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MICROSCOPIC OBSERVATIONS OF HIGH PURITY URANIUM
SUBJECTED TO THERMAL CYCLING

by

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MICROSCOPIC OBSERVATIONS OF HIGH PURITY URANIUM SUBJECTED TO THERMAL CYCLING

R. M. Mayfield and L. T. Lloyd

ABSTRACT

Observations of the microstructural changes and deformation mechanisms which occur in coarse- and fine-grained high purity uranium as a result of heat treatments between room temperature and 500°C indicate that thermal cycling growth embodies some of the features which have been observed in creep studies of aluminum. Coarse-grained specimens deformed by slip and twinning within the grains, as well as by grain boundary deformation. Intragranular slip ascribed to the $\{110\}$ planes was observed in addition to prominent slip on the (010) planes. The amount of grain interaction is dependent upon the angle between the two $[010]$ axes; as this angle increases the interaction increases. The fine-grained specimens deformed by grain interaction and grain boundary migration, together with some intragranular slip and grain boundary sliding.

I. INTRODUCTION

In the past few years, the macroscopic behavior of uranium upon thermal cycling has received considerable attention from which a vast amount of information has been compiled concerning growth behavior under varying circumstances.(1-4) The results of such studies have prompted several explanations(5-7) of the mode of deformation and of the growth behavior of polycrystalline uranium. Most investigators agree that the dimensional instability arises from the intercrystalline stresses developed during cycling which are, in turn, attributed to the anisotropic nature of the thermal expansion. Based on present information some of these explanations appear plausible; however, they all suffer from a lack of proof or confirmation by microscopic observations of the plastic deformation which is occurring during thermal cycling (grain deformation, grain boundary sliding, etc.).

Other metals have been subjected to this microscopic type analysis with significant results. Boas and Honeycombe(8-10) studied the behavior of certain noncubic metals after repeated heating and cooling over a relatively low temperature range. Three general types of phenomena were observed: (1) the smooth polished surfaces became rough and the grain

boundaries became more pronounced; (2) deformation markings appeared within the grains; the majority were brought about by slip; others were the result of twinning; and (3) migration of grain boundaries occurred in high purity material, being absent to a large degree in commercial grade material. X-ray results indicated that the deformation, which was believed to be independent of grain size, occurred throughout the whole specimen and was not restricted to the surface layers. Similar observations were obtained by Burke and Turkalo⁽¹¹⁾ from thermal cycled bicrystals of zinc. They found that when specimens were cycled between 100° and 300°C, deformation within the grains was primarily by slip, grain boundary sliding occurred between grains, grain boundary migration was evident, and in some cases recrystallization or subgraining occurred immediately adjacent to the grain boundary. When specimens were cycled between room temperature and -180°C, they observed that no grain boundary migration or sliding was evident, deformation within the grains occurred by twinning as well as slip, and in some cases cleavage occurred within the grains.

Some other interesting works on the microscopic behavior of grains and grain boundaries of aluminum tested in creep have been reported by Grant et al.⁽¹²⁻¹⁶⁾ These investigators observed effects which were similar to those noted by Boas and Honeycombe, and Burke and Turkalo.

The microscopic behavior of uranium upon thermal cycling received limited attention by Cahn⁽¹⁷⁾ who observed evidence of slip upon the surface of a specimen cycled 55 times between 20°C and 630°C. Observations of this nature should provide information for understanding the deformation phenomenon which occurs in polycrystalline uranium subjected to thermal cycling.

This report includes some experimental work of the microscopic behavior of grains and grain boundaries of thermal cycled, coarse-grained uranium as a function of orientation difference between grains. Limited observations on polycrystalline material of a finer grain size are also included.

II. EXPERIMENTAL PROCEDURE

A. Material

Both the coarse- and fine-grained specimens were of high purity uranium. The coarse-grained specimens were produced from ingot 2D-795 by rolling, swaging and subsequent grain coarsening.⁽¹⁸⁾ The fine-grained polycrystalline specimens were produced from ingot B-336 by rolling approximately 85% at 300°C, followed by vacuum annealing at 600°C for 2 hours. Both castings were prepared by melting uranium crystals produced by electrolysis of fused salts.⁽¹⁹⁾ Chemical analysis of both materials would fall in the range indicated in Table I.

B. Specimen Preparation

The coarse-grained specimens (initially about $3/4$ in. long by $3/16$ in. in diameter) were macroetched in a trichloroacetic acid electrolyte to remove the fine grains which were present at the cylindrical surfaces. After selecting and sectioning suitable areas, the specimens were mounted for polishing and subsequent metallographic examination by a special technique which subjected them to as little stress as possible. Koldweld* disks, 1 in. in diameter by about $3/8$ in. high, were formed under pressure at room temperature. Holes were then drilled in the center of the disks, filled with a paste of the liquid and powder, and the uranium specimens were inserted. The mount was allowed to harden at room temperature, usually overnight. The same general procedure was used for the fine-grained specimens, except that no macroetching was required. In either case, the samples were carefully polished by both mechanical and electrolytic (H_3PO_4) means and subjected to examination in polarized light of the initial structure. The salient features of each sample were recorded photographically. In some cases straight, orthogonal scribe marks, which were to serve as reference marks and to reveal any displacement of adjoining grains, were placed on the polished surface with a Bergsman Hardness tester. Light loads on the diamond hardness indenter were necessary to prevent undue distortion of the uranium and subsequent recrystallization upon heating.

C. Orientation Determination

The fine-grained specimens did not lend themselves to any method of individual grain orientation analysis; however, such information was obtained from the coarse-grained specimens by procuring back-reflection Laue photograms from selected grains. The latter specimens were aligned on a special micrometer stage⁽²⁰⁾ so that the X-ray beam was perpendicular to the polished surface, within the boundaries of the selected grain, and oriented with respect to a reference mark on the polished surface.

The X rays were collimated with 0.005 in. or 0.010 in. diameter pinholes so that, at a 3-cm specimen-to-film distance, the beam covered a diameter of approximately 0.015 in. or 0.030 in., respectively. Exposure times varied with the collimator size and the type of target material of the X-ray tube; they ranged from 1 to 64 hours. A typical pattern is shown in the photogram of Fig. 1.

*Trade name. A compound of the Lucite type used for the manufacture of dentures which consists of powder and a liquid polymerizer.

The relative orientations of the coarse grains in a specimen were determined by the Greninger method⁽²¹⁾ and plotted stereographically relative to the rod axis (reference mark) and normal to the polished face. Stereographic projections for the selected grains of any one specimen are included throughout the report, as in Fig. 3. Each principal pole position has the designation of the grain to which it pertains.

D. Thermal Cycling and Subsequent Examination

After the specimen has been examined metallographically and X-ray photograms had been obtained, it was removed from the Koldweld mount. The major portion of the mount was first cut away with a hot nichrome wire. The balance was dissolved from the specimen in acetone or ethylene dichloride; Na_2CO_3 was added to the solvent to free it of any small traces of acid that might damage the polished surface. After the mount material had been removed, the specimen was thoroughly cleaned of any contamination, wrapped in 2-mil tantalum sheet and sealed in Pyrex under a vacuum equal to, or better than, 5×10^{-5} mm of mercury. Needless to say, these last few steps of specimen preparation required extreme care if any satisfactory results were to be obtained upon heating the specimens to 500°C .

The thermal cycling was carried out in a small, vertical, nichrome-wound furnace. The specimen rack containing the Pyrex capsule was raised into the hot temperature zone, held for a predetermined time, and then lowered into a room temperature air blast. All test specimens were cycled between room temperature and 500°C ; temperatures were determined by means of a thermocouple mounted outside the capsule. Generally a 10-minute hot zone holding time and a 2-minute cold time were used, but a 5-minute hot holding time was used in some instances. In either case, the transfer time from hot to cold, or vice versa, was of the order of 5 seconds.

After a predetermined number of cycles the capsule was opened and the polished surface of the specimen was examined under bright field illumination. The surface roughness after cycling and the presence of a thin oxide film made the use of polarized light impractical in most cases. The various features of the metallographic surfaces were recorded photographically. In many cases oblique illumination was used to accentuate the appearance of the as-cycled surface. Some specimen surfaces were very lightly polished, both mechanically and electrolytically, to remove the roughening and to permit a detailed examination under polarized light for subgrain formation as well as other consequences of the thermal treatment.

III. RESULTS

A. Coarse-Grained Specimens

The metallographic features of five coarse-grained specimens were studied in this work. Specimens 48, 71 and 80 each had three grains on the polished faces; specimen 86 contained a total of 9 grains, but only 5 of the larger adjacent grains were used; and specimen 121 also had a total of 9 grains, all of which were considered. The results obtained from the samples are presented in relation to their condition, that is, initial, as-cycled, and as-cycled and repolished.

1. Initial Condition: Figures 2, 9, 13, 21, and 28 are sketches which show the locations of grain boundaries and scribed grid markings on the coarse-grained specimens. The arrows on each sketch relate to the reference marks used for determination of the relative orientations of the grains. The Laue photograms were taken normal to the surface represented by the sketch. The direction of the arrow is parallel to the N-S axis of the stereographic solutions, and the head of the arrow points to the N pole.

The orientations of the grains for each specimen are plotted stereographically in Figs. 3, 10, 14, 22, and 29. The axis common to each pair of adjoining grains and the magnitude of rotation (δ) about these axes, which is required to bring the two grains into the same orientation, are tabulated in Table II. The angle α was measured from the axis of rotation to the [100] axis, β and γ were measured to the [010] and [001] axes, respectively. The table also includes the angle (σ) between [010] axes of adjoining grains.

The metallographic appearances of the specimens prior to thermal cycling are shown in Figs. 4, 11, 15, 23, and 30. Some of these do not include the entire area of the specimen and the scribed grid markings shown in the sketches (Figs. 2, 9, 13, 21, and 28).

The twin trace markings observed in this condition are tabulated in Tables III and IV, depending upon their identity. Each trace is identified by the specimen number, the grain in which it appears, and an assigned number; the angular locations, clockwise (C) and counterclockwise (CC), are measured to the same reference mark used for the X-ray orientation determinations. The figure in which the traces may be seen and the method of identification are also indicated; the majority of the traces were identified by pole locus (one surface) analyses. (22)

2. As-Cycled Condition: The general appearance of the specimens after cycling between room temperature and 500°C are shown in Figs. 5, 12, 16, 24, and 31; the number of cycles to which the

specimen has been subjected is given for each figure and total cycling treatments are compiled in Table V. Because of the internal deformation and slight oxidation which occurred during cycling, structures are revealed under bright field illumination.

a. Deformation within grains: Both twinning and slip deformations occurred within the body of the grains. The new trace directions observed subsequent to thermal cycling are listed in Tables VI, VII, and VIII.

In several cases (Figs. 5 and 16) there was an obvious increase in the quantity of $\{130\}$ type twins. Frequently this appeared as thickening of twins which were present prior to cycling; however, in certain instances there was definite creation of new twins (i.e., Grains A and B, Fig. 16). Grid lines which were crossed by twins were distorted locally (Fig. 17), as would be expected from considerations of the shearing movement which occurs during the deformation. All of the new twin trace directions observed after cycling (Table VI) cannot be attributed to the thermal deformation: a portion of these may have been present prior to cycling but they were undetected in that condition; too much significance cannot be placed upon their observation after cycling.

Deformation within the grains was primarily by slip mechanisms; the most prominent of these was slip on the (010) plane. All of the coarse grains studied, with the exception of those for specimen 71, contained deformation markings which were identified as (010) plane slip (Table VII). Where grid markings were crossed by (010) slip traces, the scribed lines were found to be bent corresponding to displacement along that plane. The slip traces other than (010) are tabulated in Table VIII. Several of these markings were quite irregular in nature and had the typical appearance of cross-slip deformation observed by Cahn⁽¹⁷⁾ and by Lloyd and Chiswik.⁽²⁰⁾ A portion of the remaining traces can be accounted for by slip on the $\{110\}$ planes, a slip system which has been reported operative by Cahn.

b. Interaction between grains: The relative amount of interaction between adjacent grains is manifested by the extent of deformation (primarily slip) localized near the grain boundaries. Because of the irregular nature of the joining surface between two grains, this interaction is not uniform; however, it was possible to obtain a measure of its extent according to the arbitrary scale of deformation which is indicated in Table II. These relative values of interaction are recorded for each pair of adjoining grains in the same table.

In limited cases evidence of grain boundary sliding was observed (see Figs. 6, 17, and 18) in that there were definite offsets of

scribed lines at the boundaries. This appears to have occurred in regions where grain interaction was at a minimum. On the other hand, scribed lines which were in areas exhibiting localized deformation at the grain boundaries were bent through a continuous curve.

Differences in level between grain surfaces have resulted from the thermal cycling. These do not take the form of a sudden step, but rather the surfaces are continuously bent near the boundaries. Similar variations in height of the surfaces may be noted within the grains where heavy local deformation has occurred.

Evidence for grain boundary migration may be seen in specimen 121 (Fig. 31) at the boundaries between grains A and I and between grains G and I. Close metallographic examination indicates that there are a series of lines which represent the location of the grain boundaries at various cycles. The direction of migration appears to be such as to increase the radius of curvature of the boundary.

The above observations have been limited to the first cycling treatment of the coarse-grained samples. Their appearance after the second cycling treatment does not differ greatly, as may be seen by comparing Figs. 5 and 7. In general, increased cycling enhanced the metallographic evidence of deformation but did not result in additional mechanisms. The surface conditions of the samples were poorer, which corresponds to resealing and recycling.

3. As-Cycled and Repolished Condition: Subsequent to the final thermal cycling treatments, three of the coarse-grained samples (48, 80, and 86) were lightly polished mechanically and again electropolished. Photomicrographs of selected areas of these samples are shown in Figs. 8, 19, 20, 25, and 26.

There is a very definite change in the twins relative to their appearance before cycling. Many of the original twins have disappeared, whereas others have increased in width and have assumed irregular shapes. Comparison of these with the trace directions present prior to cycling indicates that their identity is of the $\{130\}$ twin type. Twins which were located in areas heavily deformed by the thermal cycling are bent (see Fig. 26).

The regions immediately adjacent to grain boundaries, especially those that showed considerable distortion in the as-cycled condition, exhibit subgraining; this leads to a more irregular nature of the grain boundaries. A Laue photogram taken from the area of grain C specimen #86 near the boundary with grain E (Fig. 27) indicates that the subgrains are systematically disoriented with respect to each other by a rotation about the $[001]$ axis.

B. Fine-Grained Specimens

The three specimens used for this phase of the metallographic study had an average initial grain diameter of approximately 0.05 mm. Because of this relatively small size it was impossible to determine the orientation of each individual grain. For this reason the deformations which occurred within the grains cannot be associated with specific mechanisms, nor can a measure be obtained of the effect of relative orientation upon the amount of interaction between grains. The metallographic observations, however, do permit some generalizations with respect to thermal cycling deformations.

Two of the three specimens (B-336-I and B-336-II) were cycled in the alpha-annealed condition; the remaining sample (B-336-III) was cycled after a 4-minute beta treatment at 725°C, followed by water quenching. The details of the cycling treatments are given in Table V.

Prior to cycling, the alpha-annealed specimens appeared as uniform, equiaxed grains with little evidence of internal stressing as manifested by the presence of twins. Subsequent to cycling, the metallographic surfaces appeared as shown in Figs. 32 and 33. The internal stressing of the grains is indicated by the slip and/or twin markings; this is particularly evident in B-336-II which was cycled 100 times. These deformation markings have been distorted and bent by subsequent thermal cycles, as are the originally straight scribe markings.

The interaction between grains is indicated by several factors. A subgrained structure appears to have been created not only at the grain boundaries, but within the grains themselves. This effect is clearly evidenced in Fig. 33c, where the oblique lighting delineates the subgrain boundaries. Portions of the grain boundaries appear to have migrated as a result of the thermal cycling; successive stages of the migration are shown by the series of markings. It is probable that the grain boundary left an impression for each cycle; however, it is difficult to directly correlate the number of cycles and the number of grain boundary impressions. The direction of migration of the boundaries can be determined by comparing the appearance of specimen B-336-I in the cycled condition (Fig. 32a) with that of the same area after a light mechanical polish followed by electrolytic polishing (Fig. 32b). Some metallographic evidence of grain boundary sliding is seen in these photographs, in that the various grains appear to have a difference in height and thereby cast shadows of varied width when viewed under oblique lighting (Fig. 33).

The initial microstructure of the beta-treated sample B-336-III is shown in Fig. 34. A relatively large alpha grain structure which contains extensive subgraining and some twins is evident. After cycling for 25 times, the metallographic surface appeared as shown in Fig. 35. These

photomicrographs give the appearance of considerable deformation and some increase in the quantity of twinning. The most extensive deformation has occurred at the grain boundaries although some interaction between subgrains has occurred, for they are clearly distinguishable under bright field illumination.

IV. DISCUSSION

Through proper techniques, it is possible to observe by metallography some of the changes which occur in polycrystalline uranium that has been subjected to thermal cycling treatments. The results reported above are in general agreement with the observations made by Boas and Honeycombe⁽⁸⁻¹⁰⁾ and by Burke and Turkalo⁽¹¹⁾ upon thermal cycled samples of other noncubic metals. In some respects the current observations are similar to those obtained by Hanson and Wheeler,⁽²³⁾ Grant et al.⁽¹²⁻¹⁶⁾ and Yakovleva⁽²⁴⁾ from coarse-grained samples tested in creep at elevated temperatures. In addition, certain of the results pertain to the complexity of plastic deformation which is observed in uranium.

A. Crystallographic Deformation

1. Twinning: The presence of twin traces in the microstructure of the coarse-grained samples prior to the cycling treatments can be attributed to the anisotropic nature of the linear thermal expansion of the three principal alpha-uranium crystallographic directions. It has been shown by X-ray diffraction lattice parameter measurements⁽²⁵⁾ as a function of temperature and by dilatometric measurements^(26, 27) that the coefficient of expansion in the $[010]$ direction is negative upon heating above room temperature, whereas the coefficients of expansion in the two other mutually perpendicular directions (the $[100]$ and $[001]$) are positive. If a coarse-grained sample is prepared in an internal stress-free condition at a temperature high in the alpha region, as is the case for specimens prepared by the grain-coarsening treatment, then, when this sample is cooled, stresses will be created between the adjacent grains. For a particular grain the surrounding grains may exert a component of compressive force in the $[010]$ direction, for the grain will expand in this direction upon cooling. The magnitude of this stress will depend upon the relative orientation of the adjacent grains. This latter factor will be discussed in more detail later. Deformation studies of alpha uranium⁽²⁰⁾ have shown that $\{130\}$ and $\{172\}$ type twinning can readily occur as a result of compressive forces in a direction near the $[010]$. The twin traces which are observed prior to thermal cycling belong to these two families (Tables III and IV). It is interesting to note that no twins were observed in specimen #71 in the initial condition; in this case the $[010]$ axes of the three grains were similarly oriented.

The ability of a particular grain to twin will also depend upon how it is surrounded by other grains. When twinning occurs, it is necessary that the two portions of the parent grain which are separated by the twin move in relative position with respect to each other. The direction of this movement is in the twin shearing direction, and the magnitude is dependent upon the twin thickness. If the grain which is to deform by twinning is larger in cross section than that of the surrounding material, the relative motion of the two portions of the parent grain may occur, whereas, if the grain is small in relative cross section, the shearing motion is suppressed. The coarse-grained samples represent the former situation, for the grain cross-sectional area is large in comparison to that of the surrounding fine-grained skin, while in the finer grained samples the latter situation applies. For this reason the initial structures of the coarse-grained materials show considerable twinning and the finer grained samples show only a small quantity.

Thermal cycling of the coarse-grained samples resulted in an increase in the quantity of twinning in some of the grains; this is particularly notable in specimen 80 (Fig. 16), grain A, trace directions 1 and 2, grain B, trace directions 11 and 12, grain C, trace direction 21; and specimen 86 (Fig. 24), grain D, trace direction 31, grain E, trace directions 41 and 42; all of these are $\{130\}$ type twins. Twins which were present prior to thermal cycling have increased in dimension and new twin traces have developed. The thickening of the twins appears to be discontinuous in nature; this may be the result of incremental increases in size during thermal cycles or it may be associated with thermal induced migration of the boundary between the twin and the parent. Several other grains have $\{130\}$ twins which appear thicker than they were originally; however, there is no definite increase in the number of traces. In other situations the increase in quantity of twinning appears to be localized in the grain, i.e., specimen #48, surface B, grain A, trace direction 2 near the protrusion of grain A into grain B (Fig. 5). This nonhomogeneity of twinning points to a variation of the stresses between grains, depending upon the location of the plane separating the two grains with respect to the crystallographic elements of the deformation mechanisms of the grains.

The fact that increased numbers of twins are observed indicates that one portion of the parent crystal can move with respect to another portion freely and, in turn, that there is no restraint, or at least very little, in the twin shearing direction. In some cases slip markings which are attributed to (010) plane slip appear at the intersection between two twins of the $\{130\}$ type, i.e., specimen #48, surface B, grain B (Fig. 5). This slip mechanism has been shown by Lloyd and Chiswik⁽²⁰⁾ to accommodate $\{130\}$ type twinning by relieving stresses concentrated at such intersections.

Observation of the finer grained specimens after thermal cycling indicates that little increased twinning has occurred. This is as expected for, if grains are confined on all but one surface (the plane of polish), then only those grains which have a twin shearing direction emerging from that surface will be able to twin if the surrounding stress field dictates such a deformation.

Metallographic observations of the specimens after cycling and repolishing indicated that the twin distribution noted after cycling was not exactly representative of the twinned material present in the samples. Presumably there were contaminating gases left in the evacuated capsule which were "gettered" by the hot uranium in the early stages of the cycling. Because of differences in crystallographic faces exposed to the "atmosphere," there were differences in rates of reaction and, correspondingly, there were differences in film thicknesses. These films then remained intact and resulted in an apparent existence of twins, when actually portions of these have disappeared. A good example of this is the appearance of the {130} twins of trace direction 12, specimen #48, surface B, grain B. In the initial condition (Fig. 4) these traces are well defined and parallel sided. This appearance is retained after cycling 15 times (Fig. 7). After repolishing, however, the twins are no longer seen in the area near the common edge (Fig. 8). The fact that the twins, after cycling, had the same shape as in the initial condition indicates that the markings observed in the former condition were created early in the cycling process. Further, it can be postulated that the twins were nonexistent at the end of the first cycling treatment because there is no change of their appearance as the result of the second cycling treatment. It appears that, as a result of removing the original, fine-grained skin from the coarsening sample, the stress conditions which originally created these twins were removed. Then, because of heating during the first thermal cycle, the twins were absorbed by the parent grain. Twin absorption in uranium during annealing has been discussed in detail by Madsen.⁽²⁸⁾ On the basis of these arguments it would appear that the twins disappeared after the first thermal cycle, and the film which manifests their presence must have been created during that first heating.

The possibility does exist that the surface markings which indicate the presence of the twins in the as-cycled condition may have been created by differential expansion effects. Because the twins have a different crystallographic orientation than does the parent, there will be differences in the expansions of the two volumes in a specific direction. This differential expansion will generate stress between the two volumes which could be relieved by sliding along the twin plane interface and thereby producing difference in level which could be observed under bright field illumination. If this were the case, however, one would not then expect the twins to

disappear from the structure upon further cycling, for the stresses between the two volumes would be alternately generated and relieved, and there should be no driving force for their subsequent absorption at high temperatures in the alpha region.

The twins which are shown to be thickened after cycling and repolishing (see, for example, Fig. 19) were probably generated in the following manner. The stress field around the particular grain was such as to induce further twinning in the form of separate lamella. At high temperatures in the alpha region these grew to merge in part with the twins which were already present. In the portion of the grains where the stresses did not create new twins the original twin has been absorbed by the parent orientation. The process of growth of the twins at the high temperature has created the irregular shapes which are noted.

2. Slip: Obviously, there were no metallographic evidences of slip in the coarse- or fine-grained specimens prior to thermal cycling or after the surfaces were repolished, because mechanical and electrolytic polishing remove such marking from the surface of the specimens.

In the as-cycled condition many slip deformations were noted in the body of the grains. The most prominent of these was slip on the (010) plane in the [100] direction. Apparently the stress fields in various portions of the grains were such as to result in a shear stress resolved into the slip plane and slip direction. The apparent inhomogeneities of stresses induced within a single grain again points to the dependency of the location of the plane separating the two grains with respect to the crystallographic elements of the deformation mechanisms of the grains. The (010) plane slip traces can easily be identified; as a result of geometrical relationship of the (010) plane with respect to the {130} planes, the former always angularly bisects the angle between the latter planes. These three planes all lie in the same zone and thus, on a random plane of polish, the angles between the (010) plane and the two {130} planes will be equal. Such an identification is invariably substantiated by pole-loci analyses. For the most part, well-defined areas of (010) slip are located away from the grain boundaries.

In addition to the prominent slip, there were several other types of deformation markings which have their origin in slip processes. These are compiled in Table VIII. A portion of these have the general appearance of cross-slip deformations; Lloyd and Chiswik(20) have identified the operative slip planes for this mechanism to be (010) and (001) planes and the slip direction to be [100]. The exact angular location of traces of this type cannot be measured because of their capricious nature. The deformations have been assigned trace numbers and the areas in which they appear are shown in the indicated figures. The greater portion of the cross-slip is located near grain boundaries and in regions where the deformed material is confined by the adjoining grain on more than one side.

The remaining 16 traces were of a more well-defined nature and the trace directions could be measured; however, some of these were rather short and led to some difficulty in precise identification. Ten of these were identified by pole-loci analyses as originating in $\{110\}$ plane slip. This slip plane has been reported by Cahn⁽¹⁷⁾ as operative in polycrystalline specimens deformed at elevated temperatures by either external or thermally induced internal stresses. The summation of the pole-loci for the $\{110\}$ slip traces is shown in Fig. 36. The identifications were not confused by passage near the $\{011\}$ and (001) plane poles - these are slip planes which have been reported as operative in room temperature, compressed single crystals.⁽²⁰⁾ The experimental evidence constitutes reasonable proof that $\{110\}$ plane slip does occur in alpha uranium; it also indicates that such deformation occurs at elevated temperatures or under complex intergranular stress conditions which exist in thermal cycled, polycrystalline samples.

Three of the remaining six traces were identified by pole-loci analyses as having arisen from slip on the (001) plane. Their pole-loci passed near $\{110\}$ planes as well as the (001) ; however, they were closer to the latter than they were to the former. The (001) identification does not exclude the possibility that they originate from $\{110\}$ slip. In fact the latter may be the more probable plane of slip; (001) slip has not been identified previously as being completely separated from (010) plane cross-slip. The other three traces do not lend themselves to identification as one of the known operative slip planes; however, they do pass in the proximity of $\{110\}$ plane poles. The inability to identify these six traces as (010) or $\{110\}$ slip may be attributed to one or both of two factors. Their lengths were relatively short, which makes an exact determination of the direction difficult. Also, the areas where these traces appeared were heavily distorted with respect to other portions of the grain, and it is not unreasonable to suspect that these areas might not have retained the orientation indicated by the Laue photograms taken prior to thermal cycling.

The finer grain specimens did not lend themselves to an identification of slip marking subsequent to thermal cycling. Many of the grains (see Fig. 33) contained relatively straight traces which might have been caused by slip or by twinning. The fact that these are not apparent after repolishing indicates that they probably had their origin in slip; however, their identity could not be obtained for lack of knowledge concerning the orientations of the individual grains. It is probable, because of their relatively distinct nature, that these represent (010) slip lines.

B. Interaction Between Grains

1. Coarse-Grained Specimens: The deformational interaction between two grains at their boundary is dependent upon two factors: one, the relative orientation of the two grains, and two, the orientation of the grain

boundary with respect to the crystal structures. A measure of the influence of these factors can be obtained by considerations of the relative dimensional changes of the two grains at the grain boundary.

It is necessary to make several simplifying assumptions so that such an analysis can be reduced to a simple mathematical expression. The first of these is the restraints which are imposed upon the two grains. There are four general types of restraint which may occur for two grains separated by a plane interface: one, restraint on all sides; two, restraint at the ends of the grain boundary; three, restraint normal to the grain boundary; and four, no external restraints. We will consider the latter of these conditions. This situation is one which is approximated by the thermal cycled coarse-grained samples; in general, the grains passed completely through the samples and only adjacent grains were in contact. If we limit the consideration to the material located near the grain boundary, then the differential of expansion of the two grains will be the only source of stress application. If the boundary is planar in nature there will be no component perpendicular to the boundary, for the grains are free to change dimension in that direction and the only components of stress will be parallel to the boundary plane. The mathematical analysis can be further simplified to a two-dimensional problem by assuming that the expansions in the $[100]$ and $[001]$ directions are equal. This assumption is reasonably valid in light of expansion data calculated from lattice parameter measurements of alpha uranium as a function of temperature.⁽²⁵⁾ The analysis will be confined to the plane defined by the $[010]$ axis of the two grains. The effects which would occur on a plane inclined at an angle to this plane would be a direct cosine function of the angle between the planes.

The problem is then reduced to the situation depicted in Fig. 37, where grains A and B are separated by a boundary, a o b , and are unrestrained on all external sides. In the general case, the $[010]$ axis of grain A is inclined to the boundary between grains A and B by an angle θ , and the magnitude of dimensional change as a result of a temperature increase is ΔL_b . (The dimensional change cannot differentiate between a positive or negative direction; however, it may have a positive or negative value.) The dimensional change of grain A, 90 degrees from the $[010]$ axis (ΔL_{ac}), is inclined to the boundary by an angle $(90 - \theta)$. If the angle between the $[010]$ axes of grains A and B is σ then the $[010]$ axis of grain B will be inclined to the boundary by an angle $(\theta + \sigma)$, and the dimensional change of grain B, 90 degrees from the $[010]$ axis, will be inclined to the boundary by an angle $90 - (\theta + \sigma)$. An equation that expresses the dimensional change which occurs in an elemental portion of grain A located immediately adjacent to the grain boundary can be set up in terms of the four dimensional changes of the grains as a function of temperature and the angles they make with respect to the grain boundary. This equation is:

$$\Delta L_A = -\Delta L_b [\cos \theta - \cos (\theta + \sigma)] - \Delta L_{ac} \cos [(90 - \theta) - \cos (90 - [\theta + \sigma])] ,$$

where ΔL_A is the dimensional change of A parallel to the boundary. Expansion of A relative to B will create compressive forces in A at the grain boundary, and expansion of B relative to A will create tensile forces in A at the grain boundary. A positive value of ΔL_A is considered to mean that tensile forces are created in A at the grain boundary, and a negative value of ΔL_A is considered to mean that compressive forces are created in A.

A series of curves representing ΔL_A for different values of the angles θ and σ is shown in Fig. 38. The value of ΔL_b between room temperature and 500°C was taken as -0.0027 in./in. and that of ΔL_{ac} as 0.0145 in./in. The values of ΔL_A are plotted for constant values of θ and varying values of σ . These curves, even though they represent a greatly oversimplified condition, do show some interesting features. When σ is zero, there is no interaction between the grains (single crystal); as σ increases, the stresses upon A, as indicated by the differential expansion, increases. These stresses attain their maximum values when σ is 90 degrees. (Values of ΔL_A for angle of θ and σ greater than 90 degrees are symmetrical about the 90 degree values, because ΔL_b and ΔL_{ac} do not know directionality.) The curves also indicate that statistically the majority of the stresses are tensile stresses. Finally, the curves indicate that for certain different combination of the angles θ and σ there will be no stress applied on A at the grain boundary. This latter fact is easily observed experimentally; along the length of the irregular grain boundaries in the coarse-grained samples there are regions where no apparent interaction is observed within a specific grain. Of course it is necessary to exceed a certain value of stress, which depends upon the crystallographic orientation of the grains with respect to the direction of stress application before plastic deformation can occur.

A direct correlation of the above-calculated dependency of the stresses induced within a grain upon the relative orientation of the two grains, and the position of the boundary between them with that observed experimentally in the coarse-grained specimens, is impossible because of the simplifying assumptions which were made. The situation is further complicated by the fact that in most cases the boundaries between two grains are not planar but rather are curved surfaces. This results in components of stress normal to the boundary as well as parallel to it which, in turn, induces local inhomogeneities of deformation along the length of the boundary.

In spite of these localized inhomogeneities of deformation the relative interaction of two adjacent grains can be described quantitatively on an arbitrary scale as indicated in Table II. Although the number of tests was small, the results indicate that the amount of interaction depends

upon the relative orientation of the grains as measured by the angle (σ) between the $[010]$ axes of grains, as would be expected from the above analysis. When the orientation of the $[010]$ axes of the two grains are quite close, the interaction at the grain boundaries is at a minimum and increases with an increase in value of the angle σ . Rotation of the other two principal poles [the (100) and (001)] about the $[010]$ (where σ is small for two adjoining grains - specimen #71) appears to have little effect upon the deformation behavior.

The crystallographic nature of the deformation which occurs adjacent to the grain boundaries can best be understood by observations of the cycled specimen after repolishing. All of the three samples (#48, #80, and #86) which were subjected to this treatment showed areas of subgrains in these localities. The extent of this subgraining appears to be directly correlated with the apparent amount of deformation as observed in the same area prior to repolishing and subsequent to thermal cycling. Presumably this structure was created by a phenomenon similar to that of polygonization. As the samples were heated, stress fields were established at the grain boundaries such as to bend the lattice and create imperfections within it. Upon further heating these imperfections became mobile and diffused to form low angle boundaries or subgrains. Further heating and cooling would enhance the appearance of these areas. The fact that these subgrains are related to crystallographic deformations and thereby to slip deformations is indicated by the regularity of their misorientation. The Laue photogram shown in Fig. 27 was obtained from grain C of specimen #86 near the boundary with grain E. There is a definite indication that the Laue spots are misoriented by a rotation about the (001) plane pole (or $[001]$ axis). This is the direction which lies perpendicular to the slip direction and in the slip plane for the $(010) - [100]$ slip system and is the direction about which lattice rotation will occur as a result of slip on that system. It is interesting to note that the $[001]$ direction also satisfies the same condition for the $(110) - [1\bar{1}0]$ slip system which was reported by Cahn(17) and which has been observed in the present work. The regularity of the misorientation of the subgrains may be observed metallographically in Figs. 20 and 26. The subgrains are arranged in rows and there is a regular change in observed tones of gray across these rows.

Two other types of boundary interactions were observed in the coarse-grained specimens; they are grain boundary sliding and grain boundary migration. The evidence for grain boundary sliding was rather limited (see Figs. 6, 17, and 18); however, there were definite sharp offsets of some of the scribed lines at the grain boundary. It is probable that this type of plastic deformation occurred only in those portions of the grain boundary where the grain interactions had a component only in the plane of the surface separating the two grains. There was only one clear case of grain boundary migration which was observed in the coarse-grained specimens. The boundary separating grains A and I of specimen 121 migrated

so as to increase its radius of curvature. This is the normal phenomenon observed during grain growth of metal specimens. In this case the migration was probably enhanced by local distortions of grain I, caused by interaction with grains A, H, and G.

2. Fine-Grained Specimens: It has been stated above that the finer grained specimens did not readily show plastic deformations which are related to crystallographic deformation mechanisms. It is difficult to bridge the gap of knowledge concerning deformation which occurs in grains essentially confined on one surface to that which will occur in more completely surrounded grains. There are, however, some similarities between the behavior of semiunrestrained coarse grains and restrained fine grains; these are the formation of subgrains and grain boundary migration.

The occurrence of grain boundary migration is well illustrated by the photographs in Figs. 32 and 33. Successive locations of the boundaries are indicated by the series of lines appearing at some grain boundaries (see, for example, the boundaries of grain I in Fig 32). Presumably each line represents the location of the grain boundary during an individual cycle, and the number of lines should equal the number of cycles. The direction of migration of the boundaries can be ascertained by comparing the structure in the repolished condition (Fig. 32b) to that of the as-cycled condition. The observations at this point become somewhat confusing, for some of the changes which have occurred in the grain boundaries appear to be opposite to what one would expect from normal equilibrium heating of a specimen. Instead of increasing the radius of curvature of the grain boundaries some of these have been decreased; in one case (the lower grain boundary) the direction of growth is away from its center of curvature, and some of the angles between grain boundaries have changed from near equilibrium values of 120° to values larger and smaller than this. These behaviors can only point to inhomogeneities of deformations of the various grains during heating.

The occurrence of subgraining within individual grains is illustrated in Figs 33b and 33c where low angle boundaries, as indicated by slight surface tilts, can be seen. The mechanism of subgrain formation is probably similar to that proposed for the formation of subgrains in coarse-grained samples; however, in the fine-grained samples these are not confined to the grain boundary areas.

The metallography of the fine-grained samples does not permit positive identification of grain boundary sliding. The photographs of the scribed line on the surface of specimen B-336-II show only one possible boundary where sliding has occurred (see right-hand end of scribed line in Fig. 33a). At all the other grain boundaries the line is continuously bent across the boundary and there is no indication of sharp offsets. The differences in height of various grains, as shown by the shadow cast under

oblique illumination, indicates that relative motion along these boundaries has occurred; however, such variations in level could be created by extremely localized lattice bending near the boundary plane. One would expect that such localized deformation would be associated with grain boundary migration or even possibly recrystallization. It may well be that boundary sliding does occur. On a purely statistical basis it is reasonable to suspect that some of the boundaries will be subjected to stresses which are primarily shearing stresses in the plane of the boundary.

The metallographic observations of the thermal cycled, beta-treated material tend to substantiate the viewpoint that the randomization of orientation (large variation in σ angle values) would result in more liberal localized deformation, but because of the lack of any well-defined texture, there is no directionality of macrodeformation. Upon continued cycling the size of the subgrains would probably be changed until such time as they reached a magnitude dictated by the mobility of lattice imperfections which, in turn, would be controlled by the maximum heating temperature and the time at temperature.

C. General Observations

The results reported here agree well with those observed by Boas and Honeycombe(8-10) and by Burke and Turkalo(11) in that the same general types of deformational mechanisms are observed. Specifically, these mechanisms are: intragranular slip, twinning, subgrain formation, boundary migration, and boundary slip. A difference in the mechanisms of deformation with grain size, however, does appear to exist and is generally in agreement with the results of Grant et al.⁽¹³⁾ on the creep of 2S aluminum. Greatly oversimplified, slip is the dominant mechanism in coarse-grained specimens, with boundary slip playing a very minor role, whereas in the fine-grained specimens the latter becomes more prominent, because statistically there are more loci for that type of deformation. The results also find agreement with the creep studies(13, 15) in that subgrain formation and grain boundary migration occur in both types of tests.

While the present tests were performed on high purity material, other investigators have indicated that boundary movement is hampered by the presence of impurities. Chang and Grant(14) point out that this restriction will eventually give rise to cracks at or in the vicinity of the boundary. A similar comparison has been made in the thermal cycling of high purity vs. commercial grade uranium(4) (both fine grained); the commercial grade was found to be full of intercrystalline cracks after a prescribed number of cycles, while the high purity had none. Boundary mobility in the high purity material prevented intercrystalline failure.

From all the microscopic observations made throughout these experiments there was little visual evidence of a directional deformation of any form that would produce a macroscopic change in dimensions of the

test piece, with the possible exception of a component of slip within the grain being resolved into the direction of elongation - usually longitudinally. Consequently, the explanations of any gross dimensional change would have to be predicated on what changes were observed. These observations would tend to substantiate creep relaxation theories and associated phenomena. Textural characteristics of uranium and, more specifically, the disorientation between individual grains provide the stress upon cycling. Whether the texture be (010) and (110), (140) and (010), or other combinations with considerable scatter, the stress generators are present. The resultant effect of these textures, normally present in fabricated uranium, is to produce results upon thermal cycling that amount to creep in the longitudinal direction. Additional investigative work will be required to substantiate in more detail the creep behavior associated with thermal cycling.

V. SUMMARY

Thermal cycling of high purity uranium specimens of both large and small grain size results in the appearance of relief on the polished surface, accompanied by the disclosure of grain boundaries as well as other surface effects of deformation. The boundary deformation and subsequent subgrain-ing that became evident upon cycling the coarse-grained specimens may be ascribed to the amount of disorientation between adjoining grains. This explanation probably also accounts for the deformation noted in the fine-grained material, but orientation data were lacking and consequently only qualitative statements can be made in this regard. Coarse-grained specimens revealed two slip systems upon cycling, (010) plane in the [100] direction being the more predominant, with a secondary system of the (100) - $[1\bar{1}0]$ type. Twinning plays a minor role in the deformation of the coarse-grained material, but is almost nonexistent in the fine-grained specimens. The fine-grained specimens deformed by slip, grain boundary migration, and some grain boundary slip. Beta-treated material revealed pronounced deformation upon cycling which may be the result of randomization, with the consequence of large disorientation between adjoining grains.

The deformation of uranium upon thermal cycling produced results in microstructure very similar to those obtained in Al under creep conditions. The macroscopic elongation of uranium upon cycling may therefore be closely associated with creep relaxation phenomena; however, further work of a more quantitative nature will be required to more clearly define the mechanism producing actual macroscopic dimensional changes.

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Table I
CHEMICAL ANALYSIS OF
HIGH PURITY INGOTS

Element	Analysis (ppm by weight)
C	15 - 40
Al	<10
Fe	<5
Si	10 - 15
Nitrogen	<10
Oxygen	Not determined
Others	At or below limit of detection

Table II
ORIENTATION RELATIONSHIP DATA FOR COARSE-GRAINED SPECIMENS

Spec. No.	Grains	Angle between common axis and (degrees)			Rotation required about com- mon axis, δ -(degrees)	Angle be- tween [010] axes, σ -(degrees)	Degree of grain boundary deforma- tion*
		[100]- α	[010]- β	[001]- γ			
48	A-B	72	56	40	84	63	3
48	B-C	77	24	70	85	48	3
80	A-B	59	31	85	44	22	2
80	B-C	88	21	69	88	29	2
71	A-B	88	8	82	47	4	0
71	B-C	82	6	85	47	9	0
71	C-A	89	2	89	83	4	0
86	A-B	84	6	84	84	12	0-1
86	B-E	50	50	64	51	40	2
86	B-C	60	82	31	17	17	0-1
86	C-E	67	47	52	54	40	2
86	C-D	39	60	69	50	42	2
86	D-E	27	83	64	60	60	3
121	A-B	87	8.5	82	83	11	1
121	A-C	83	10	83	90	13.5	1
121	A-D	70	25	77	66.5	21	0-1
121	A-F	33	60	80	15	22	2
121	A-G	12	90	78	56	35	3
121	A-H	79	17	76	82	23	2
121	A-I	86	11	78	56	22	1
121	B-C	61	35	73	12.5	8	0-1
121	C-D	80	20	78	27	8	0
121	D-E	84	23.5	63	58	20	1
121	D-F	69	58	40	84	4	1
121	E-F	88	19	71	78	21	2
121	F-G	57	62	45	21	19	3
121	F-I	89.5	1	89.5	68	1	0
121	G-I	86	4	89	58	20	1
121	H-I	82	8	90	24	3	1

*Arbitrary scale of deformation. (Deformation values were assigned after the first cycling treatment of each sample.)

0 - negligible; 1 - very slight; 2 - moderate; 3 - pronounced

Table III

{130} TYPE TWIN TRACES OBSERVED PRIOR TO THERMAL CYCLING

Specimen No.	Grain	Surface	Trace	Angular location with respect to reference mark (degrees)*	Figure illustrating trace
48**	A	A	1	72C	4
48	A	E	2	86C	4
48	A	A	3	49.5CC	4
48	A	B	4	24CC	4
48	B	A	11	49.5C	4
48	B	B	12	75C	4
48	B	A	13	84.5C	4
48	B	B	14	38C	4
48	C	A	21	52C	4
48	C	B	22	72.5C	4
48	C	A	23	61.5CC	4
48	C	B	24	51CC	4
80	A	-	1	67CC	15
80	A	-	2	44C	15
80	E	-	11	63CC	15
80	B	-	12	68C	15
80	C	-	21	68C	15
80	C	-	22	53CC	15
86	A	-	1	83C	23
86	A	-	2	73.5CC	23
86	B	-	11	69C	23
86	B	-	12	41.5CC	23
86	C	-	21	82.5C	23
86	C	-	22	30CC	23
86	D	-	31	61C	23
86	D	-	32	39CC	23
86	F	-	41	62.5CC	23
86	E	-	42	49C	23
121	A	-	1	62.5CC	30
121	A	-	2	82C	30
121	B	-	11	66.5C	--
121	B	-	12	55CC	--
121	C	-	21	58C	30
121	C	-	22	58CC	--
121	D	-	32	63.5CC	30
121	D	-	33	48.5C	30
121	E	-	41	55.5CC	30
121	E	-	42	77.5C	30
121	F	-	51	62C	30
121	F	-	52	69.5CC	30
121	H	-	71	69CC	30
121	H	-	72	55C	30
121	I	-	81	71C	30

* C - Measured clockwise from the reference mark.

CC - Measured counterclockwise from the reference mark.

**The twin traces in specimen No. 48 were identified by two surface analyses; all other traces were identified by pole-loci analyses.

Table IV

{172} TYPE TWIN TRACES OBSERVED PRIOR TO THERMAL CYCLING

Specimen No.	Grain	Surface	Trace (**)	Angular location with respect to reference mark (degrees)*	Figure illustrating trace
48	C	A	25	59C	4
48	C	A	26	75CC	4
48	C	B	27	81C	4
48	C	B	28	75.5CC	4
48	C	A	29	85CC	4
80	A	-	3	66CC	15
80	A	-	4	88CC	15
80	A	-	5	84CC	-
80	B	-	13	69C	15
80	B	-	14	90	15
80	B	-	15	85CC	15
86	A	-	3	64CC	23
86	B	-	13	63CC	23
86	B	-	14	85.5C	23
86	D	-	33	87C	23
86	E	-	43	86.5CC	23
86	E	-	44	74.5C	23
86	E	-	45	66C	23
121	D	-	31	68C	30

* C - Measured clockwise from reference mark.

CC - Measured counterclockwise from reference mark.

** All trace identities were determined by pole-loci analyses.

Table V

CYCLING HISTORIES OF SPECIMENS

(Temperature range of cycling - room temperature to 500°C)

Specimen No.	Treatment No.	Number of cycles	Room temperature holding time (minutes)	Transfer time (seconds)	500°C holding time (minutes)
48	1	15	2	5	10
48	2	15	2	5	10
71	1	50	2	5	10
80	1	25	2	5	5
80	2	25	2	5	5
86	1	25	2	5	5
86	2	25	2	5	5
121	1	50	2	5	10
B-336-I	1	25	2	5	10
B-336-I	2	25	2	5	10
B-336-II	1	100	2	5	10
B-336-III	1	25	2	5	10

Table VI

NEW TWIN TRACES OBSERVED AFTER THERMAL CYCLING TREATMENTS

Specimen No.	Grain	Trace	Angular location with respect to reference mark (degrees)*	Identity	Figure illustrating trace
80	C	23	87CC	{172}	18
80	C	24	62C	{172}	16,18
80	C	25	64CC	{172}	18
121	C	23	82.5CC	{172}	31
121	G	61	88C	{130}	31

* C - Measured clockwise from reference mark.

CC - Measured counterclockwise from reference mark.

Table VII

(010) SLIP TRACES OBSERVED AFTER THERMAL CYCLING TREATMENTS

Specimen No.	Grain	Surface	Trace	Angular location with respect to reference mark (degrees)*	Figure illustrating trace
48	A	A	5	87CC	5
48	A	B	6	58.5CC	5
48	B	A	15	62.5C	5
48	B	B	16	54C	5
48	C	A	30	84C	5
48	C	B	31	76CC	5
80	A	-	6	77C	16
80	B	-	16	88CC	16, 17
80	C	-	26	87.5C	16
86	A	-	4	87CC	24
86	B	-	15	74.5CC	24
86	C	-	23	64CC	24
86	D	-	34	84CC	24
86	E	-	46	86C	24
121	A	-	3	76.5CC	31
121	B	-	13	86CC	31
121	C	-	24	89.5C	31
121	D	-	34	82.5C	31
121	E	-	43	84CC	31
121	F	-	53	86C	31
121	G	-	62	69.5C	31
121	H	-	73	83C	31
121	I	-	82	88C	31

* C - Measured clockwise from reference mark.

CC - Measured counterclockwise from reference mark.

Table VIII

SLIP DEFORMATION TRACES OTHER THAN (010) PLANE SLIP WHICH
WERE OBSERVED AFTER THERMAL CYCLING TREATMENTS

Specimen No.	Grain	Surface	Trace	Angular location with respect to reference mark (degrees)*	Figure illustrating trace
(010) - (100) Cross-Slip					
48	A	A	7	?	5
48	B	B	17	?	5
48	B	A	18	?	5
80	B	-	19	?	16, 18
80	C	-	27	?	16, 18
121	A	-	4	?	31
121	F	-	54	?	31
121	G	-	63	?	31
121	I	-	83	?	31
{110} Plane Slip					
48	A	B	8	3.5C	5
48	B	A	20	35.5CC	5
48	C	B	32	23CC	5
48	C	A	33	27.5C	5
80	A	-	7	15C	16, 17
80	B	-	17	30.5CC	16, 17, 18
80	B	-	18	30.5C	16, 18
86	B	-	16	42C	24
86	C	-	24	53C	24
86	E	-	47	14.5C	24
(001) Plane Slip					
48	A	A	9	18.5CC	5
86	A	-	5	0.5CC	24
86	D	-	35	33.5CC	24
Slip Traces of Unknown Identity					
48	B	B	19	59CC	5
48	C	A	34	33C	5
121	H	-	74	17C	31

* C - Measured clockwise from reference mark.

CC - Measured counterclockwise from reference mark.

Figure 1. Typical Laue Photogram Taken Prior to Thermal Cycling (Specimen #86 Grain D)

Figure 2. Sketch of Grain Shapes of Two Polished Surfaces of Specimen #48 (Included angle between surfaces equals 121°)

19,074

15X

Figure 3. Relative Orientations of Grains in Specimen #48 (Surface A)

19,062

Figure 4. Composite Photomicrograph of Two Polished Surfaces of Specimen #48 Prior to Thermal Cycling

Figure 5. Composite Photomicrograph of Two Polished Surfaces of Specimen #48 after 15 Cycles from Room Temperature to 500°C

19,078

Bright Field

30X

Figure 6. Grain Boundary Sliding in Specimen #48 after 15 Cycles (Note offset of nearly vertical scribed lines)

19,077

Bright Field

200X

Figure 7. Composite Photomicrograph of Two Polished Surfaces of Specimen #48 after Second Thermal Cycling Treatment of 15 Cycles between Room Temperature and 500°C

19,076

Bright Field

30X

Figure 9. Sketch of Grain Shapes on Polished Surface of Specimen #71

19,080

15X

Figure 10. Relative Orientation of Grains in Specimen #71

○ - (100)
 ○ - (010)
 ● - (001)

19,063

Figure 11. Surface of Specimen #71 Prior to Thermal Cycling

19,081

Polarized Light

50X

Figure 12. Surface of Specimen #71 after 50 Cycles from Room Temperature to 500°C

19,082

Bright Field

50X

Figure 13. Sketch of Grain Shapes on Polished Surface of Specimen #80

19,083

15X

Figure 14. Relative Orientations of Grains in Specimen #80

19,064

Figure 15. Surface of Specimen #80 Prior to Thermal Cycling

19,084

Polarized Light

60X

Figure 8. Composite Photomicrograph of Two Polished Surfaces of Specimen #48 after Repolishing Subsequent to Thermal Cycling

19,079

Polarized Light

30X

Figure 16. Surface of Specimen #80 after 25 Cycles from Room Temperature to 500°C

19,085

Bright Field

60X

Figure 17. Microstructure Near Boundary between Grains A and B of Specimen #80 after 25 Cycles from Room Temperature to 500°C

19,086

Bright Field

200X

Figure 18. Microstructure Near Boundary between Grains B and C of Specimen #80 after 25 Cycles from Room Temperature to 500°C

19,087

Bright Field

200X

Figure 19. Microstructure of Specimen #80 after Repolish Subsequent to Thermal Cycling

19,088

Polarized Light

50X

Figure 20. Microstructure Near Boundary between Grains B and C, Specimen #80, after Repolish Subsequent to Thermal Cycling

15,833

Polarized Light

100X

Figure 21. Sketch of Grain Shapes on Polished Surface of Specimen #86

19,089

15X

Figure 22. Relative Orientations of Grains in Specimen #86

19,065

Figure 23. Composite Photomicrograph of Polished Surface of Specimen #86 Prior to Thermal Cycling

19,090

Polarized Light

20X

Figure 24. Microstructure of Specimen #86 after 25 Cycles from Room Temperature to 500°C

19,091

Bright Field

40X

Figure 25. Microstructure of Specimen #86 after Repolish Subsequent to Thermal Cycling

19,092

Polarized Light

50X

Figure 26. Subgrain Located near Grain Boundaries of Specimen #86 after Repolishing Subsequent to Thermal Cycling

a.

15,838

Polarized Light

150X

b.

15,837

Polarized Light

150X

Figure 27. Laue Photograph of Subgrains of Grain C Specimen #86 Located near Boundary with Grain E. [Note rotational asterism of the spots about the (001) pole.]

18,203

1X

Figure 28. Sketch of Grain Shapes on Polished Surface of Specimen #121

19,093

15X

Figure 29. Relative Orientations of Grains in Specimen #121

19,066

Figure 30. Composite Photomicrograph of Polished Surfaces on Specimen #121 Prior to Thermal Cycling

19,094

Polarized Light

20X

Figure 31. Composite Photomicrograph of Polished Surface of Specimen #121 after 50 Cycles from Room Temperature to 500°C

19,095

Bright Field

20X

Figure 32. Polycrystalline Specimen #B-336-I
after 25 Cycles from Room Temperature to 500°C

a. As removed from capsule

15,593 Bright Field 300X

b. Same area as above after repolishing to remove surface effects

15,615 Polarized Light 300X

Figure 33. Appearance of Specimen B-336-II after
100 Cycles from Room Temperature to
500°C

a.

15,928

Bright Field (Oblique Illumination)

250X

b.

15,929

Bright Field (Oblique Illumination)

250X

Figure 33. (continued)

c.

15,931 Bright Field (Oblique Illumination) 250X

d.

15,930 Bright Field (Oblique Illumination) 250X

Figure 34. Initial Microstructure of Specimen B-336-III after Beta Treatment (4 min at 725°C, water quench). The two photographs were taken from the same area; one is at a slightly different rotation than the other.

a.

16,382

Polarized Light

50X

b.

16,381

Polarized Light

50X

Figure 35. Microstructure of Specimen B-336-III
after 25 Cycles from Room Temperature
to 500°C (same area)

a.

16,383

Bright Field

50X

b.

16,380

Bright Field

100X

Figure 36. Summation of Pole-Loci of
(001) and {110} Slip Traces

19,096

Figure 37. Schematic Representation of Thermally Induced Dimensional Changes Acting upon Grain Boundary

19,427