and complex movements within the region of rapid plastic motion. Some idea of the magnitude of this effect can be obtained by a careful examination of photomicrographs of the reference patterns in the affected areas recorded after creep testing (see fig. 7). Once erratic readings had begun, they continued for the balance of the test.

A rather surprising characteristic of spasmodic displacement was revealed by a series of temperature control failures, by reason of which the specimen temperature was permitted suddenly to increase several hundred degrees for a brief time. When this occurred during the rest period the grain boundary did not become active but remained at rest for the normal time, resuming gliding days or weeks later as though the temperature had remained constant. If, on the other hand, motion was in progress, or was about to start, the extent of displacement during the period of overheating was usually very large indeed, as shown in figure 8. In this illustration it is shown that a brief rise of 100° C during the progress of gliding increased the displacement in that cycle, but when the temperature was adjusted upward 50° C during a subsequent rest period the state of rest continued.

It has been found from later studies that cyclic motion, as well as the previously discussed induction period, can be observed only if the measurements are made in a direction other than that of the applied stress. As mentioned before, if measurements equivalent to those at points A in figure 3 are made vertically instead of horizontally, an uninterrupted extension is found, its rate being somewhat larger than that measured over the lengths B_1, B_2, and C.

Influence of Time on Rate of Grain-Boundary Displacement

That the overall rate of grain-boundary displacement decreases with the time may be seen by a casual inspection of any of the time-displacement curves, such as that of figure 4. Such curves become approximately straight lines when the displacement is plotted as a function of the cube root of the time. Each curve, however, has a different slope, the value of which depends upon the specific orientation relationship existing between the pair of crystals concerned. If a large number of such curves all pertaining to a single temperature and stress are averaged, there is obtained a nearly smooth straight line, the slope of which approximates the average for all possible combinations of crystal orientation (see fig. 9). Irregularities in the curves arising from spasmodic gliding are largely smoothed by averaging because the cycles are of somewhat diverse duration in different samples. An impression of the spread in the slope of the curves included in the average is given by the breadth of the shaded areas in figure 9.

The instantaneous rate of motion during the active portion of each cycle of gliding is, of course, much greater than the average gross rate referred to above. Puttick and King (ref. 15) suggested that the initial displacement in tin bicrystals is not only very rapid but also linear with the time. The circumstances of the present studies make it difficult to decide whether the initial rate is, in fact, linear, because the present method of measurement is too cumbersome to yield a sufficiently rapid succession of early readings. The measurements usually show that a major portion of the displacement has occurred between the first two readings and that gliding then gradually dies out in a matter of days or hours, depending upon whether the temperature is low or high.

Influence of Temperature on Rate of Grain-Boundary Displacement

The influence of temperature upon the overall rate of displacement may, in principle, be isolated experimentally either by fixing the crystallographic orientation or by employing an average of measurements in which the orientation effect is well randomized. The latter procedure is best adapted to the present results. Using average displacement readings taken from graphs such as those of figure 9, temperature-displacement curves of the type of that given in figure 10 are obtained. The curve in figure 10 has the form that is typical of chemical-rate processes and its slope yields a heat of activation of 11,000 ± 1,000 calories per mole. This value is slightly higher than that previously reported (ref. 1), though perhaps not significantly so.

It was reported previously (ref. 1) that the extent of the grain-boundary displacement in the first surge of motion is a linear function of the temperature (see fig. 11). This result was obtained by the direct comparison of a series of samples of similar crystallographic orientation so selected as to yield a high gliding rate. When the corresponding findings from all samples that were tested are averaged irrespective of orientation, the result is less sharp but still indicates a linear relationship between the step height and the temperature, at least for the first cycle of activity (see table II). Because of the smearing out of subsequent cycles at the higher temperatures, it is not possible to extend this analysis beyond the first cycle. It is apparent from table II, however, that the displacement in each surge decreases progressively with the time.

Influence of Stress on Rate of Grain-Boundary Displacement

The influence of the stress upon the rate of displacement is again isolated by the method of averaging out the orientation effects. When, for a testing temperature of 300° C, the logarithm of the average time required to reach a displacement of 25 microns is plotted as a function of the stress, the curve reproduced in figure 12 is obtained. While this curve might be considered straight over modest ranges of the stress (i.e., 100 to 400 psi) the curve as a whole is distinctly not linear. Neither is a linear curve obtained by plotting the stress directly as a function of the time for unit displacement.

Qualitatively, it appears that the stress relationship is subject to limits in both directions. Below a certain stress (somewhere between 10 and 50 psi) no displacement was found within an experimentally feasible time, even at temperatures approaching that of melting, while at high stress values (e.g., 1,600 psi) grain-boundary displacement failed to develop before the bicrystals necked down and ruptured.

Influence of Relative Crystal Orientations on Rate of Grain-Boundary Displacement

The influence of the relative crystal orientations upon the cumulative rate of displacement is both complex in form and large in magnitude. Many attempts to relate the crystal orientation and the rate of gliding were made without finding more than vague trends before the scheme illustrated in figure 13 was found to yield a loosely quantitative relationship that holds equally at all temperatures at which grain-boundary gliding was measured. To apply this relationship it is necessary first to identify the operative slip planes in both crystals, either by calculation or by direct observation, and also the operative slip direction in each family of active slip planes. The angle between the operative slip directions of the two grains is designated θ, and that between the operative slip planes, as measured in the plane of the grain boundary, is ω. Empirically, the relationship between the displacement rate D, the average displacement rate Da (as read from the graphs in fig. 9), and the angles θ and ω is

$$D \approx Da \frac{(\theta + \omega)}{c}$$

where the angular constant c is found to have a value in the neighborhood of 65. About half of the measured rates fall within 5 percent of the values so estimated; very much larger deviations are sometimes encountered, probably because of the operation of more than one slip system in one or both of the crystals. Bicrystals having a twin relationship never exhibited any displacement upon the boundary, but it should be noted that none were tested with the boundary lying at other than 45° to the tension axis.

A schematic representation of the relationship between the displacement rate and the two angles (θ and ω) is presented in figure 14. An impression of the spread of real data points from a regular succession, when plotted in the same manner, may be had from figure 15, which is fairly typical of experimental results.

Similar plots (figs. 16 and 17) corresponding to relatively short testing intervals show in addition that the duration of the induction period tends to vary inversely with the sum of the angles θ and ω. In these graphs, points plotted as dots in the zero-displacement plane represent the states of samples that have not yet begun to glide; the difference in the number of points so denoted in figures 16 and 17 corresponds to the number of samples having induction intervals of between 300 and 450 hours' duration under the stated conditions of testing. In this example the $\theta + \omega$ angles fall in the range $50^\circ \pm 10^\circ$; the samples with larger angles for the most part exhibited shorter induction periods, while those of smaller angle mostly had longer induction intervals.

Further, it is to be noted that the magnitude of the displacement in the first complete cycle of gliding is related qualitatively to the crystallographic orientation in the same manner as is the overall gliding rate. There are in this instance fewer data available for comparison, but, insofar as these permit the testing of the relationship, it appears that the correspondence is about equal to that cited for the case of the overall displacement rate.

Microstructural Observations

Microstructural observations made chiefly subsequent to the completion of creep testing relate mainly to two conditions: (1) The breadth of the active zone and (2) the heterogeneity of movement throughout this and adjacent zones. At the lowest temperature at which gliding is observed, the path of shear remains narrow and apparently sharp (fig. 18). That this zone has finite width, however, is readily seen by comparison with a corresponding photomicrograph (fig. 19) of a similar sample tested at a higher temperature. Much the same effect is produced by increasing the stress (fig. 20). The width of the active zone is not a constant at any fixed values of temperature and stress but varies somewhat from sample to sample, being the narrowest where the gliding rate is highest and the broadest where the rate is least; moreover, the width always increases with the progress of the test.

Further evidence of the finite thickness of the shear layer is given by examination of the contour of the edges of the samples, where the motion is not parallel to the external surface (fig. 21). In figure 21 the uppermost diagonal trace reveals grain-boundary displacement, and those below are gross slip, which occurred simultaneously and in a nearly parallel direction. It is clearly apparent that slip produces a relatively sharp step at the edge surface, while grain-boundary gliding has resulted in the formation of a rounded terrace. Moreover, if the distribution of the original oxide skin upon the disrupted edge surface is examined, it is found that none of this oxide is retained upon the surfaces of steps exposed by slip, whereas a substantial quantity of oxide is distributed across the terrace produced by grain-boundary motion, the distribution being uniform, though sparse, in a band corresponding in thickness and location to the darkened grain-boundary trace upon the broad face of the sample.

Grain-boundary migration of the kind found by Grant and others in polycrystalline aluminum has not been observed in any of the aluminum bicrystals with straight grain boundaries. Occasionally, shearing was upon other than the originally etched grain boundary (e.g., fig. 22), but, once a path of shearing was established, motion continued upon this path throughout the balance of the test. In other words, the original grain boundary of figure 22 must have migrated to a new position in immediate response to the establishment of the temperature and stress of testing and prior to any displacement along the boundary. Occurrences such as this were relatively uncommon.

The heterogeneity of the motions that result in grain-boundary displacement are perhaps most vividly described by the use of an analogy, which, because of its obvious limitations, is tendered with diffidence. The observer of the motion is reminded of fluid flow in which the linear flow that is occurring throughout both crystals gives way to turbulent flow in the immediate neighborhood of the grain boundary. The transition, though not following any smooth line of demarkation, occurs more or less abruptly, and the degree of turbulence appears fairly uniform within the active zone. An impression of the violent rotations that occur within this zone can be had from figure 19, where a portion of a reference square in the upper left of the picture has been rotated through an angle of 45°; evidently this square is upon a relatively undistorted fragment of metal that was caught in the middle of the stream of turbulence. Other occurrences of this kind were seen. Ordinarily, however, the reference marks are so fragmented and dispersed in this region that they can no longer be identified. One of the most usual reasons for terminating the tests short of fracture was the obliteration of the reference marks by this action.

If, after creep testing, the bicrystal is once more electropolished and examined, the resulting appearance of the reference surface may be as in figure 23. A dominant feature of this structure is the presence of a uniform distribution of subgrains, recently remarked upon by Hunter and Robinson (ref. 25) and others. The grain boundary is preserved with remarkable sharpness although its location sometimes deviates in detail from that of the etching trace produced before creep testing, as has been noted above. The two crystal faces, now lying at different levels, are connected by a sloping section adjacent to the boundary, which, though apparently crystallographically continuous with the lower crystal, exhibits an obvious difference in the texture of its subgrains. A rather similar structure has been described by Gifkins (ref. 19) as occurring in lead-thallium alloys. This detail of the microstructure correlates with the findings of the X-ray diffraction studies.

Outside the zone of "turbulence" the plastic motion is by no means uniform. This is shown clearly in figure 7 where adjacent reference squares outside the most active zone are elongated and distorted in strikingly different ways and amounts. These effects are most pronounced during the late portion of tests in which the crystals have exhibited large extensions. Metallographic examination tells little of how these heterogeneous distortions progress with time, but displacement readings made upon reference points that are within this region of high (but not turbulent) activity show clearly that the reference squares squirm this way and that in an erratic manner. At high temperature, or stress, the region of obviously heterogeneous motion eventually extends all across both crystals; greatly elongated reference squares are then seen to occur in bands which seem to correspond to the kink bands observed by Ramsey (ref. 26) and others in aluminum during creep.

X-Ray Diffraction Examination

The X-ray diffraction examination supports the metallographic observations in all respects. After testing, both crystals were always found to have been reduced to fragments, which had rotated a few degrees out of the original orientation of the parent crystal in a manner reported by virtually all recent investigators of the creep process (see fig. 24(a)). In the initial publication on this research (ref. 1), the axis of rotation was identified as an octahedral direction in the parent crystal. This type of relationship has recently been confirmed and described in further detail by Gervais, Norton, and Grant (ref. 27) as one of two mechanisms of kink-band formation in aluminum; the other mechanism is a rotation about a [211] axis.

Within the zone of turbulence along the grain boundary the rotations become so large that the Laue pattern often loses all trace of kinship with the parent crystal (fig. 24(b)). This effect is most readily displayed by a Laue photogram made with a microbeam directed upon the terrace at the end of an operated grain boundary, such as that in figure 21. If a similar photogram is made with the beam directed upon one of the steps exposed by slip, no greater disorganization of the parent crystal is registered than that shown in figure 24(a). Thus, it is apparent that the structural changes accompanying grain-boundary displacement are distinctly greater in magnitude than those accompanying slip-band formation. It is further apparent that the microstructure displayed in figure 23 may not be interpreted as a return to anything resembling the predeformed states of the two grains of the bicrystal; substantial orientation divergence must exist among the subgrains, even though this has not destroyed the apparent sharpness of the grain boundary.

Concurrent Mechanical Effects

Concurrent mechanical effects observed in the course of the present studies concern the mode of elongation of the individual grains and the manner of ultimate rupture. For the most part, the stress levels employed were so low that the lineal extensions of the individual crystals were barely perceptible through much of the course of each test. In nearly all cases a rapid initial rate of extension ceased before more than 1 or 2 percent of extension had occurred. There followed, then, a long period during which the extension was extremely small, its rate being approximately constant. Occasionally, the rate of extension of one of the crystals abruptly became large, though it was usually constant at the higher rate; this event always preceded rupture in that crystal by from 500 to 1,000 hours (see fig. 4). Failure always occurred in the rapidly extending crystal at a considerable distance from the grain boundary; failure was usually midway between the grain boundary and the specimen grip and occasionally very close to the grip but never close to the grain boundary.

Such behavior is contrary to that of polycrystalline aluminum which is generally found to rupture intergranularly. In the bicrystals the grain boundary appeared to act as a stiffener, preventing necking from occurring in its vicinity. Perhaps the most remarkable feature of these observations is that the accelerated extension rate foretelling rupture was felt over a short gage length (0.32 centimeter) adjacent to the grain boundary and rarely less than 2 centimeters from the site of visible necking and ultimate rupture.

It is worthy of note that there was never any suggestion of a time correlation between the onset of surges of grain-boundary gliding and the rate of extension of either crystal, or of the rate of total extension. Neither was there any evidence that the rate of grain-boundary motion was changed by an alteration in the rate of crystal elongation. Only when both rates were changed by a change in the testing temperature were they altered simultaneously.

Comparison of Behavior of Cast and Recrystallized Grain Boundaries

The behavior of cast versus recrystallized grain boundaries was compared by the additional testing of a short series of bicrystals made by controlled progressive freezing. These samples were geometrically equivalent to the strain-annealed bicrystals in all respects. When similarly tested they exhibited all of the behavior characteristics of the recrystallized bicrystals with the single exception that the rates of both grain-boundary displacement and crystal extension were only about one-third those of comparable recrystallized bicrystals.

With the cast bicrystals, it was possible to produce several test bars from the same casting, thus affording an opportunity to investigate the reproducibility of test results. The outcome of a comparison among three identical cast bicrystal samples tested similarly, but in different equipment and at different times, is presented in figure 25. While the time-displacement curves for the three samples do not superimpose, they have very similar form and the overall rate derived from any one of them would be within the range of experimental error considered probable for these studies. The fact that the cycles of activity do not coincide exactly in the early stages of the tests seems less significant when it is observed that, later, the three samples settle down to almost coincident cycles of activity of very similar magnitude.

DISCUSSION OF RESULTS

The foregoing findings demonstrate, beyond reasonable doubt, that grain-boundary gliding is not a viscous process, nor is it even quasi-viscous. Neither of the major characteristics of viscous behavior, which are constancy of the rate and direct proportionality of the rate to the stress, is exhibited in grain-boundary motion. Here, by "rate" is meant the instantaneous slope of the average time-displacement curve, this average including a random distribution in bicrystal orientations. Once again the amorphous-metal hypothesis is found wanting and reasons for the existence of an apparently uniform creep rate must be sought elsewhere than in the postulated viscous flow upon grain boundaries.

The decaying rate of grain-boundary displacement stands also as an argument against grain-boundary "migration" being an essential part of the displacement process. If, as Grant and coworkers have suggested, the capacity of a grain boundary to support flow becomes permanently exhausted, then the reported migration of the grain boundary to fresh sites should tend to maintain a constant average rate of gliding. From the fact that this did not occur in the bicrystals of the present investigation, it may be inferred both that the grain boundary continues to occupy the same path and that there is a permanent change in the crystalline matter along that path, a change which is capable of decreasing the rate of flow but not of stopping it, except temporarily. Both metallographic and X-ray diffraction demonstrations pointing to the same conclusion have already been cited; gliding appeared always to continue upon the same path, which, with the passage of time, became broader and more highly fragmented and disoriented. The sharp appearance of the grain-boundary trace upon reetching after testing probably comes from the fact that the rotated subcrystal fragments from each grain have a common axis of rotation.

With gliding continuing upon a single path, it becomes important to consider what processes may be responsible for the alternation of periods of rest and motion. There has been established a qualitative correlation between the extent of the induction period and the crystal orientation, the initial delay being the longer the smaller the angles of mismatching between the conjugate grains. Since this relationship becomes most clearly apparent when the orientation difference is viewed in terms of differences in the orientations of the respective slip planes and slip directions in the two crystals, it may be inferred that the onset of gliding is somehow related to the development of crystallographic disturbances at points where crystallographic slip impinges upon, or crosses, the grain boundary. The more violent the disturbance the briefer the induction or rest period. It is worth recalling in this connection that the original initiation of grain-boundary motion is always preceded by a substantial degree of general extension of the specimen, as measured over short gage lengths within both crystals and across the grain boundary; such extension is almost certainly effected by slip, even though visible slip lines may not have developed (refs. 28 to 33).

Thus, it appears that a bending of the crystalline matter immediately adjacent to the grain boundary is probably prerequisite to gliding, although it does not necessarily follow that grain-boundary shear occurs within deformed metal. Indeed, reason directs that quite the opposite must be true, because the very fact of gliding suggests localized yielding within a band of metal that is softer than the metal deeper within the bodies of the conjugate grains. It seems possible, therefore, that the development of a disturbed zone along the grain boundary is itself prerequisite to localized softening by some kind of recovery process, such as polygonization. Support for this hypothesis is to be derived not merely from the X-ray evidence but most particularly from the suddenness with which motion begins. Were yielding to occur preferentially within a zone of disturbed metal, it should be expected that gliding would begin gradually as the disturbance was built up.

Let it be supposed, accordingly, that yielding occurs by slip within the softened grain-boundary band and in a direction as closely approximating parallelism with the grain boundary as the subgrain orientation relationships will allow, this being the only direction in which slip could proceed very far without encountering resistance from the less soft metal outside the active zone. This action would, of course, result in a gross shear nearly parallel with the grain boundary. It will be noted, however, that the amount of gliding that occurs in any one cycle is obviously many times more than enough to work-harden the grain-boundary zone to equality with the hardness of the bodies of the crystals. This may mean that the total displacement results from a rapid succession of alternations of slip and recovery in the grain-boundary zone. Such alternations would have to be well coordinated, with slip occurring simultaneously all along the active zone and then recovery occurring everywhere at nearly the same instant.

There is no difficulty in visualizing a coordinated beginning of the process just described, because, at the outset, the bending of the crystals should be uniform all along the grain boundary and recovery should occur everywhere at about the same instant. It is not plausible, however, that the alternations of slip and recovery should remain in phase through a very large number of repetitions, where the slip must be transmitted through a chain of subgrains that inevitably differ somewhat in their orientations from place to place along the grain boundary. As some of the regions get out of phase with the others they should tend to block gliding, and when a major part of the metal in the active zone is no longer yielding in phase there ceases to be any reason to expect the average extension to be parallel to the grain boundary; gliding should cease to be apparent and the deformation within the subgrains along the grain boundary should become very similar to that everywhere else with each crystal, which is to say that their resultant strain should be altogether in the direction of the applied stress.

This provides a reason not only for the subsidence of grain-boundary flow into a state of rest but also for the erratic displacement readings commonly obtained as gliding slackens and ceases, for the evidence of turbulence provided by the metallographic and X-ray diffraction studies, and for the fact that extension in the direction of the applied stress continues in the grain-boundary zone during the rest period. With slip occurring locally in a sequence of short bursts, each time in a reformed subgrain of slightly different orientation, the local slip direction may change drastically from time to time, with the result that a surface reference point will describe a far from regular path of motion and the grain-boundary zone will appear as though in a turbulent state.

At the termination of a cycle of motion the bicrystal must have returned to much the same state as that in which it existed before grain-boundary gliding had occurred, except for the existence of a narrow zone along the grain boundary where the subgrains are rather more disoriented than elsewhere. Transgranular slip or its equivalent may again predominate and, once more, bending of the crystalline matter may develop more or less uniformly all along the grain boundary where slip impinges or crosses. If this leads to simultaneous recovery all along the grain boundary, a new wave of coordinated slip and recovery, that is, a new cycle of gliding, will follow. The main difference that is expected, in this second event, is that the newly activated zone should be somewhat thicker than the original one, owing to interference from the disoriented zone already present, and out-of-phase behavior should therefore develop sooner, thus decreasing the extent of the displacement in each successive cycle. The time between cycles (i.e., the length of the rest periods) should also increase progressively, because more slip will be necessary each time to condition the ever thickening active zone for recovery.

Lest it be inferred that the authors are claiming originality for the idea of alternating slip and recovery, as such, it should perhaps be pointed out that this concept was put forward by Bailey (ref. 34) in 1928 and again in a form closely approximating the present proposal by Cottrell and Aytekin (ref. 35) in 1950.

Although the nature of the mechanism just described is such as to discourage attempts to express it in mathematical terms, its basic concept is relatively simple, namely, that plastic motion, which is sustained in a unit of crystalline matter by an alternation of slip and recovery, becomes equivalent to a gross shear whenever a number of units yield in unison in a physically continuous chain; the role of the grain boundary is simply that of a nucleator of such a chain. This mechanism merits credence to the extent that it accommodates the characteristics of grain-boundary flow that have been observed experimentally, to wit: The direction of flow is insensitive to the crystal orientation, while the rate of flow is responsive to orientation; an induction period, which is orientation sensitive, precedes gliding; the displacement begins at its maximum rate and dies out altogether after a little time; the subgrains are more disoriented along the grain boundary than elsewhere; the identity of the grain boundary is preserved; no gliding occurs at temperatures below that at which recovery may take place; momentary temperature changes fail to interrupt the rest period but do affect the amount of the displacement if they occur while active gliding is in progress. The rate characteristics require more detailed consideration.

The overall rate of grain-boundary gliding is determined by the amount of the displacement in each cycle and the frequency of the cycles. If it be true that the active part of the cycle represents the occurrence, within a brief time interval, of a rather definite number of alternations of slip and recovery prior to the development of blocking by out-of-phase regions, the displacement achieved during this interval must be mainly a matter of how much slip can occur before the conditions requisite to recovery are developed. Whether this should be a linear function of the temperature, as the experiments seem to indicate, would appear to be much the same question as whether there is a linear increase with the temperature in the amount of plastic deformation, by rolling at high temperature, that is required to induce recrystallization in a piece of aluminum. The authors have been unable to find a clear answer to this question in the literature, but such sketchy indications as have been found at least do not deny the possibility that such a relationship exists.

The cycle frequency is presumed to be the inverse of the time required to accumulate enough crystal bending at the grain boundary to induce recovery. As a manifestation of polygonization, recovery involves an activation energy and the amount of stored energy of crystal bending required to excite recovery should be associated with the temperature in accordance with the Arrhenius relationship. Because the effect of temperature upon the extent of slip prior to recovery may be expected to influence the overall rate of grain-boundary gliding in opposite ways during the active and during the rest period (i.e., higher temperature increases gliding linearly during the active period, while it may be expected to delay the termination of the rest period correspondingly by requiring that more general extension of the metal occur prior to the reestablishment of coordinated yielding along the grain boundary) it seems that the effect of temperature upon the extent of slip should not influence the shape of the overall rate curve. This would leave recovery as the rate-controlling process. In this connection it is interesting to note that Fazan, Sherby, and Dorn (ref. 21), using measurements from the present studies, have calculated an activation energy for grain-boundary flow which is similar to the activation energy of self-diffusion in aluminum.

It is difficult to justify the cube-root law for the isothermal rate of gliding except qualitatively. As has been pointed out, the thickness of the active zone along the grain boundary increases progressively. This is expected to decrease both the displacement in successive cycles and the frequency of the cycles. Thus, the overall rate must diminish with time.

Total Process of Creep

A relationship with the total process of creep is now apparent, although its nature is not at all of the kind that had been expected. The initial proposition that grain-boundary flow has a linear rate and is responsible for the linear rate of secondary creep is denied by the decaying rate of grain-boundary gliding. There does remain a clear link with secondary creep, however, in the subgrain rotation that accompanies both processes and the apparent heterogeneity of the localized rotations and extensions.

Surprisingly, the direct association of grain-boundary flow seems to be with primary creep. The rates of both processes are constant with the cube root of the time; the variation in the rate is an Arrhenius type function of the temperature; and, within limited ranges of stress, the logarithm of the rate is nearly a linear function of the stress. Such extensive conformity can scarcely be coincidental. An obvious interpretation is that primary creep at elevated temperatures represents a stage during which gliding, similar in some respects, at least, to that at the grain boundary, is occurring along isolated zones scattered through the crystals. McLean, (ref. 17), among others, has described a process by which kink-band formation within the grains can lead to a region of local softening by polygonization, and Hino, Shewman, and Beck (ref. 36) have shown that the subgrain structure that develops during the early stages of creep occurs in bands of greatly differing subgrain size that cross the grains at intervals. The essence of the present proposal is that these zones should be isolated and should operate individually as coordinated chains, so that the same factor that is presumed responsible for the decaying rate of grain-boundary flow, namely, the lateral growth of the active zone coupled with increasing subgrain rotation, may operate to control the decay of the rate of primary creep. It seems necessary to postulate, also, that the number of the active zones is established at the outset and that these increase only in volume and not in number; otherwise the rate of flow would be modified by the rate of appearance of new active zones. This view seems consistent with the findings of Wood and Scrutton (ref. 37) to the effect that the disorientation of the subgrains gradually becomes detectable by X-ray diffraction methods during the primary stage of creep.

When the active zones finally impinge upon one another, their growth must cease and the rate of extension due to alternating slip and recovery should remain constant so long as these active zones retain their identity. This is secondary creep. Some indication that secondary creep does involve an integration of many localized cyclic-yielding operations, such as this proposal demands, is to be derived from observations reported by Chang and Grant (ref. 13). It is interesting to note also that Gervais, Norton, and Grant (ref. 27) remarked that kink bands do not grow in width (presumably during secondary creep).

Passing now to third-stage creep (accelerating creep) and rupture, the present experiments offer three new observations of possible significance. First, the grain boundaries are not inherently weak in pure aluminum and rupture does not occur along them unless forced to do so; Chang and Grant (ref. 11) described a process of polycrystalline rupture in which stresses concentrated at grain-boundary "triple points" nucleate rupture which then spreads as a crack from triple point to triple point along the most direct path available, which usually happens to approximate the grain boundary. Second, the increasing rate of tertiary creep is felt all across the crystal and not merely in the locality where necking is incipient; necking appears to be a consequence rather than a cause of accelerated creep. Third, in single crystals accelerating creep is not continuously accelerating but is rather a new constant rate that is considerably higher than that of secondary creep. It seems reasonable to assume that the progressive acceleration that is characteristic of third-stage creep in polycrystalline metal is a consequence of this stage being attained at different times in different grains.

Metallographically, the main feature that becomes apparent with the onset of third-stage creep, besides heavy slip bands, is the development of long surface ridges upon which the reference squares are much more elongated than in the intervening valleys (see figs. 7 and 26). This suggests that, somehow, the same sort of rapid chain yielding that occurs along grain boundaries develops in extended zones across the grains themselves. Just why this should happen abruptly and concurrently all across a grain is not presently apparent, but this does suggest that the essential difference between secondary and tertiary creep lies in the degree of organization of the unit operations of alternating slip and recovery.

These proposals are best summed up by reference to typical creep curves shown schematically in figure 27. At a temperature below that at which recovery may occur, the alternation of slip and recovery is not available, so that creep must proceed by slip alone. The rate will diminish as the metal work-hardens. If the stress is of the order of a quarter of the ultimate tensile stress, or larger, extension may persist until necking can occur and rupture follows a brief period of localized accelerating extension.

At higher temperature the process begins in the same manner, but, in a moment, scattered zones of kinking begin to polygonize and the recovery effected thereby prevents the rate of extension from declining as rapidly as it otherwise would. Presently, cyclic slip in polygonized regions becomes the dominant mechanism of extension and the rate declines linearly with the cube root of the time. When the activated zones impinge, the rate ceases further to decrease and is constant over a period of time until long continuous bands of kinking develop, whereupon coordinated slip-recovery yielding establishes a new and much higher rate of extension.

Meanwhile, and also much earlier, grain-boundary shear has been in progress. This ultimately develops, at three- and four-grain junctions, local tensile-stress concentrations that are so high that cracks develop and spread to produce failure.

Development of Alloys With High-Temperature Creep Resistance

Since the ultimate objective of this research is the practical one of pointing out the most promising avenues toward the development of alloys of superior high-temperature creep resistance, it is appropriate to consider what bearing the foregoing findings and interpretations have upon this subject. It seems to the present writers that, to be effective, any measure to increase the high-temperature creep resistance must accomplish one or more of the following aims: (1) Reduce the rate of secondary creep, (2) delay the onset of tertiary creep, and (3) minimize coordinated slip-recovery yielding both at grain boundaries and elsewhere within the metal.

Solid-solution-forming alloy additions should, in general, increase both the resistance to plastic flow and the recovery temperature. Thus, the most specific effect of solid-solution alloying is expected to be the elevation of that temperature minimum below which no slip-recovery creep can occur at a specified stress level. Even above this temperature, however, all solid-solution additions which raise the recrystallization temperature should decrease somewhat the rate of slip-recovery yielding in all of its aspects; the uniform creep rate should be lower, grain-boundary gliding should be slower, and tertiary creep should be both slower and of later occurrence.

The effect of an addition which produces a new phase in the alloy may be expected to vary widely depending upon the mechanical properties of the new phase, its relative volume, and the manner of its distribution in the alloy structure. Thus, a fully random distribution of small platelike hard particles may be expected to prevent (or greatly delay) the occurrence of long kink bands and thereby the onset of tertiary creep. However, it is conceivable that platelike particles laid down along a plane in the crystal, as precipitate particles, sometimes develop upon a slip plane, and these could have the opposite effect by assisting the coordination of slip-recovery yielding along that plane. It seems unlikely that hard particles situated upon a grain boundary would accelerate gliding, but it is altogether possible that they would have no significant effect. The essential contribution of the grain boundary is seen to be that of providing a physically continuous path for coordinated slip-recovery yielding; second-phase particles are expected to interfere only if they make the path of flow so devious that the effective shear in each cycle of gliding is reduced. These few samples may serve to illustrate the manner in which the principles that have been proposed might be applied to the prediction, or interpretation, of alloying effects.

It is apparent also that the arrangement of the crystals in a piece of metal should have an effect upon the occurrence and extent of grain-boundary gliding. Thus, preferred orientations that minimize the orientation difference from grain to neighboring grain may be expected both to reduce the contribution of grain-boundary flow to the overall extension and to increase the time to rupture. There seem to be scant grounds for hope that preferred orientations would greatly affect the rates of either secondary or tertiary creep, although modest effects are perhaps to be anticipated.

CONCLUSIONS

As a result of creep studies conducted upon aluminum bicrystals in the temperature range of 200° to 650° C and the stress range of 10 to 1,600 psi the following major characteristics of grain-boundary gliding have been established:

1. Gliding upon the grain boundary is spasmodic and begins with an induction period.

2. The direction of gliding is determined by the direction of the maximum shear stress parallel to the grain boundary and is independent of crystal orientation.

3. The velocity of gliding increases with the angular difference θ between the operative slip directions in the conjugate crystals and in addition with the angular difference ω between the active slip planes as measured between their traces upon the grain boundary

$$\text{Displacement} \approx d_a \frac{(\theta + \omega)}{65}$$

where d_a is the average displacement for all orientations under stated conditions of temperature and stress.

4. The average displacement for all orientations is constant with the cube root of the time.

5. The logarithm of the rate of gliding is a linear function of the reciprocal of the absolute temperature, the slope giving an activation energy of 11,000 ± 1,000 calories per mole.

6. Considering the detail of spasmodic gliding, the displacement in one cycle is a linear function of the temperature prevailing during the period of activity and the length of the rest period between cycles decreases with rising temperature.

7. For small stress ranges, the logarithm of the time for unit gliding is almost proportional to the stress, but no simple means has been found to relate the displacement and the stress over large ranges.

8. Gliding continues indefinitely in a sequence of cycles upon the same path, which becomes broader with time and broadens the more rapidly the higher the temperature and the higher the stress.

9. The active zone along the grain boundary is composed of subgrains that are rotated (about a [110] axis, among others) through rather larger angles than are the subgrains of the body of the crystal, the direction of rotation reversing many times during a single cycle.

10. Rupture does not occur through the grain boundary in bicrystals; the fracture is always transcrystalline.

11. Preceding necking and rupture, the crystal that is destined to fail abruptly increases its rate of extension to a faster, but linear, rate that obtains throughout that crystal.

12. Cast bicrystals exhibit about one-third the rate of extension and grain-boundary gliding that strain-annealed bicrystals of the same composition exhibit.

13. From the above observations it is concluded that grain-boundary gliding consists in an alternation of coordinated slip and recovery operations in a physically continuous chain of subgrains lying along the grain boundary. Coordinated motion is believed to be activated by the accumulation of energy, in the form of crystal bending, as transgranular slip impinges upon the grain boundary and to decay as the alternations of slip and recovery drift out of phase from point to point along the grain boundary.

14. Because primary creep has the same rate characteristics as grain-boundary gliding, it is proposed that the same kind of mechanism operates in transgranular bands in primary creep; when the bands grow, so as to include the whole volume of the metal, the rate becomes constant; tertiary creep follows when large groups of active bands become coordinated, perhaps by gross kinking.

Some examples of the manner in which these concepts could be employed in guiding the search for creep-resistant alloys have been cited.

Carnegie Institute of Technology,
Pittsburgh, Pa., April 15, 1954.

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TABLE I

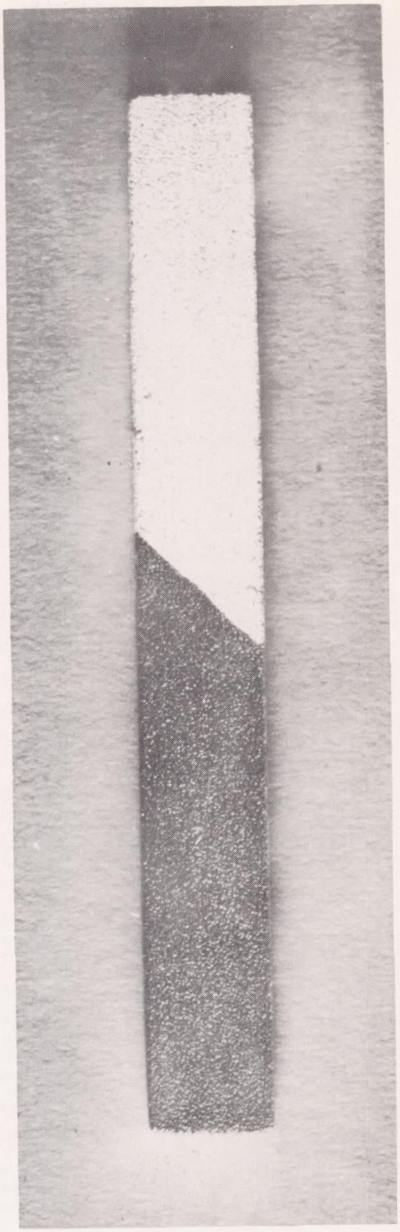
NUMBER OF ALUMINUM BICRYSTALS TESTED AT VARIOUS TEMPERATURES AND STRESSES

Temperature, °C	Number of bicrystals tested at stress, psi, of -						
	Less than 50	50	100	200	400	800	1,600
200			3				
300		2	28	7	14	2	1
400		1	18	1	1		
500		1	6				
600	2		1				
650			1				

TABLE II

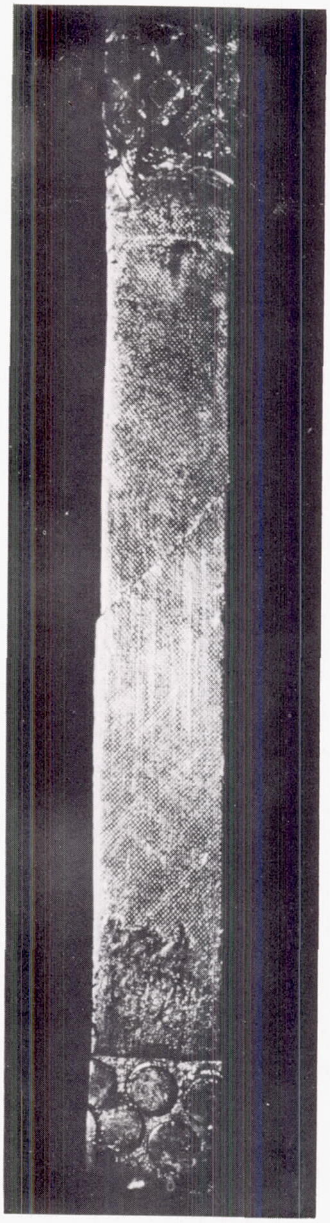
AVERAGE GRAIN-BOUNDARY DISPLACEMENT IN EACH CYCLE

Temperature, °C	Average displacement in each cycle, mm			
	First cycle	Second cycle	Third cycle	Fourth cycle
300	27×10^{-3}	22×10^{-3}	17×10^{-3}	13×10^{-3}
400	70	63	35	30
500	100	---------	---------	---------



(a) Typical bicrystal deeply etched to show crystal orientation difference; Tucker's etch; magnification, X3.

Figure 1.- Typical bicrystals.



(b) Typical bicrystal after tested 1,176 hours at 300° C under a stress of 100 psi exhibiting grain-boundary displacement, reference pattern, and, at each end, indentations from specimen grips; magnification, X3.

Figure 1.- Concluded.

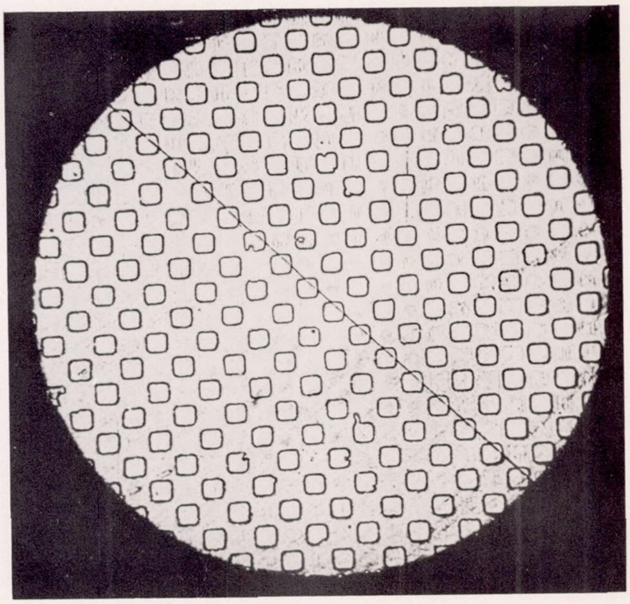


Figure 2.- Reference pattern on face of a bicrystal; magnification, X30.

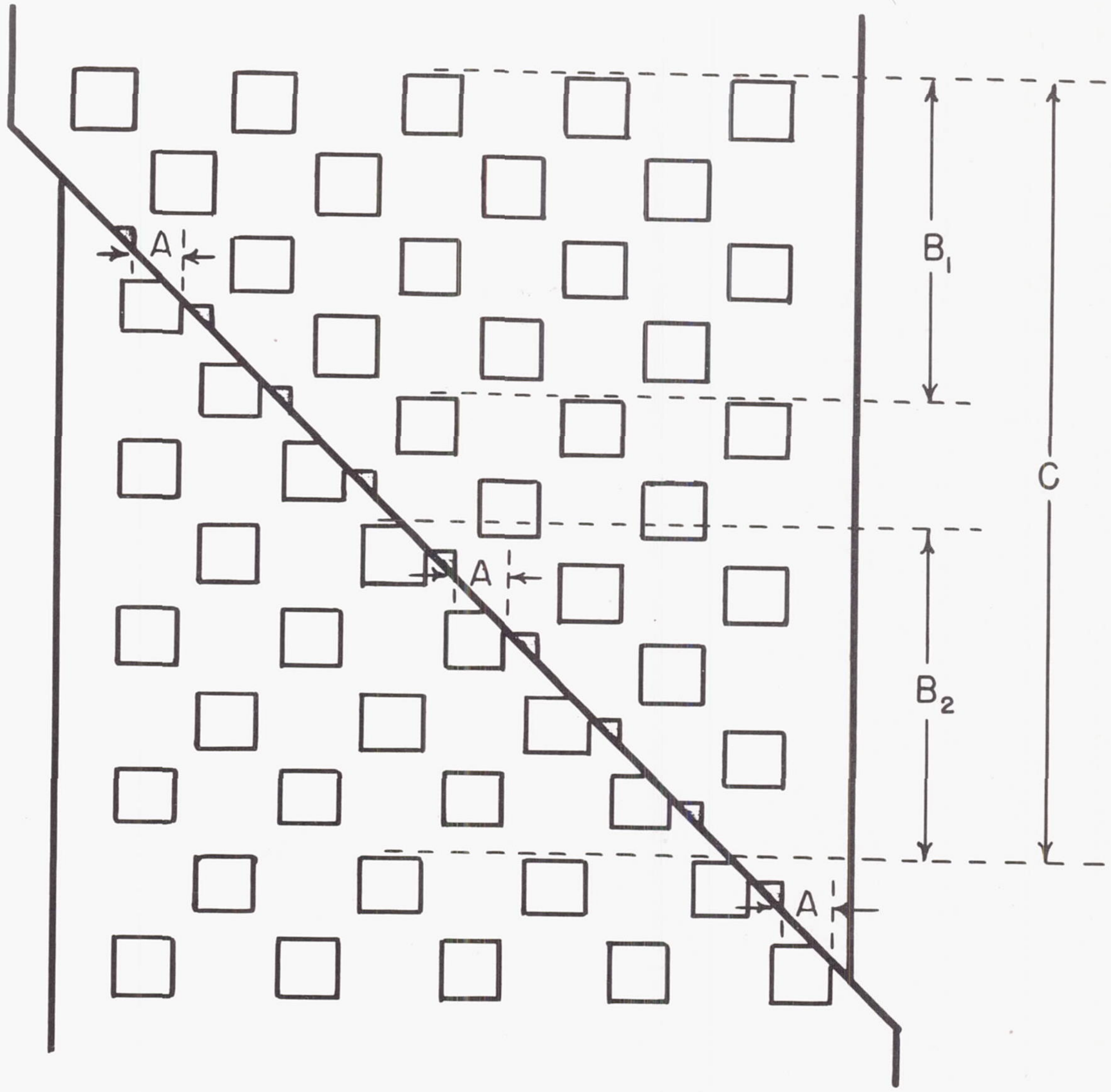


Figure 3.- Schematic plan of measurements made upon bicrystals during creep testing.

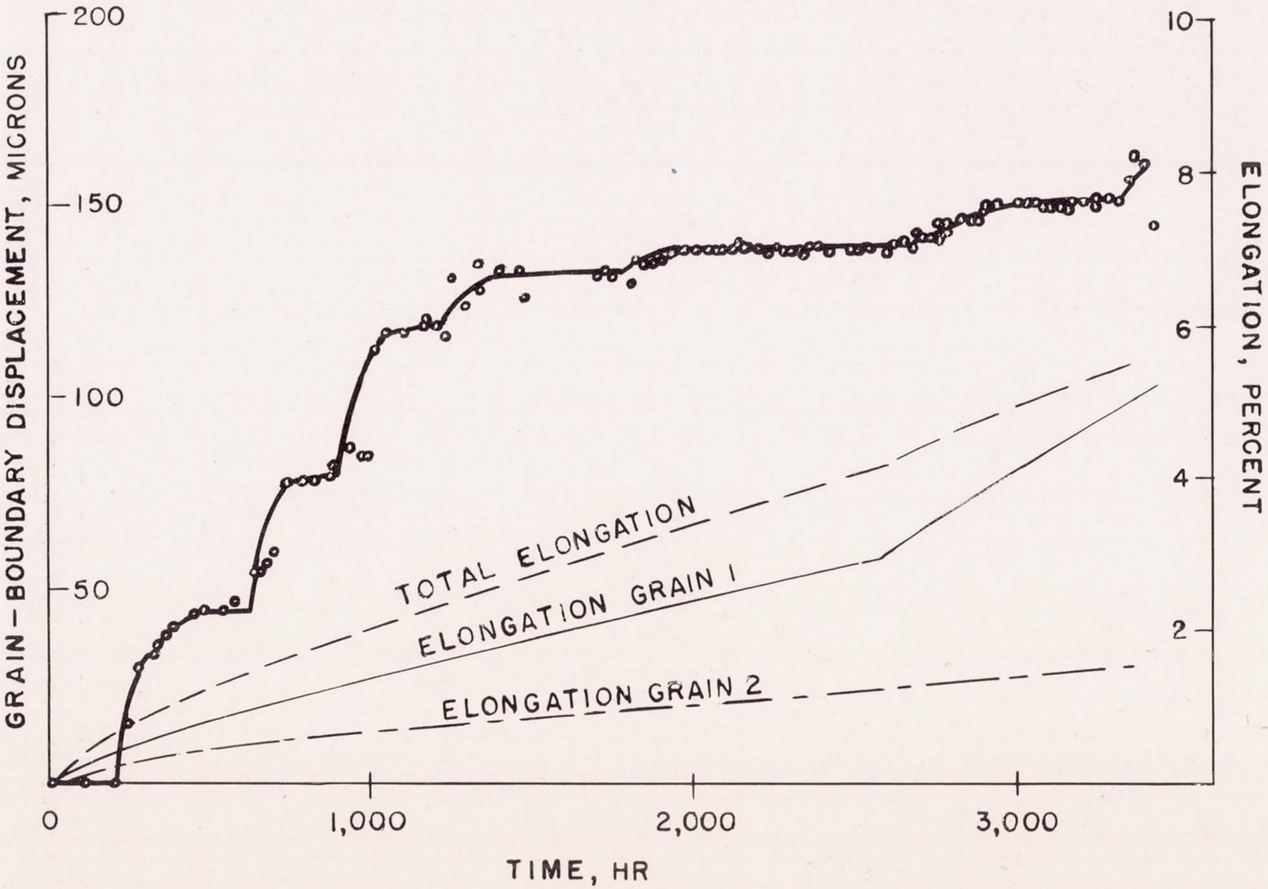


Figure 4.- Typical curve of grain-boundary displacement versus time with accompanying plots of concurrent elongation of each crystal and total extension; specimen tested at 300° C under a stress of 100 psi.

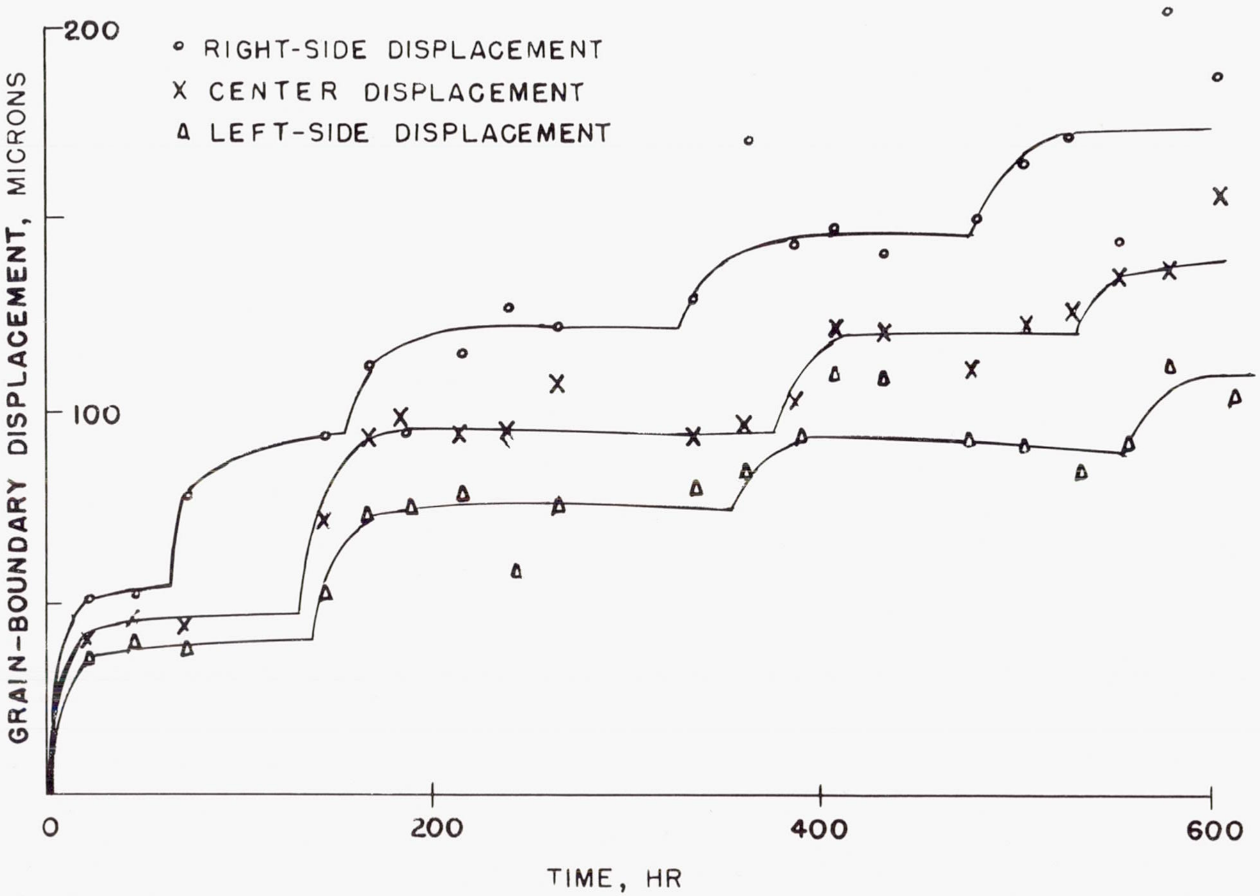


Figure 5.- Grain-boundary displacement versus time at 300° C and a stress of 200 psi, in which separate curves are plotted for each of three reference points along grain boundary. Notice that cycles cease to be coincident after a time and that an average of the three curves would show no cycles after first one or two.

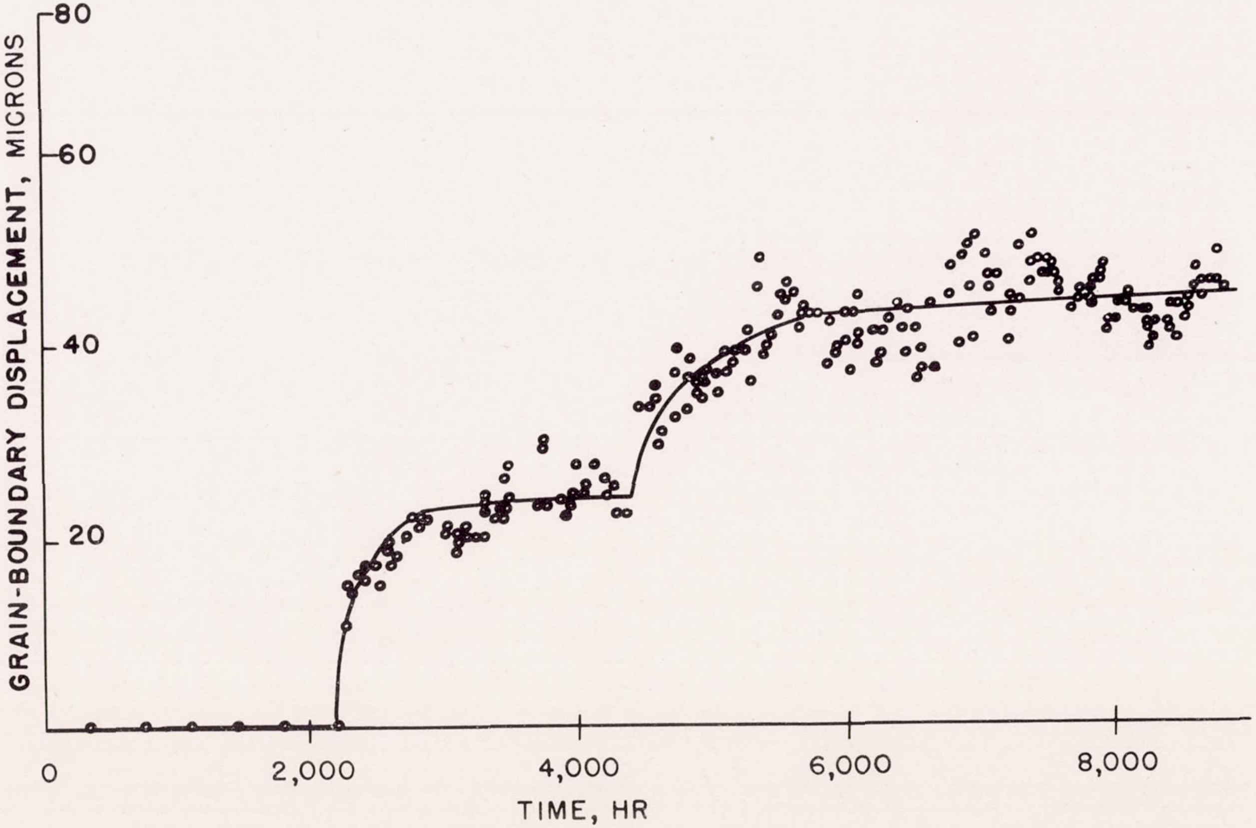


Figure 6.- Example of erratic readings sometimes obtained in a measurement of grain-boundary displacement versus time; specimen tested at 300° C under a stress of 50 psi.

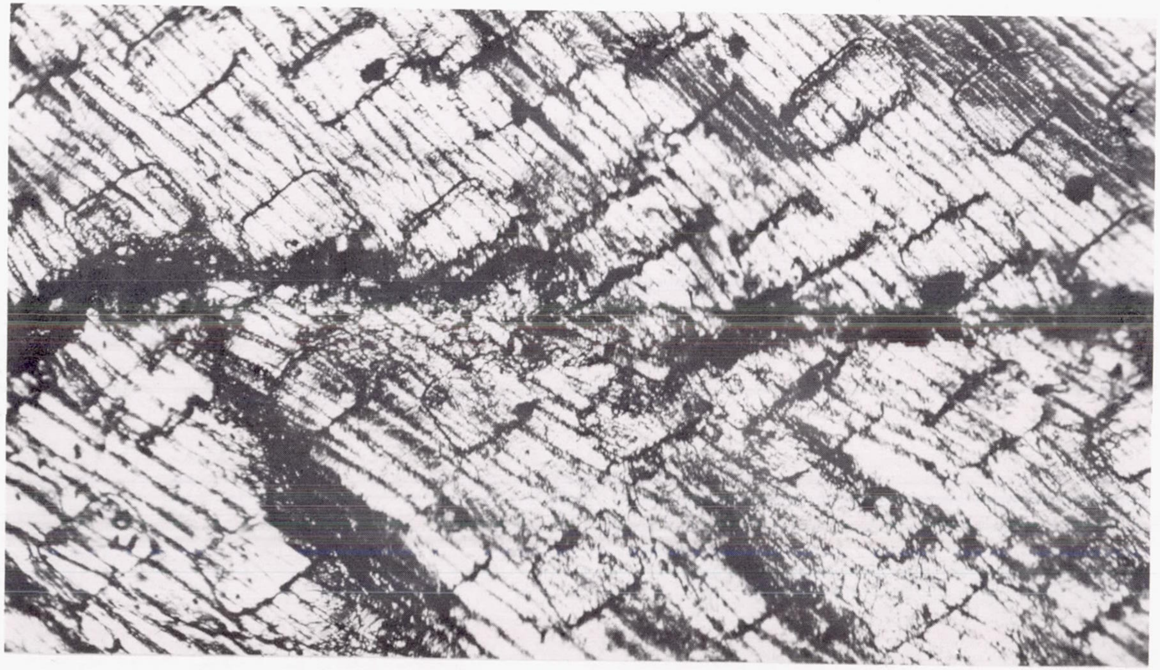


Figure 7.- Photomicrograph of bicrystal subjected to stress of 200 psi for 19 hours at 400° C; magnification, X100; besides a highly distorted grain-boundary zone, gross distortion in adjacent crystals is shown; kink-band ridges are those along which reference squares have been most drastically elongated.

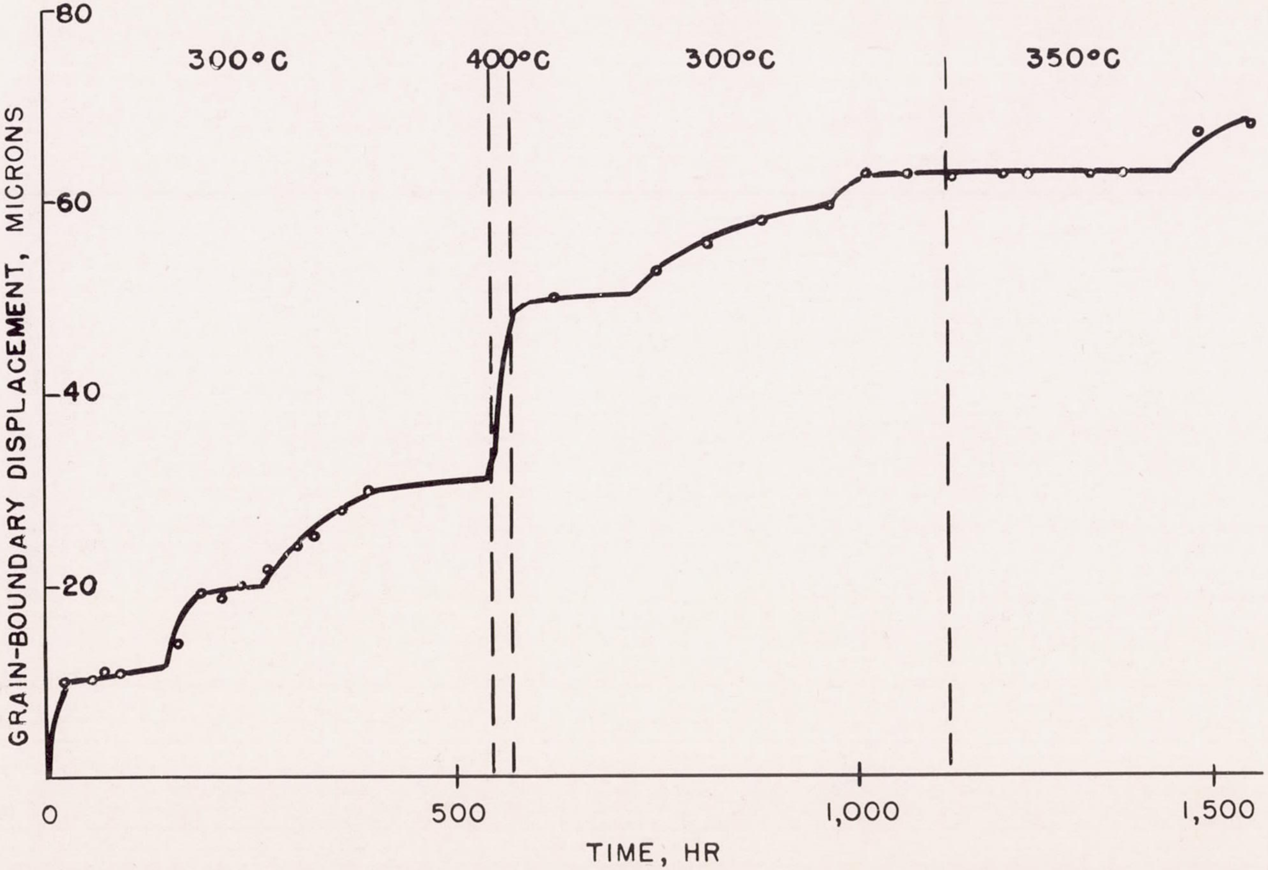


Figure 8.- Some effects of temperature variation upon progress of grain-boundary displacement. An accidental overheating of 100° C occurring at end of a rest period has caused a very large displacement to occur. When temperature was returned to its original level of 300° C, a normal rate of gliding with normal cycling was resumed. Later temperature was deliberately increased by 50° C during early part of a rest period, and rest period continued unaffected for another 2 weeks. This is typical of many such experiences.

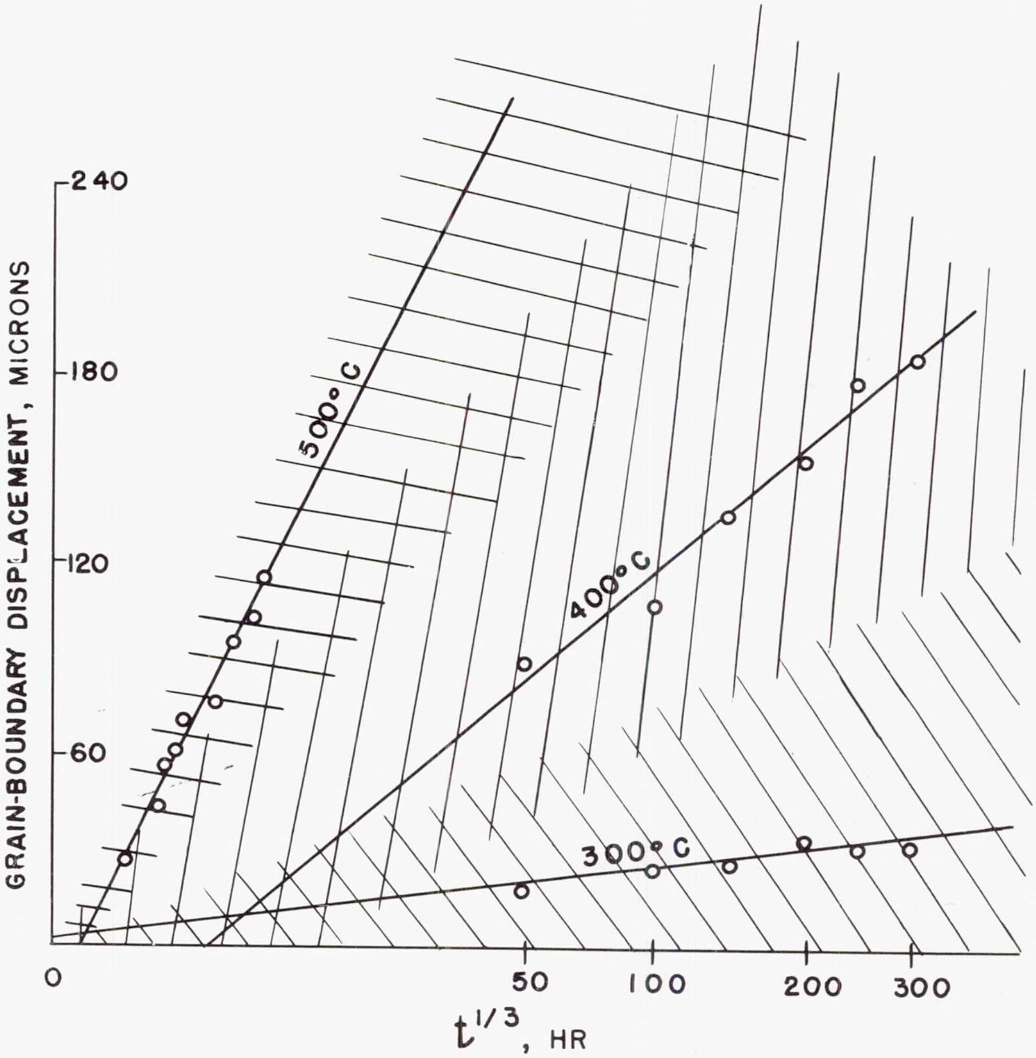


Figure 9.- Grain-boundary displacement averaged for all tests at 300°, 400°, and 500° C, respectively, under a stress of 100 psi and plotted as a function of cube root of time; shaded areas indicate spread of data points employed in averages.

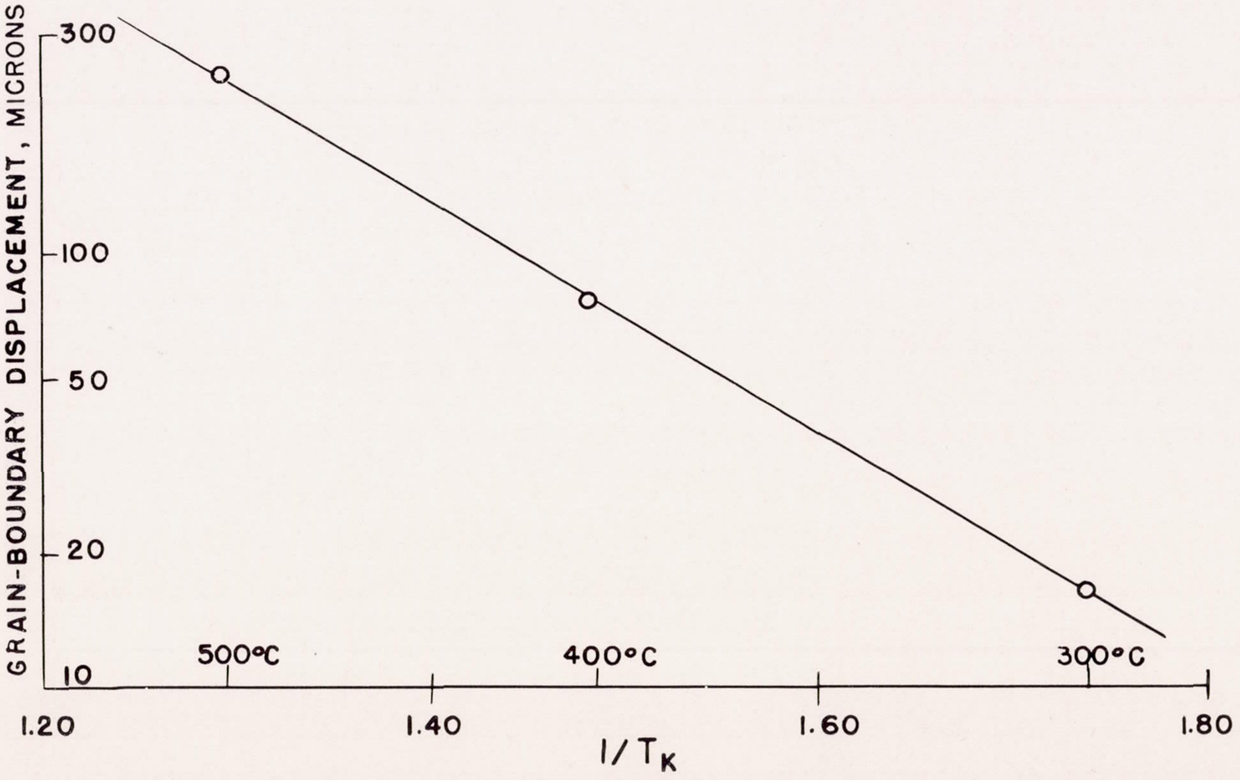


Figure 10.- Logarithm of average displacement in 50 hours as a function of reciprocal of absolute temperature T_K.

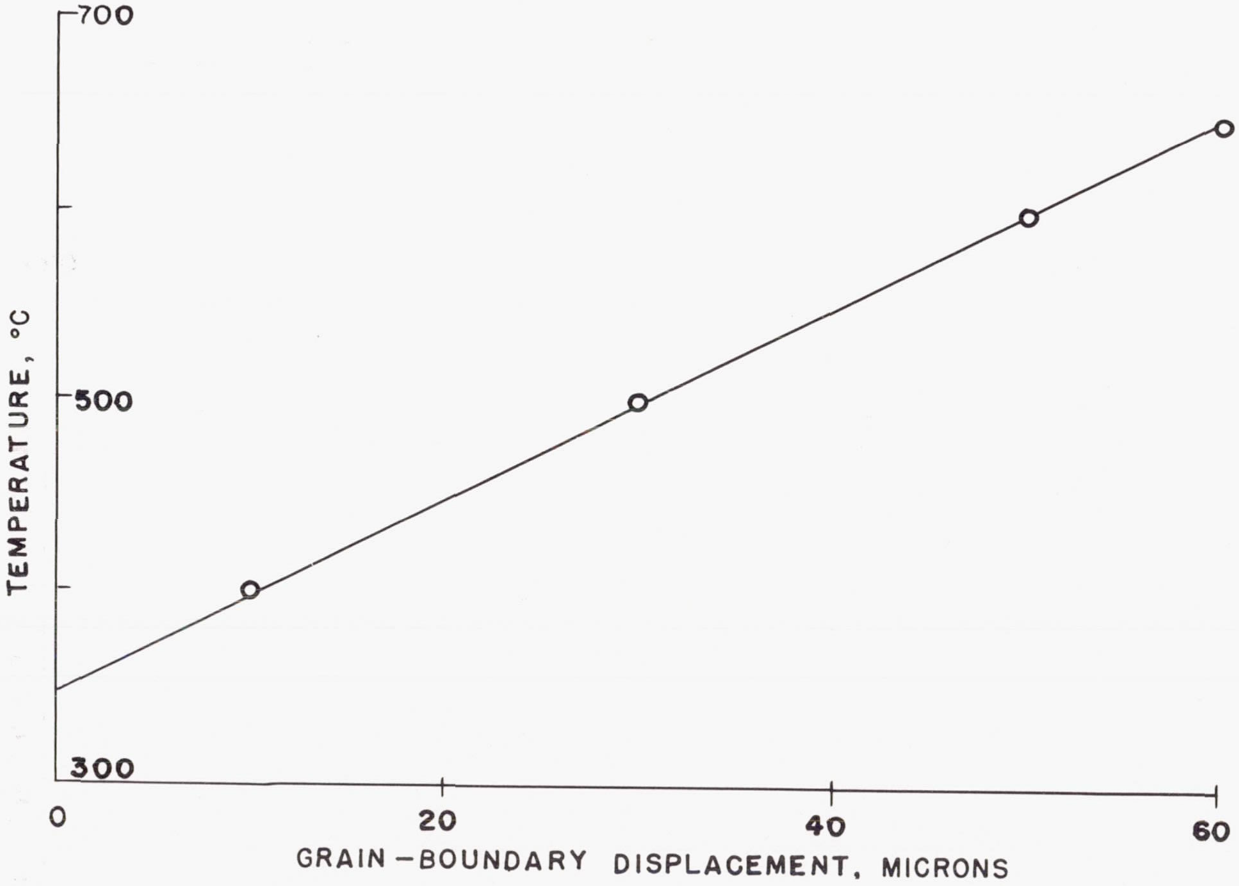


Figure 11.- Grain-boundary displacement during first cycle in four specimens having similar crystallographic-orientation relationships tested at four different temperatures under a stress of 100 psi.

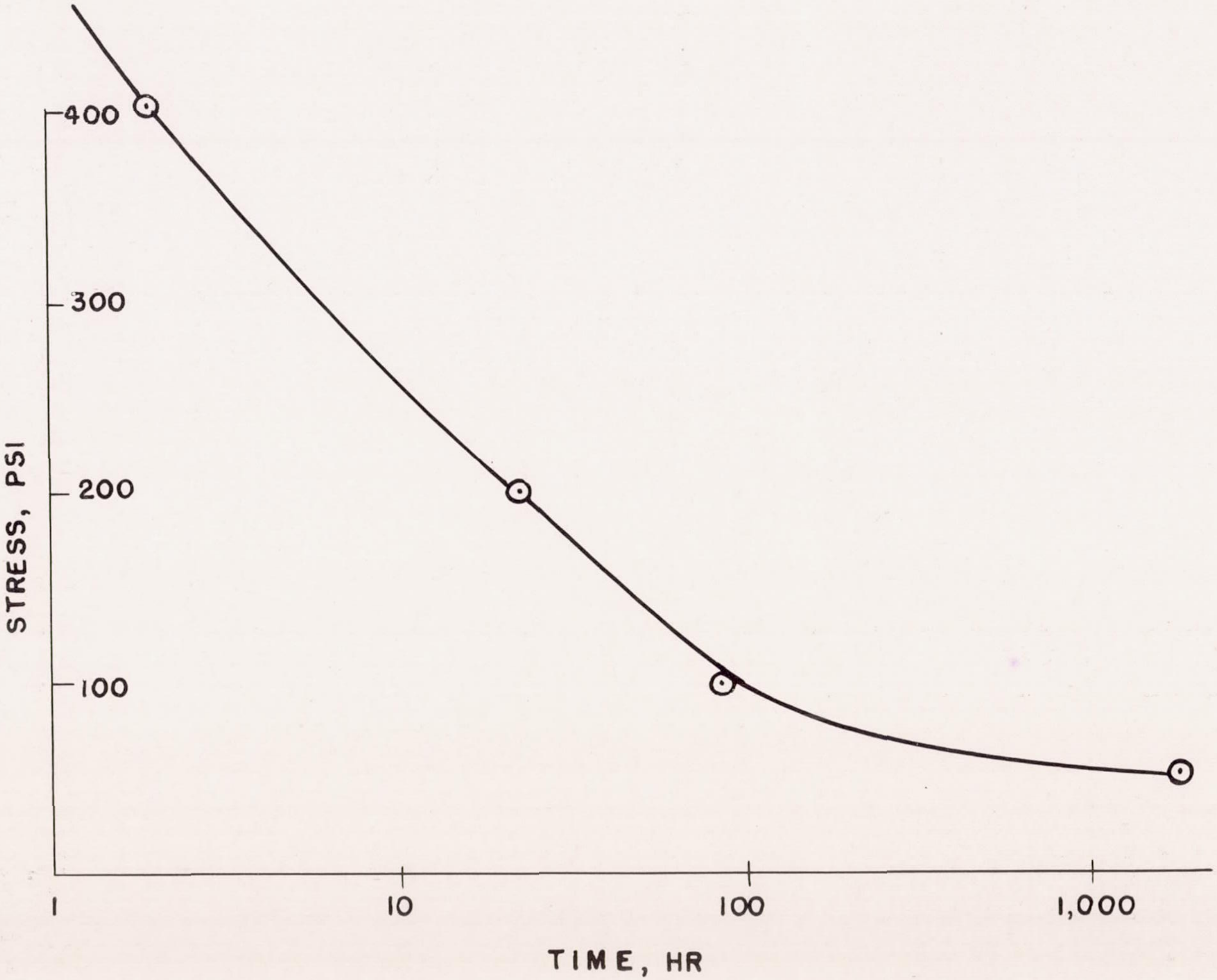


Figure 12.- Stress needed to produce an average grain-boundary displacement of 25 microns at 300° C as a function of logarithm of time.

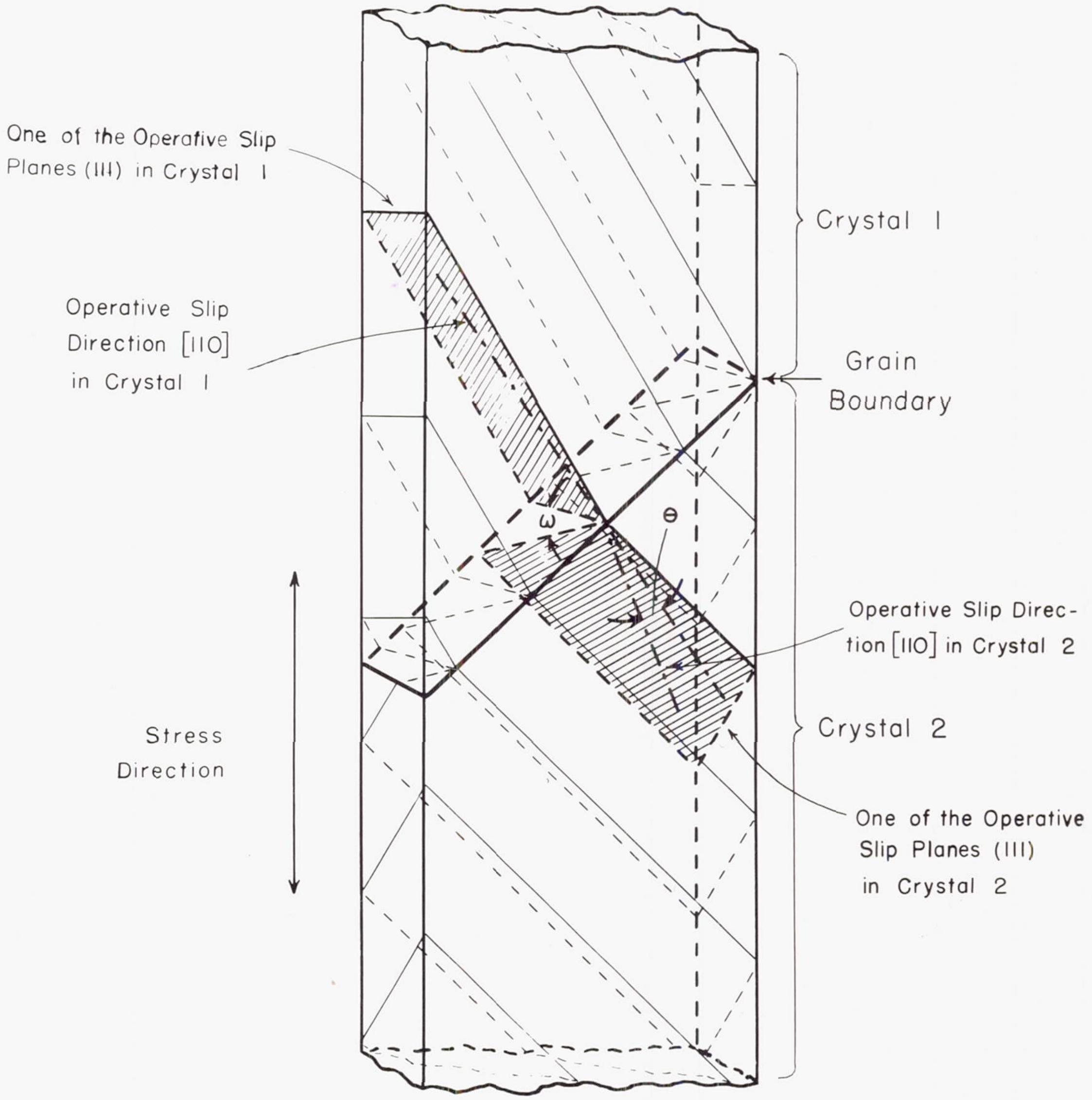


Figure 13.- Schematic representation of a bicrystal showing grain boundary heavily outlined and slip planes of both crystals lightly outlined. Angle between slip directions is θ; that between slip planes, as measured in plane of grain boundary, is ω.

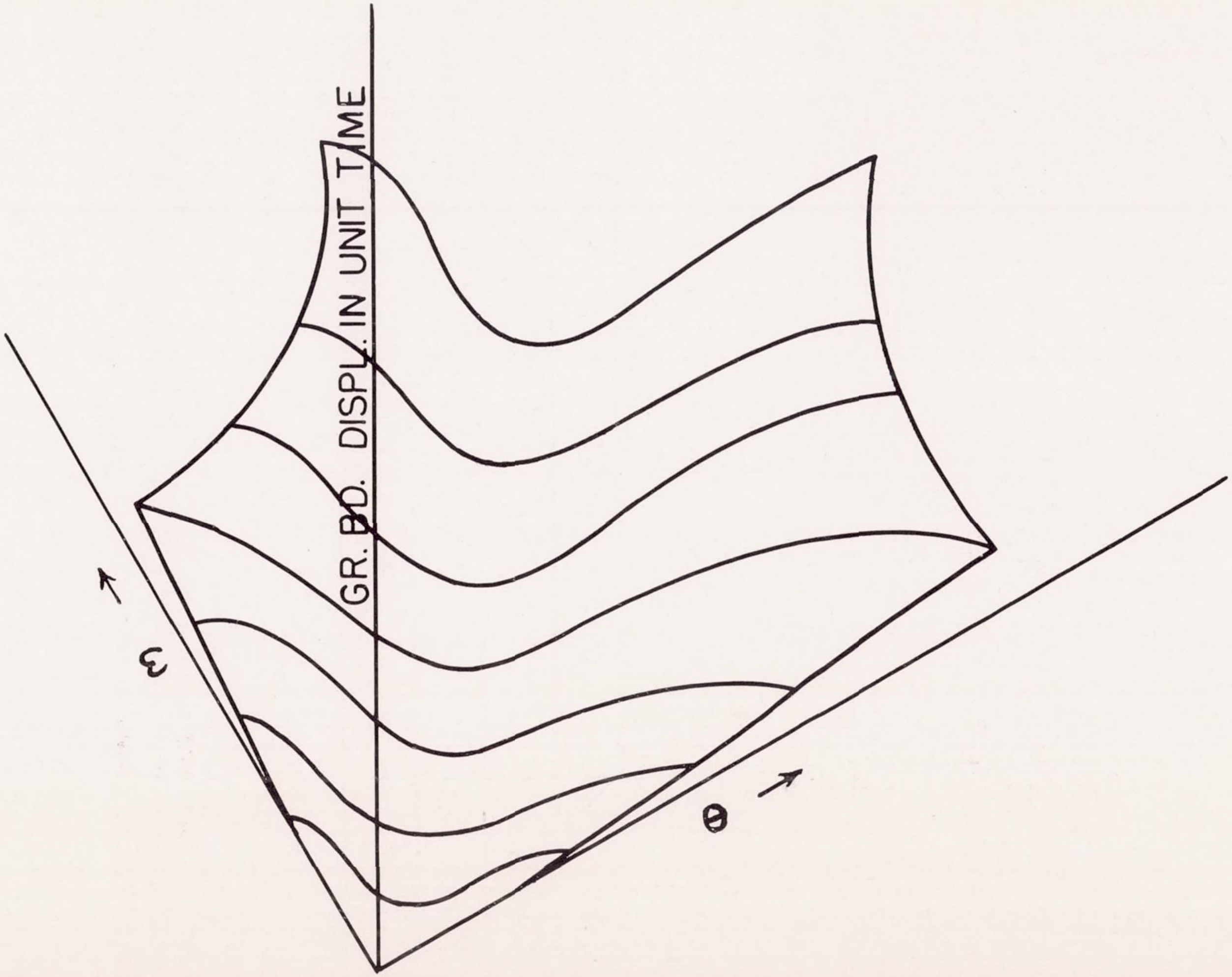


Figure 14.- Schematic plot of relationships between grain-boundary displacement and angles θ and ω. ω, angle between traces of favorite slip planes intersected with grain boundary; θ, angle between favorite slip directions of two crystals.

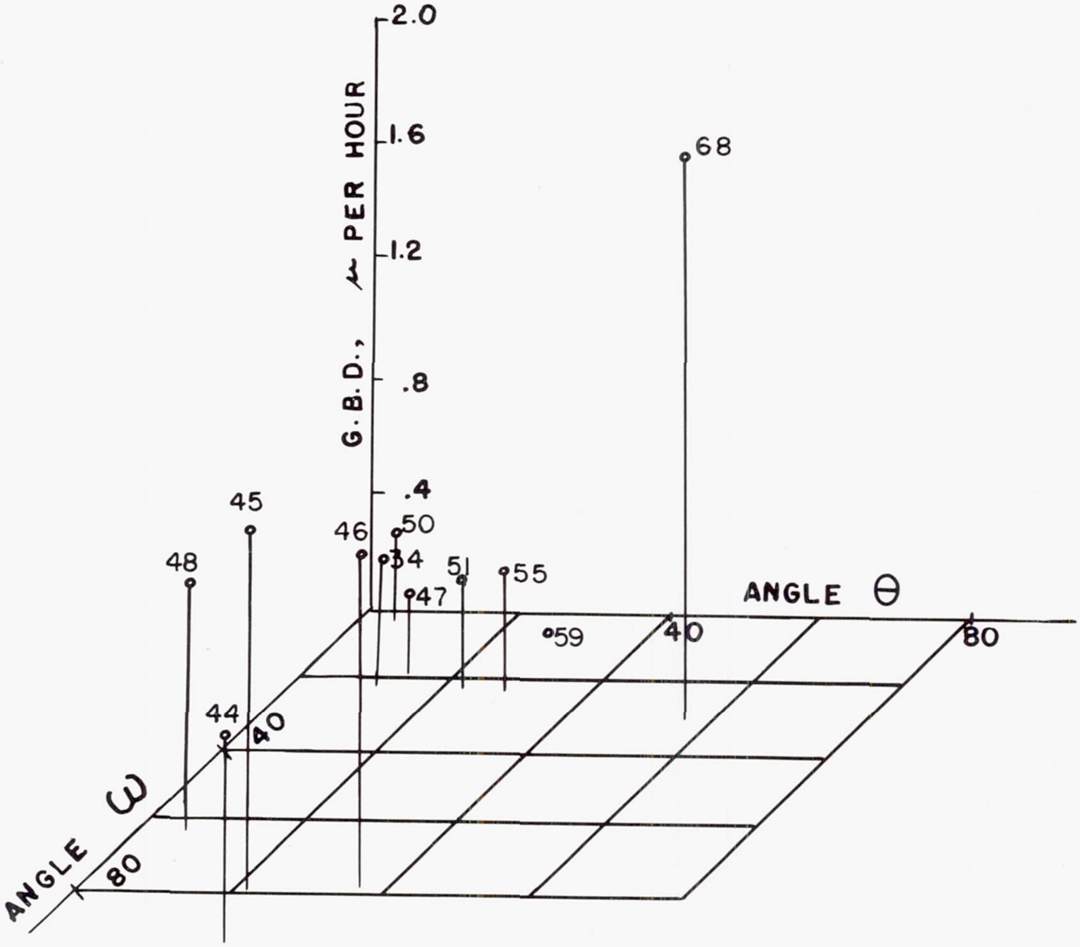


Figure 15.- Grain-boundary displacement in 200 hours in bicrystals of various orientation relationships tested at 400° C under a stress of 100 psi; figure shows increase in displacement with an increase in $\theta + \omega$.

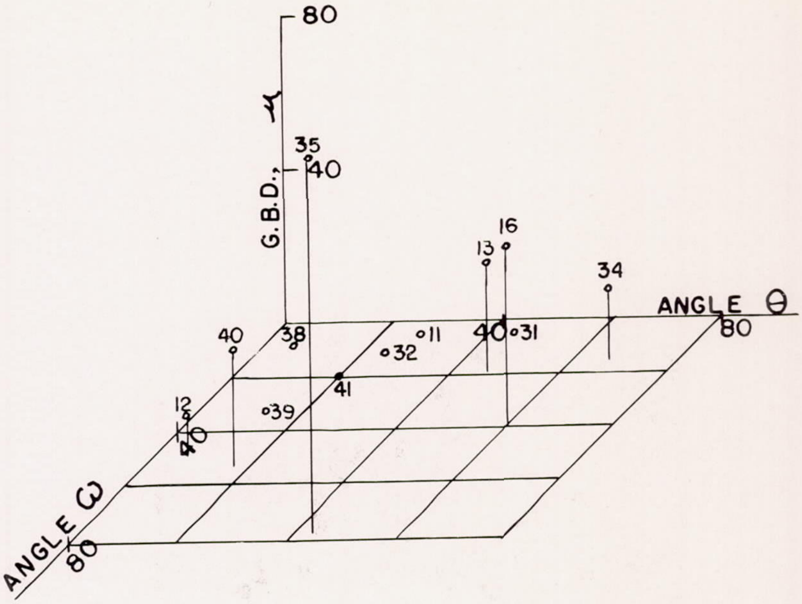


Figure 16.- Grain-boundary displacement in 300 hours in bicrystals of various orientation relationships tested at 300° C under a stress of 100 psi; note that specimens 39 and 41 are among those which have not yet started to glide.

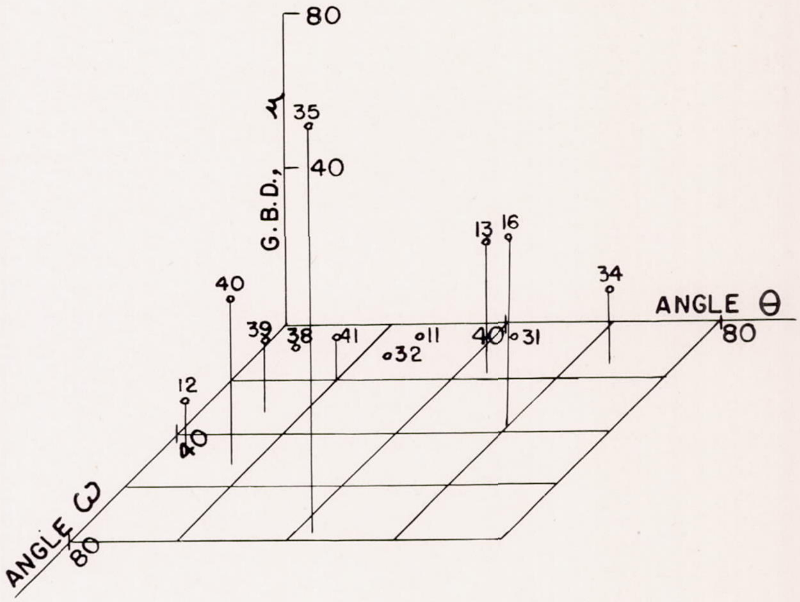


Figure 17.- Grain-boundary displacement in bicrystals of various orientation relationships; same as figure 16 but 150 hours later; note that specimens 39 and 41 exhibit displacement, while those of smaller angle are still dormant.

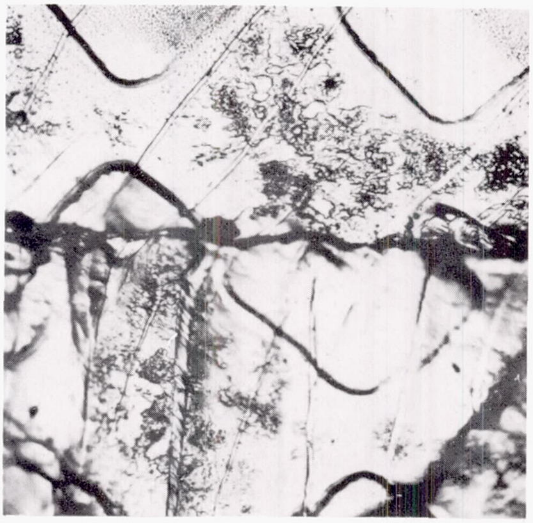


Figure 18.- Photomicrograph of grain-boundary displacement of 150 microns after 1,600 hours at 300° C under a stress of 100 psi; magnification, X250; notice that disturbed zone is scarcely 0.001 millimeter in width.

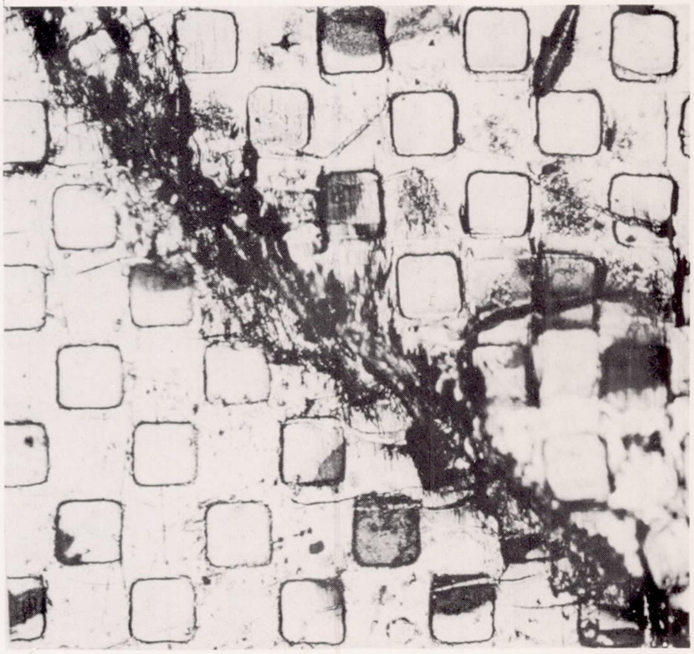


Figure 19.- Photomicrograph of grain-boundary displacement of 350 microns after 750 hours at 400° C under a stress of 100 psi; magnification, X75; disturbed zone along grain boundary is here about 0.100 millimeter in width.

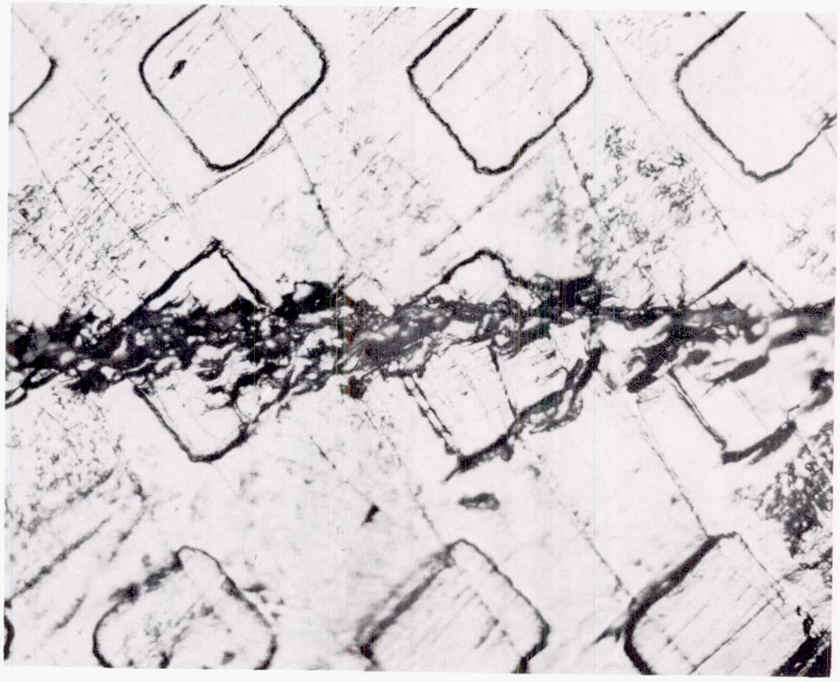


Figure 20.- Photomicrograph of grain-boundary displacement of 110 microns after 936 hours at 300° C under a stress of 200 psi; magnification, X200; disturbed zone is about 0.100 millimeter in width.

Figure 21.- Photomicrograph of grain-boundary displacement of 300 microns and gross slip after 864 hours at 400° C under a stress of 100 psi; magnification, X5; upper diagonal trace is grain boundary, while those below are slip bands; compare rounded-edge contour at ends of grain boundary with sharp steps at ends of slip bands; also note that grain-boundary displacement has been parallel to reference surface, while slip has produced ledges upon reference surface.

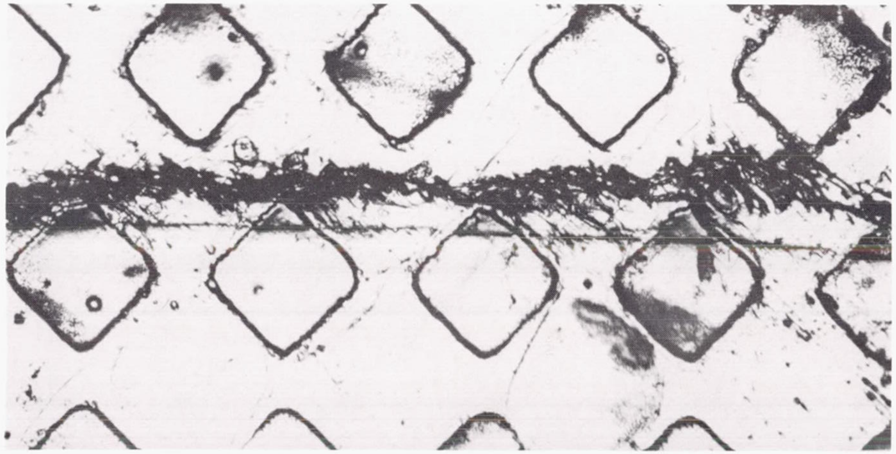


Figure 22.- Photomicrograph of grain-boundary displacement along a path which is not coincident with original grain boundary upon which there has been no displacement; grain-boundary migration evidently preceded gliding, but path of gliding then remained constant through 2,700 hours and 50 microns of displacement; magnification, X160.

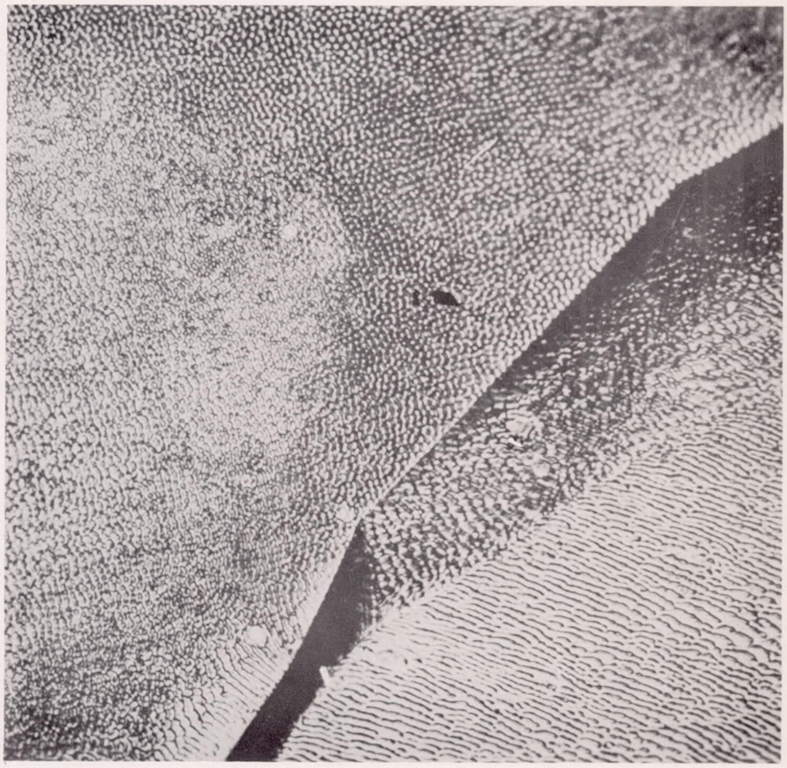
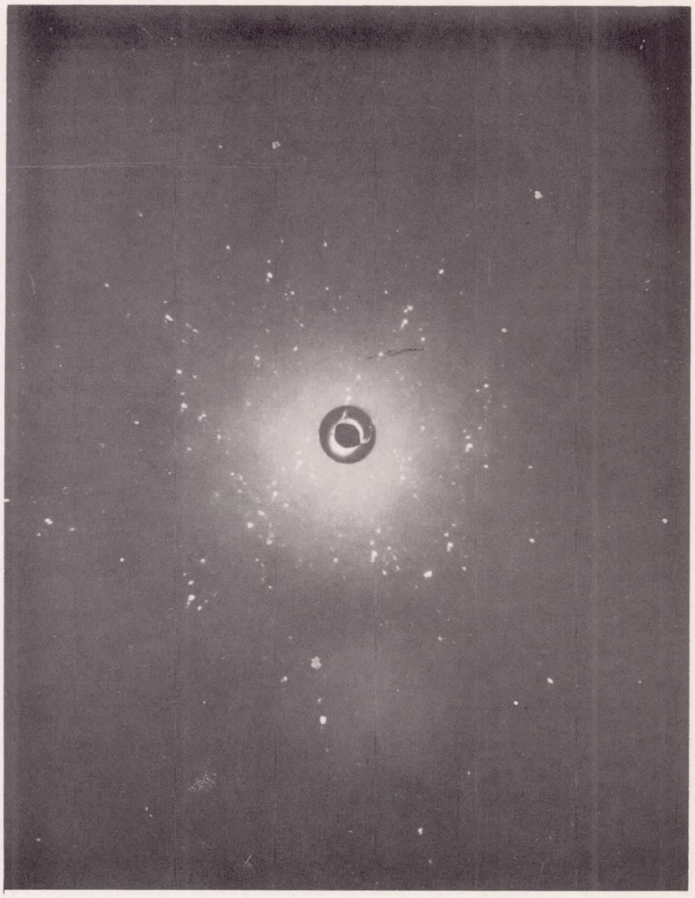


Figure 23.- Short segment along grain boundary of specimen illustrated in figure 20 deeply electropolished and etched after testing; magnification, X400; notice that grain boundary is sharp and that subgrains are uniform except in a band that separates lower grain from actual boundary.

(a) Laue back-reflection pattern of one of grains of a bicrystal tested at 400° C under a stress of 100 psi showing fragmentation of crystal accompanied by a rotation of subgrains by a few degrees with respect to orientation of parent crystal.

Figure 24.- Laue back-reflection patterns.



(b) Laue back-reflection pattern of grain-boundary ledge of a bicrystal similarly tested except for only 50 hours showing that metal along grain boundary has been broken up into minute crystallites whose orientations are more widely dispersed than those of original crystal.

Figure 24.- Concluded.

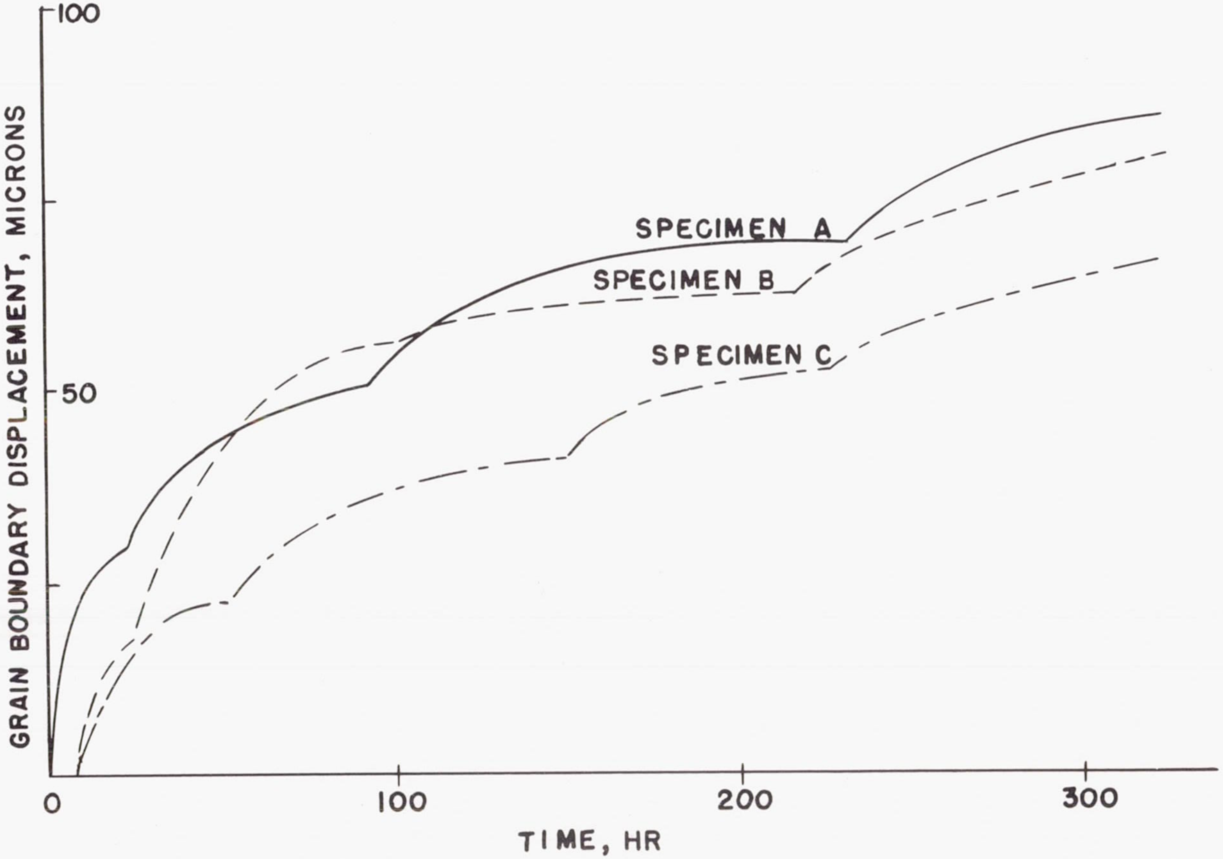


Figure 25.- Grain-boundary displacement versus time for three identical cast bicrystals tested at 300° C under a stress of 400 psi.

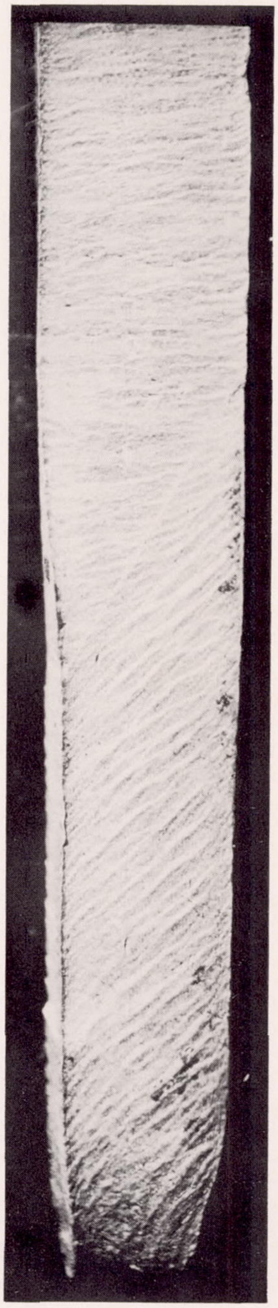


Figure 26.- Same bicrystal as that shown in figure 7; magnification, X3.5; surface wrinkles are presumed to be result of kink banding; lower end of this specimen is fractured end.

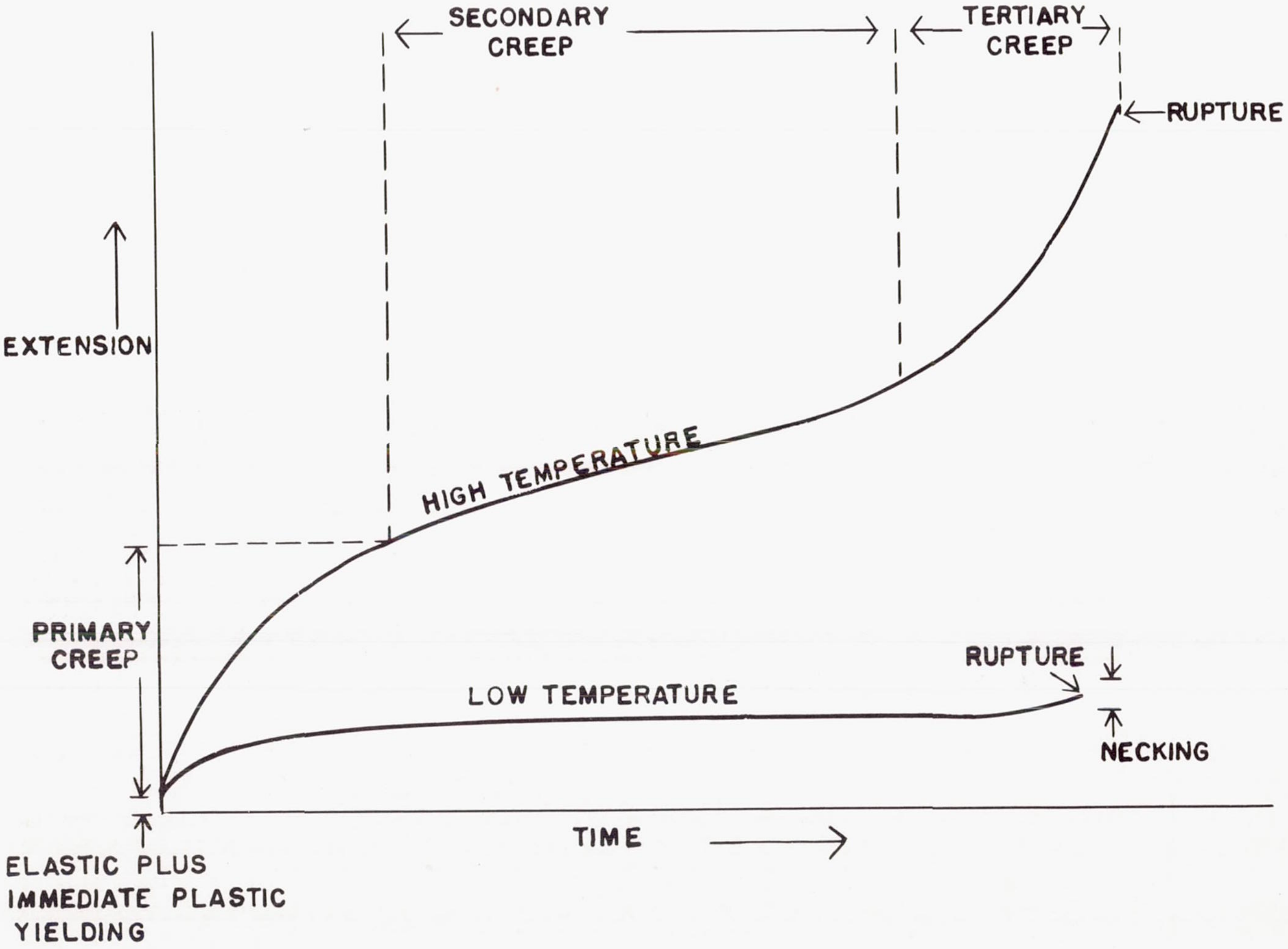


Figure 27. - Schematic representation of ideal creep curves.

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