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TECHNICAL NOTE 2746

PREVIEW OF BEHAVIOR OF GRAIN BOUNDARIES IN CREEP OF ALUMINUM BICRYSTALS

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Carnegie Institute of Technology

Washington
July 1952

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PREVIEW OF BEHAVIOR OF GRAIN BOUNDARIES IN CREEP OF ALUMINUM BICRYSTALS

By F. N. Rhines and A. W. Cochardt

SUMMARY

The gliding of one metal crystal with respect to another, along their mutual grain boundary, has been studied during high-temperature creep tests conducted with bicrystals of pure aluminum. Stresses of from 1 to 100 psi and temperatures from 200° to 650° C were employed. The direction of motion depends only upon the direction of the maximum resolved shear stress in the plane of the grain boundary. The magnitude of the motion increases with the magnitude of the resolved shear stress, the temperature, and the degree of mismatch between the orientations of the two crystals. The gliding rate is cyclic, increasing directly with the temperature at the beginning of the cycle but subsequently obeying a chemical-rate law, with a computed activation energy of 9700 calories per mole. There is for each temperature a threshold stress below which gliding does not occur. At lower temperature, and where the orientation difference between the grains is small, an induction period precedes the onset of gliding. A substantial thickness of crystalline metal participates in the gliding motion and becomes fragmented and extensively disoriented in the process. During creep, the portions of the grains away from the boundary also degenerate into blocks (or fibers) that move with respect to each other, parallel to the tension axis, and that rotate about octahedral axes of the original crystal.

INTRODUCTION

One phase of the important problem of developing better high-temperature alloys is the effort to gain a more detailed understanding of the nature of creep in metals. It has been appreciated for some time that creep is a complex process and that, among other complications, displacement sometimes occurs along grain boundaries at the same time that the grains themselves are deforming. The present research constitutes an effort to isolate and examine the grain boundary displacements. Information obtained in this way should make it possible, ultimately, to describe and predict the contribution to creep that is made by the

grain boundaries. It is expected that, as a byproduct, these studies will contribute also to the general knowledge of the nature of grain boundaries, which knowledge is basic to the understanding of other characteristics of metals.

This paper is offered as a preview of a study, which, because of the time-consuming nature of its experimentation, is not likely to reach maturity for some time. The results at hand appear of sufficient interest to warrant publication, even though continuing research may require the modification of some of the interpretations and conclusions.

Interest in grain boundary behavior in creep arose originally from efforts to account for steady-state creep and for the effects of grain size upon the creep rate. Probably the earliest observation of steady-state creep was that of Howe in 1885 (reference 1); but it remained for Andrade in 1914 (reference 2) to associate this phenomenon with the grain boundaries. Realizing that steady-state flow is a characteristic of viscous materials that is difficult to reconcile with the limited slip behavior which is characteristic of crystalline substances, Andrade proposed that this type of deformation occurs by a viscous flow of amorphous metal resident in the grain boundaries and upon slip planes that have operated previously. Further need for the assumption of a grain boundary flow was presented in 1931 by Clark and White (reference 3), who found that, at elevated temperatures, certain metals exhibit higher rates of creep as the grain size is reduced. The first direct evidence of grain boundary displacement came in 1938, when Moore, Betty, and Dollins (reference 4) observed, in creep specimens, the lateral displacement of polishing scratches where these crossed grain boundaries. Meanwhile, numerous observations of grain boundary gliding have been reported and the effect has become accepted to the extent that Wood, Wilms, and Rachinger (reference 5), in their recently propounded mechanism of creep, have ascribed the major part of the extension at low loads and high temperatures to displacement at the grain boundaries.

Since there have been, up to now, no extensive investigations into the characteristics of grain boundary glide as a distinct phenomenon, there was, at the inception of the present research, no adequate basis for planning the studies that would be required to reveal the nature of the process. It was necessary to engage in a series of purely exploratory experiments, for the purpose of finding a suitable approach for more intensive investigation. The details of these preliminary sorties are unimportant to the present account, but a summary of the somewhat provisional conclusions derived from them will serve to explain the selection of experimental methods, as well as to indicate some of the more evident aspects of the process.

This work was conducted at the Carnegie Institute of Technology under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

PRELIMINARY EXPLORATION

In coarsely polycrystalline aluminum specimens, observed in creep at various low loads and elevated temperatures, it was found that movement was rarely uniform along any one grain boundary but tended to be rapid at some places and slow, or negligible, at others. The most rapid movement occurred only where a boundary lay at an angle of approximately 45° to the axis of stress, little if any movement occurring where the boundary lay perpendicular to, or parallel with, the tension axis (see fig. 1). It did not always happen, however, that movement took place where the grain boundary lay near 45° to the stress direction. Frequently, the entire length of boundary between two grains remained firm throughout the period of testing, regardless of its orientation.

Thus, it was concluded that, for a given boundary between a specific pair of grains, the magnitude of the displacement increases with the resolved shear stress, as might well have been expected. However, it was also concluded that the degree of the response to stress differs from boundary to boundary in a manner such as to suggest control by the difference in crystal orientation obtaining between the members of each of the pairs of grains forming the boundaries.

Nonuniformity was observed also in the direction of motion. Between single pairs of grains, where the boundary was curved, or irregular, it was usually found that the direction of the displacement changed markedly from point to point along the boundary (see fig. 2). This means, clearly, that the direction of flow is insensitive to crystal orientation; it depends, instead, upon the local orientation of the boundary surface with respect to the tension axis.

With local variation in both the magnitude and direction of the grain boundary displacement, between each pair of grains, it is inevitable that the grains themselves should be subjected to distortion of a highly complex character and that this distortion should react upon the boundary movement by superimposing unpredictable secondary stresses.[1] That such was the case was indicated also by the finding that the progress of local movement was frequently erratic with respect to both magnitude and direction.

It was made apparent, thus, that the behavior of the grain boundaries could not be studied uniquely in polycrystalline bodies. The research that is about to be described was conducted, therefore, by the use of bicrystal creep specimens, having flat grain boundaries oriented

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[1]Some excellent illustrations of the interplay of grain boundary glide and grain distortion have been presented in a very recent paper by Betteridge and Franklin (reference 6).

at 45° to the tension axis. Variation in the motion from point to point was largely eliminated by using a flat boundary. This assisted also in minimizing the irregular distortion of the crystals, as did the removal of restraints at the edges of the boundary. Orienting the boundary at 45° insured a maximum displacement, thereby minimizing errors of measurement. The superposition of a further requirement, that the grains of the bicrystals have identical, or preselected, differences in orientation, was necessary for the determination of the effects of such external factors as temperature and applied stress upon motion at the boundaries.

EXPERIMENTAL MATERIAL

Pure aluminum (99.95 percent) was selected for these studies, partly because of the abundance of pre-existing knowledge of the creep behavior of this metal, but mainly because of the advantage that it offered in the ease of specimen preparation and the convenience of suitable testing temperatures and loads. Exhaustive spectrographic analyses showed no significant difference in composition between the two lots of aluminum that were used. 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 present in spectrographically detectable amount.

EXPERIMENTAL METHOD

Bicrystal test specimens were made by cutting bars, with suitably oriented straight grain boundaries, from very coarse-grained aluminum plates that had been produced by strain annealing (see fig. 3). The alternative method, of growing bicrystals from the melt, was not adopted, because of uncertainty concerning the importance of possible effects of the cast lineage structure and the concentration of impurties at the cast grain boundary.

Aluminum plate, rolled to a thickness of approximately 1/8 inch, was annealed and then alternately stretched 1.7 percent and heated to just under the melting point of aluminum until grains of usable size had developed. For the most part, the grain boundaries lay perpendicular to the surface in these plates, so that it was necessary only to find sections of straight grain boundary and to lay out upon them test bars with their long axes at 45° to the boundary trace. The bars, which were usually about $1\frac{1}{2}$ inches long by $\frac{3}{8}$ inch wide, were cut out with a jeweler's saw. Distorted metal was removed by alternate polishing and etching. The

appearance of a typical specimen, etched to produce grain contrast (Tucker's etch), is shown in figure 4. At this stage, the orientations of the two crystals were determined either by the etch-pit method, described by Barrett (reference 7), or by the analysis of Laue back-reflection photograms with Greninger charts (reference 8).

Before testing, these specimens were electropolished to produce a bright surface with the grain boundary delineated as a very fine line. A reference pattern was then photoengraved upon the polished surface. The latter operation was accomplished by the use of a photoengraver's cold-top process, involving exposure through a screen with 133 lines to the inch, re-electropolishing after development of the image, and removal of the residual cold top with a suitable solvent. This produced a pattern of squares that protruded minutely from the polished surface, but did not mask any portion of the surface or introduce distortion (see fig. 5). The electropolish produced a fairly heavy, although transparent, oxide film, which could be largely removed by washing with a solvent, but which was usually left in place as a deterrent to tarnishing during testing at elevated temperatures.

The testing apparatus consisted of conventional dead-weight loaded creep machines with resistance furnaces under automatic control and open to the atmosphere. File-faced grips of light construction were used to hold the specimens. A specially designed long-focusing Gaertner measuring telescope with horizontal and vertical micrometer scales, a protractor, and focusing scale was used for measuring the grain boundary displacement and elongation of the specimen.

The general plan of the measurements is shown schematically in figure 6. The displacement of the two halves of bisected reference squares was measured horizontally at three points, such as those designated A in the diagram, averaged, and translated into displacement parallel to the boundary. At the same time, elongation was measured within each crystal, parallel to the stress axis, over the lengths C and D which were initially 0.32 centimeter long. Finally, a total extension B was measured across the grain boundary between reference points about 0.7 centimeter apart.

Specimens were selected for the various tests in accordance with the relative orientation differences between their crystals; no attempt was made to control the crystal orientations with respect to the direction of the tension axis. For the study of the effect of orientation differences upon the rate of grain boundary displacement, a series ranging from only 5° difference to a maximum of about 45° was selected and tested at constant temperature and stress. Samples of almost the same orientation difference, usually near $45^{\circ} \pm 5^{\circ}$, were chosen for the examination of the effects of varying temperature and stress.

The conduct of the typical test consisted in placing the sample in the creep machine, adjusting the temperature, making an initial set of readings, and then applying the load very gently. Another set of readings was made as soon after loading as possible; that is, as soon as the vibrations caused by loading had been dissipated. Thereafter, readings were made at increasing time intervals until they were spaced 24 hours apart, when daily readings were begun. The tests were terminated at various times, up to an exceptional maximum of 6 months, for one or another of the following reasons: (1) Rupture had occurred (always transgranular), (2) the surface had become so tarnished that further readings could not be made, (3) the pattern of behavior of the test had become sufficiently evident, or (4) the specimen had been damaged by some accident, such as a failure of the temperature control.

After creep testing the specimens were again examined to find any unusual characteristics that might have escaped earlier notice. Such examinations included, at most, a microscopic survey of the surface and X-ray diffraction studies at several locations.

PRECISION

The measuring microscope was designed to read directly to 0.001 millimeter. Various operators could not check each other within this precision, however, and the exact location of reference points became increasingly difficult as the specimens became tarnished and distorted. It is probably safe to assume that the grain-boundary-displacement readings are reliable to within 0.005 millimeter. Assuming this same error in grain-elongation measurements, the error, in terms of percentage elongation, may have been as high as 0.5 percent for the single-grain values and 0.2 percent for the total elongation. In some of the early tests (low specimen numbers) larger errors were present.

Aside from periods of actual control failure, the temperature variation did not exceed $\pm 5^{\circ}$ C. Although thermal gradients of as much as 5° C within the specimen length were found, it is unlikely that the difference along the length of any one grain boundary exceeded 1° C. The testing temperatures reported were read by means of an auxiliary thermocouple placed close to the specimen at the grain boundary. Errors in this reading, resulting from calibration drift, probably have not exceeded 3° C.

The two methods used for determining crystal orientation are both capable of a precision of 0.5°. As operated, however, the optical method was probably more reliable. An error of as much as 1° is likely in the X-ray determinations.

Loads were calculated upon the basis of the measured dimensions of the samples and were made up in shot that was carried in a tare bucket. No good means is available for assessing the error in this case, but it seems improbable that it could have exceeded 1 percent.

RESULTS

Examples of grain boundary gliding are shown in figures 7 and 8. Since the grain boundary was very nearly perpendicular to the reference surface, the translation has proceeded almost exactly parallel with this surface. It is evident both that the grain boundary has remained straight on a coarse scale and that the distortion of the crystals has been slight. Viewed in greater detail, as in figure 8, the boundary appears somewhat irregular, but not so much so as to interfere seriously with the measurement of the relative displacement of the reference squares. The results that follow have been obtained from the observation of the behavior of about 30 such bicrystal samples.

Grain Boundary Glide Rate

Typical examples of the progress of grain boundary glide with time are presented in figures 9 and 10. Common to all of the cases at hand is a cyclic variation of the rate. In tests conducted at 300° C with a stress of 100 psi, the length of the cycles ranged from 100 to 1000 hours (fig. 10). The motion is rapid at the beginning of the cycle, decreases gradually, sometimes coming to a virtual stop, and then resumes at a rate that is sometimes greater, sometimes less, than that at the start of the first cycle. As many as four distinct cycles have been found in a single test at 300° C. At higher temperatures the cycles seem less distinct (see fig. 11) but the survey at temperatures other than 300° C has been too limited to permit any definite conclusion in this respect.

The resumption of gliding at the end of each rate cycle seems to be altogether spontaneous. There were cases of a coincidence between temperature change and the resumption of gliding, as illustrated by the behavior of specimen 12 (fig. 9) at 550 hours, when a temperature-control failure caused an increase of 100° C overnight. But this same specimen failed to respond to an intentional increase in the temperature at 1250 hours, while specimen 34 (fig. 10(a)) resumed gliding spontaneously, without temperature variation, after a similar lapse of time.

Another, and possibly related, characteristic of the grain-boundary-displacement curves is the occurrence of an induction period preceding

initial gliding in some of the samples tested at 300° C (see fig. 10). This effect was absent in specimens having a large orientation difference between their grains and in all specimens tested at 400° C and above. Its duration, when it occurred, seemed to increase as the angular disregistry between the two grains of the bicrystal became smaller; thus specimen 34 (fig. 10(a)), with a moderately large orientation difference, had an induction period of about 60 hours, while specimen 31 (fig. 10(b)), with a small angular difference, delayed nearly 5 months before the first rapid gliding began.

In sharp contrast with the test histories involving induction periods were others in which grain boundary gliding began immediately. As soon as the load had been applied, the portions of the reference pattern on the two sides of the grain boundary could be seen to be in relative motion. During the first quarter minute, or so, the gliding was too rapid to permit the making of readings, but had usually subsided sufficiently at 1/2 minute to allow measurements to be taken. Some of the samples tested at 300° C and all tested at higher temperatures exhibited this behavior.

The elongation of the grains themselves has been minimized in these tests by the low load employed (100 psi), so that the extension that occurred, after the first moment of rapid response, was often within the limits of the experimental error. As well as can be determined from the data at hand, however, the behavior of the individual grains was normal and altogether unresponsive to rate changes occurring at the grain boundary. For the most part, the individual crystal creep appears to have been in the second (steady-state) stage throughout the major part of the testing period; occasionally one of the crystals entered upon third-stage (accelerating) creep and ruptured. All such fractures were transcrystalline. Considerable distortion of the grain boundary sometime preceded such rupture (e.g., see fig. 20).

Influence of Orientation Difference between Grains

The effect of crystal orientation difference upon the ease of motion along the grain boundary has been studied at one temperature (300° C) and stress (100 psi) (see fig. 12). In order to express the orientation difference between the two crystals of each bicrystal, unambiguously, it is necessary to use two angles, α and β. It is possible, of course, to express the angular difference with one number, but this does not describe a unique relationship. The procedure in the present case has been to identify the pairs of a, b, and c axes of the two crystals as those pairs which would be brought to mutual registry by the smallest single rotation that would bring the two crystals to registry. In general, the original angular difference between members

of each of these pairs will have been different. The smallest angle between corresponding members of the three pairs, so identified, is designated α, the next smallest β. Thus, the α angle is always smaller than the β angle and all possible orientation differences will fall within a triangular plot limited by $\alpha = 0^\circ$, $\beta = 0^\circ$; $\alpha = 0^\circ$, $\beta = 45^\circ$; and $\alpha = 45^\circ$, $\beta = 45^\circ$.

At 144 hours the extent of gliding is indicated as a function of orientation difference, in figure 12, by the lengths of vertical lines each having an $\times$ at the top and a circle at the bottom. Where no displacement was found at 144 hours the $\times$ and circle are superimposed. Had a longer testing interval been selected for this comparison, much the same relative movements would have been reported, with the exception that some of the samples that here show no displacement would have passed their induction periods and have begun to move. The latter statement applies specifically to two points in the diagram of figure 12, namely, one at $\alpha = 15^\circ$, $\beta = 43^\circ$, which began to glide at 375 hours, and the other at $\alpha = 12^\circ$, $\beta = 15^\circ$, which began to glide at 3600 hours.

From the diagram of figure 12 it can be seen that the tendency to glide increases with the angular difference between the grains of the bicrystals, with the exception that no movement was ever observed on a twin boundary, although several specimens of this orientation type were tested. The twin relationship is, of course, one of perfect lattice matching, so that the exception can be avoided by saying that the rate of glide increased with the degree of mismatching of the two crystals at their mutual boundary.

Influence of Temperature

Although, as pointed out above, the instantaneous rate of grain boundary gliding is not necessarily altered by a change in temperature, the total movement, measured over a sufficient interval of time, increases with temperature rise. Two distinct modes of response to temperature change have been found, one corresponding to the immediate gliding that commonly occurs within the first minute after loading the specimen and the other corresponding to the long-time displacement.

In order to examine the temperature effects, a series of bicrystals of similar orientation difference ($\alpha = 38^\circ$ to 43°, $\beta = 43^\circ$ to 48°) was tested in creep at various temperatures from 200° to 650° C, using a stress of 100 psi (see fig. 13). All of these tested above 300° C exhibited rapid initial displacement, which had virtually terminated after 1/2 minute. If the magnitude of the immediate glide is plotted as a function of the temperature, a straight line is obtained (fig. 14).

It should be noted that this line intersects the zero displacement axis at about 350° C and that the samples tested at 300° and 200° C exhibited no immediate grain boundary movement, the one tested at 300° C having an induction interval.

If, on the other hand, the total glide displacement at 1 hour is related to the temperature, an altogether different relationship is found. A straight line is obtained in this case, by plotting the logarithm of the displacement (i.e., the logarithm of the rate) as a function of the reciprocal of the absolute temperature (see fig. 15). The slope of this line gives the activation energy of the process, which is computed to be 9700 calories per mole. The fact that the samples tested at 300° and 200° C do not lie upon this line should not be a matter of concern, because 1 hour is evidently too short a time, in terms of cycles of activity, to establish the rate at these low temperatures.

Influence of Applied Stress

In a brief study of the effects of stress upon grain boundary glide, specimens of similar orientation difference have been tested over a temperature range with applied loads of 1, 10, 50, and 100 psi. The procedure for the lower loads was to look for grain boundary gliding at an established temperature, for a period of 48 hours, or more, and, if no motion was detected, to raise the temperature 50° or 100° C for another period of observation. The bars stressed at 1 and 10 psi were tested at temperature intervals all the way to actual melting without any indication of displacement at the grain boundary. Gliding began in the sample stressed 50 psi when the temperature was changed from 400° to 500° C (see fig. 16). At 100 psi displacement along the grain boundary occurred in comparable samples at 300° C and above, while none was ever found at 200° C.

Clearly, there is for each temperature a minimum stress below which the grain boundary remains inactive. In other words, there is a threshold stress for grain boundary gliding. It may be noted, as should be anticipated, that the magnitude of both the initial and subsequent gliding at 500° C and 50 psi (fig. 16) is considerably less than that at 500° C and 100 psi (fig. 13).

Structure of Operated Grain Boundary

Microscopic studies suggest that the motion of grain boundary glide is distributed over a zone of substantial thickness. The intensity of motion appears maximum along the original grain boundary trace and

decreases rapidly with distance from this center. The evidence is of several kinds. Fortuitously, the transparent oxide film that covers the specimen behaves as a "stress coat," developing cracks that lie approximately normal to the axis of elongation of the metal (see fig. 17). In the photograph in figure 17, as in others, it is apparent that the nearly horizontal stress cracks curve rather sharply toward the grain boundary as they approach it. This means that the metal adjacent to the boundary has moved in the direction of the displacement and that the degree of this motion has been greatest next to the boundary.

Another kind of evidence was obtained by examination of the protruding grain boundary ledge of the sample pictured in figure 7. This ledge is shown in detail in figures 18 and 19, the first of which exhibits the lip as seen from the front of the sample and the second shows the surface of the lip as viewed from one side of the specimen. In the latter picture the surface of the lip appears as a mottled light and black band. The light areas are islands of the original oxide film and the black areas represent clean metal that exhibits no surface detail whatever; that is, the exposed metal is mirrorlike. Above and below the lip the usual stress cracks can be seen in the surface oxide. It is reasoned that, had the two grains slipped apart upon a single plane, none of the original oxide film would have been found adhering to its face. Hence, the lip must have developed by the expansion of a finite area of original external surface of the sample, as would happen if a definite thickness of metal had participated in gliding. Yet further evidence of the operation of a zone of appreciable thickness is obtained from the fact that the edges of the lip are not sharp where they join the original side surface of the specimen.

It has been shown by X-ray diffraction studies that the metal along the boundary has been broken up into minute crystallites whose orientations are widely dispersed from that of the original crystal. This disorganization is much more severe than that in the grains themselves at a distance from the boundary (cf. figs. 22(a) and 22(b)).

By a fortunate chance, another sample tested at 400° C had in one of its crystals a system of slip planes exactly parallel to the grain boundary. This specimen extended rapidly both by grain boundary gliding and by very extensive slip, so that broad lips were produced, both at the grain boundary and where slip bands intersected the edge of the specimen (see figs. 20 and 21). The Laue photograms of figures 22(a) and 22(b) were, in fact, made from this sample. Another photogram of the surface of one of the lips produced by slip is given in figure 22(c) and a fourth exposure (fig. 22(d)) of the same crystal prior to testing is included for comparison. Although displacement upon the slip band has, in this instance, been greater than upon the grain boundary, the disorganization of the crystal at the slip band is minor compared with

that at the grain boundary; indeed, there is no more disorganization at the slip band (fig. 22(c)) than in the body of the crystal (fig. 22(a)). The protruding lips of the slip bands (fig. 21) can be seen to be composed of many smaller steps, doubtless representing individual slip planes. This stepped surface is to be contrasted with the smooth surface of the grain boundary.

Structural Changes Within Grains

Several of the specimens, which were tested at the higher temperatures and which had experienced large elongation leading to rupture, exhibited, in their more severely deformed regions, an interesting degeneration of the reference pattern (see fig. 23). The squares of the pattern were elongated into rectangles and the horizontal margins of the rectangles had become serrated. Upon close examination, it can be seen that the terrace that originally formed the horizontal boundary of each square has broken into segments that have moved with respect to one another parallel to the tension axis, that is, perpendicular to the front of the terrace. At 600° C the segments that are formed (fig. 23(a)) are distinctly coarser than those formed at 500° C (fig. 23(b)). No slip system in either of these crystals lies closer to the tension axis than 15°.

A Laue photogram taken in the same neighborhood, on the specimen tested at 600° C, is reproduced in figure 24. This shows that the original crystal has been broken into blocks (or fibers) that have rotated a few degrees about the octahedral axis of the crystal. In more than a dozen cases examined in this way the center of rotation of the blocks appeared always to be about an octahedral pole. It is not clear whether this observation is compatible with the metallographic effects cited above.

DISCUSSION

The foregoing results make it clear that grain boundary gliding is a far from simple process. Provisionally, it may be concluded that the finding of two distinct temperature relationships of the glide rate denotes the cooperative action of at least two different mechanisms. The first rate process has the characteristics of a linear temperature response and a threshold energy requirement. The second appears to be of the chemical-rate type. There may be two such processes, because those portions of the time-displacement curves which exhibit chemical-rate behavior include both the slackening of the glide rate, as by the development of an impedance, and a reactivation of the glide process at intervals.

The first process is reminiscent of interfacial friction in several respects. Corresponding to the threshold force required to overcome static friction is the threshold force of gliding. At the energy level required to start the process, the immediate glide rate is relatively rapid, as in the transition from static to sliding friction. The material adjoining the interface is violently disturbed in both cases. Whether both processes have the same temperature function is open to question, because the available measurements of friction against temperature are inconclusive. Some of the most trustworthy observations (reference 9), however, indicate both negative and positive deviations from linearity, suggesting that grain boundary glide and sliding friction may be alike in their temperature response.

Perhaps the most obvious parallel to the slackening and stopping of glide, in the latter part of each cycle, is to be found in slip behavior, where motion on each plane ceases after a certain amount of displacement has occurred. Otherwise, however, the similarity between slip and gliding, if such exists, is not apparent in the present results. Slip is not accompanied by anything like the same degree of disorganization of the adjoining crystalline matter. Slip occurs on one plane at a time, whereas gliding appears to involve a continuum of motion, at a varying rate, over a finite thickness of metal. It seems rather more likely, therefore, that the impedance to gliding is associated somehow with the crystallographic disarrangement of the metal adjacent to the boundary.

Having experimentally eliminated external disturbances as a cause for the reactivation of gliding, it is concluded that the new cycle must be started by an accumulation of energy resulting from some continuing process within the metal. The most evident activity within the specimen is the continued deformation of the individual crystals in creep. Since it is known that crystals of different orientation creep at different rates, it is to be expected that a stress will develop at the grain boundary as a result of a growing difference in the lateral dimensions of the grains. The rate of accumulation of this stress energy and, hence, the frequency of reactivation of gliding may be expected to follow the chemical-rate law, which is obeyed by the creep process itself.

Another kind of change, which might be progressive with time irrespective of the continuation of motion at the boundary, is the gradual conversion of deformed crystalline matter to the disarranged crystallites that were observed in the boundary at the end of the test. Conceivably, their growth might decrease the impedance to gliding and permit the resumption of motion. This also would be a chemical-rate process, if it depended upon the rate of crystallite growth.

The variation of the glide rate with difference in grain orientation appears at least crudely in accord with the assumption that the effect

is related directly to the grain boundary energy. All of the several determinations now on record, for example, Greenough and King (reference 10), show the energy starting at zero, at zero angular difference, and attaining a maximum somewhere near 45°. A return to zero energy at the twin relationship is commonly recognized. There is, however, one noteworthy difference between the angular variation of the grain boundary energy and the angular variation of grain boundary gliding at 100 psi and 300° C, namely, that no gliding occurs until an angular difference in excess of 10° (sometimes more) exists. This discrepancy is doubtless to be associated with the induction period which becomes very long at low angles. Presumably, all of the samples would have exhibited some gliding after sufficient time, but, judging from the trend in the relationship between the induction interval and the orientation difference, the times for some of the samples to show movement might well be of the order of years.

In summary, it is proposed that gliding proceeds under the influence of two constant sources of energy, the resolved shear stress and the energy of crystal mismatching, plus an energy, resulting from differential creep rates, that varies cyclically with time, and minus an opposing energy, appearing as an impedance to gliding, that also varies with time. When the positive total exceeds the threshold requirement, corresponding to the resistance of static friction, gliding begins and, since the resistance of the dynamic friction is less, proceeds at a high initial rate. The subsequent decrease in the rate is thought of as resulting from the exhaustion of the energy of the differential creep rates plus the growth of an impedance. With gliding at a near standstill the energy of the differential creep rates may again accumulate and reactivate gliding or crystallite growth may relax the impedance. An induction period is expected when the sources of constant energy are alone inadequate to overcome the static friction. It is suggested that the linear temperature function of the initial gliding rate is a frictional characteristic that is soon masked by chemical-rate processes that determine the magnitude of the rate cycles.

Considering the complexity of the process, it is difficult to assign the measured activation energy to any single mechanism. Quite possibly this energy represents a combination of two or more cooperative processes. At any rate, the value (9700 cal/mole) appears rather low to be associated with any single simple process, such as grain boundary self-diffusion.

CONCLUSIONS

In order to study the gliding of one metal crystal with respect to another along their mutual grain boundary, creep tests at 200° to 650° C

and from 1 to 100 psi were conducted on bicrystals of pure aluminum. The experimental observations indicate that:

1. The magnitude of grain boundary gliding is related to the intensity of the shear stress resolved into the plane of the boundary.

2. The direction of grain boundary gliding is independent of the crystals forming the boundary and is determined only by the direction of maximum resolved shear stress in the plane of the boundary.

3. The rate of gliding is cyclic.

4. The greater the mismatch between the crystals forming the boundary, the greater the glide displacement in unit time.

5. Within certain ranges of temperature (about 300° C), grain orientation difference (low), and possibly stress (low) an induction period precedes the onset of gliding.

6. The initial gliding rate varies directly with the temperature.

7. The logarithm of the subsequent gliding rate varies directly with the reciprocal of the absolute temperature; that is, a chemical-rate law applies.

8. The activation energy of long-time gliding is 9700 calories per mole.

9. There is a threshold stress, below which grain boundary gliding does not occur at any specific temperature.

10. Grain boundary gliding occurs as a motion of varying intensity that operates within a relatively thick zone of metal upon both sides of the boundary trace.

11. The disorganization of the crystals is more severe upon the operated grain boundary than upon operated slip planes, or elsewhere within the crystals that have participated in creep.

12. Slipless flow, within the grains, appears to be accompanied by the degeneration of the crystals into blocks or fibers that move relative to one another in a direction parallel to the axis of stress.

13. The crystals degenerate into blocks or fibers that rotate about an octahedral axis during creep. There is X-ray evidence of this degeneration. It is not clear whether this observation is compatible with conclusion 12 above.

Carnegie Institute of Technology
Pittsburgh, Pa., December 21, 1951

REFERENCES

1. Howe, H. M.: The Patients of Copper and Silver as Affected by Annealing. Trans. Am. Inst. Min. Eng., vol. 13, 1885, pp. 646-656.

2. Andrade, E. N. daC: The Flow of Metals under Large Constant Stresses. Proc. Roy. Soc. (London) ser. A., vol. 90, no. 619, July 1, 1914, pp. 329-342.

3. Clark, C. L., and White, A. E.: Properties of Non-Ferrous Alloys at Elevated Temperatures. Trans. A.S.M.E. vol. 53, no. 8, May-Aug. 1931, pp. 183-189.

4. Moore, H. F., Betty, B. B., and Dollins, C. W.: Investigation of Creep and Fracture of Lead and Lead Alloys for Cable Sheathing. Bull. No. 306, Eng. Exp. Station, Univ. of Ill., vol. 35, no. 102, Aug. 19, 1938.

5. Wood, W. A., Wilms, G. R., and Rachinger, W. A.: Three Basic Stages in the Mechanism of Deformation of Metals at Different Temperatures and Strain Rates. The Jour. Inst. Metals, vol. 79, pt. 3, May 1951, pp. 159-172.

6. Betteridge, W., and Franklin, A. W.: An Investigation of the Structural Changes Accompanying Creep in Tin-Antimony Alloys. The Jour. Inst. Metals, vol. 80, pt. 3, Nov. 1951, pp. 147-150.

7. Barrett, Charles S.: Structure of Metals. First ed., McGraw-Hill Book Co., Inc., 1943, pp. 173-177.

8. Greninger, A. B.: Determination of Orientations of Metallic Crystals by Means of Back-Reflection Laue Photographs. Trans. Am. Inst. Min. and Met. Eng., vol. 117, 1935, pp. 61-71.

9. Bowden, F. P., and Hughes, T. P.: The Friction of Clean Metals and the Influence of Absorbed Gases. The Temperature Coefficient of Friction. Proc. Roy. Soc. (London), ser. A., vol. 172, no. 949, Aug. 1939, pp. 263-279.

10. Greenough, A. P., and King, R.: Grain Boundary Energies in Silver. The Jour. Inst. Metals, vol. 79, pt. 6, Aug. 1951, pp. 415-428.

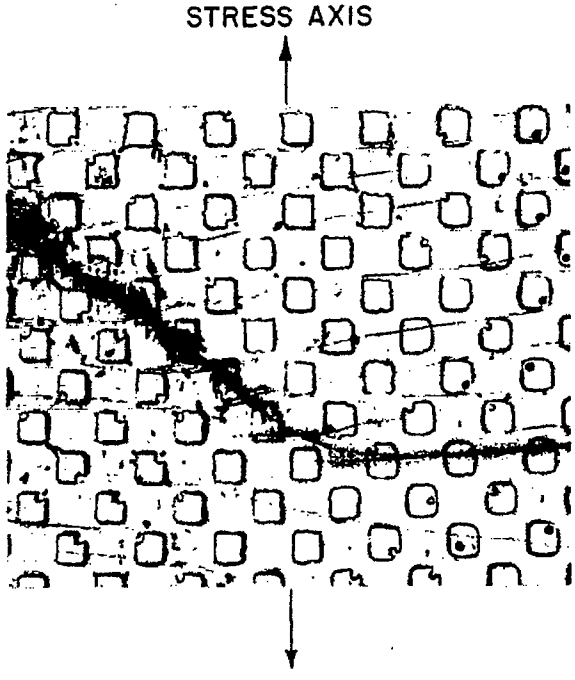
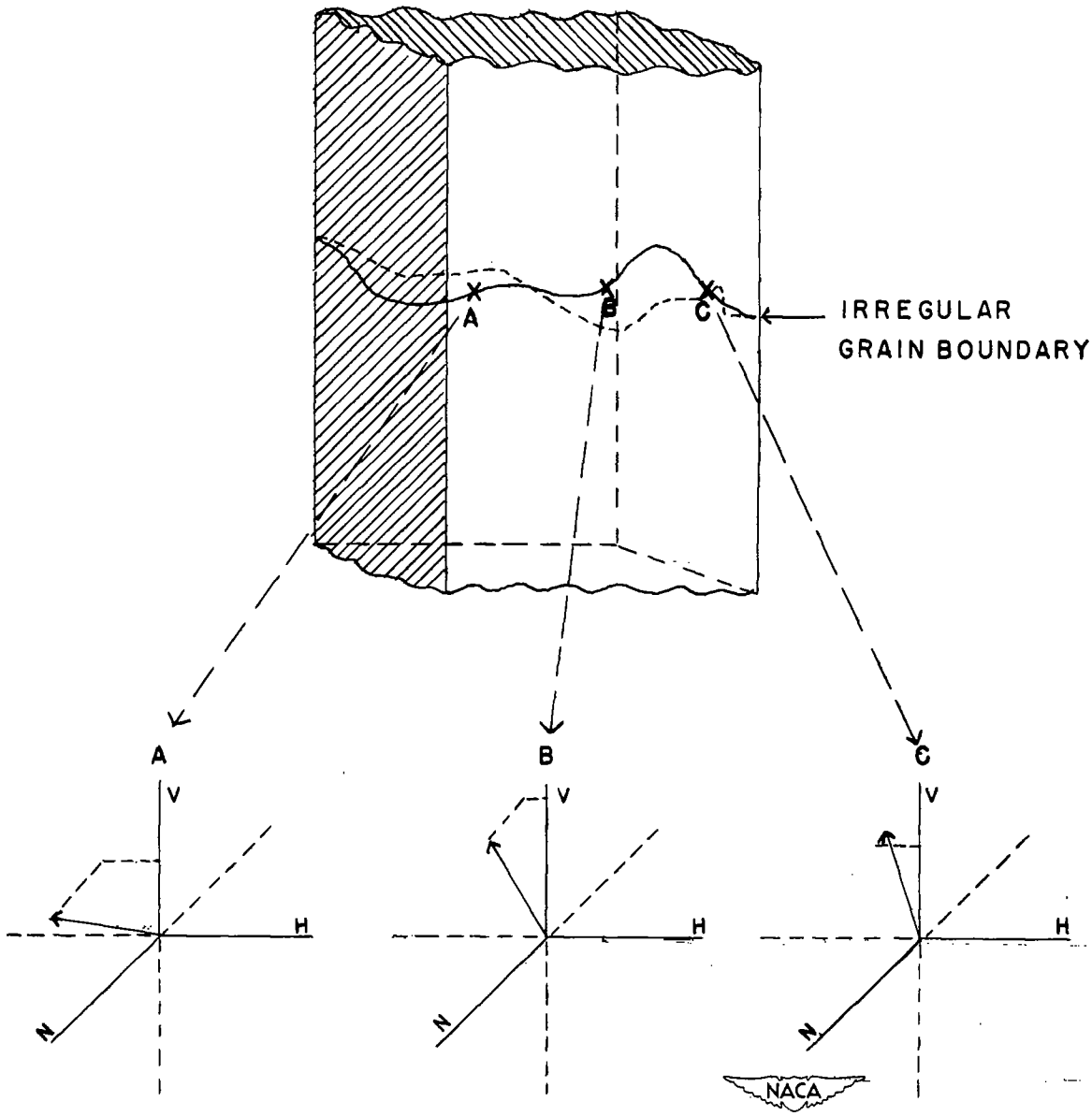


Figure 1.- Varying extent of grain boundary glide along a bent grain boundary in coarse-grained aluminum. Temperature, 384° C; stress, 120 psi; orientation difference, 40°. X35.



V-(VERTICAL) POSITIVE FOR EXTENSION
H-(HORIZONTAL) POSITIVE FOR MOVEMENT OF UPPER GRAIN TO THE RIGHT
N-(NORMAL) POSITIVE FOR UPPER GRAIN MOVING FORWARD

Figure 2.- Change in direction of displacement from point to point along an irregular boundary between a pair of grains.

Figure 3.- Coarse-grained aluminum plate made by alternate straining and annealing. Typical bicrystal specimen shown laid out (black rectangle) for cutting. Tucker's etch. Full size.

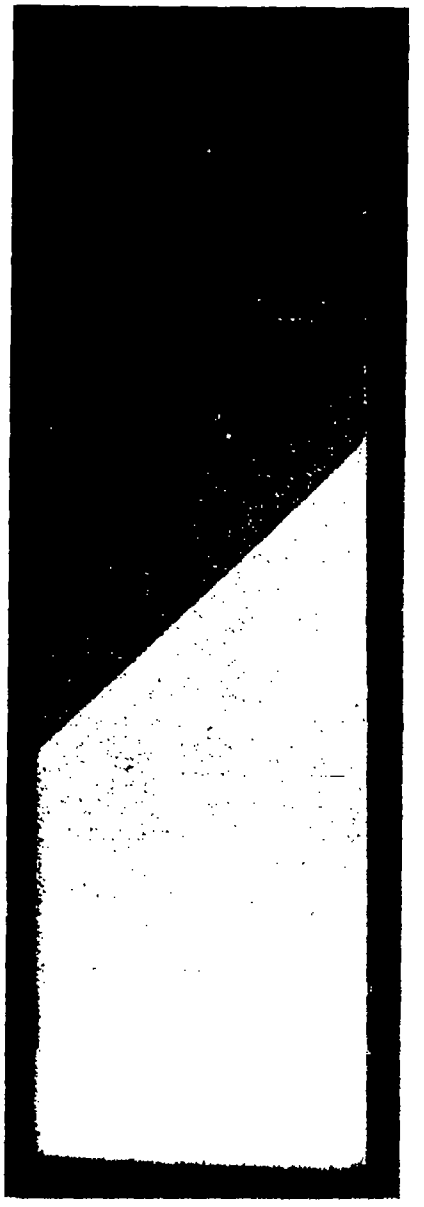


Figure 4.- Typical bicrystal cut, with grain boundary at 45°, from coarse-grained aluminum plate. Tucker's etch. X4.

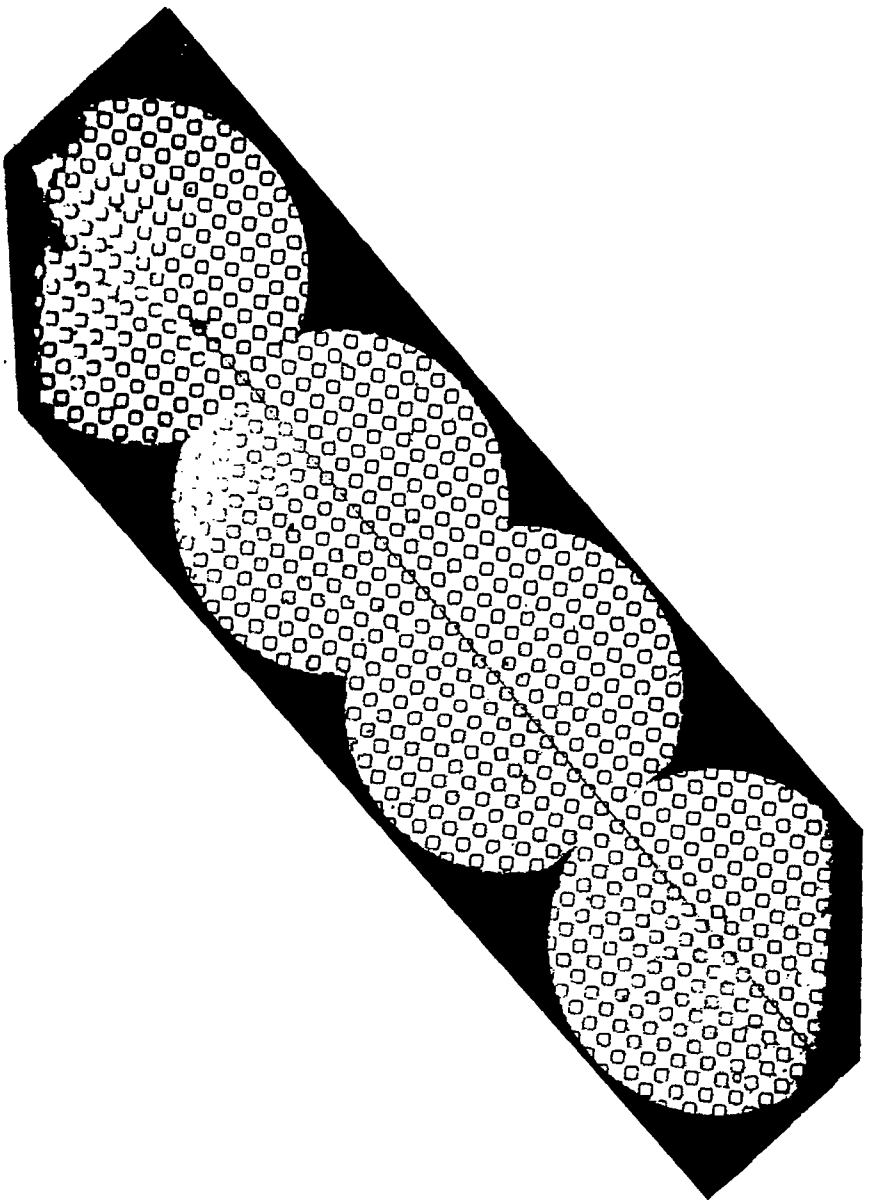


Figure 5.- Typical appearance of photoengraved reference pattern superimposed upon a grain boundary (fine straight line) in an aluminum bicrystal. X25.

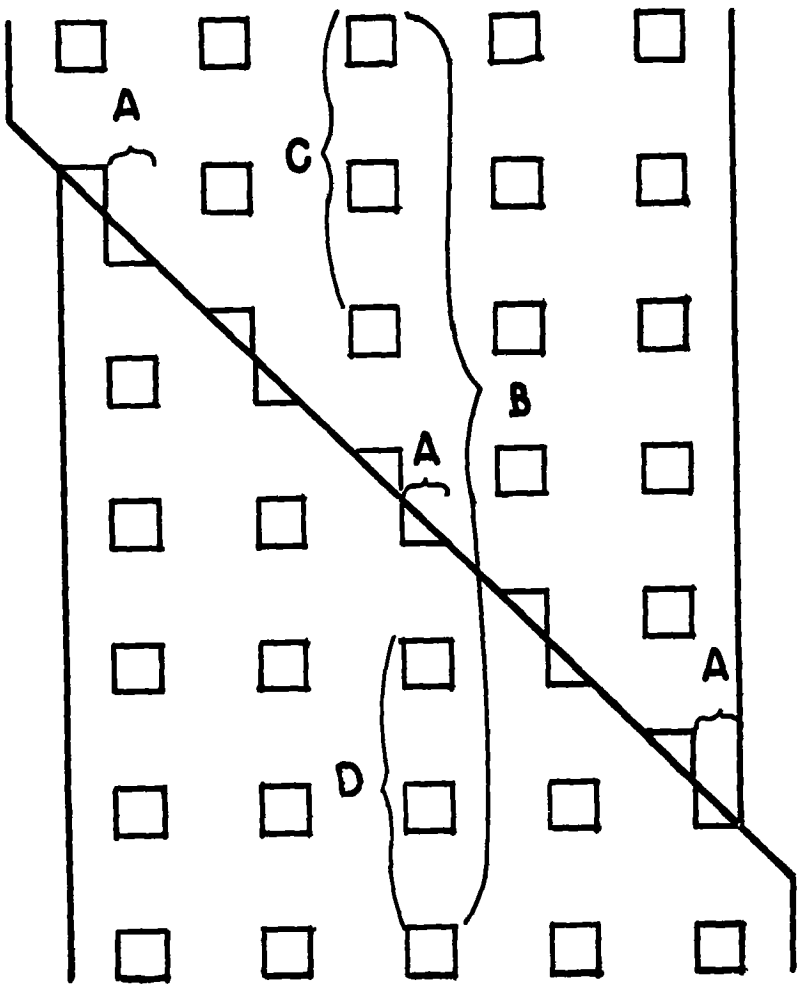


Figure 6.- General plan of measurements. Displacement was measured at points A, averaged, and translated into displacement parallel to boundary; elongation within each crystal was measured over lengths C and D; and total extension was measured over length B.

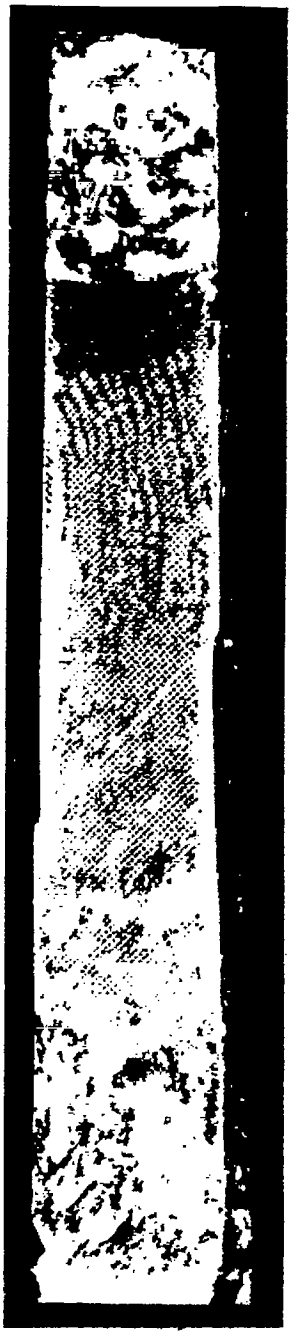


Figure 7.- Typical bicrystal specimen after testing showing displacement of the two grains at grain boundary. The fine "calico" design is the reference pattern; white spots at top and bottom are tooth marks made by specimen grips. Time, 1500 hours; temperature, 300° C; stress, 100 psi; $\alpha = 33^{\circ}$, $\beta = 38^{\circ}$. $X3\frac{1}{2}$.

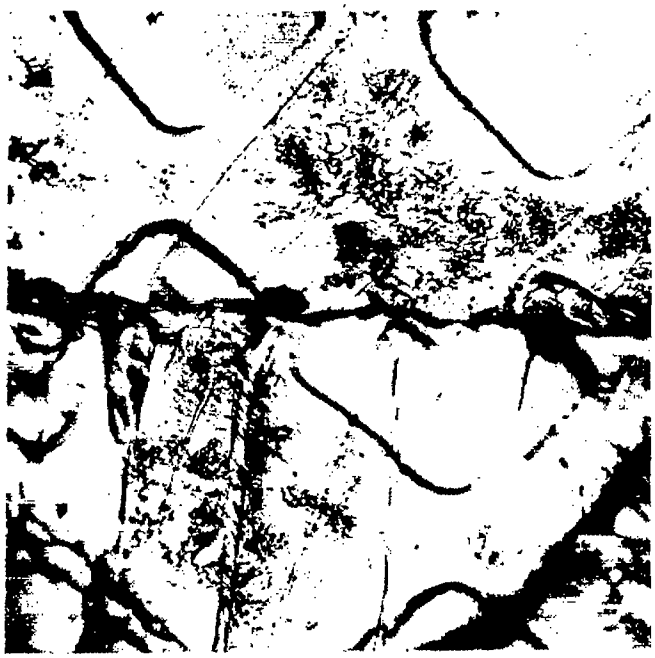


Figure 8.- Gliding along grain boundary shown by displacement of portions of reference squares that lie astride the boundary. Vertical and diagonal streaks are stress cracks in surface film of aluminum oxide; "flowery" patterns are tarnish. Time, 1600 hours; temperature, 300° C; stress, 100 psi; $\alpha = 27^{\circ}$, $\beta = 35^{\circ}$. X250.

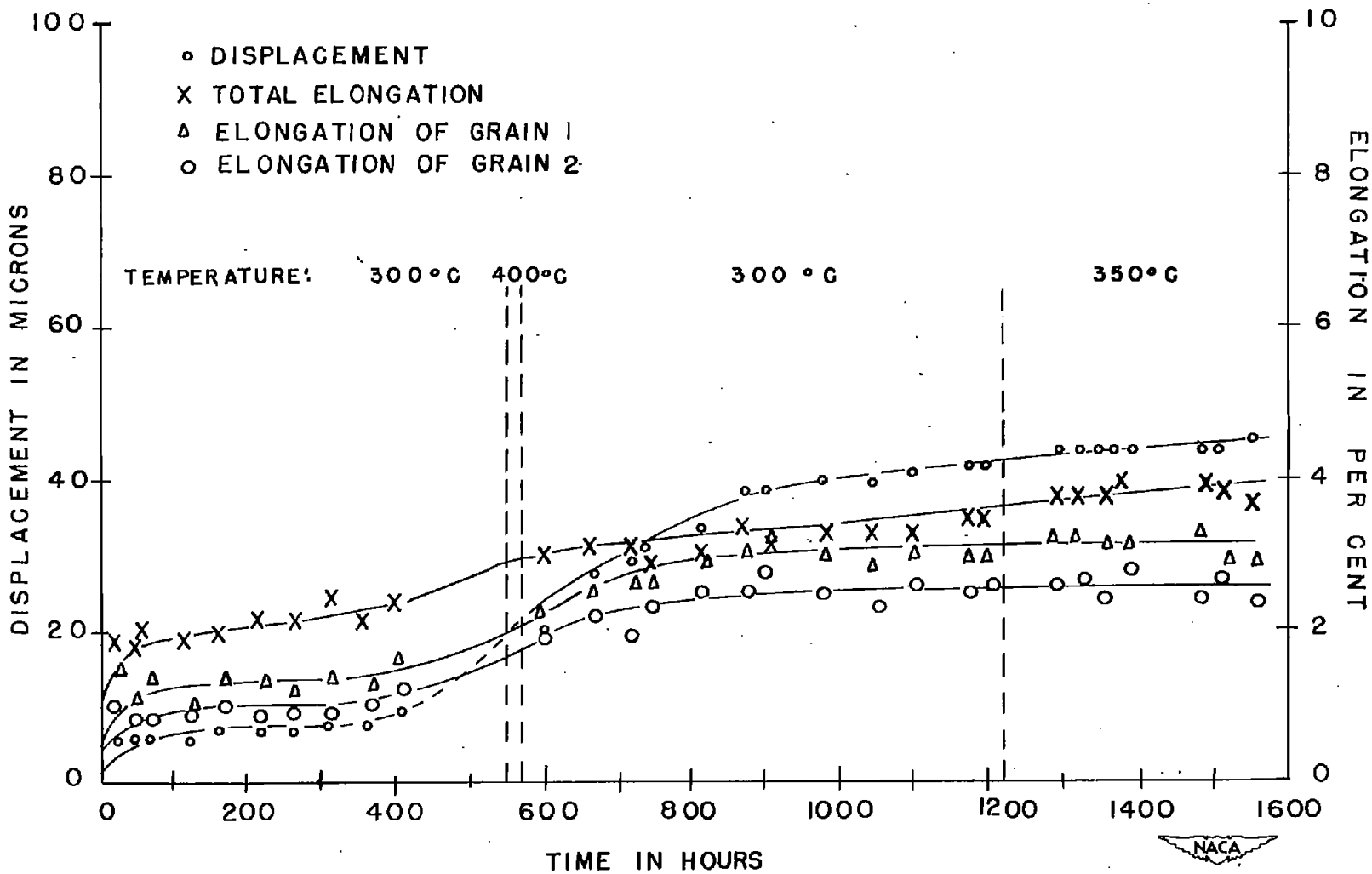
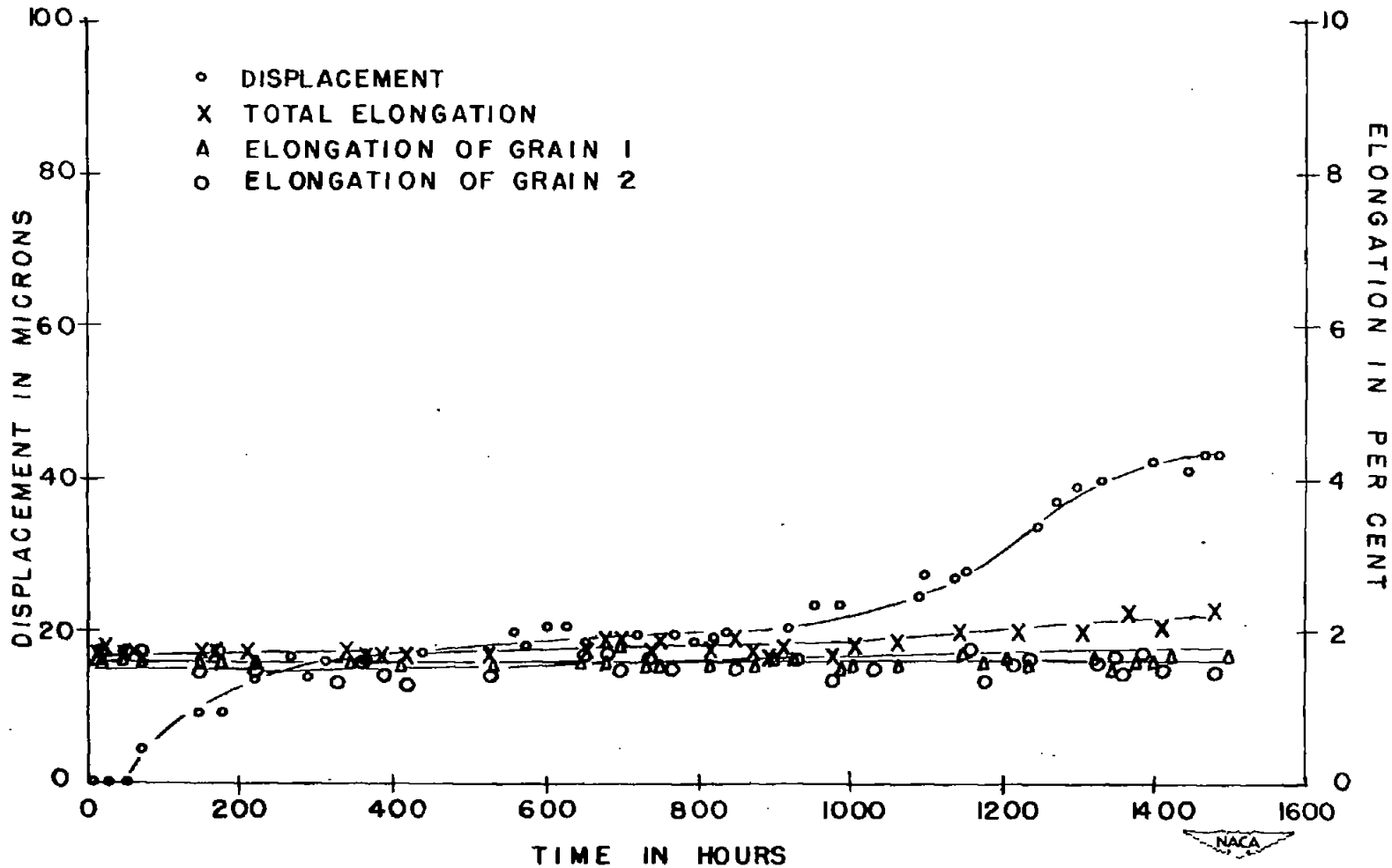
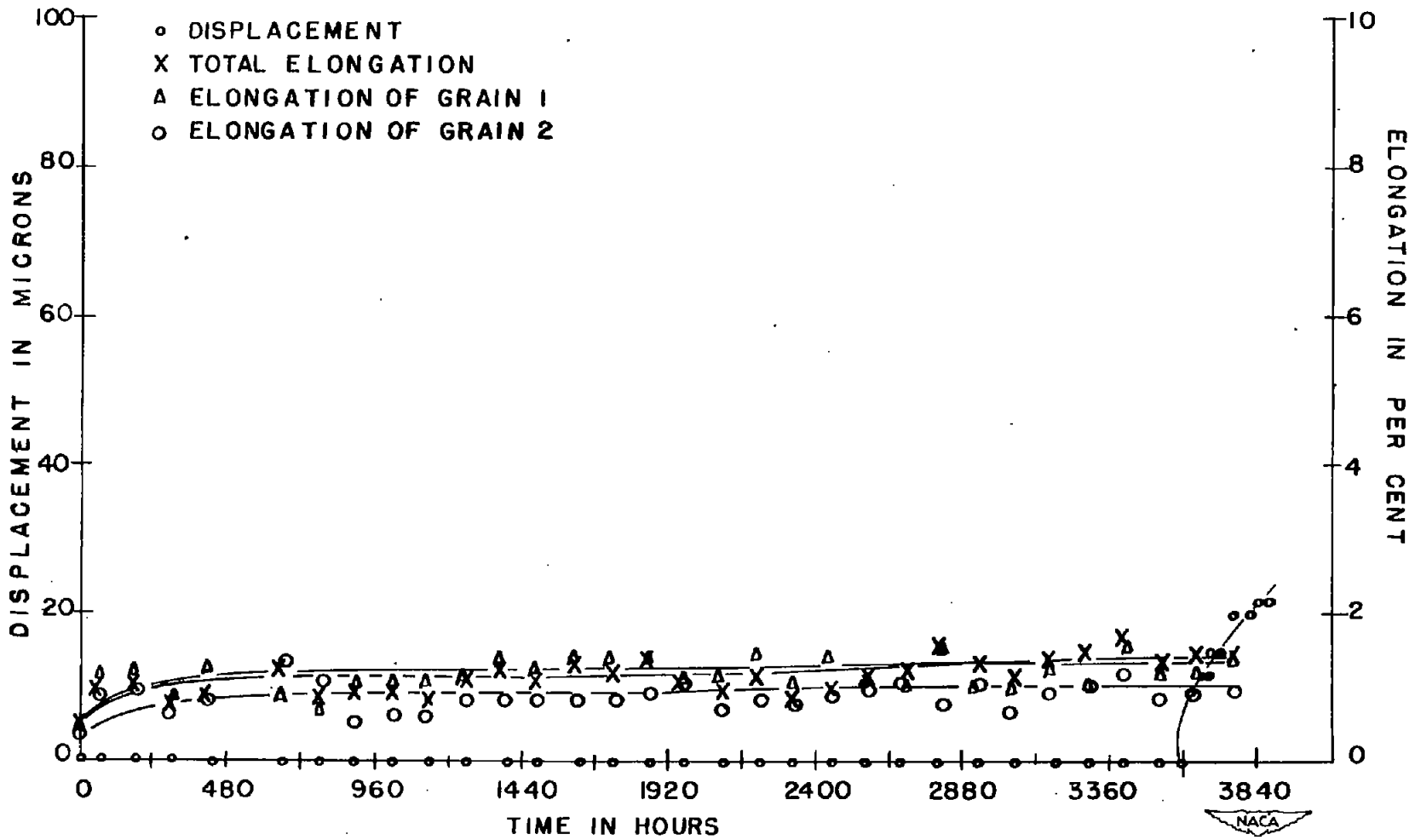


Figure 9.- Rate of grain boundary displacement and of elongation at various temperatures and a stress of 100 psi. Specimen 12; $\alpha = 18^{\circ}$, $\beta = 20^{\circ}$.



(a) Specimen 34; $\alpha = 27^{\circ}$, $\beta = 31^{\circ}$.

Figure 10.- Rate of grain boundary displacement and of elongation at 300° C and 100 psi.



(b) Specimen 31; $\alpha = 12^{\circ}$, $\beta = 15^{\circ}$.

Figure 10.- Concluded.

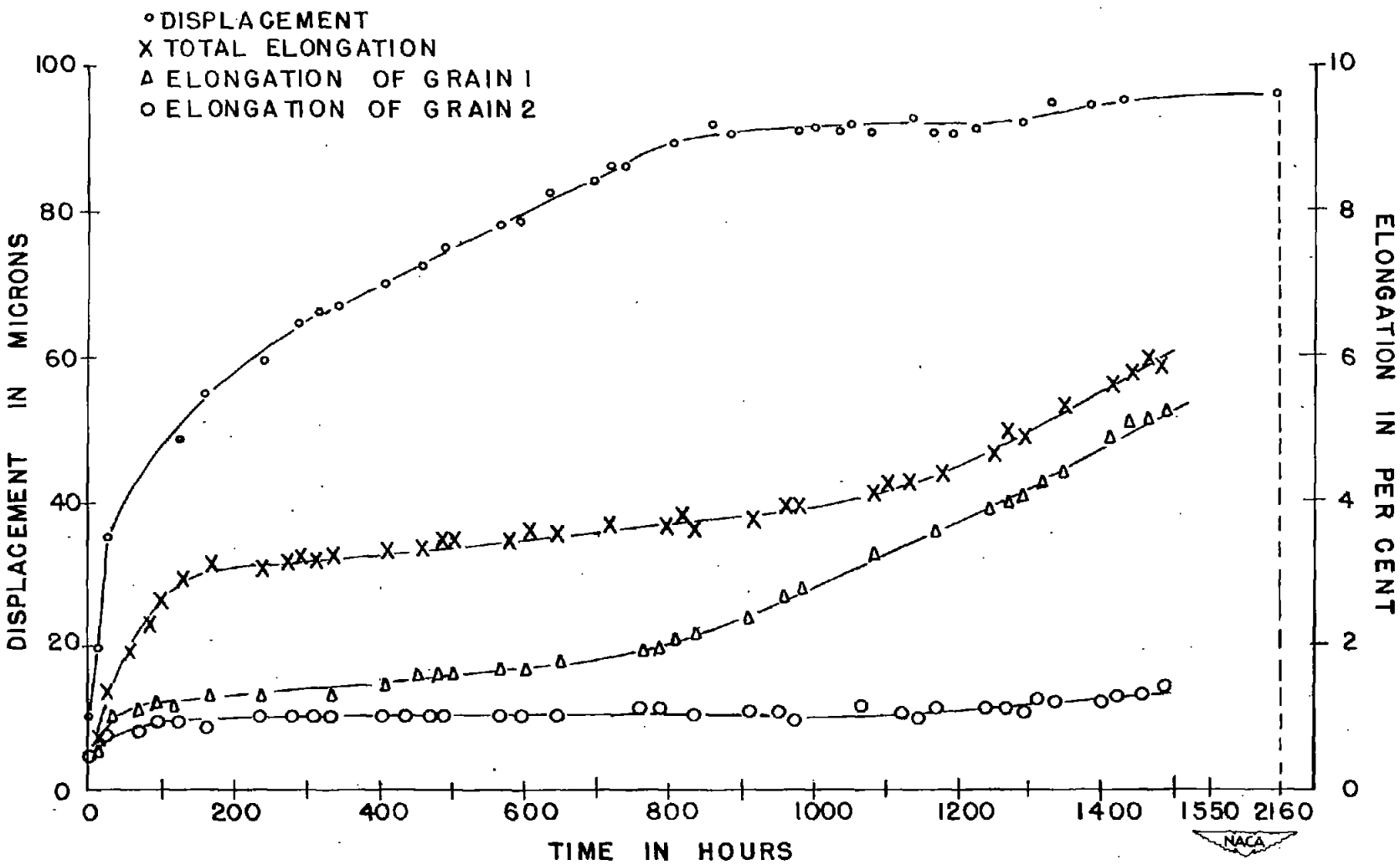


Figure 11.- Rate of grain boundary displacement and of elongation at 400° C and 100 psi. Specimen 24; $\alpha = 43^{\circ}$, $\beta = 48^{\circ}$.

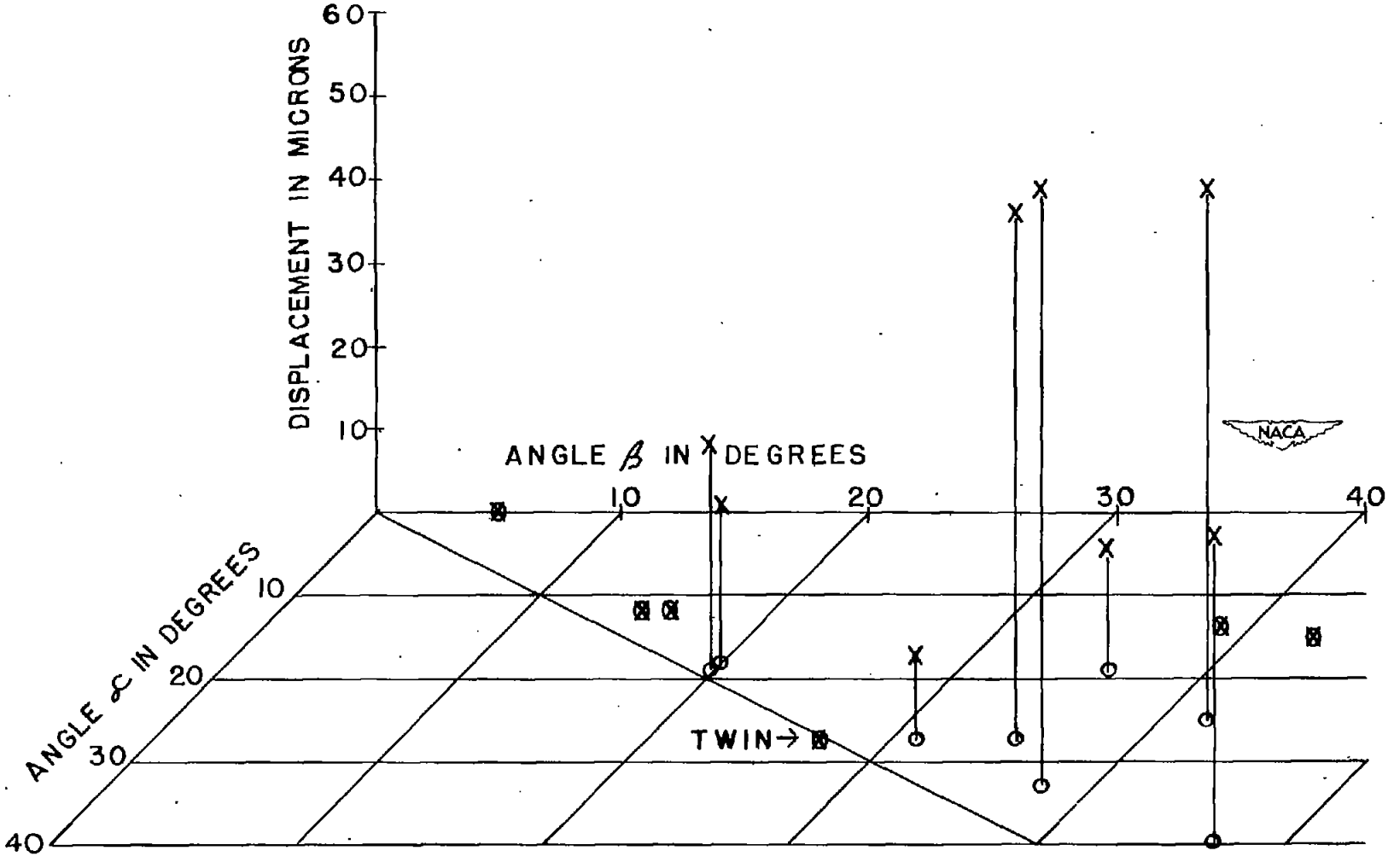


Figure 12.- Effect of orientation difference on grain boundary displacement. Time, 144 hours; temperature, 300° C; stress, 100 psi.

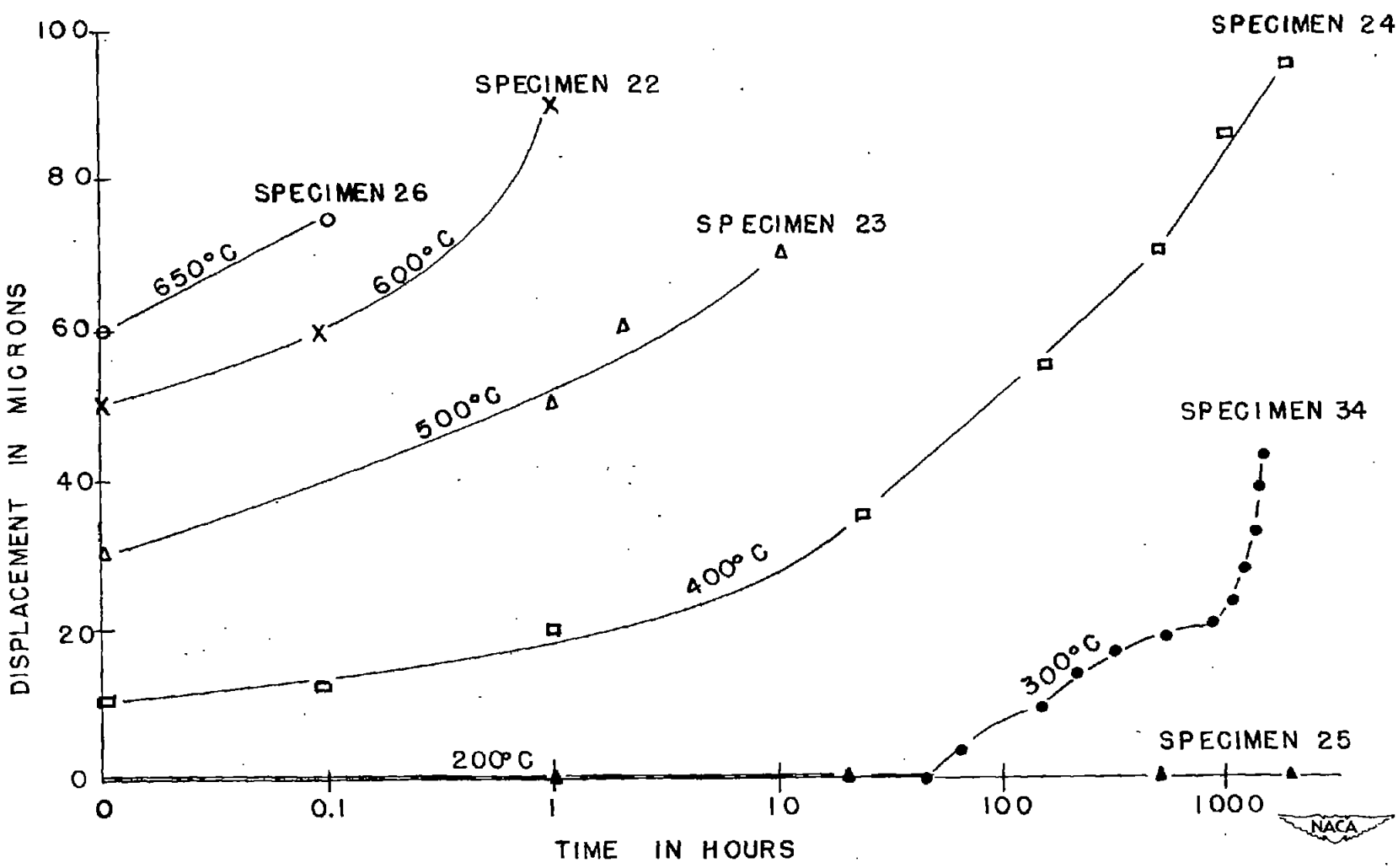


Figure 13.- Effect of temperature on grain boundary displacement. Stress, 100 psi; $\alpha = 38^{\circ}$ to 43°, $\beta = 43^{\circ}$ to 48°.

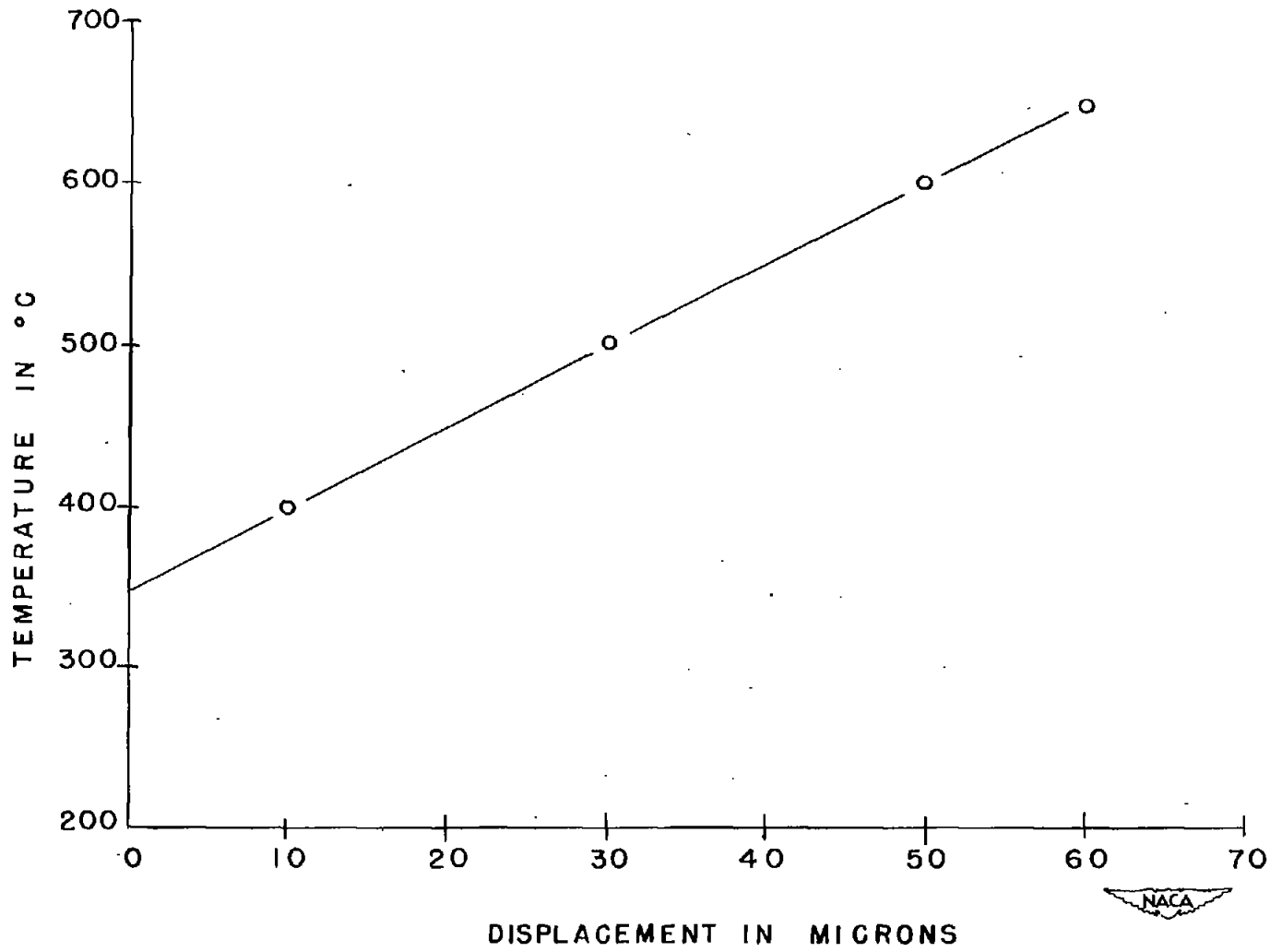


Figure 14.- Effect of temperature on magnitude of initial grain boundary displacement. Time, 0 to 1/2 minute; stress, 100 psi; $\alpha = 38^{\circ}$ to 43°, $\beta = 43^{\circ}$ to 48°.

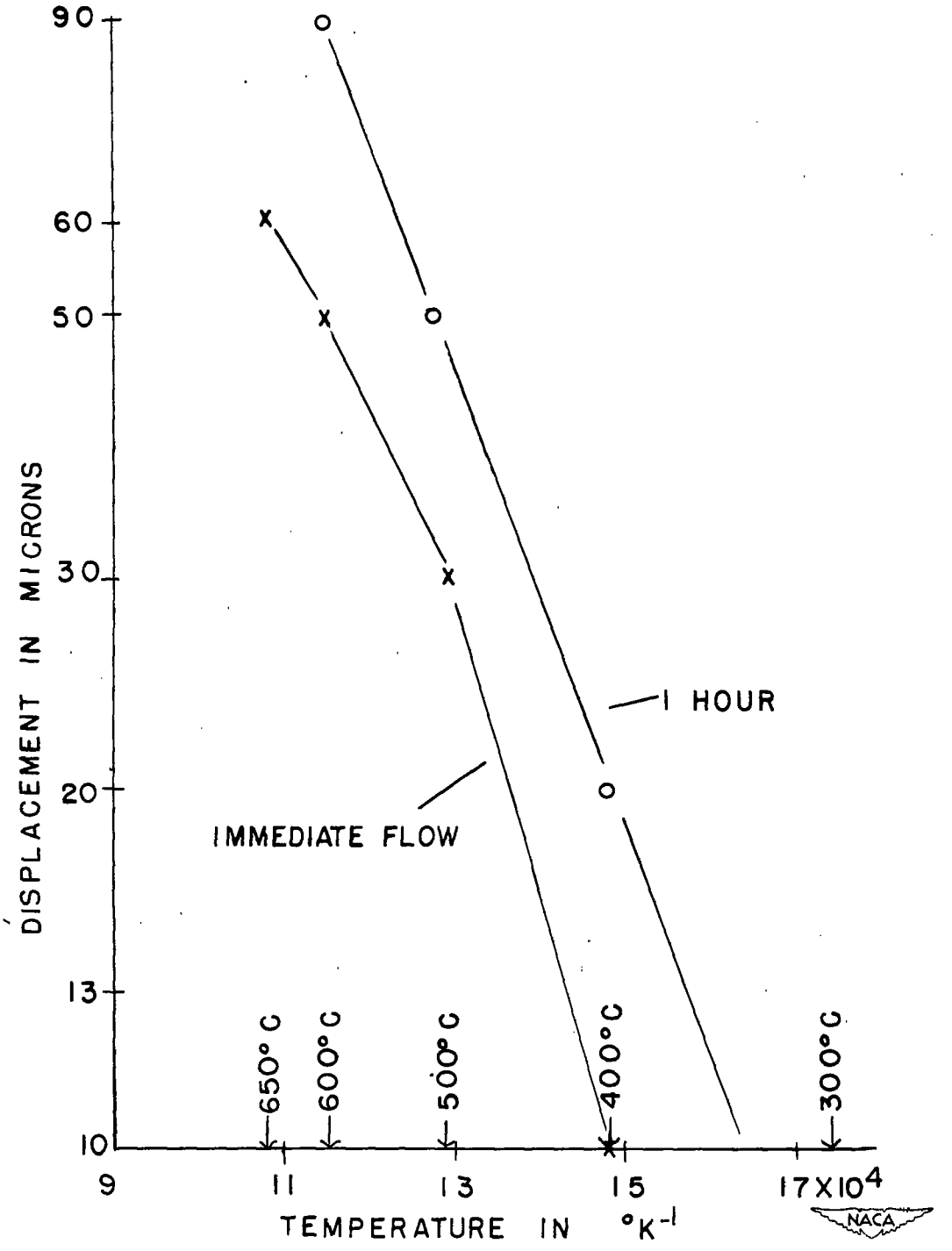


Figure 15.- Comparison of effects of temperature on initial displacement and on total displacement after 1 hour. Stress, 100 psi; $\alpha = 38^{\circ}$ to 43°, $\beta = 43^{\circ}$ to 48°.

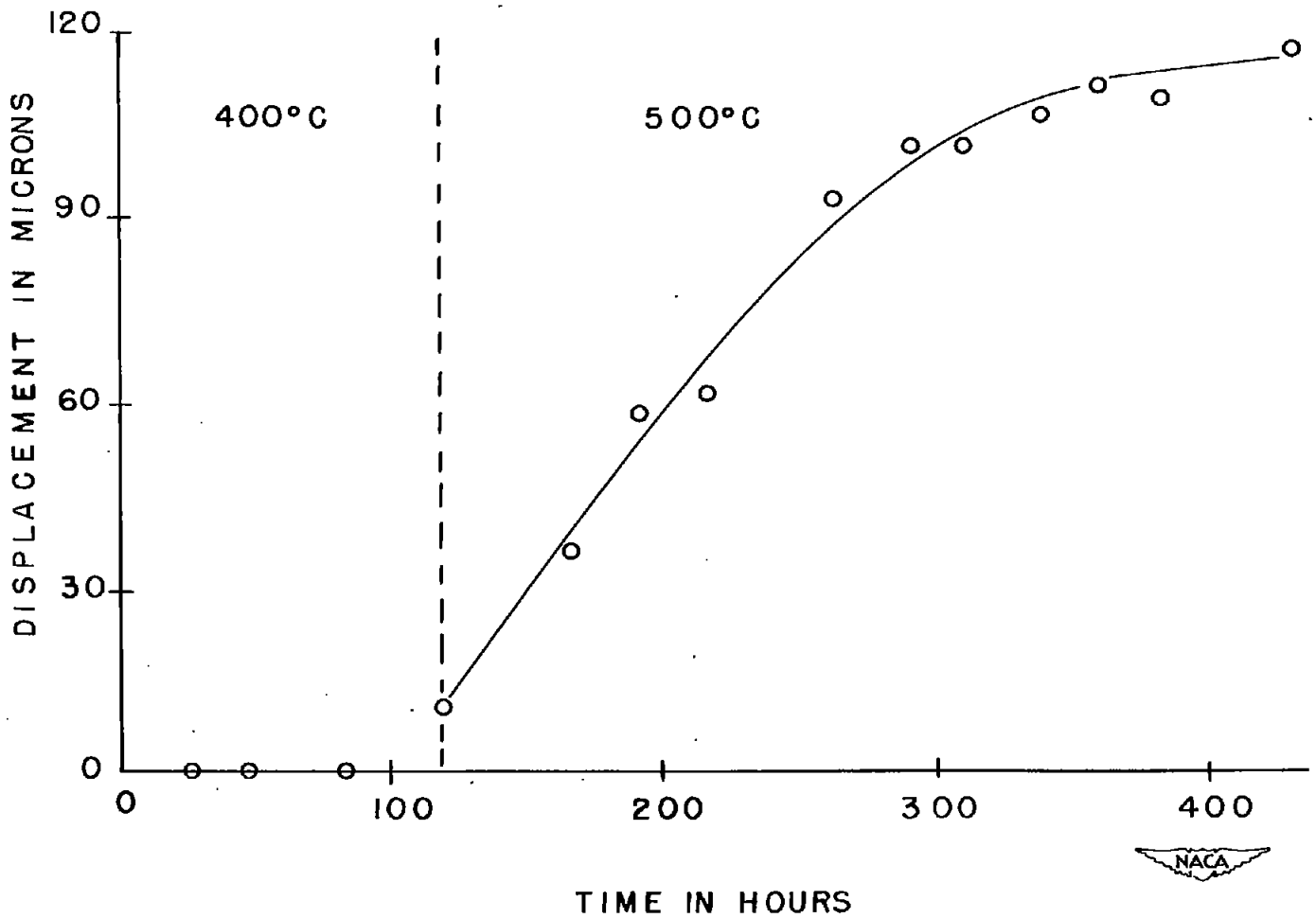


Figure 16.- Effect on grain boundary displacement of an applied stress of 50 psi over a range of temperature. Specimen 19; $\alpha = 40^{\circ}$, $\beta = 45^{\circ}$.

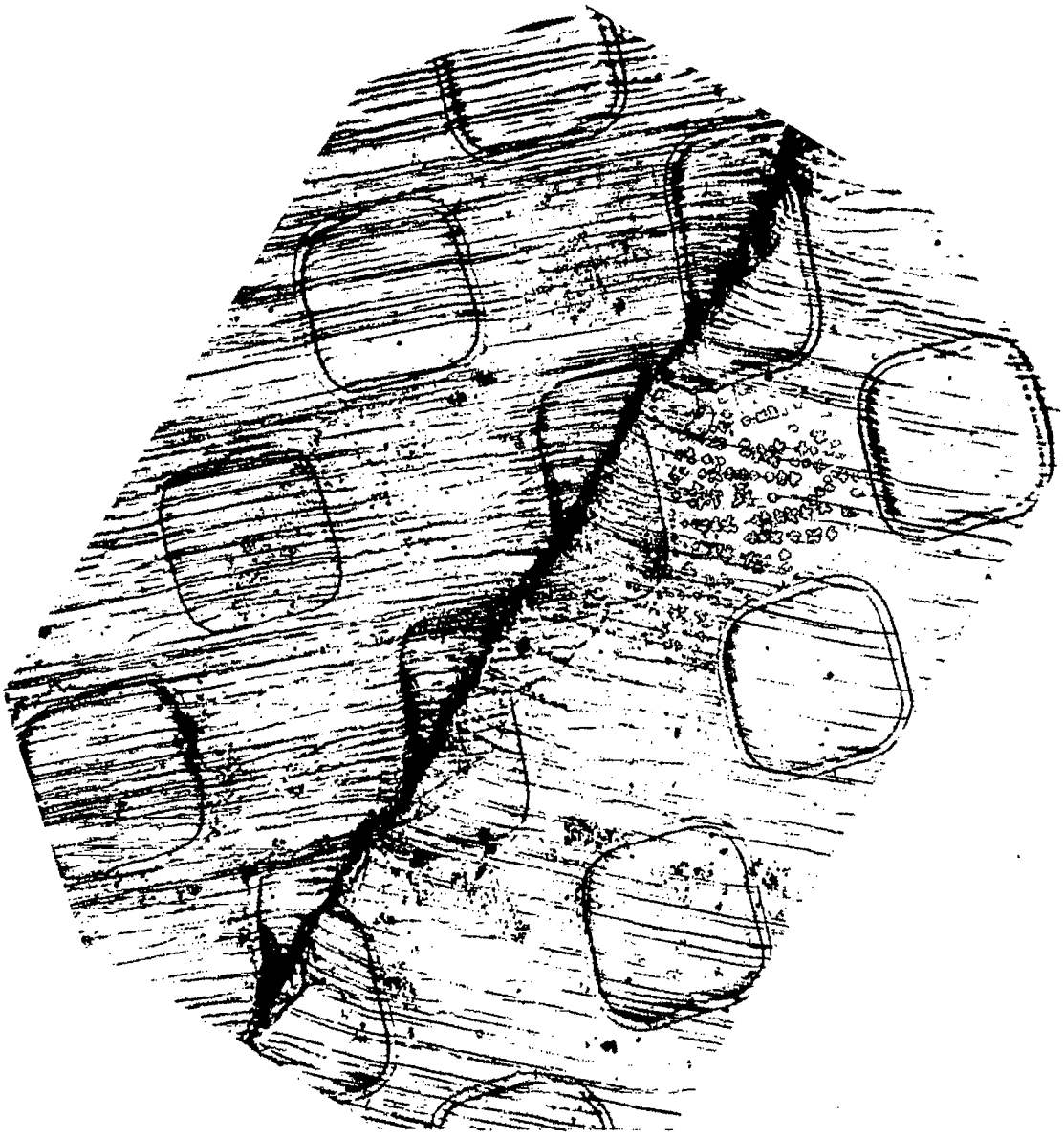


Figure 17.- Detail of displacement in a tested specimen. Fine lines are stress cracks in surface oxide skin. Time, 250 hours; temperature, 300° C; stress, 100 psi; $\alpha = 25^{\circ}$, $\beta = 42^{\circ}$. X200.

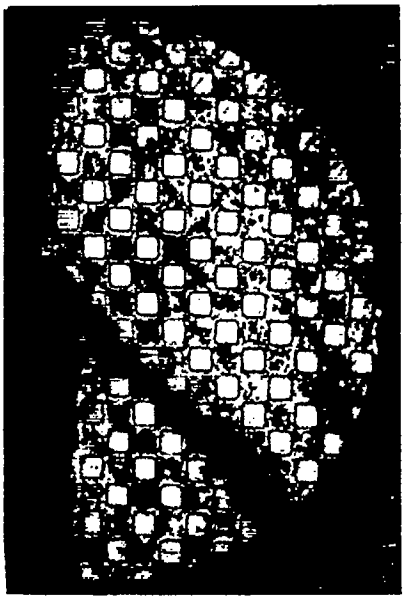


Figure 18.- Detail of specimen in figure 7 showing displacement at grain boundary at one side of specimen producing a ledge (or lip). X35.

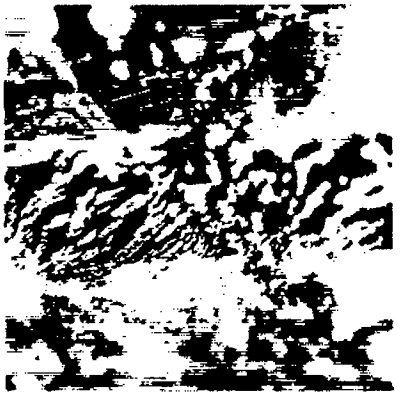


Figure 19.- Exposed grain boundary surface in figure 18. Light areas are fragments of original oxide skin; dark areas are exposed clean metal showing no surface detail. Fine lines above and below ledge are stress cracks. X50.

Figure 20.- Portion of broken specimen 36. Lowest white line is grain boundary; white lines above are operated slip bands. Time, 48 hours; temperature, 400° C; stress, 100 psi; $\alpha = 32^{\circ}$, $\beta = 37^{\circ}$. X15.

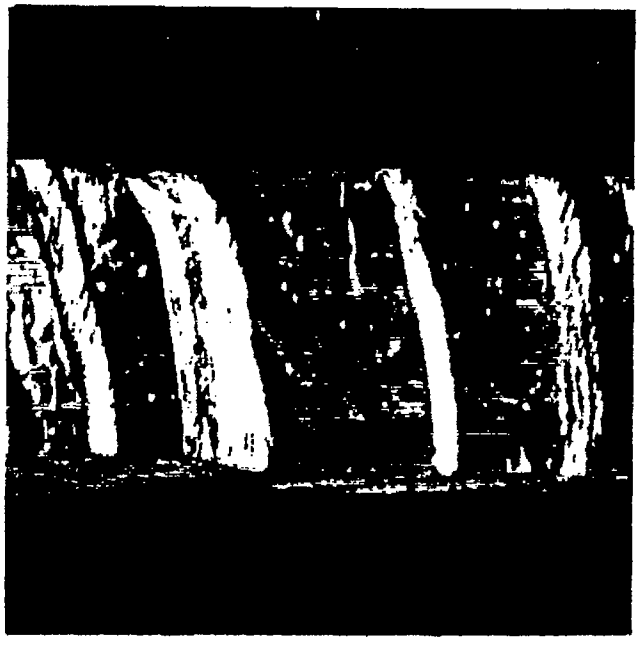
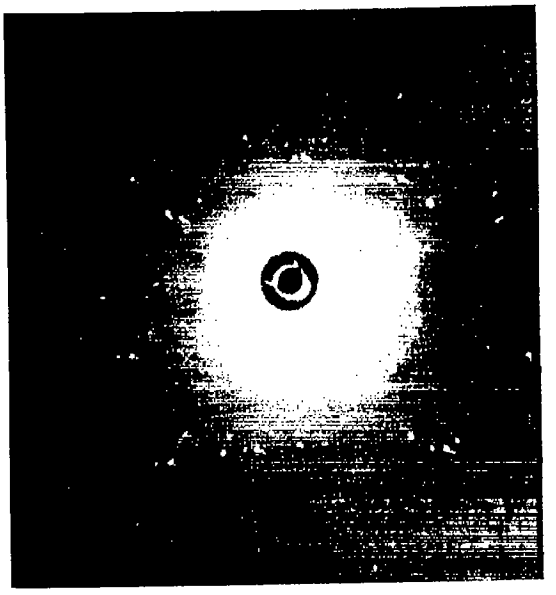
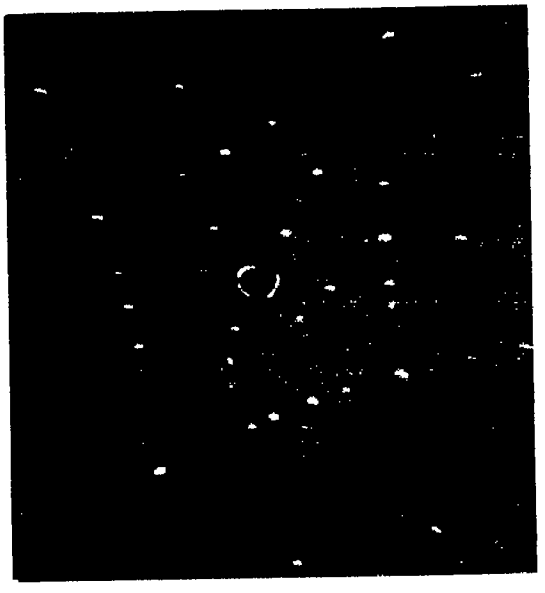


Figure 21.- Detail of one side of specimen 36 in figure 20. Lighter areas are exposed surfaces of slip bands; steps on surface of each ledge are probably individual slip planes. X15.

(a) Grain after testing. Time, 48 hours; temperature, 400° C; stress, 100 psi.



(b) Surface of grain boundary ledge after testing. Time, 48 hours; temperature, 400° C; stress, 100 psi.



(c) Surface of slip band ledge after testing. Time, 48 hours; temperature, 400° C; stress, 100 psi.

(d) Grain before testing.

Figure 22.- Laue back-reflection patterns (molybdenum radiation) of one grain of specimen 36. $\alpha = 32^{\circ}$, $\beta = 37^{\circ}$.

(a) Time, 1 hour; temperature, 600° C; stress, 100 psi; $\alpha = 42^{\circ}$, $\beta = 43^{\circ}$. X180.

(b) Time, 14 hours; temperature, 500° C; stress, 100 psi; $\alpha = 43^{\circ}$, $\beta = 46^{\circ}$. X90.

Figure 23.- Oxide stress cracks, diagonal slip lines, and degeneration of horizontal lines of reference pattern in severely deformed regions of specimens.

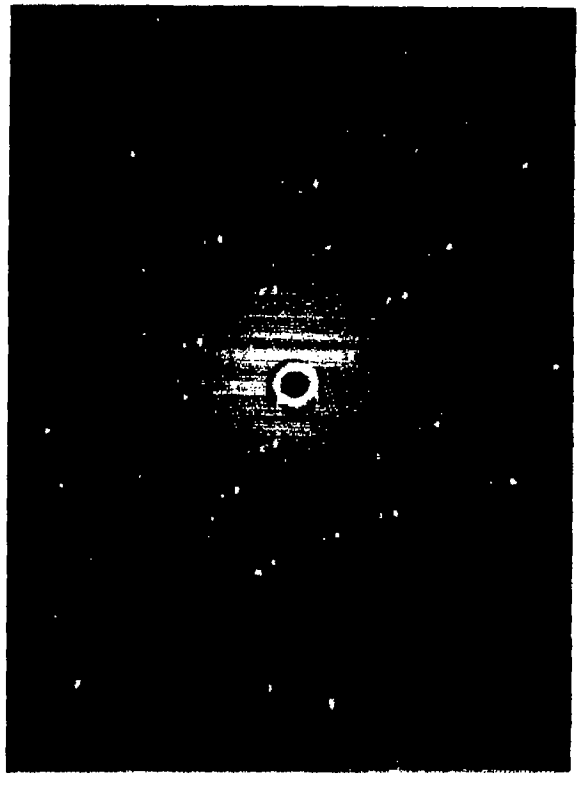


Figure 24.- Laue back-reflection pattern of specimen and area in figure 23(a).