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THE REMOVAL OF SUB-STRUCTURE FROM ALPHA URANIUM

BY

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A

THESIS

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ABSTRACT

The possibility of obtaining larger, perfect single crystals of alpha uranium by a modified strain-anneal method has been investigated. A -196°C tensile deformation was performed on pseudo single crystal rods which had been prepared by the phase transformation method. The specimens were subsequently annealed at 650°C.

It was found that by utilizing pseudo single crystals with a prescribed crystallographic orientation with respect to the tensile axis that recrystallized crystals of comparable size and perfection to those prepared by the grain coarsening method could be produced. Grain coarsening is the presently accepted process for preparing perfect single crystals of alpha uranium.

The higher purity of the perfect crystals prepared by the strain-anneal method described in this paper is definitely an improvement over the purity of the crystals produced by the complex grain coarsening technique, which requires intentional additions of foreign elements.

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I. INTRODUCTION

The purpose of this investigation was to determine a method by which the sub-structure(1) might be removed from alpha uranium pseudo single crystals which have been produced by the phase transformation method.(2)

Large, perfect single crystals are essential in the study of certain properties of uranium; especially those that are affected by the presence of grains and grain boundaries, such as, deformation mechanisms, diffusion coefficients, electrical and thermal properties, and elastic constants.

The possibility of producing larger grains of perfection comparable to those from the grain coarsening technique(3) by performing a tensile strain and subsequent anneal on alpha uranium pseudo single crystals was suggested by Cahn(4) and attempted by Lacombe and Calais.(5)

There are several definite reasons for supporting such an approach, they are the following.

1. "Polygonization of a deformed metal occurs when recrystallization has not yet taken place.(1)(6)(7) But when the annealing temperature is high enough, recrystallization can take place. The new crystals then develop at the expense of the polygonized crystals and are 'perfect'; they are no longer divided into sub-grains."(1)

The terms sub-graining, polygonization, and "recrystallization in situ" have been used synonymously in the literature. Some authors consider sub-graining to be large-scale polygonization, but the two phenomena are considered equivalent throughout this investigation. Briefly, Cahn(6) describes polygonization in terms of an excess number of edge dislocations of one sign being arranged into low-angle boundaries perpendicular to the slip planes thus forming "polygons" between the boundaries. Polygonization is generally observed by the X-ray Laue back-reflection technique. The Laue spots, which are elongated by asterism due to the deformation, are broken up into a large number of striae.

2. The strain and anneal approach would not involve the intentional additions of an impurity element as is required in the grain coarsening method, and thus could yield a higher purity single crystal.

3. Furthermore, the application of critical cold-working to pseudo single crystals results in a more homogeneous deformation of the material, and at the same time eliminates the nucleating effects of the grain boundaries, which must be large for polycrystalline uranium. Localized deformation of pseudo uranium crystals does not bring about grain growth.(8)

Lacombe and Calais(5) concluded, "The soundness of the method we propose has been proved, but to grow perfect crystals with large dimensions, we still have to define all the possible orientations of the phase-change crystals, the deformation mechanisms of which would be most appropriate for the growth of large perfect crystals."

Further investigations have been undertaken at their laboratory, the Center of Metallurgical Research of the School of Paris Mines. The influence of the deformation on the recrystallization of tensile strained alpha uranium single crystals has been reported, as well as certain general conditions, such as, crystallographic orientation, process of deformation, and mode of annealing which might promote the growth of large, perfect, single crystals.(9, 10)

L. T. Lloyd of the Argonne National Laboratory proposed to perform a technique similar to that of Lacombe and Calais, except with various refinements.(8) The most notable variation suggested was the application of the stress at lower than ambient temperature -- or liquid nitrogen temperature, -196°C -- in order to obtain a greater advantage from the cold-working.

"Cold-work appears to be more effective when operated at a lower temperature than a higher one, since the temperature of minimum recrystallization is lower in the former case. For a given deformation, the higher the temperature at which annealing is carried out, the larger will be the size of grains after annealing."(11)

"The cold-worked condition is thermodynamically unstable relative to the unstrained one, and a worked crystal undergoes spontaneous changes to reduce the extra free energy it has gained from the work done on it. The most striking of these changes is 'recrystallization', in which new crystals grow from nuclei and replace the strained ones. The growth is 'incoherent', and takes place by the advance of the large-angle boundaries separating the new crystals from their surroundings. Worked crystals can also soften without recrystallizing, or altering the orientation, by a[n] [undesirable] process called 'recovery'.(12)

Since the annealing temperature is limited to approximately 650°C due to the alpha-beta transformation at 668°C(13); the low-temperature deformation effectively increases the temperature range over which recrystallization and grain growth may occur.

R. O. Teeg(14) reports, "The critical shear stress for slip increases with decreasing temperature; while the stress required to initiate twinning is considered to be relatively insensitive to temperature." D. Calais(10) reveals, "The crystals deformed preponderantly by twinning give on recrystallization perfect crystals having optimum dimensions and having orientational characteristics closely related to those in the original crystal."

The availability of relatively large pseudo single crystals (2 to 4½ inches in length) in rods of 0.08, 0.16, and 0.25 inch diameters at the Argonne National Laboratory prompted their use, instead of sheet material which has been used by other investigators.

"No phase transformation is required in the strain-anneal method, and therefore there is no discontinuous volume expansion. Furthermore, ..., the $\{010\}$ preferred orientation supports the growth of $\{010\}$ crystals upon recrystallization; hence even the thermal expansion directions of the parent material and growing crystal are matched during crystal growth. One may conjecture, therefore, that more perfect crystals could be grown in a sheet, with its very restricted preferred orientation, than in fiber-textured rod, with its extra degree of orientation freedom." according to Holden.(15) However, it must be remembered that in this investigation pseudo single crystals were employed, not polycrystalline material with its fiber-texture.

Because the specimens and low-temperature apparatus were more satisfactorily suited for tensile testing, and information was available on the tensile deformation mechanisms at room temperature,(9) which would aid in the prediction of the mechanisms at -196°C; the strain was effected accordingly.

II. REVIEW OF THE LITERATURE

Known attempts to grow single crystals of alpha uranium date back to 1948, when Holden(16) employed the Bridgeman crystal-growing technique(17). This process involves a slow cooling of the metal from the molten state, and in this case single crystals were not produced. The approach failed principally because of the anisotropic behavior of the thermal expansion of alpha uranium, and the consequent confinement of the mold. The presence of phase transformations, per se, was not reason enough to discredit the possibility of growing alpha uranium crystals; since crystals of alpha iron(18) have been produced by this technique.

Since this first rather elementary attempt, several other approaches have been tried. Basically, these have been the grain coarsening method, the strain-anneal method, and the phase transformation method.

A. <u>The Grain Coarsening Method</u>.

This technique, developed for alpha uranium by Fisher(3), produces what is considered the most desirable results of any known approach. A number of perfect single crystals (4 millimeters in diameter by 5 to 10 millimeters in length) have been grown by this technique. The method, which was adopted from Beck's(19) work with copper, aluminum, and manganese, depends upon grain coarsening induced by the gradual decrease in the grain growth inhibiting influence of dispersed inclusions in a fine grained recrystallized matrix. A discontinuous type of grain growth occurs during which a few grains in the fine grained recrystallized matrix grow to large grain sizes at the expense of the matrix.

The rather lengthy and complex grain coarsening process is summarized below in detail.

Step 1. An ingot of high purity, vacuum-melted electrolytic uranium was warm-rolled to a rod of convenient starting diameter (0.337 inch).

Step 2. A short length of rod was sealed into an evacuated Vycor glass capsule, heated at 1000°C for 5 hours, and then water quenched. During the Vycor glass anneal, silicon and other foreign elements are apparently transferred to the rods by vaporization from the glass and diffuse into the uranium to form a concentration gradient of impurities.

Step 3. In order to adjust the concentration gradient of the inhibiting particles, the rod was wrapped in tantalum foil to prevent further contamination and again sealed in an evacuated Vycor capsule for a second annealing at 1000°C, and water quenched.

Step 4. For each lot there existed a particular final temperature between 800°C and 1000°C which produced upon water quenching the best final yield. This step was believed necessary in reducing the solute retained by quenching and, consequently, reducing the number of fine particles at all points of the concentration gradient during alpha annealing.

Step 5. The as-quenched rod was then reduced in diameter to 0.16 inch by 300°C swaging with intermediate recrystallization anneals. A step reduction schedule was effective in producing a uniformly fine grain size prior to the occurrence of grain coarsening.

Step 6. The rod was cut into 1 inch lengths, one end of which was machined to a 30° taper.

Step 7. The specimen was sealed into an evacuated capsule and recrystallized at 475°C by a 30-minute anneal. Then it was placed in a vertical annealing tube so that a temperature gradient of 50°C per inch existed over the specimen length, the point being at a maximum of 650°C for several hours. The specimen was then lowered into a uniform temperature of 655°C and annealed for 48 hours.

Step 8. After removal from the furnace those crystals which were of sufficient size were isolated by chemically removing the adhering small surface grains and by grinding away the larger grains.

The important factors in the method are described as follows:

The original material must be of a high purity composition similar to that prepared by the consolidation melting(20) of electrolytically prepared crystals(21). Such materials are better suited for producing uniformly dispersed fine particles upon heat treating so that grain coarsening may occur.

The tendency of grain coarsening is significantly influenced by the variation in gamma phase heat treatment prior to fabrication. This is attributed to the effect of the heat treatment on the dispersion of grain growth inhibiting inclusions in the primary recrystallized structure. Grain coarsening is attributed to the gradual coalescence or dissolution of these inclusions during prolonged annealing.

The largest coarse grains result when the concentration of fine inclusions is some optimum value, the primary recrystallized grains are of uniformly fine grain size, and the final annealing process is carried out at the highest possible temperature.

The crystals grown by this technique, although small, contain no sub-structure, but are of higher silicon and other impurity concentration than crystals prepared by other modes. Such crystals as these have been used in mechanical deformation studies.

B. The Strain-Anneal Method.

The following technique has been investigated by several experimenters. Holden(15) and Cahn(14) each made a preliminary study, however Fisher(22) has probably completed the most extensive investigation of the strain-anneal approach. Fisher's study consisted of a large number of experiments with specimens of differing purity, yet no single crystals were produced by the technique. However, aggregates of large perfect grains were produced.

In this mode, the following factors for growing single crystals are considered important:

The uranium used should be of relatively high purity. This is a prime factor, because inclusions impede the grain growth.

A fine, uniform alpha grain size, obtained by the recrystallizing of heavily worked rod or sheet is desirable. A preferred orientation seems to be required. A minimum critical strain is necessary for recrystallization to occur and is usually the order of 1 % elongation, compared to 2 to 3 % for other metals.

An annealing method should be carried out at as high a temperature in the alpha range as practicable, or approximately 650°C.

The specimen shape has some influence on the results. A sheet should be more conducive to the growth of single crystals than a rod.

The lack of success of this method can be attributed to the following reasons:

Deformation by twinning causes copious nucleation, and the difficulty in attaining the optimum conditions of the previously mentioned factors makes the probability of obtaining large crystals of uranium small, if not impossible.

There are a number of metals, of less complicated structure than uranium, which have produced single crystals upon being strain-annealed(23), among them are aluminum and iron.

Various strain-relieving mechanisms were believed to have occurred; they were continuous grain growth, grain coarsening, strain-induced boundary migration, and growth of strain-free nuclei into strained structures.

In his discussion Fisher(22) states, "The failure to prepare single crystals in this investigation is attributed largely to the occurences of strain-relieving mechanisms other than nucleation and growth. On this basis, any further experiments should include high-purity specimens of relatively large penultimate grain sizes, since tendencies for continuous grain growth and coarsening are both decreased with increasing matrix grain size."

"However, a number of factors exist which would make the optimum penultimate grain size very critical or non-existent. First of all, the rate of growth of the recrystallization nuclei will probably decrease with increasing matrix grain size(24). Since the annealing temperature is limited, a reduced rate of growth of the first nucleus means a greater chance for subsequently formed nuclei to grow large enough to resist consumption. Secondly, the strain necessary to foster recrystallization will probably increase with increasing grain size. Larger strains will probably entail more twinning and, consequently, more regions of high local stresses which as proposed by Cahn(4), result in simultaneous nucleation at many points in the structure(22).

Additional techniques have been suggested in order to produce larger crystals. These are the utilization of tapered specimens with a reduced center cross-section, the application of the anneal in a slowly rising temperature, and the incorporation of a temperature gradient across the specimen. These tricks of the crystal-growing art are designed to limit the number of nuclei that grow, and thus make the crystals larger.(15)

C. Phase Transformation Method.

Burke and Turkalo(25), Cahn(4), and Fisher(2) all attempted to grow single crystals by using a modified Bridgeman technique which involves slowly passing a specimen from temperatures at which the beta phase is stable into the alpha phase. In all cases it was found that pseudo, or imperfect, single crystals were produced by employing very slow transformation rates in a moving temperature gradient that ranged on both sides of the alpha-beta phase change temperature. The crystals contained no large-angle boundaries; however, they were profuse with small-angle boundaries parallel to the growth direction that divided the crystals into many long sub-grains.

To utilize the Bridgeman technique in such a manner, certain conditions are of utmost importance, such as material composition, rate of specimen travel, and nature of the temperature gradient. Additional factors include the size, shape, and penultimate grain size of the samples. These conditions are crucial in this method for the same reasons as were pointed out for the other techniques.

Lehr(26) made a study of the allotropic transformations of uranium and the structural consequences. In his investigation he noted the effects of nucleation and growth of the alpha phase in a temperature gradient, the degree of perfection of crystals obtained by phase change, and the dilatometric behavior of uranium single crystals prepared by this method.

Crystals prepared by the phase transformation method are composed of two general types of sub-grains. There are columnar shaped sub-grains which are in general aligned parallel to the rod axis, and there are equi-axed sub-grains -- the former type being the more predominant.

By observing the striation, or scatter, of the spots of Laue back-reflection photograms, the degree of perfection of the crystals may be determined. Two adjacent sub-crystals rarely vary in orientation by more than 1°, but two widely separated sub-crystals which are in the same crystal may vary by as much as 10° to 15°.

"It has been suggested(4) that these sub-grains are formed because of the large volume change accompanying the transformation. It was proposed that the alpha phase, which is softer than the beta phase at the transformation temperature, suffers distortion during the transformation; this distortion is a form of creep which, at the high temperature concerned results in the formation of 'creep cells' such as are formed in aluminum during creep. This process may also be regarded as a strain-induced polygonization. The faster the rate of cooling through the transformation temperature, the faster is the effective rate of creep of the alpha grains, and the smaller should be the mean size of creep cells. This is in fact observed," according to Cahn and Tomlinson(27).

This explanation of the source of sub-structure in uranium has been substantiated by the 1.12 % decrease in volume for the beta to alpha change calculated by Chiotti, Klepfer, and White(28).

Pseudo single crystals such as those formed by the phase transformation process may be employed for experiments where sub-structure is not critical, such as for determining electrical conductivity, thermal conductivity, radiation damage, fission product diffusion, and radiation growth, which have all been studied using pseudo single crystals.

D. Combinations of the Three Major Methods.

Any method of combining the principal modes in order to produce perfect single crystals involving grain coarsening would be disregarded; since the addition of impurities is undesirable.

The application of the phase transformation method followed by a strain-anneal appears to be the most feasible combination and was first attempted by Lacombe and Calais(5).

The large size of the pseudo single crystal resulting from the phase transformation method indicates that such crystals would be better suited for an attempt to obtain perfect crystals by strain-anneal than would polycrystalline specimens; because deformation is likely to be more homogeneous throughout the specimen due to the absence of intergranular interactions. The deformation mechanisms should also be more readily related to the most favorable crystal for producing large recrystallized grains.

Calais, Lacombe, and Simenel(9) investigated the deformation mechanisms of imperfect uranium single crystals in sheets strained in tension at room temperature, as a function of orientation. They also determined values of critical strain for commercial grade uranium as a function of the orientation. Due to the effects of impurities, the critical strains they reported would be larger than corresponding values for high purity material.

Calais(10) has done the most recent and perhaps the most extensive study of the various methods of applying plastic deformation to induce the growth of alpha uranium single crystals. Included among the means of producing plastic deformation are rolling, bending, compression, and tension.

Calais(10) concludes, "Our observations enabled us to select the imperfect crystals produced by phase transformation whose orientation will be the most favorable to obtain large perfect crystals by cold-working and annealing. They are the crystals which are deformed by slip or major twins."

E. Deformation Mechanism Studies.

The numerous modes of deformation in alpha uranium -- 3 slip systems, 5 twin systems, cross-slips, and deformation bands (or kinking)(9) -- as well as the additional complications due to the fact that uranium has corrugated planes, covalent bonding and a high degree of anisotropy(15), make it difficult to activate a single mode during critical cold-working. For the same reasons it is difficult to deform the entire volume of a crystal homogeneously.

Cahn(29) investigated the deformation mechanisms of large grained polycrystalline orthorhombic alpha uranium. The main slip system was identified as the $\{010\}$ with a $\langle 100\rangle$ slip direction, and a minor slip system on the $\{110\}$ planes with an undetermined direction was reported. The slip planes were determined by the pole loci method(30) from a single polished surface. Experimentally found twinning systems included the $\{130\}$ and the irrational $\{172\}$ planes. Indirect evidence led to the identification of $\{112\}$ and $\{121\}$ twin planes. Kinking and cross-slip were also observed in conjunction with the $\{010\}$ slip system.

Cahn hypothesized, "It is possible that if the critical deformation is imparted to the specimen at a temperature high enough to obviate twinning (about 350°C for uranium(29)) larger crystals can be made."(4)

Frank(31) made a comparison of the crystal structures of alpha uranium and zinc and discussed the reciprocal shears and twinning modes.

Lloyd and Chiswik(32), using single crystals prepared by the grain coarsening method and deformed in compression at room temperature, substantiated the occurrence of {010} ⟨100⟩ slip. These data were obtained by conclusive two-surface analysis. The resolved critical shear stress for this slip system was found to be 0.34 kg/mm^2. Employing the pole loci method, they found {011} slip, with an estimated ⟨100⟩ direction. This type was believed to occur primarily to accomodate twins and not as a principal means of deformation. Fine cross-slip was attributed to {001} slip in the ⟨100⟩ direction.

The most frequently observed twinning was of the {130} plane. The second most frequently observed twinning system had an irrational composition plane, assigned the indices of {172}. An irrational {176} twinning system was also observed.

The kinking mechanism is similar to that in most metals in that it occurred mainly when the deformation direction was nearly parallel to the principal slip direction ⟨100⟩ and was associated with a lattice rotation about an axis contained in the slip plane and normal to the slip direction -- [001] for uranium.

Two very important conclusions were made from Lloyd and Chiswik's study. First, {110} slip at room temperature is questionable. Second, there is no critical temperature in the vicinity of 400°C above which no twinning will occur and where the deformation is entirely by slip.

Many single crystals indicated that twinning was always preceded by slip. If so, twinning must be preceded by the formation of high stress concentrations at points of localized deformation or at lattice discontinuities of one form or another.

The deformations mechanisms at -196°C and 350°C were studied by Teeg(14). Deformation at liquid nitrogen temperature gives $\{110\}$ slip with a $\langle 001\rangle$ direction, which is not the closest packed direction. No evidence of the $\{010\}$ $\langle 100\rangle$slip system was found in any crystal deformation at -196°C. Twinning on $\{130\}$, $\{176\}$, and $\{172\}$ was observed with $\{130\}$ by far the most active at both -196°C and 350°C. Teeg and Ogilvie(33) have reported the results of continued investigation of the deformation mechanisms.

A study was made(34) using compression at 600°C, and $\{110\}$ $\langle 001\rangle$ slip was observed.

In their study, Calais, Lacombe and Simenel(9) observed the previously confirmed modes and labeled them "major" or "minor" according to the volume of crystal that they affect. The orientations of the tensile axes giving rise to the different deformation modes were shown on stereographic projections.

Calais(10) has compiled a resume' of the investigations of the deformation mechanisms of alpha uranium; however, he made no contribution to the results.

The deformation processes may perhaps be best summarized by the use of a stereographic projection similar to that of Lloyd and Chiswik$^{(32)}$.

In compression (see Figure 1) a crystal with an axis within area I will deform by {130} twins and {010}⟨100⟩ slip; within II will deform by {130} and {172} twins and {010} ⟨100⟩ slip; within III by {176} twins and by {011} ⟨100⟩, {001} ⟨100⟩, and {010} ⟨100⟩ slips; within IV by kinking involving the {010} ⟨100⟩ slip system; and within V by {010} ⟨100⟩ slip alone.

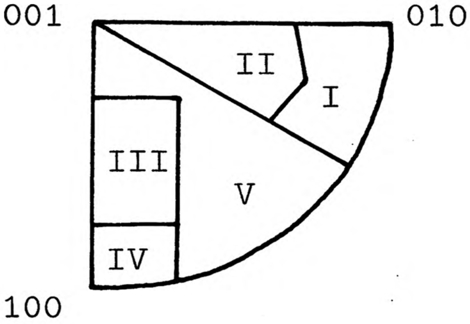

Figure 1. The deformation mechanisms upon compression.

"A diagram similar to Figure 1 may be constructed for tensile deformation, the principal difference being in the regions in which twinning will occur. In general, twinning occurs in regions near [010] in compression but near [100] in tension. Slip, on the other hand, is reversible, and those directions where it occurs in compression will permit slip in tension."$^{(15)}$

F. Recrystallization of the Deformed Uranium Single Crystals.

An investigation(35) was made of the orientations of the large grains, which were produced in a rod of high purity uranium in an attempt to prepare a single crystal by the strain-anneal method. The orientations were determined by the Greninger(36) method, using back-reflection Laue photograms obtained from the individual grains and were reported relative to the rod axis and to a longitudinal plane of examination parallel to the rod axis. The results showed a preferred orientation of (010) poles in the direction of the rod axis with a preponderance of the grains having the (001) poles within a 90° range of rotation around the rod axis.

Lloyd(37) analyzed the orientations of crystals from the compression deformation study and the corresponding recrystallized grains following anneal, by using the Greninger method for Laue back-reflection patterns.

The orientation relationship between the recrystallized grains and the deformed orientations in the crystals before anneal can be expressed as a 27.5° rotation about axes located 70.0° from the [100], 63.5° from the [010] and 34.0° from the [001]. The rotations being clockwise in the first and third quadrants of a (001) standard projection and counter-clockwise in the second and fourth quadrants. The grains were considered to originate from twin orientations and from the parent crystal. The twins necessary to produce the recrystallized grains corresponded exactly with the observed twins.

Calais, Lacombe, and Simenel(9) reported orientation data for the single crystals deformed by tension; however, their orientations were related to the deformed crystal only. The average rotation was 19° about axes located 82.5° from the [100], 52.5° from the [010] and 38.5° from the [001].

Calais, Mandin, and Lacombe(38) reported observations of a similar nature and gave a magnitude of rotation of 30° with the axes located close to those of Lloyd(37). Thus, all three findings were in fair agreement. The magnitude of rotation about the axis common to deformed and recrystallized orientations for orthorhombic alpha uranium agrees well with cubic and hexagonal metals, namely 20° and 40°, respectively.

The recrystallization relationship does vary from that for higher symmetry metals because rotation is not about an axis normal to the most densely populated (and slip) planes(37).

The several different investigators noted that if a pseudo single crystal was deformed by twinning, annealing would cause recrystallization by three different processes.

1. When two different twin systems are active, recrystallized nuclei form at their intersection.

2. If the major deformation is due to {172}, {112} or {121} twins, a correlation exists between the behavior of the twins and the sub-boundaries of the pseudo crystal. Twins in certain sub-grains coarsen and coalesce while twins in other sub-grains are re-absorbed upon annealing. The inter-facial energy of twin boundaries is a minimum for a mechanical twin, and after annealing it is increased and allows the migration of the boundaries normal to the composition plane.

3. Following more extensive strains, coalescence is evident. That is, the simultaneous disappearance of twins and growth of new crystals. In such a case, crystals grow parallel to the twin composition plane at the expense of twinned regions.

There are two hypotheses at present to describe the mechanism of recrystallization in metals. Beck(39) believes that recrystallization textures originate from "selective growth" and Burger(40) relates the effect to the preferential "oriented nucleation" with respect to the deformed crystal.

"The recrystallization of uranium can proceed by either of these mechanisms, depending on the major deformation mechanism. If deformation bands are the major mechanism, recrystallization proceeds more by 'oriented nucleation'. If the major deformation is by slip, recrystallization occurs by 'selective growth'. After deformation by twinning as the major mechanism, recrystallization proceeds without doubt by a combination of the two mechanisms."(9)

III. DISCUSSION

A. Preliminary Work.

Alpha uranium pseudo single crystals had previously been prepared by the phase transformation method(2) at the Argonne National Laboratory. A nichrome wound tube furnace(2) with a temperature gradient of approximately 150°C per inch for a maximum temperature of 850°C was employed. The rods were sealed in an evacuated Vycor capsule, and the capsule was gradually lowered through the full length of the furnace at 0.5 centimeter per hour by a Telechron clock system. The specimens were designated with arabic numerals, which were followed in some cases with an "R" and a sub-script. "R" indicates that the specimen was re-run once through the phase transformation technique before a suitable pseudo single crystal was obtained, "R_1" --re-run twice, and "R_2" -- three times.

The rods had been prepared by a swaging technique to various diameters (0.08, 0.16, and 0.25 inch) and were from 3.5 to 5 inches in length. The samples were of high purity (see Table I) material obtained from four different lots of consolidated electrolytically produced sponge(20). Some of the specimens had been machined to form a 30° taper on one end; however, this was not found to improve the growth of pseudo crystals and the practice was discontinued. Several of the rods were machined to a 10 % reduction of area at the center of the specimen in order to induce a strain gradient along the axis upon elongation.

The pseudo single crystal portion of the rods ranged from 2 to 4.5 inches. The orientations of the pseudo single crystals with respect to the rod axes had been determined by the Greninger(36) method of interpreting Laue back-reflection patterns and were recorded on a (001) stereographic projection (see Figure 16).

A study of the deformation mechanism literature for uranium single crystals was made by the author in order to determine the most desirable orientation for large crystal growth upon recrystallization following tensile strain performed at -196°C.

From the information, which is presented in the Review of the Literature of this paper, concerning Figure 1 and the corresponding deformation mechanisms plus the tensile deformation mechanism study(9) it was decided that a crystal with an axis near [100] would be the best choice for deformation under tension at liquid nitrogen temperature. A slight amount of strain should produce {130} twinning, perhaps on a two twin plane, as the lone deformation mechanism. Calais' thesis(10) confirmed this choice of orientation for optimum results.

The orientations of pseudo crystals produced by the phase change method are reported as being randomly orientated, according to Calais, Lacombe, and Simenel(9). Not as many crystals prepared at Argonne were oriented near [100] as was predicted and thus limited the number of specimens of favorable orientation which could be used in this study. This deviation was perhaps brought about by the use of a vertical instead of a horizontal furnace.

B. Experimental Procedures.

The following is the outline of the steps performed on each specimen in this investigation:

1. The specimens were chemically macro-etched, by steps, in concentrated hydrochloric acid, washed in water, etched in concentrated nitric acid, washed in water, rinsed with acetone, and air dried.

2. The etching procedure revealed the pseudo single crystals and the related sub-structure upon visual examination. Figure 2 is of a specimen, 70R, which had been deformed, but retained its original polygonized appearance upon annealing.

3. The macro-structure was then drawn in order to record the position of the original crystals. The rods were handled usually with a tissue, but on occasion with bare fingers -- the alpha radiation hazard was a matter of concern only around open wounds or with the possibility of ingestion.

4. Fiducials were made on the specimens with a crayon pencil in order that the actual elongation of the crystals might be determined, and the regions to be X-rayed were marked by a circle so that even after strain and anneal

relatively the same points could be X-rayed again. (It was noted that by taking Laue back-reflection photograms of the bright regions of the crystals the (001) poles would be included in the patterns.)

5. To preserve the surface from oxidation and to shorten the times necessary for X-ray exposure, the specimens were anodically electropolished using an aqueous sulfuric acid electrolyte (45 % H_2SO_4, 10 % glycerin). The current density was not critical and the surfaces were usually lustrous after approximately a 30 second treatment. The rods were air dried after being washed in water and rinsed with acetone.

6. X-ray patterns, designated as alpha, were taken to indicate the extent of sub-structure, or polygonization, by the striation of the spots; and to confirm the previously determined orientations. The unfiltered radiation from a copper target tube, operated at 18 milliamperes and 45 kilovolts, was collimated through a 10 mil pinhole. The specimen to film distance and exposure times were 3 centimeters and approximately 2 hours, respectively. The specimens were mounted so that their surfaces were normal to the X-ray beam and their rod axes were parallel to one side of the film. Typical alpha Laue back-reflection photograms are seen in Figures 3c, 4c, 5c, 6b, and 7c.

7. In this study the major portion of the specimen deformations were effected by tensile elongation performed at liquid nitrogen temperature, -196°C. Some preliminary

work was done at room temperature, and several specimens were deformed by compression at room temperature.

An Instron tensile testing machine was utilized to produce the desired elongations. This is a screw machine with an amplidyne drive and a weighing system which incorporates a SR-4 strain gauge to measure the elastic deformation of a stainless steel member. The tensile apparatus was adapted to do low temperature work by a special rig (see Figure 10).

All components of the grips were made of stainless steel for sufficient strength. The bearing surfaces of the grips were thoroughly lubricated with molybdenum disulphide in order to prevent galling. The ends of the specimens were etched as in step 1. and coated with glycerin before being tightened in the jaws in order to provide a better grip at liquid nitrogen temperature.

The elongations of the specimens were given by the gauge length dial on the machine, which measured the distance of travel of the cross-head, and were compared to the determinations of the permanent elongations by measurements of the fiducial changes and the two results were found to be in reasonable agreement (within one per cent).

A chart of applied load versus cross-head travel -- elongation, if the grips did not fail to hold -- was automatically made on the testing machine for each specimen. Relief mechanisms, which should have been twins in most cases, were quite evident, in Figures 11, 12, 13, 14, and 15, which are reproductions of such charts for specimens 42, 52, $55R_1$, 64R, and 111 R.

8. Individual specimens were placed in Vycor glass tubes which were closed at one end. Then the tubes were evacuated to pressures of 10^{-5} to 10^{-6} millimeters of mercury and sealed with an oxy-hydrogen flame.

9. Anneals were performed by placing the encapsulated samples in a stainless steel tube immersed in a lead pot furnace and held at $650 \pm 5^{\circ}C$ for 48 hours. As a rule, the encapsulated specimens were heat treated by placing them directly into a uniform maximum temperature region. Several specimens were lowered through a temperature gradient of $200^{\circ}C$ to $400^{\circ}C$ in a four inch length, then into a uniform $650^{\circ}C$.

10. The capsules were then removed from the furnace and allowed to slowly cool to room temperature.

11. The tubes were then broken and the samples removed and macro-etched in the same manner as in step 1., careful attention being made to note the position of the circle denoting the spot to be X-rayed.

12. Visual examinations were made to determine if recrystallization and grain growth had occurred or if the appearances had been altered in any manner.

13. Any changes in macro-structure were then recorded and photographs of newly recrystallized grains or unusual specimens taken. Figures 3a, 3b, 4a, 4b, 5a, 5b, 6a, 7a

and 7b are photographs of specimens which upon being strain-annealed recrystallized and produced relatively large perfect crystals. The specimens were 52, $55R_1$, 64R, 80R, and 111R. The "a" and "b" photographs were of opposite sides of the same specimen. Figure 8 is of specimen 42 which locally deformed upon elongation and recrystallized without substantial grain growth in the strained region when annealed. Figure 9 shows the finely recrystallized structure of specimen 43 following this technique.

14. The original positions of the X-ray circles were re-marked in the cases in which no recrystallization occurred, and the larger grains were marked on the recrystallized samples.

15. Electropolishes similar to step 5. were performed.

16. X-ray patterns, designated as omega, were taken to indicate changes in the extent of sub-structure. Similar conditions to step 6. were observed, except when in certain cases a 25 mil collimating system was employed so the unpolygonized structure of the recrystallized grains might be better shown. Typical omega Laue back-reflection photograms of non-recrystallized portions of recrystallized specimens 64R and 111R are Figures 5e and 7f, respectively. Figures 3d, 3e, 3f, 4d, 4e, 4f, 5d, 6c, 7d and 7e represent the perfect structures of recrystallized crystals in specimens 52, $55R_1$, 64R, 80R, and 111R.

The orientations of the newly recrystallized grains were determined by the Greninger method(36) and the rod axes of the recrystallized grains were plotted on a (001) standard projection (see Figure 17) similar to the (001) standard projection of the original pseudo crystal orientations (Figure 16). Stereographic projections showing the locations of the three principal poles of the five original crystals and their corresponding recrystallized grains with reference to the rod axis were plotted (see Figures 18, 19, 20, 21, and 22).

17. The samples were again macro-etched according to step 1.

18. The specimens were encapsulated as described in step 8. in order to prevent surface oxidation and to provide for safe storage.

The schedule for each individual specimen is condensed in Table II. Such information as extent of plastic deformation, maximum applied load, and anneal method is included.

C. Experimental Results

In a preliminary study, prior to the completion of the construction of the low temperature apparatus, 12 cylindrical pseudo single crystal specimens, 19, $21R_2$, 22, 23, $31R_2$, $33R_2$, 34, 38, 39, 41, 42, and 43, were elongated at room temperature. These specimens were chosen by the size of their pseudo portions predominantly, not their crystallographic orientations. Only five of these, $33R_2$, 34, 39, 42, and 43, exhibited any recrystallization following a 650°C anneal, and the absence of grain growth was evident by the small grained appearance of all the specimens. Specimen 42 exhibited an anomalous behavior for a uranium rod deformed by tension, because it locally deformed at one end of the pseudo crystal (see Figure 8).

A total of 15 cylindrical specimens, 5, 9B, 10A, $15DR_2$, 18, 25, $29R_2$, 37, 52, $55R_1$, 64R, 70R, 80R, 87R, and 111R, underwent the procedure peculiar to this investigation -- that is the elongation at liquid nitrogen temperature and subsequent 650°C anneal. Special attention was paid to assure that all pseudo crystals with the prescribed [100] orientation were included. Recrystallization occurred in six specimens -- pronounced grain growth was apparent in four, 52, $55R_1$, 64R, and 111R, (see Figures 3a and b; 4a and b; 5a and b; and 7a and b), but two, $15DR_2$ and 80R, remained finely grained. Specimen 80R failed at one end and recrystallized at the other end without recrystallizing at the point of fracture (see Figure 6a).

Tapered rods with a 10 % reduction in area at the center were fabricated. However, only two specimens T116 and T120, had relatively favorable pseudo crystal orientations for the study following the phase transformation technique. Specimen T 120 fractured during -196°C elongation and yielded medium sized grains near the broken portion after anneal.

Specimen 12C which had been accidently bent to a radius of curvature of approximately 2 inches was annealed in order to determine if such a deformation might induce a sufficient strain to cause recrystallization and grain growth. Recrystallization did not occur.

Deformation by compression was performed on six specimens, 96-1, -2, -3, -4, -5, and -6, which had been produced from one pseudo single crystal, 96. All of the specimens recrystallized; however, they were finely grained. The size of the grains decreased with increasing plastic deformation.

D. Discussion of Results

The results of this investigation established one very definite fact -- the orientation of the original pseudo single crystal is the cardinal factor involved in the removal of the sub-structure from alpha uranium by a -196°C tensile deformation followed by a 650°C anneal. Specimens 52, $55R_1$, 64R, and 111R were oriented such that their rod axes were situated in the spherical triangle formed by the

(001), (100), and (110) poles of a (001) standard projection (see Figure 16) and these produced the largest crystals. Preferential initial orientation, near the [100], supposedly allowed the metal to deform by a single deformation mechanism -- {130} twinning. Thus the deformation was relatively homogeneous in the pseudo crystal and no conflicting modes were present to produce nucleation centers at their intersections with the twins.

Crystals in specimens 55R, and 111R (Figures 4a and b, and 7a and b) definitely were brought about by coalescence -- the crystals grew parallel to the twin planes with the consumption of twinned regions. The recrystallization mechanism operative in these samples was a combination of "selective growth" and "oriented nucleation".

The largest crystals were produced in specimens which had been deformed at liquid nitrogen temperature. However, the exact effect of this factor could not be evaluated, since the specimens involved were those of the most favorable orientation. To determine the effectiveness of the introduction of the -196°C strain, two specimens of relatively the same favorable orientation should be strain-annealed, one elongated at -196°C, the other at room temperature. Nevertheless, this would pose the problem of straining the two specimens the same amount. Uranium is extremely brittle at the lower temperature and the elastic effect is pronouncedly different. Therefore for a given applied load the strains would be apt to vary widely, likewise for a given elongation. Such an investigation was not made during the course of the effort due to the lack of desirably oriented pseudo crystals. However, the author believes the incorporation of the -196°C elongation was beneficial to the results of the technique.

Deformation by bending of orthorhombic alpha uranium crystals of high purity does not promise the favorable results which were found for face-centered cubic aluminum of high purity by Leighly and Perkins(41). The occurrence of a multiplicity of deformation mechanisms for uranium and the consequent numerous nucleation centers is the primary basis for objection raised against such a treatment.

The specimens which were deformed by compression were originally at an orientation which would have given {130} twins and {010} ⟨100⟩ slip. The slip system could have been eliminated if the compression would have been performed at -196°C, according to Teeg(14), but suitable apparatus for low temperature compression was not available. The constraint of the platens upon the movement of the ends of the specimens might have introduced additional deformation mechanisms. Recrystallization occurred in all of the specimens obtained from pseudo crystal 96. Decreased grain size with increased compression did not necessarily mean that a critical strain for optimum grain growth had been exceeded, even in the least deformed specimen, but rather the nucleating effects of the various interacting mechanisms became more pronounced with increasing deformation. The author considers the means of performing the deformation, either by tension or compression, to be of relatively minor importance, if the proper apparatus is available so that end constraints do not exist to a detrimental degree.

The utilization of tapered specimens and the employment of a slowly rising temperature gradient anneal both appear to be factors, but of minor nature, in producing larger single crystals by this technique; although the experimental results related to these effects were limited in this investigation.

The primary purpose of the Laue back-reflection photograms was to confirm the perfection of the recrystallized grains. However, they were interpreted and the orientations of the recrystallized grains and original crystals were plotted on stereographic projections with respect to a (001) standard projection (see Figures 16 and 17), and with respect to the rod axis (see Figures 18 - 22).

Correlations between the recrystallized grain orientations and the original and deformed pseudo single crystal orientations were not calculated. Lloyd's(37) determination of a discrete rotation relationship involved many precise Laue back-reflection patterns from grains on planar surfaces within a single specimen. He performed various crystallographic operations, reflections and rotations, in order to group the principal poles of the recrystallized grains with respect to the original crystal orientation in a (001) standard projection. An average position for each group of poles was determined and the average orientation relationship was calculated. Noting Figures 18 - 22, it is doubtful that a reliable rotation relationship could be found, because there are so few grains for each original crystal, and they are extremely scattered.

IV. CONCLUSIONS

The sub-structure may be removed from alpha uranium pseudo single crystals by performing a -196°C tensile deformation and a subsequent 650°C anneal. Specimens $55R_1$ and 111R (see Figures 4a and b, and 7a and b, respectively) recrystallized to produce several perfect crystals which were larger than any reported by other strain-anneal investigators, and which were comparable in size and perfection to the less pure crystals produced by the grain coarsening method. Other specimens confirmed the feasibility of this approach, however, they exhibited only moderate grain growth.

Perfect crystals may be best produced by the technique if the original pseudo single crystals, which are produced by the phase transformation method, have their rod axes oriented near [100].

The introduction of the liquid nitrogen temperature strain is believed to increase the size of subsequently recrystallized perfect crystals. Tapered rods, slowly rising temperature anneals, and thermal gradient anneals are felt to be only of minor importance, but perhaps worthwhile refinements to the technique.

The prospects for future attempts to grow larger perfect single crystals of alpha uranium will probably also involve some means of propagating large-angle grain boundaries through pseudo single crystals. Fraser, Gold, and Mullins(42) have recently reported a technique by which the grain boundaries of locally deformed bismuth were induced to migrate by the incorporation of a strong magnetic field (11.88 X 10^4 gauss) in the annealing step. The magnetic pressure which was exerted on the mobile boundaries was related to the anisotropic behavior of the magnetic susceptibilities. Bismuth crystals four centimeters in length and 0.4 centimeters in diameter have been produced by their method.

Anisotropic magnetic susceptibility values have not been reported for alpha uranium, but Gordon(43) suggests the probability of such a behavior. Although it is likely that the susceptibility differences would be less marked for uranium than bismuth, the application of a strong magnetic field during the annealing step of the procedure employed in this investigation might produce still larger single crystals.

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TABLE I

COMPOSITION OF SPECIMEN LOTS USED IN THE STUDY OF THE REMOVAL OF SUB-STRUCTURE FROM ALPHA URANIUM

(Composition in p.p.m.)

Impurity	Lots					
	43T*	43B	60T	60B	100	158
Ag	L 1.0**	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0
Al	5.0	5.0	L 5.0	L 5.0	5.0	L 5.0
As	L 10.0	L 10.0	L 10.0	L 10.0	L 10.0	L 10.0
B	L 0.1	L 0.1	L 0.1	L 0.1	L 0.1	0.2
Be	L 0.5	L 0.5	L 0.5	L 0.5	L 0.5	L 0.5
Bi	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0
C	3.5	6.0	7.0	4.0	7.5	12.0
Ca	L 20.0	L 20.0	L 20.0	L 20.0	L 20.0	L 20.0
Co	L 5.0	L 5.0	L 5.0	L 5.0	L 5.0	L 5.0
Cr	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0
Cu	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0
Fe	5.0	4.0	2.0	2.0	2.0	2.0
K	L 50.0	L 50.0	L 50.0	L 50.0	L 20.0	L 50.0

* T - top, B - bottom (same ingot)
** L - less than

TABLE I (continued)

Impurity	Lots					
	43T	43B	60T	60B	100	158
Li	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0
Mg	2.0	1.0	L 1.0	L 1.0	L 1.0	2.0
Mn	L 1.0	L 1.0	L 1.0	L 1.0	2.0	2.0
Mo	L 20.0	L 20.0	L 20.0	L 20.0	L 20.0	L 20.0
N	L 10.0	13.0	15.0	16.0	L 10.0	L 10.0
Na	L 10.0	L 10.0	L 10.0	L 10.0	L 10.0	L 10.0
Ni	L 5.0	L 5.0	L 5.0	L 5.0	L 5.0	L 5.0
O	19.0	45.0	29.0	27.0	13.0	29.0
P	L 50.0	L 50.0	L 50.0	L 50.0	L 50.0	L 50.0
Pb	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0
Sb	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0	L 1.0
Si	10.0	10.0	15.0	10.0	15.0	10.0
Sn	L 5.0	L 5.0	L 5.0	L 5.0	L 5.0	L 5.0
Ti	L 50.0	L 50.0	L 50.0	L 50.0	L 50.0	L 50.0
Zn	L 50.0	L 50.0	L 50.0	L 50.0	L 50.0	L 50.0

TABLE II

INDIVIDUAL SPECIMEN SCHEDULES

			Length		Deformation			Anneal		Remarks
No.	Lot	Dia. (in)	Rod (in)	Pseudo (in)	%	Load (kg)	Temp. (°C)	Time (hr)	Temp. (°C)	
5	43	0.08	2.4	1.0	L' 1.	20	-196	48	645	
9B	43	0.08	3.9	3.6	0.8	44	-196	48	650	F*
10A	43	0.08	3.9	3.1	L 1.	22	-196	47	645	
12C	43	0.08	3.8	3.8	bent	--	RT#	50	650	
$15DR_2$	43	0.16	3.8	1.7	8.	200	-196	41	640	F; R** - small grains
18	43	0.25	4.0	3.0	1.	335	-196	48	650	F
19	43	0.25	4.2	2.6	1.	760	RT	50	650	

'L - less than
*F - specimen fractured during deformation
#RT - room temperature
**R - specimen recrystallized during anneal
^{1}TG - 200°C in 4 inches - thermal gradient
"q - liquid nitrogen quenched - then deformed
^{2}TG - 400°C in 4 inches - thermal gradient
◊T - tapered specimen

TABLE II (continued)

			Length		Deformation			Anneal		Remarks
No.	Lot	Dia. (in)	Rod (in)	Pseudo (in)	%	Load (kg)	Temp. (°C)	Time (hr)	Temp. (°C)	
$21R_2$	100	0.25	3.9	2.0	3.	540	RT	50	650	
22	100	0.25	4.0	2.5	240.	310	RT -196	94	650	
23	100	0.25	3.8	2.2	1.	500	RT	50	650	
25	100	0.25	4.0	2.5	1.	420	-196	48	650	F
$29R_2$	100	0.25	3.7	2.8	1.	200	-196	48	650	
31R	100	0.25	3.5	2.4	L 1.	175	RT	94	650	
$33R_2$	100	0.25	4.4	3.0	1.	305	RT	54	650	R - small grains
34	100	0.25	4.8	4.0	8.5	375	RT	66	650	R - small grains
37	100	0.25	4.8	4.0	L 1.	250	-196	65	650	
38	100	0.25	4.9	4.5	10.	550	RT	48	650	
39	100	0.25	3.8	2.5	10.	235	RT	91	650	F; R - very fine grains
41	100	0.25	4.7	4.0	6.	225	RT	49	650	
42	100	0.25	4.9	4.1	6.	430	RT	48	650	R - small grains - local
43	100	0.25	4.6	2.5	6.5	350	RT	48	650	R - very fine grains

TABLE II (continued)

			Length		Deformation			Anneal		Remarks
No.	Lot	Dia. (in)	Rod (in)	Pseudo (in)	%	Load (kg)	Temp. (°C)	Time (hr)	Temp. (°C)	
52	100	0.25	4.8	3.5	2.	185	-196	97	650	R - medium (0.1 - 0.3 in.)
55R$_1$	60	0.25	3.2	2.2	3.	222	-196	22 90	T6[1] 650	R - medium (0.1 - 0.3 in.)
64R	60	0.25	3.7	2.4	3.	140	-196	48	645	R - large (0.2 - 0.5 in.)
70R	60	0.25	3.6	2.6	3.	172	-196	47	TG[1]	
80R	60	0.25	3.5	2.5	4.	500	-196	48	645	F; R - small grains
87R	60	0.25	3.5	3.0	2.	235	-196	48	645	
96	60	0.25	4.2	3.1	--	--	--	--	--	Original rod-compression samples
96-1	60	0.25	0.5	0.5	c 2.4	156	RT	44	650	R - small grains
96-2	60	0.25	0.5	0.5	c 3.2	187	RT	44	650	R - smaller grains
96-3	60	0.25	0.5	0.5	c 3.8	190	RT	44	650	R - smaller grains
96-4	60	0.25	0.5	0.5	c 5.6	222	RT	44	650	R - fine grains

TABLE II (continued)

			Length		Deformation			Anneal		Remarks
No.	Lot	Dia. (in)	Rod (in)	Pseudo (in)	%	Load (kg)	Temp. (°C)	Time (hr)	Temp. (°C)	
96-5	60	0.25	0.5	0.5	c 1.6	175	-196q"	44	650	R - small grains
96-6	60	0.25	0.5	0.5	c 7	250	-196q"	44	650	R - very fine grains
111R	60	0.25	4.1	2.5	2	83	-196	27 64	TG^2 650	R - large (0.5 in.)
T116	158	0.25	4.0	2.0	1	300	-196	48	650	
T120	158	0.25	4.0	4.0	2.5	382	-196	48	650	F; R - medium grains

Figure 2. Specimen 70R - example of substructure in upper pseudo single crystal. 1X.

Figure 3a. Figure 3b.

Figure 3a. Specimen 52 - recrystallized grain distribution following modified (-196°C) strain-anneal. 1X.

Figure 3b. Specimen 52 - rotated about rod axis 180°. 1 X.

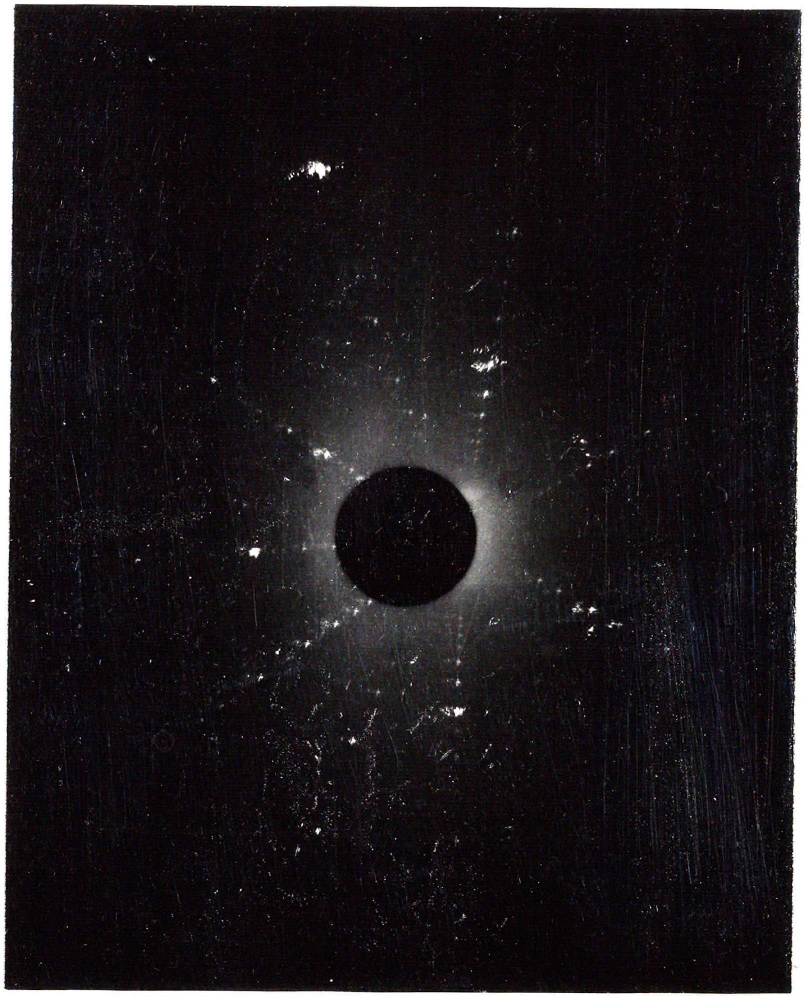

Figure 3c. Specimen 52 - alpha Laue back-reflection photogram - striated reflections from planes close to (001). Cu target, 2 hour exposure, 10 mil pinhole, 3 cm.

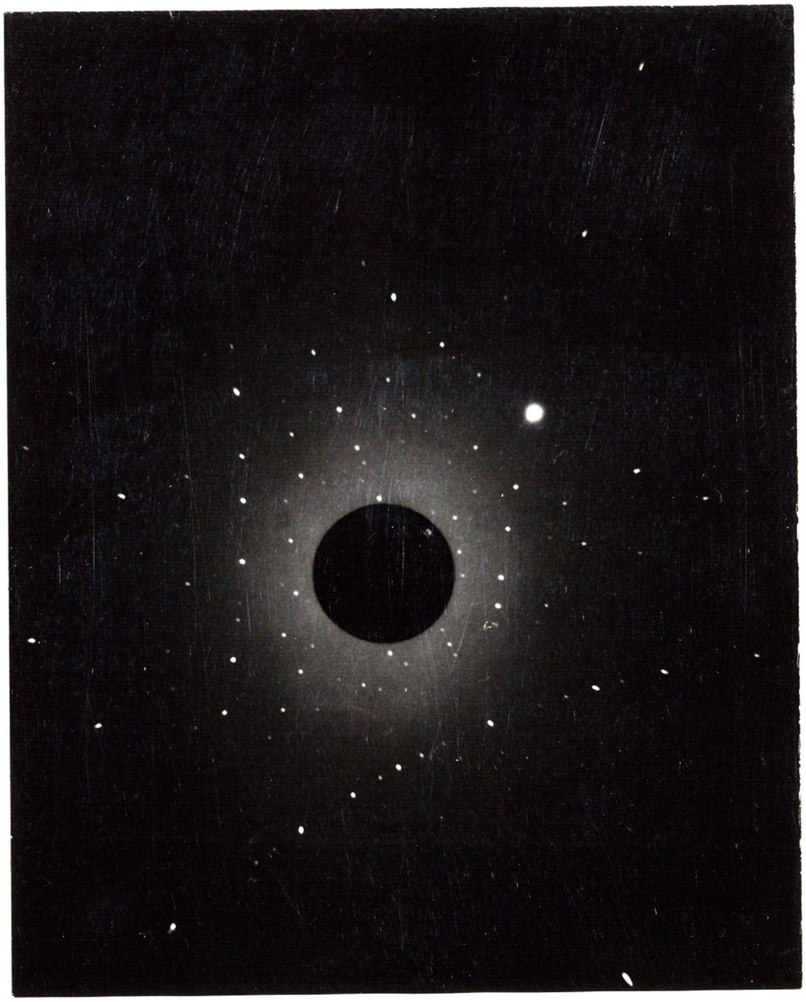

Figure 3d. Specimen 52, grain #1 - omega Laue back-reflection photogram - perfect structure reflections from planes near (100). Cu target, 2 hour exposure, 10 mil pinhole, 3 cm. distance.

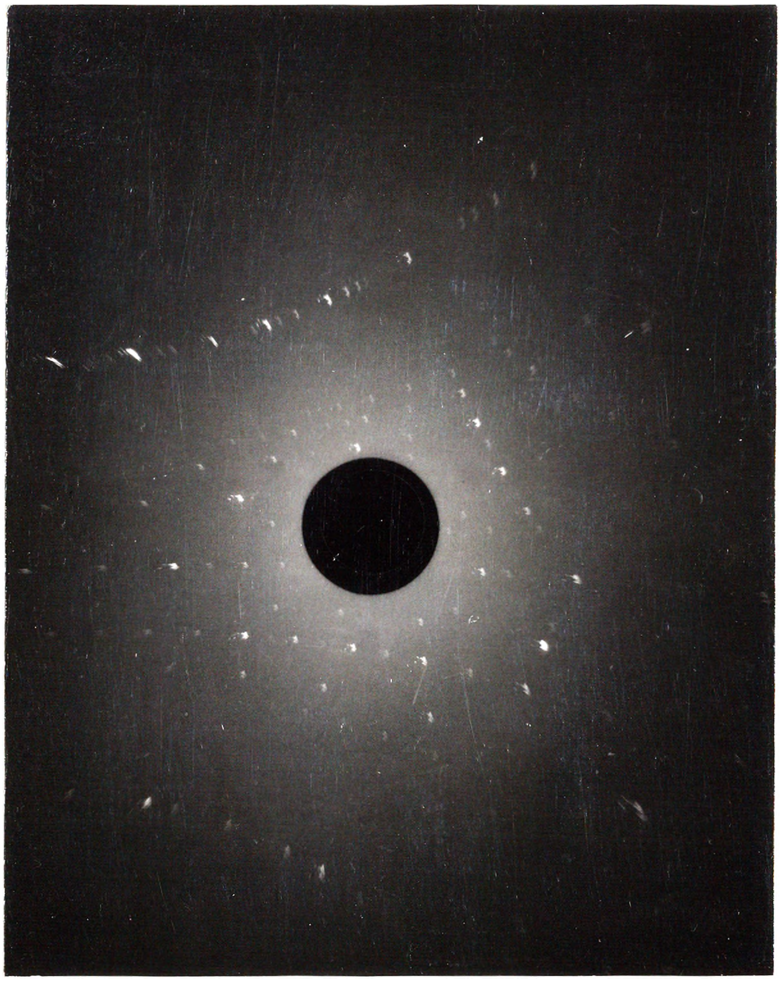

Figure 3e. Specimen 52, grain # 2 - omega Laue back-reflection photogram - near-perfect structure reflections from planes near (201). Cu target, 2 hour exposure, 10 mil pinhole, 3 cm. distance.

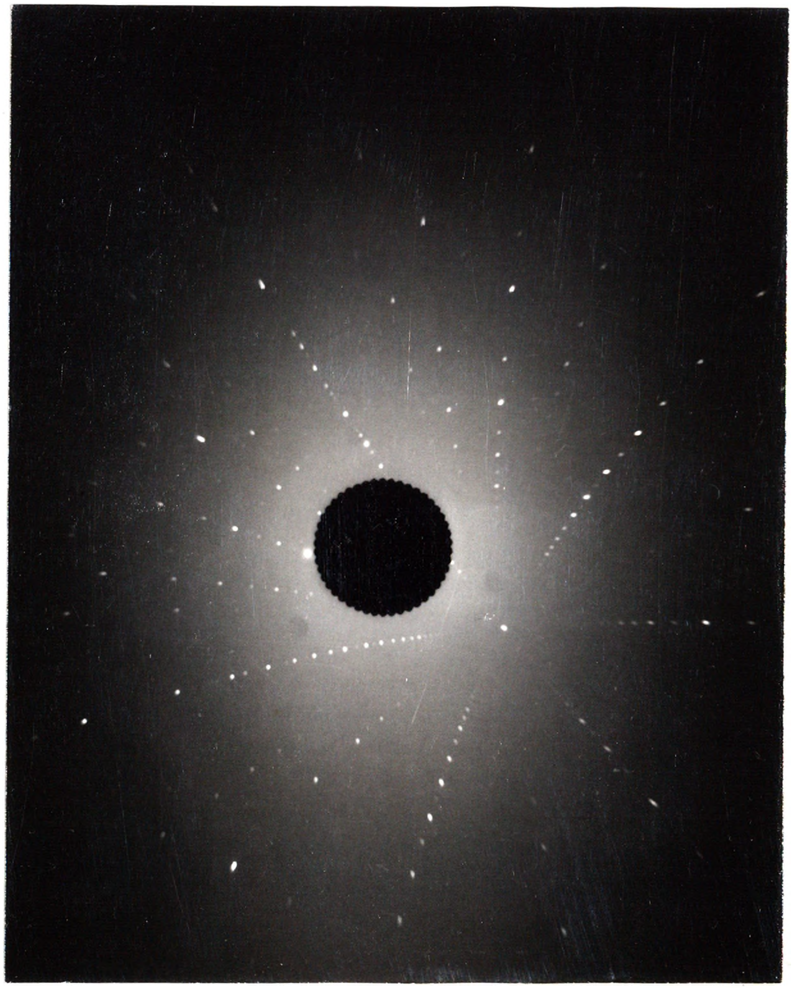

Figure 3f. Specimen 52, grain # 3 - omega Laue back-reflection photogram - perfect structure reflections from planes near (001). Cu target, 2hour exposure, 10 mil pinhole, 3 cm. distance.

Figure 4a.

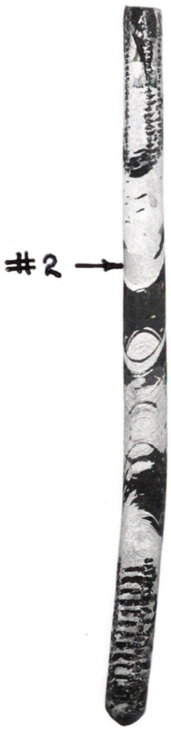

Figure 4b.

Figure 4a. Specimen $55R_1$ - recrystallized grain distribution following modified (-196°C) strain-anneal. (Note residual twin traces.) 1X.

Figure 4b. Specimen $55R_1$ - rotated about rod axis 180°. 1X.

Figure 4c. Specimen $55R_1$ - alpha Laue back-reflection photogram - striated reflections from planes close to (001). Cu target, 2 hour exposure, 10 mil pinhole, 3 cm. distance.

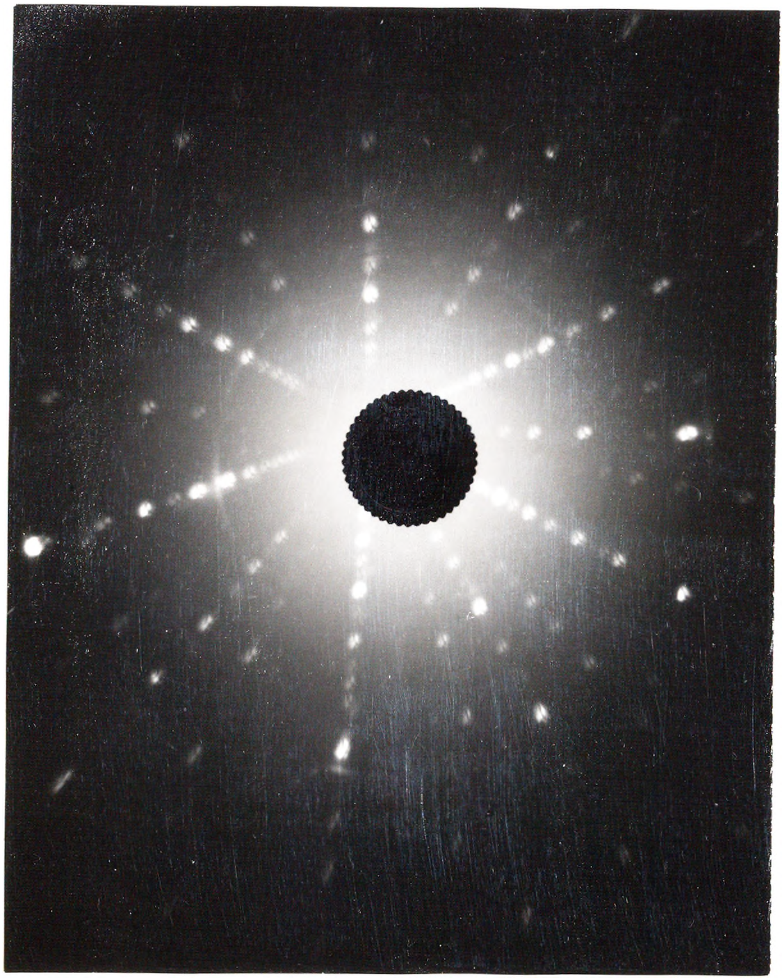

Figure 4d. Specimen $55R_1$, grain # 1 - omega Laue back-reflection photogram - near-perfect, but twinned structure reflections from planes near (001). Cu target, 2 hour exposure, 25 mil pinhole, 3 cm. distance.

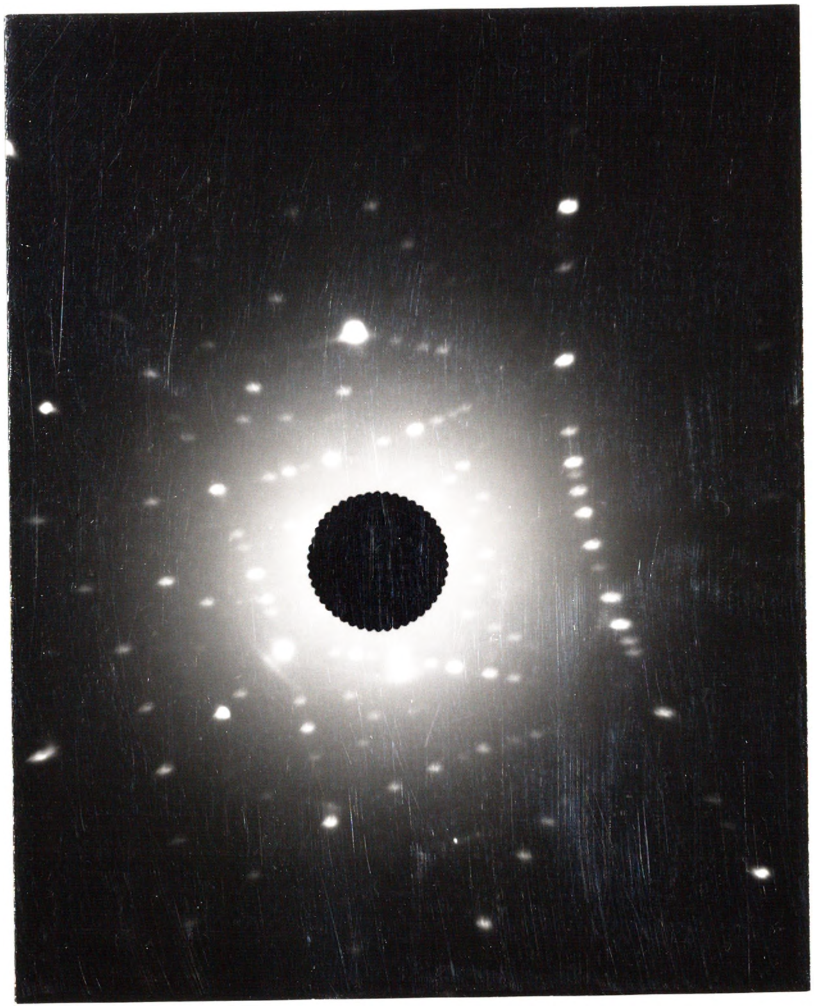

Figure 4e. Specimen $55R_1$, grain # 2 - omega Laue back-reflection photogram - perfect structure reflections from planes between (010) and (132). Cu target, 2 hour exposure, 25 mil pinhole, 3 cm. distance.

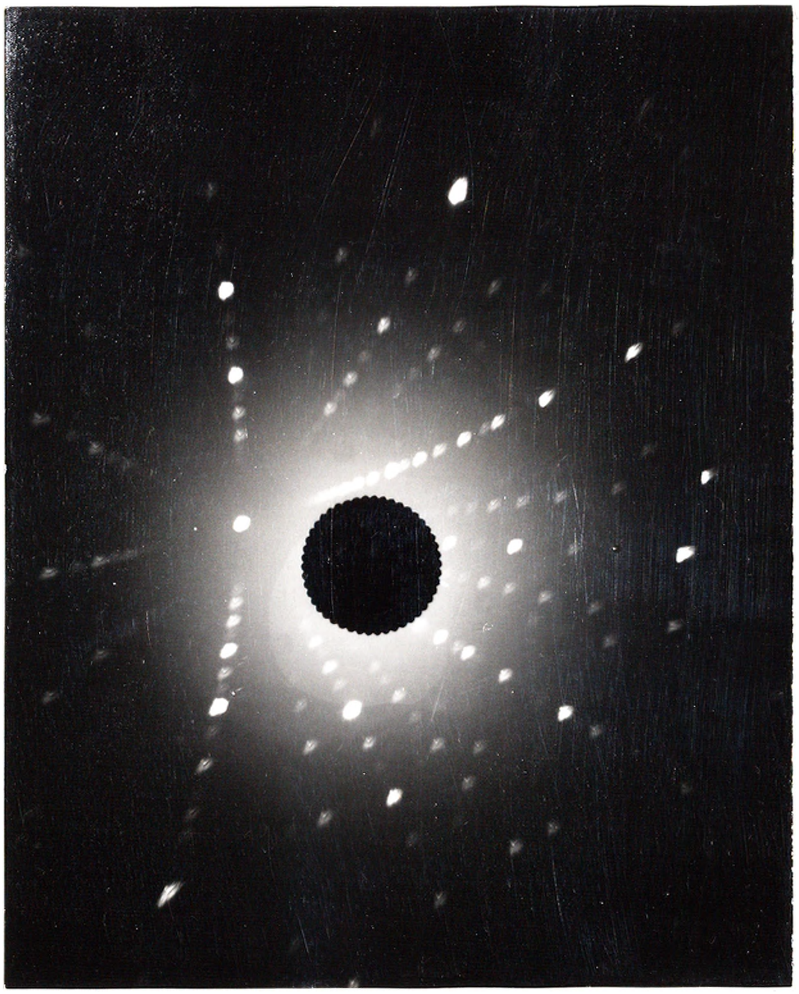

Figure 4f. Specimen $55R_1$, grain # 3 - omega **Laue** back-reflection photogram - perfect structure reflections from planes near (001). Cu target, 2 hour exposure, 25 mil pinhole, 3 cm. distance.

Figure 5a.

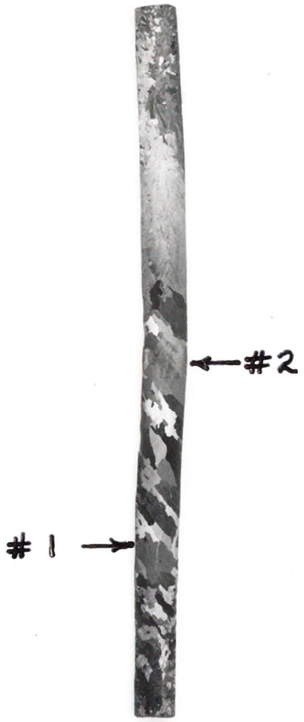

Figure 5b.

Figure 5a. Specimen 64R - recrystallized grain distribution following modified (-196°C) strain-anneal. 1X.

Figure 5b. Specimen 64R - rotated about rod axis 180°. 1X.

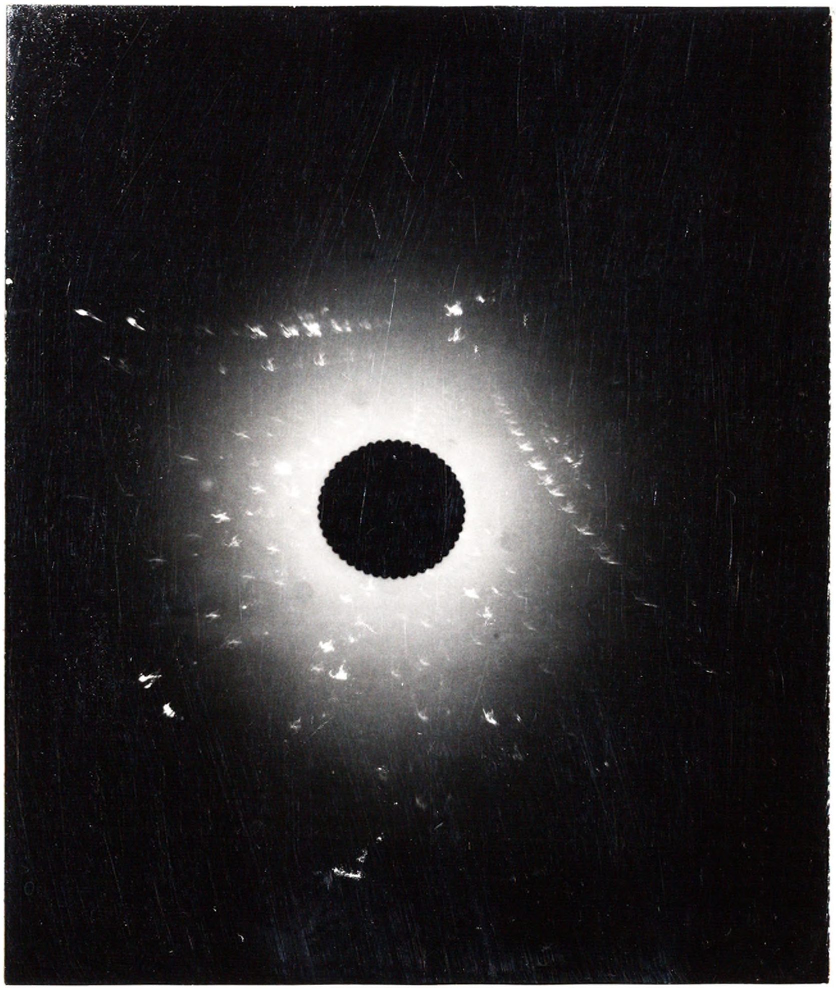

Figure 5c. Specimen 64R - alpha Laue back-reflection photogram - striated reflections from planes near (001). Cu target, 2 hour exposure, 10 mil pinhole, 3 cm. distance.

Figure 5d. Specimen 64R, grain #1 - omega Laue back-reflection photogram - perfect structure reflections from planes near (010). Cu target, 2 hour exposure, 10 mil pinhole, 3 cm. distance.

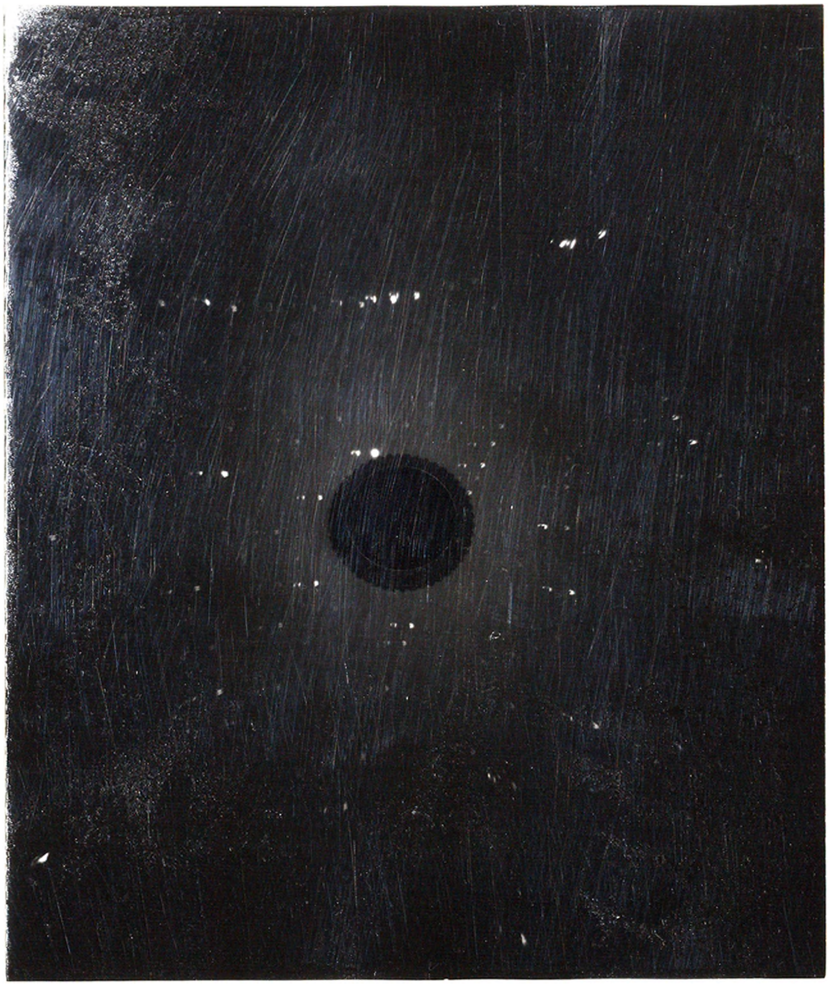

Figure 5e. Specimen 64R, grain # 2 - omega Laue back-reflection photogram - striated reflections from planes near (001). Cu target, 10 mil pinhole, 2 hour exposure, 3 cm. distance.

Figure 6a. Specimen 80R - recrystallized grain distribution following fracture in modified (-196°C) strain-anneal. (Note absence of recrystallization at point of failure.) 1X.

Figure 6b. Specimen 80R - alpha Laue back-reflection photogram- radically striated reflections from planes near (001). Cu target, 4 hour exposure, 10 mil pinhole, 3 cm. distance.

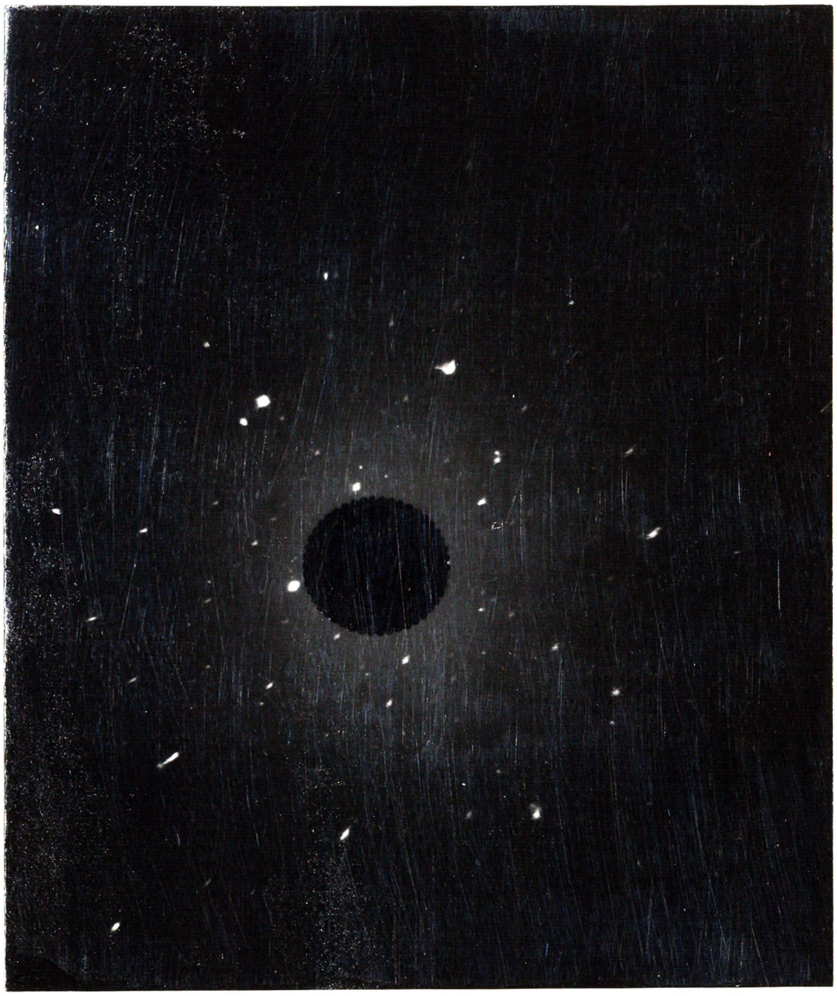

Figure 6c. Specimen 80R, grain #1 - omega Laue back-reflection photogram - perfect structure reflections from planes near (110). Cu target, 2 hour exposure, 10 mil pinhole, 3 cm. distance.

Figure 7a.

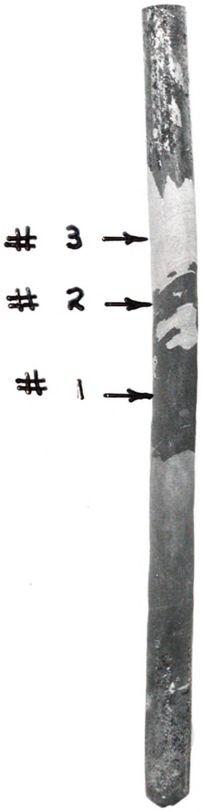

Figure 7b.

Figure 7a. Specimen 111R - recrystallized grain distribution following modified (-196°C) strain-anneal. (Note the grain growth by coalescence.) 1X.

Figure 7b. Specimen 111R - rotated about rod axis 180°. 1X.

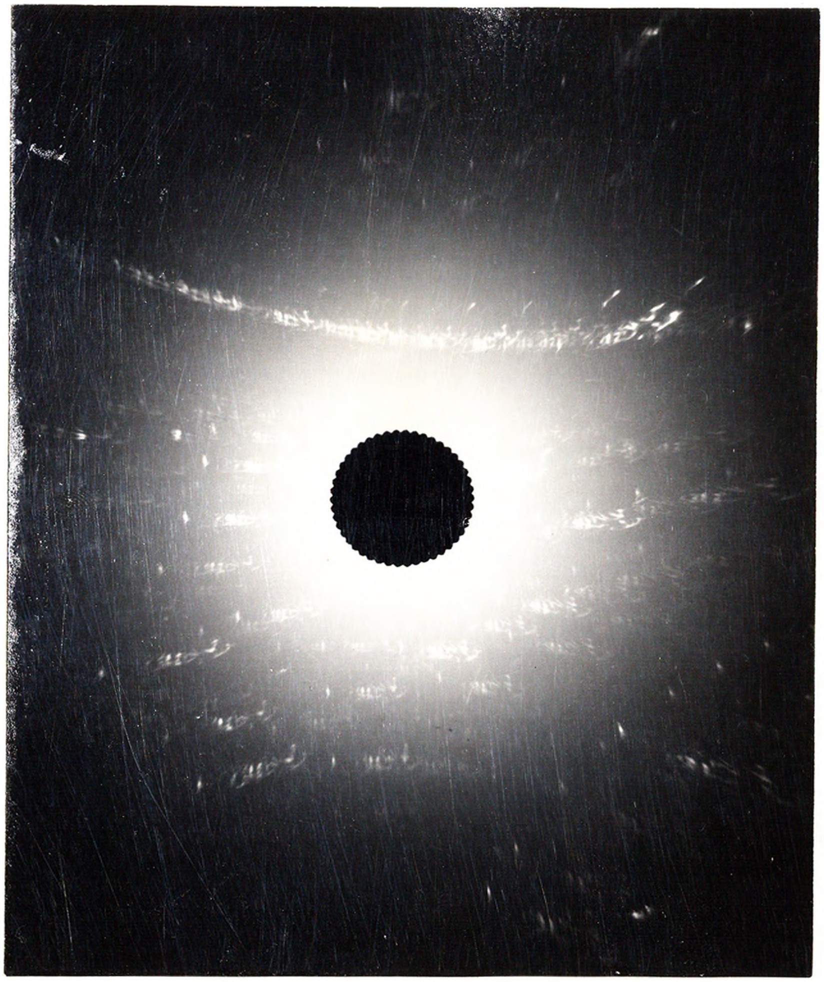

Figure 7c. Specimen 111R - alpha Laue back-reflection photogram - radically striated reflections from planes between (131), (021), and (111). Cu target, 4 hour exposure, 10 mil pinhole, 3 cm. distance.

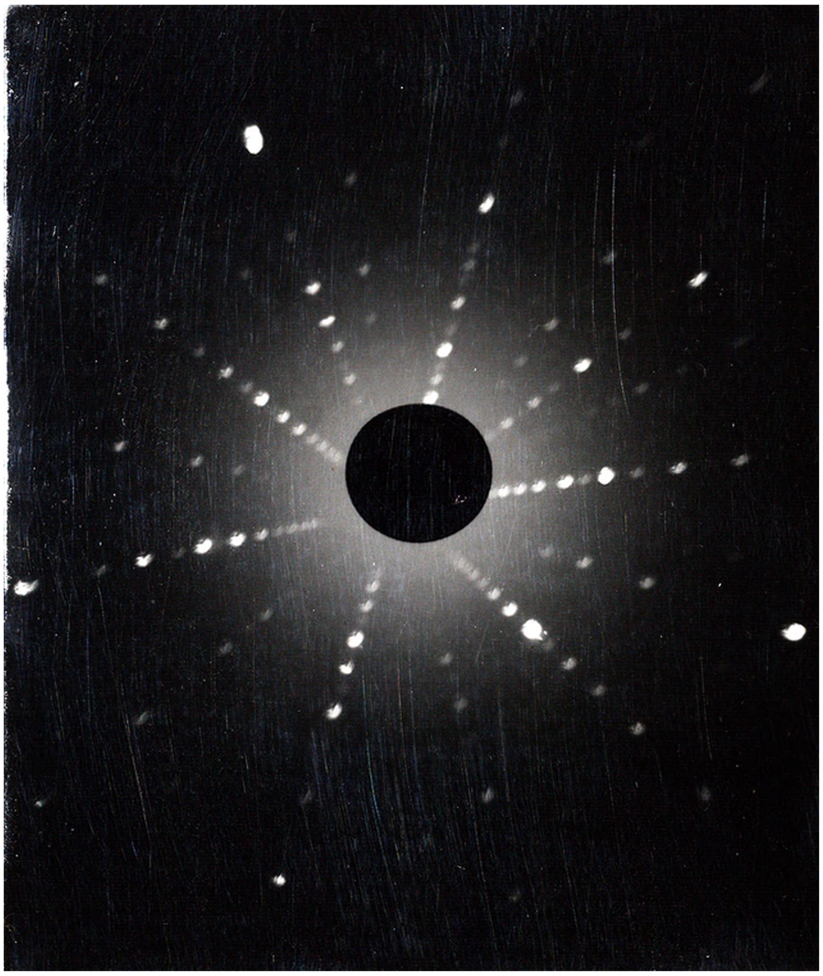

Figure 7d. Specimen 111R, grain # 1 - omega Laue back-reflection photogram - near-perfect structure with slight twinning, reflections from planes near (001). Cu target, 2 hour exposure, 25 mil pinhole, 3 cm. distance.

Figure 7e. Specimen 111R, grain # 2 - omega Laue back-reflection photogram - perfect structure reflections from planes close to (001). Cu target, 2 hour exposure, 25 mil pinhole, 3 cm. distance.

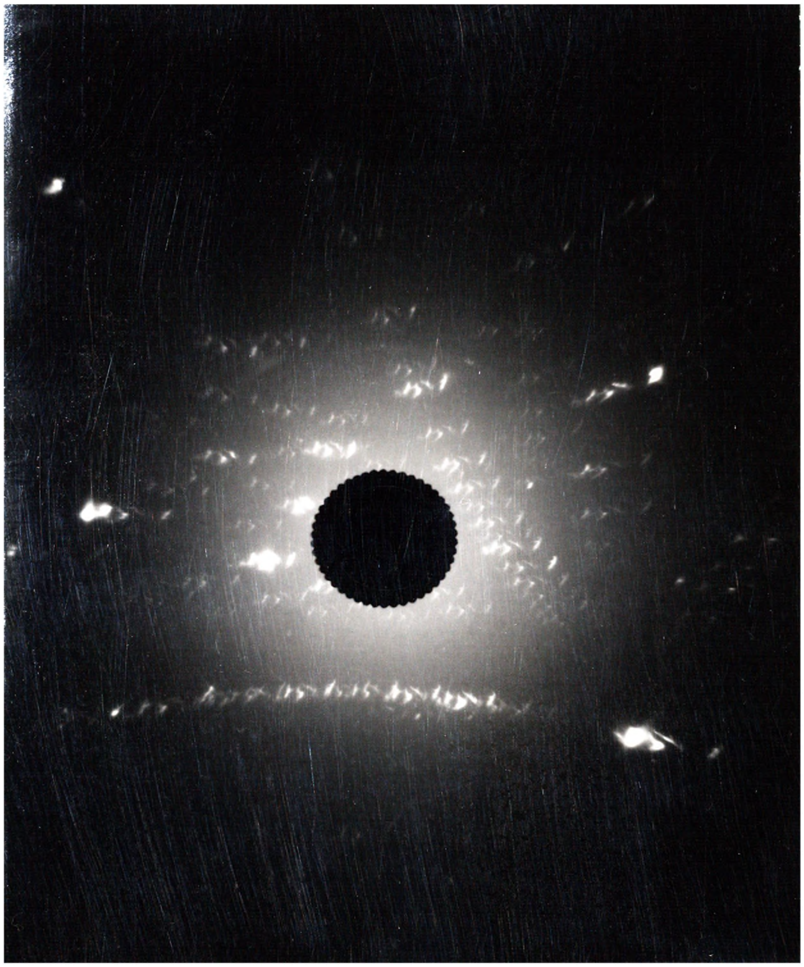

Figure 7f. Specimen 111R, grain # 3 - omega Laue back-reflection photogram. - still polygonized; reflections from planes near (100). Cu target, 2 hour exposure, 25 mil pinhole, 3 cm. distance.

Figure 8.

Figure 9.

Figure 8. Specimen 42 - recrystallized grain distribution at locally deformed portion following modified (-196°C) strain-anneal. 1X.

Figure 9. Specimen 43 - very fine recrystallized grain distribution following modified (-196°C) strain-anneal. 1X.

Figure 10. Low temperature rig incorporated with the Instron tensile testing machine - Argonne National Laboratory.

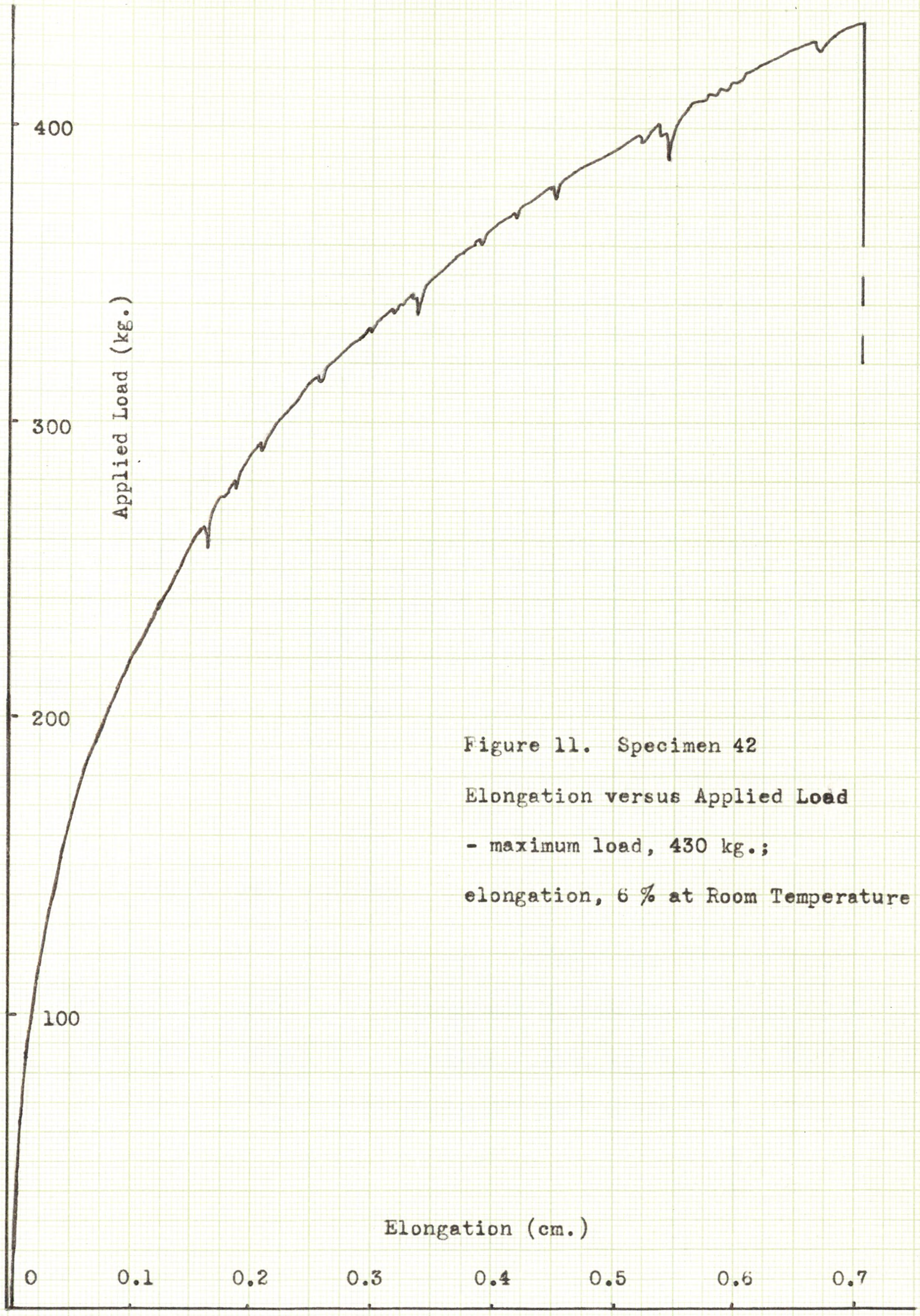

Figure 11. Specimen 42

Elongation versus Applied Load

- maximum load, 430 kg.;

elongation, 6 % at Room Temperature

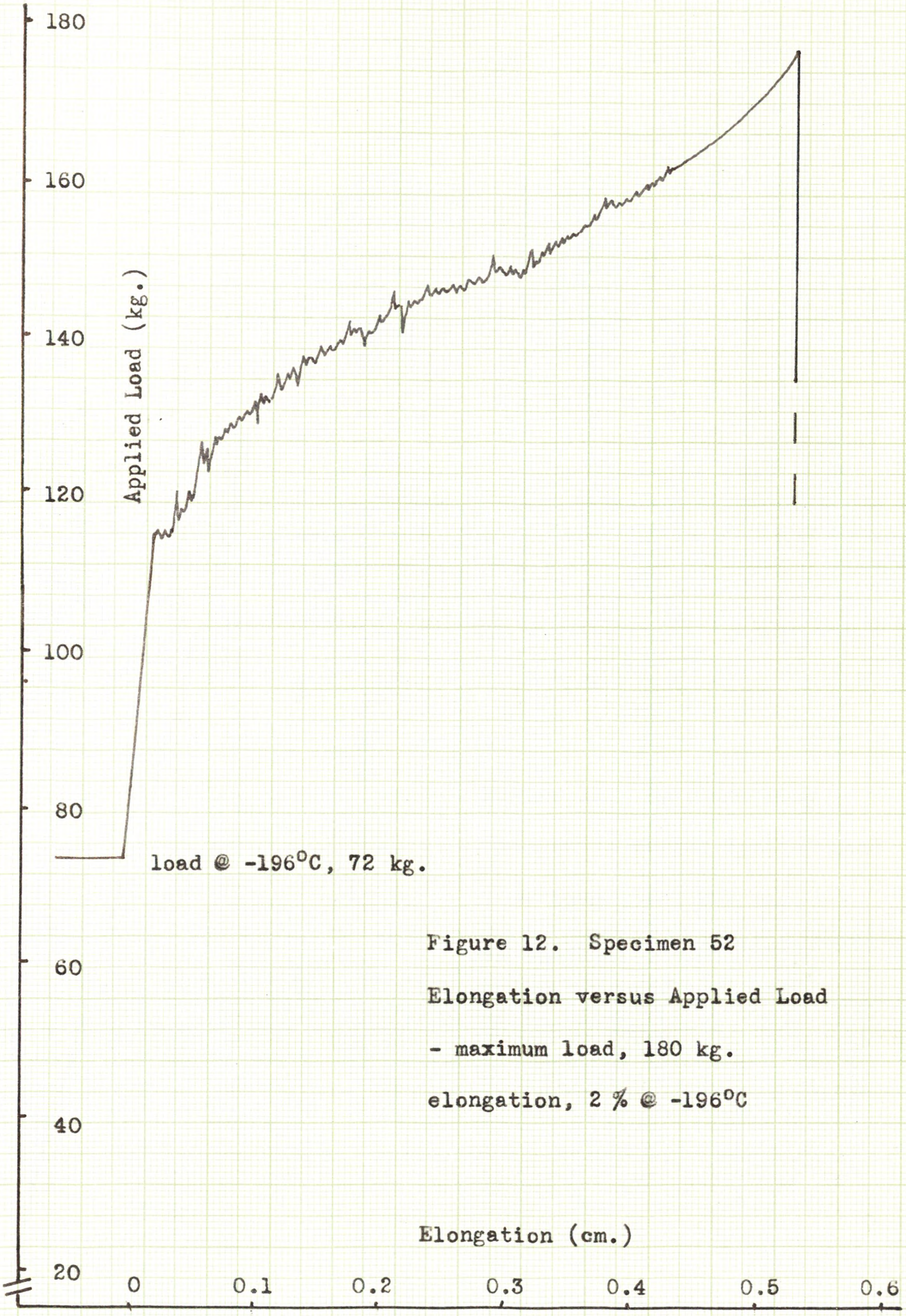

Figure 12. Specimen 52

Elongation versus Applied Load

- maximum load, 180 kg.

elongation, 2 % @ -196°C

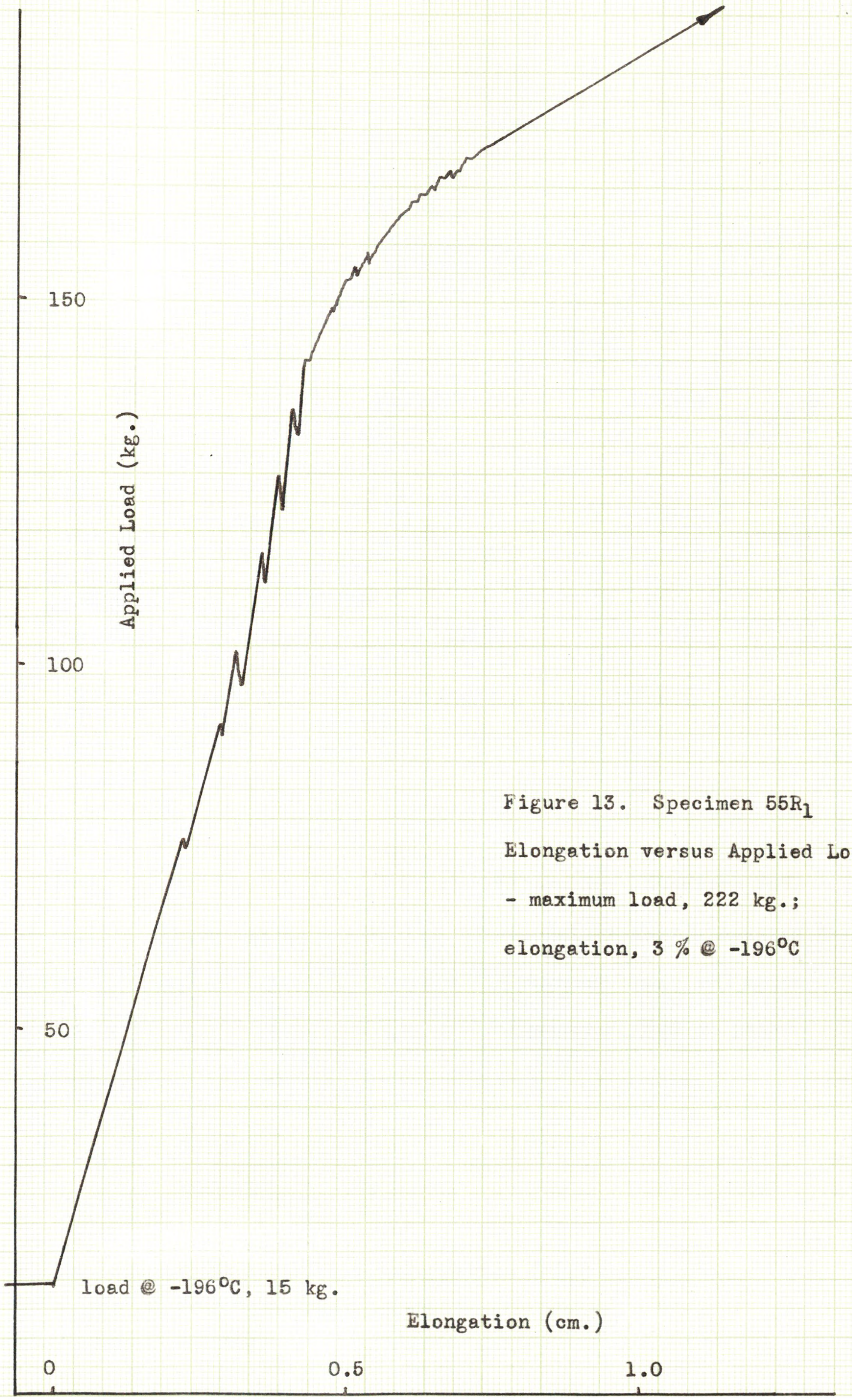

Figure 13. Specimen $55R_1$

Elongation versus Applied Load

- maximum load, 222 kg.;

elongation, 3 % @ -196°C

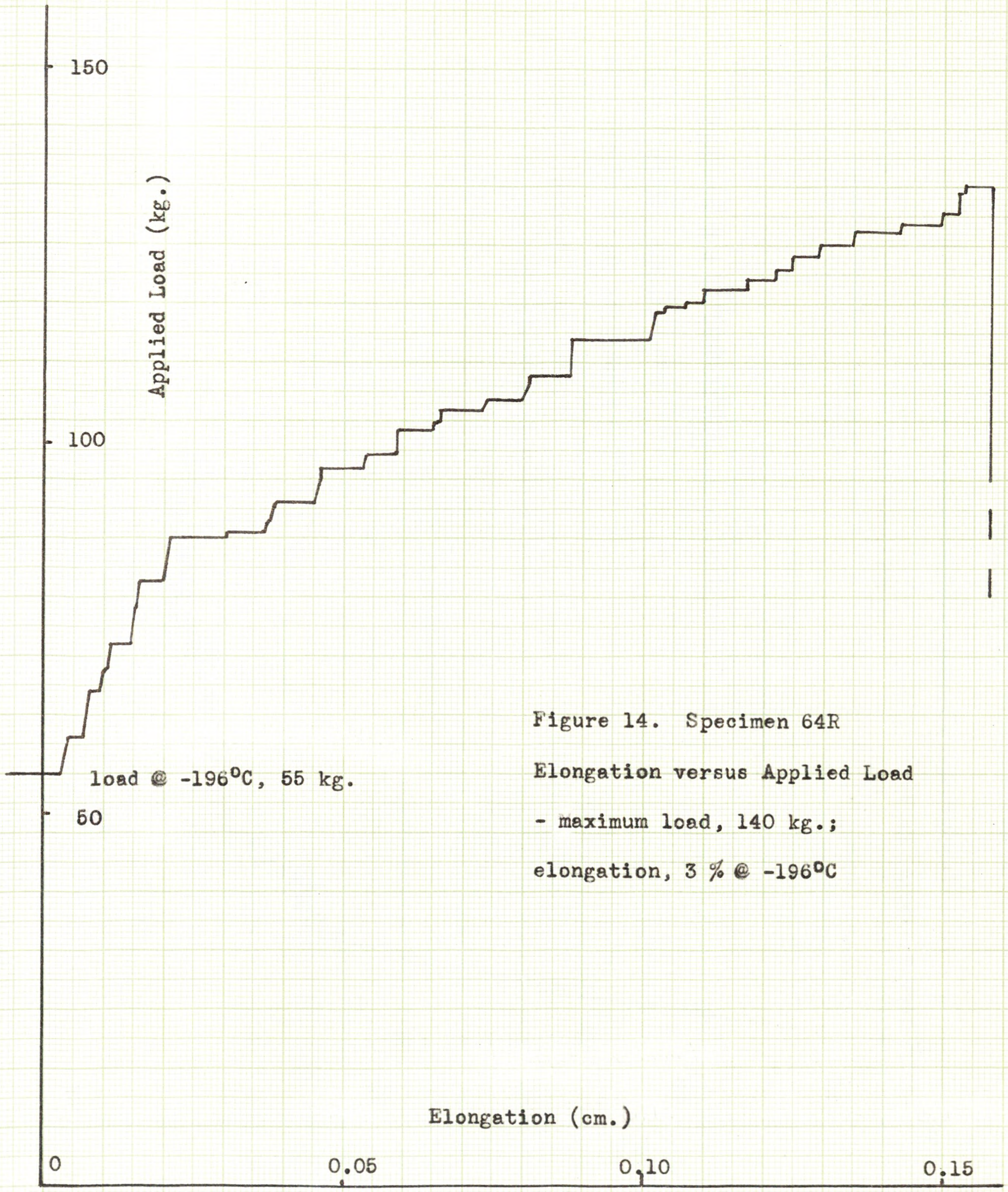

Figure 14. Specimen 64R

Elongation versus Applied Load

- maximum load, 140 kg.;

elongation, 3 % @ -196°C

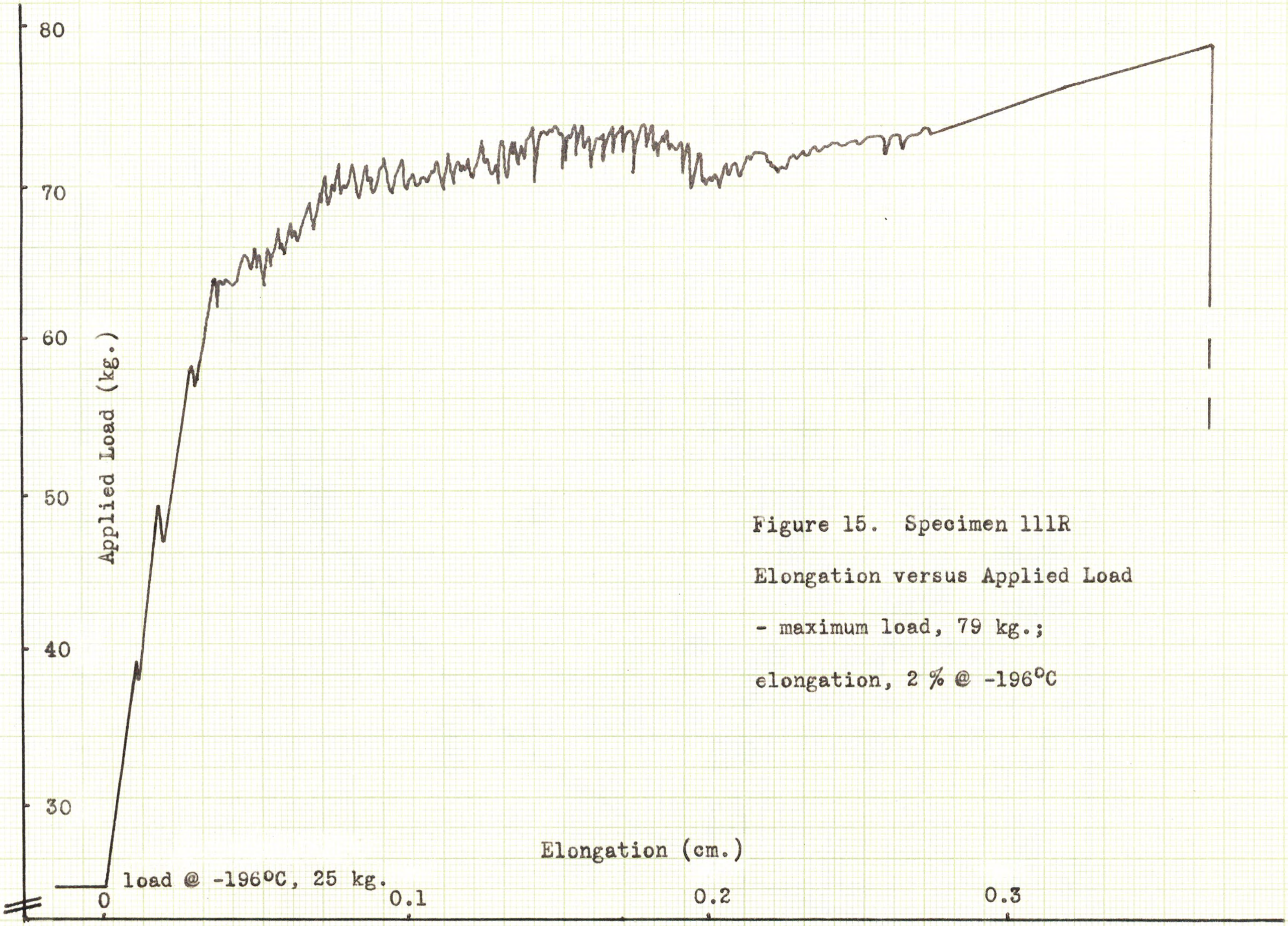

Figure 15. Specimen 111R

Elongation versus Applied Load

- maximum load, 79 kg.;

elongation, 2 % @ -196°C

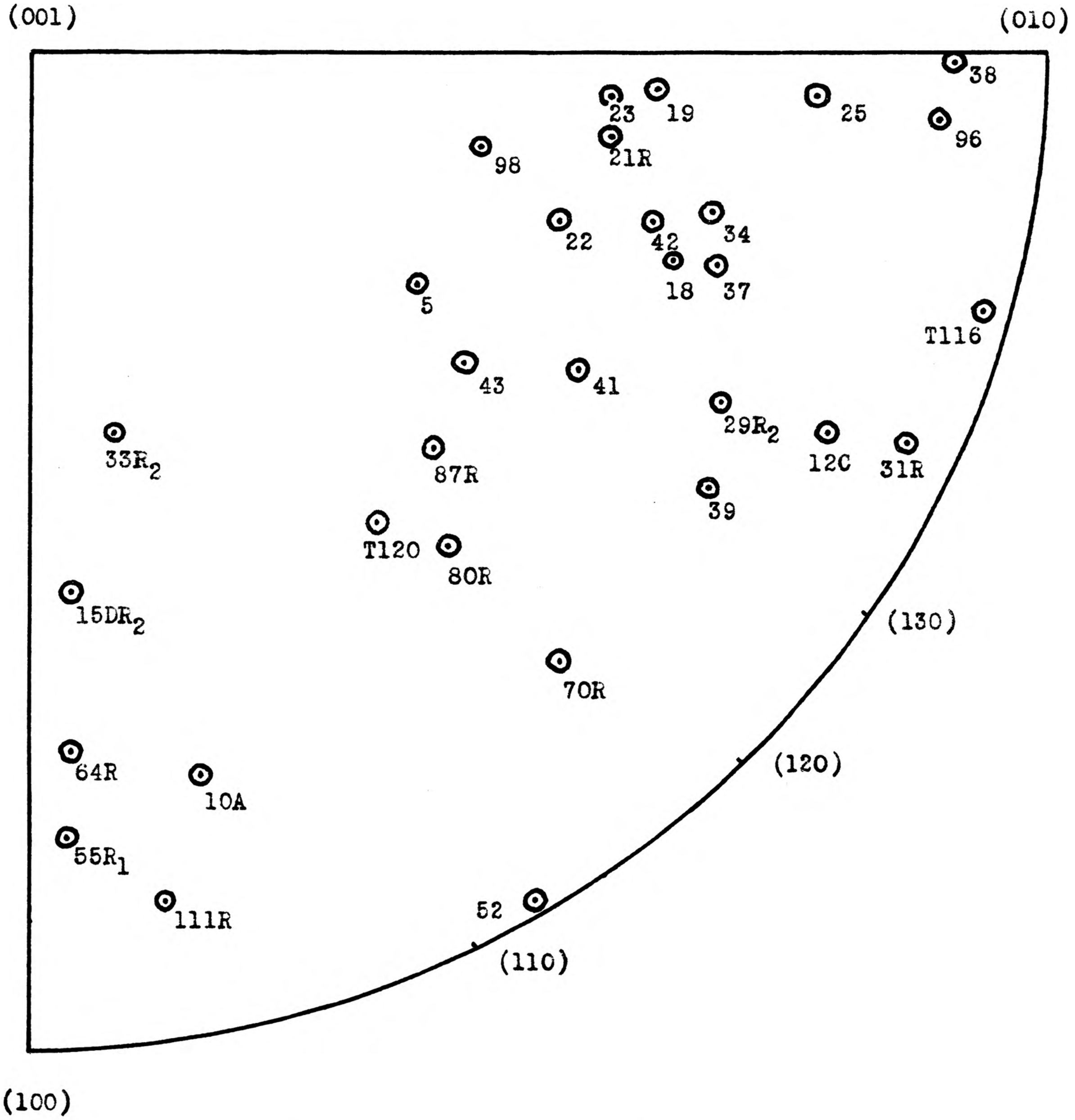

Figure 16. Grouping of the Rod Axes of the Original Pseudo Crystals Utilized in This Investigation in (001) Standard Projection.

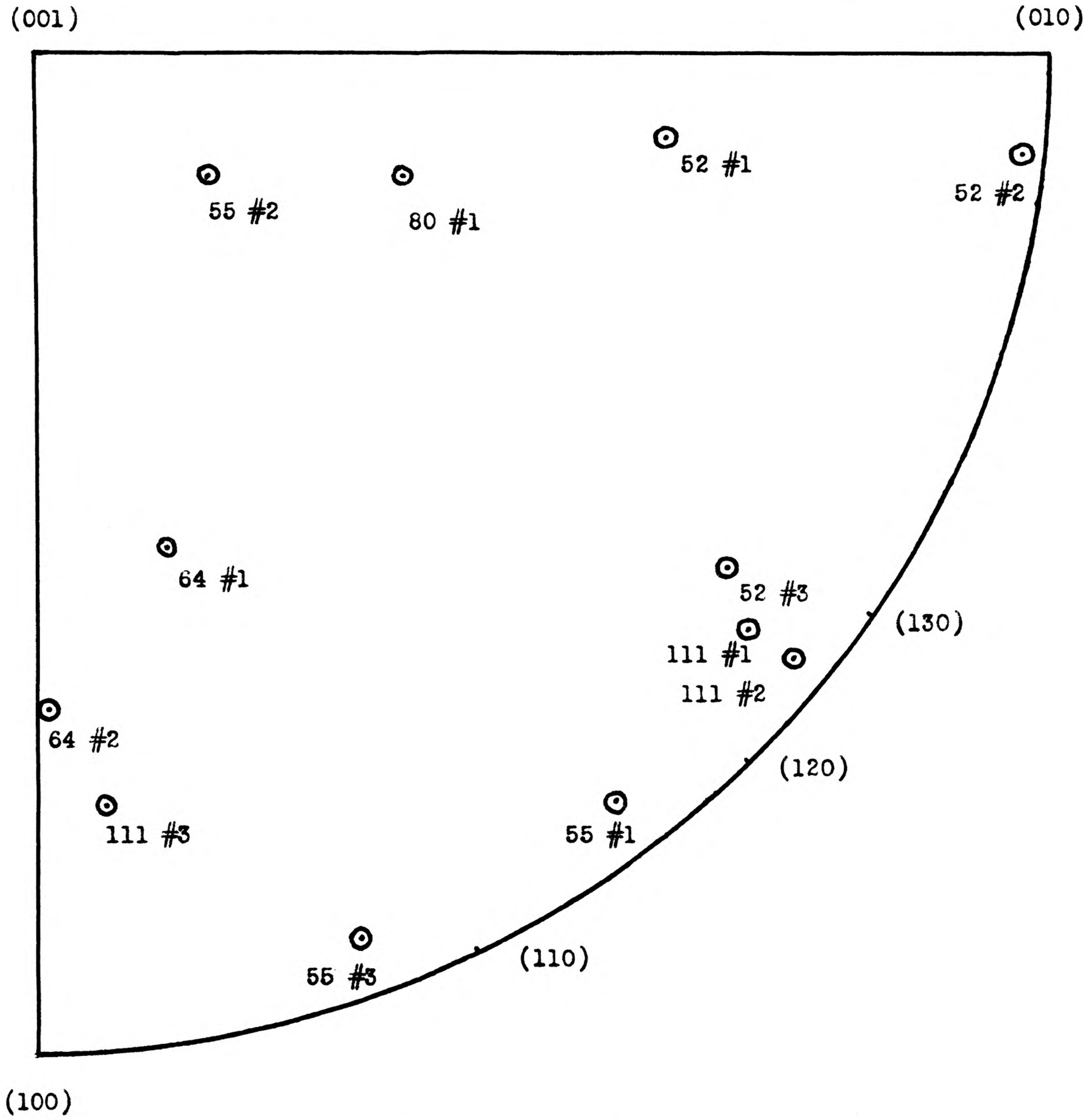

Figure 17. Grouping of the Rod Axes of the Recrystallized Grains Produced in the Investigation in (001) Standard Projection.

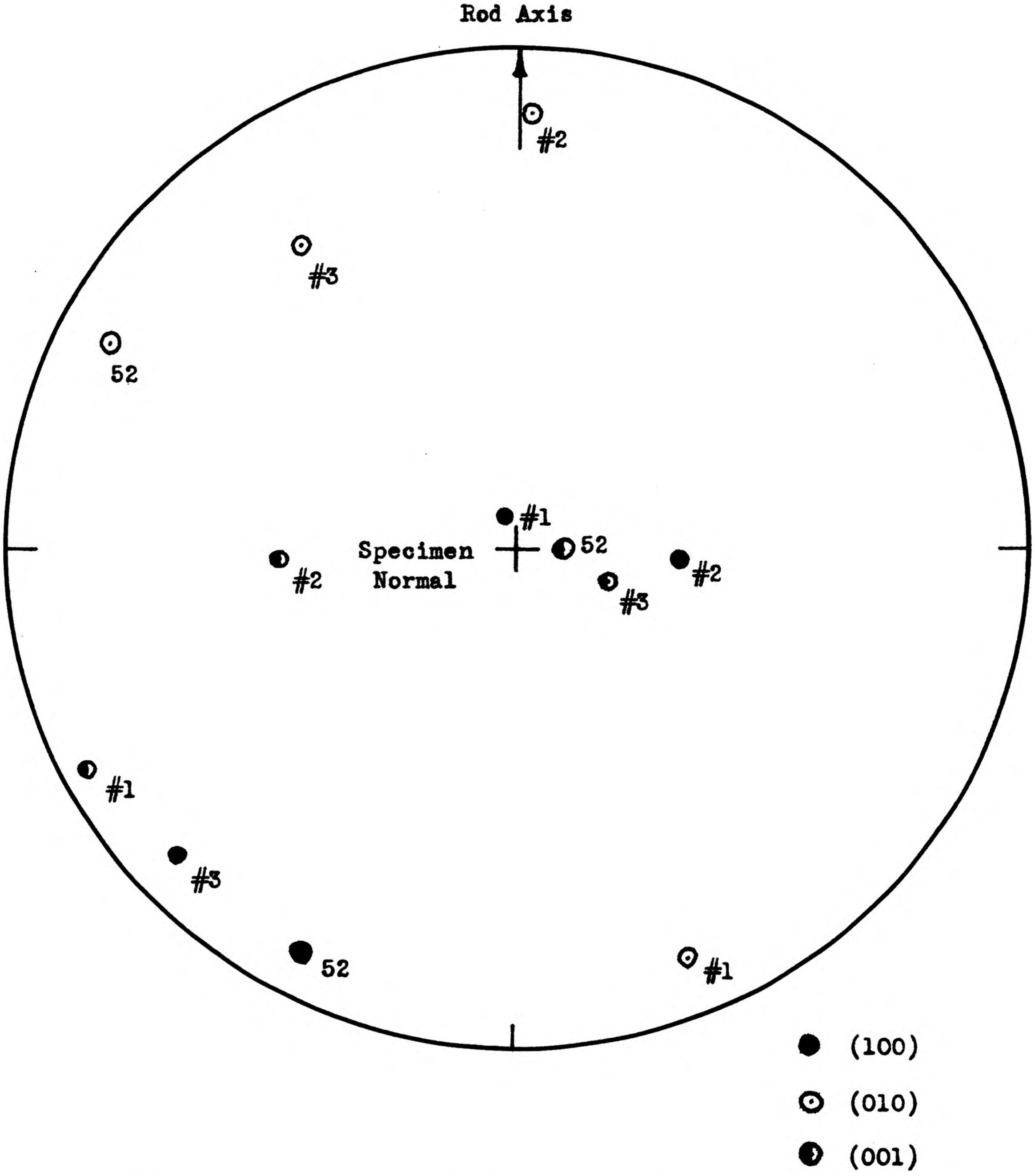

Figure 18. Stereographic Projection of Specimen 52 Showing Locations of the Three Principal Poles of the Original and Recrystallized Grains with Reference to Rod Axis and Specimen Normal.

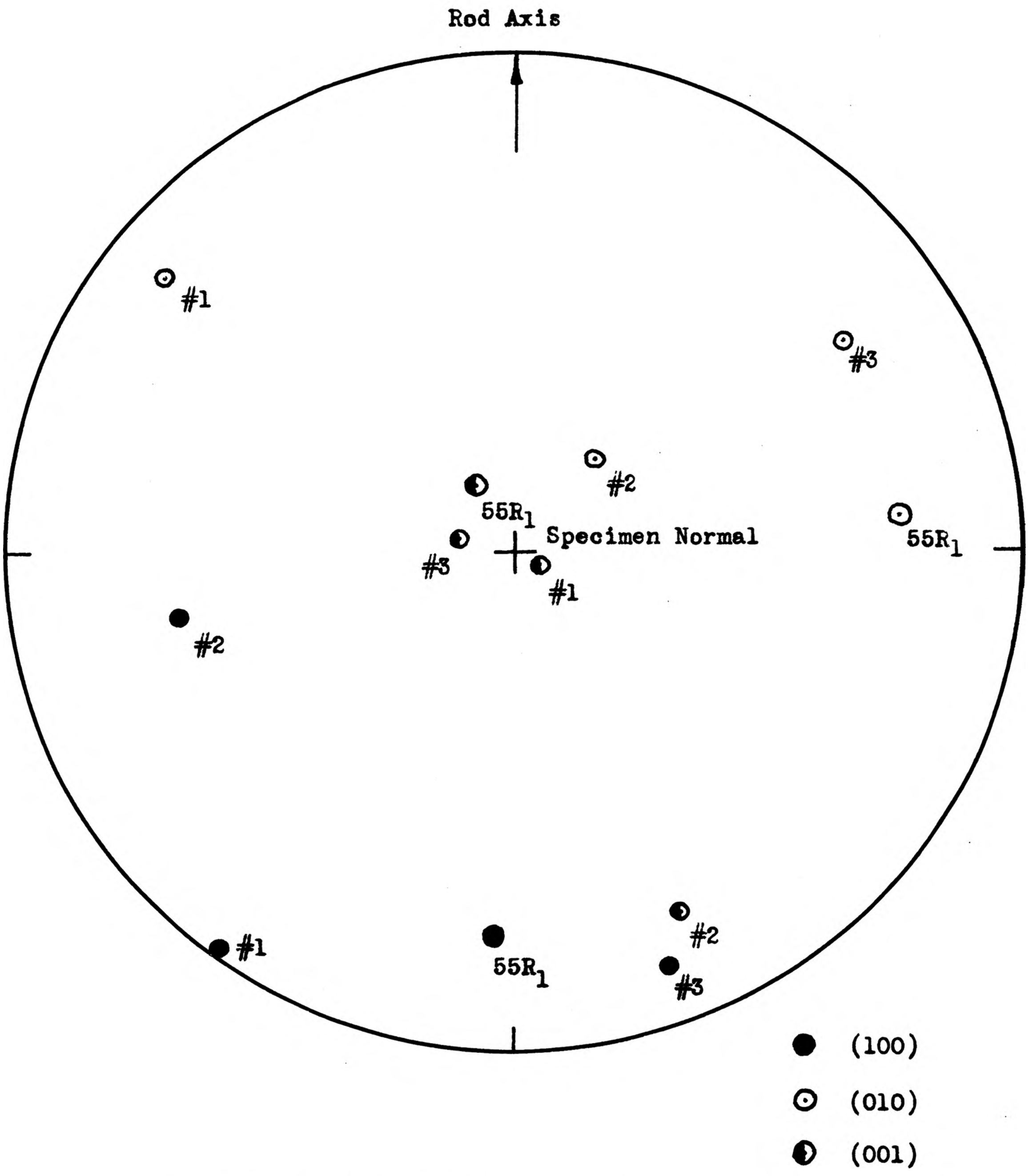

Figure 19. Stereographic Projection of Specimen $55R_1$ Showing Locations of the Three Principal Poles of the Original and Recrystallized Grains with Reference to Rod Axis and Specimen Normal.

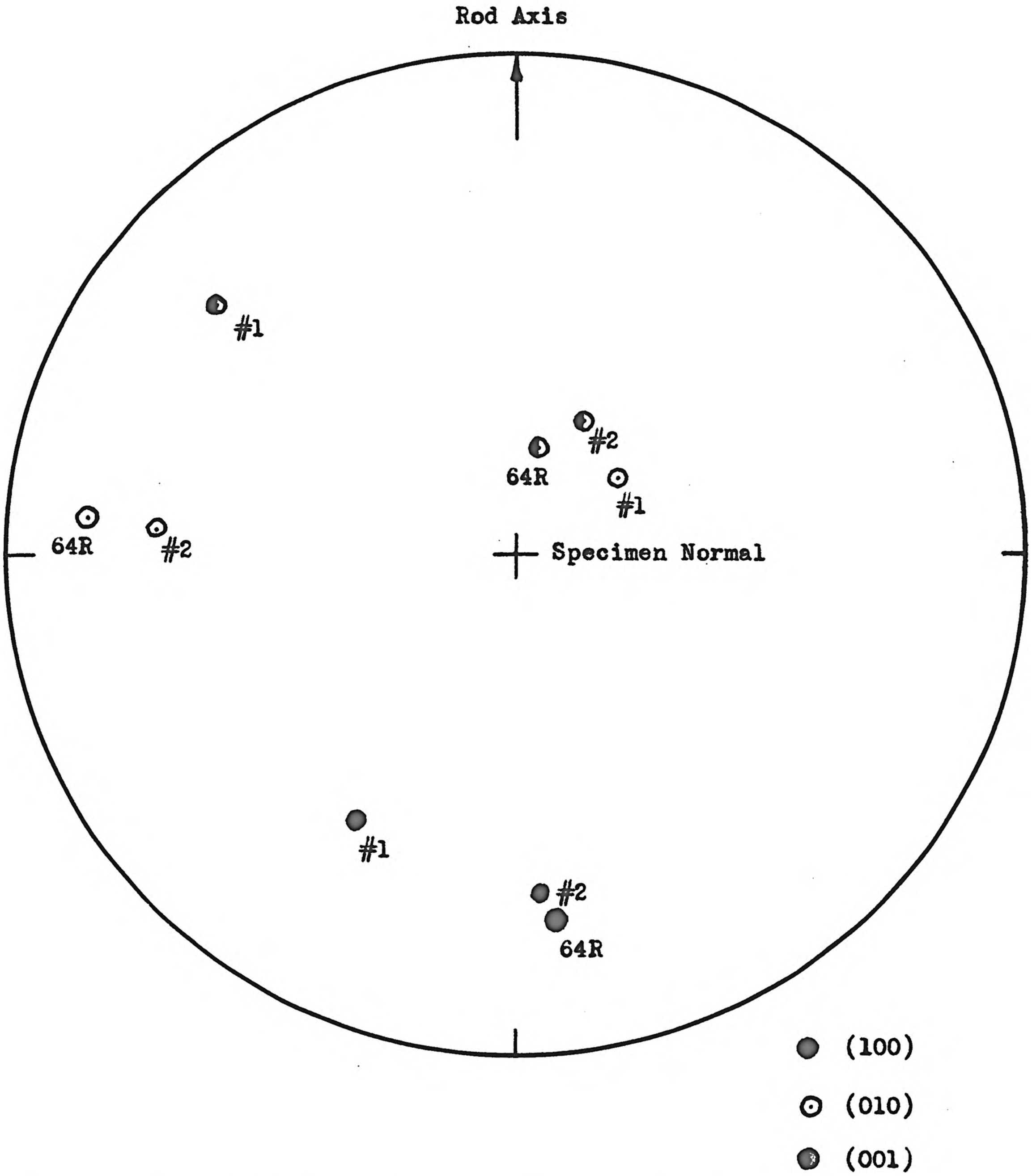

Figure 20. Stereographic Projection of Specimen 64R Showing Locations of the Three Principal Poles of the Original and Recrystallized Grains with Reference to Rod Axis and Specimen Normal.

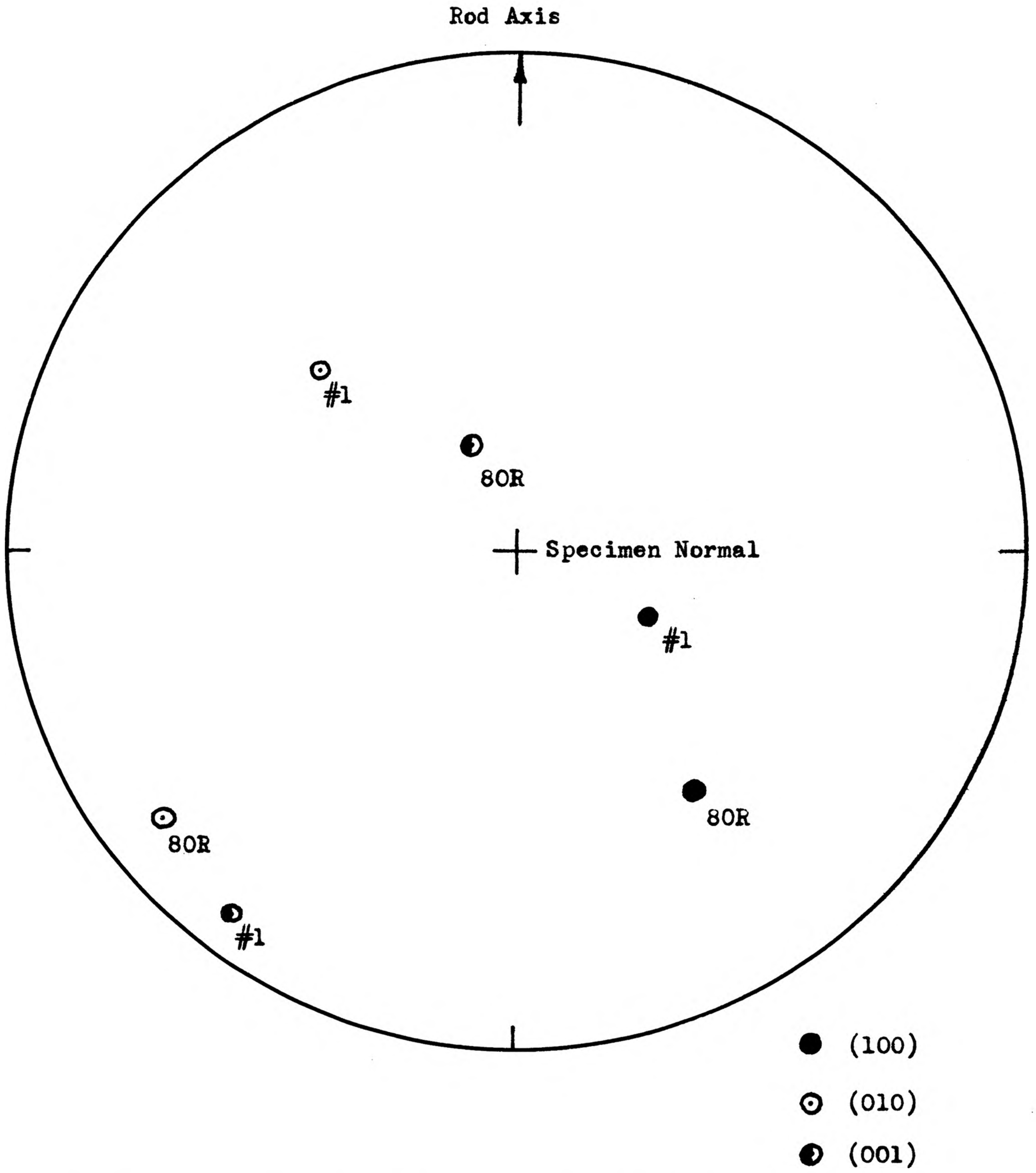

Figure 21. Stereographic Projection of Specimen 80R Showing Locations of the Three Principal Poles of the Original and Recrystallized Grains with Reference to Rod Axis and Specimen Normal.

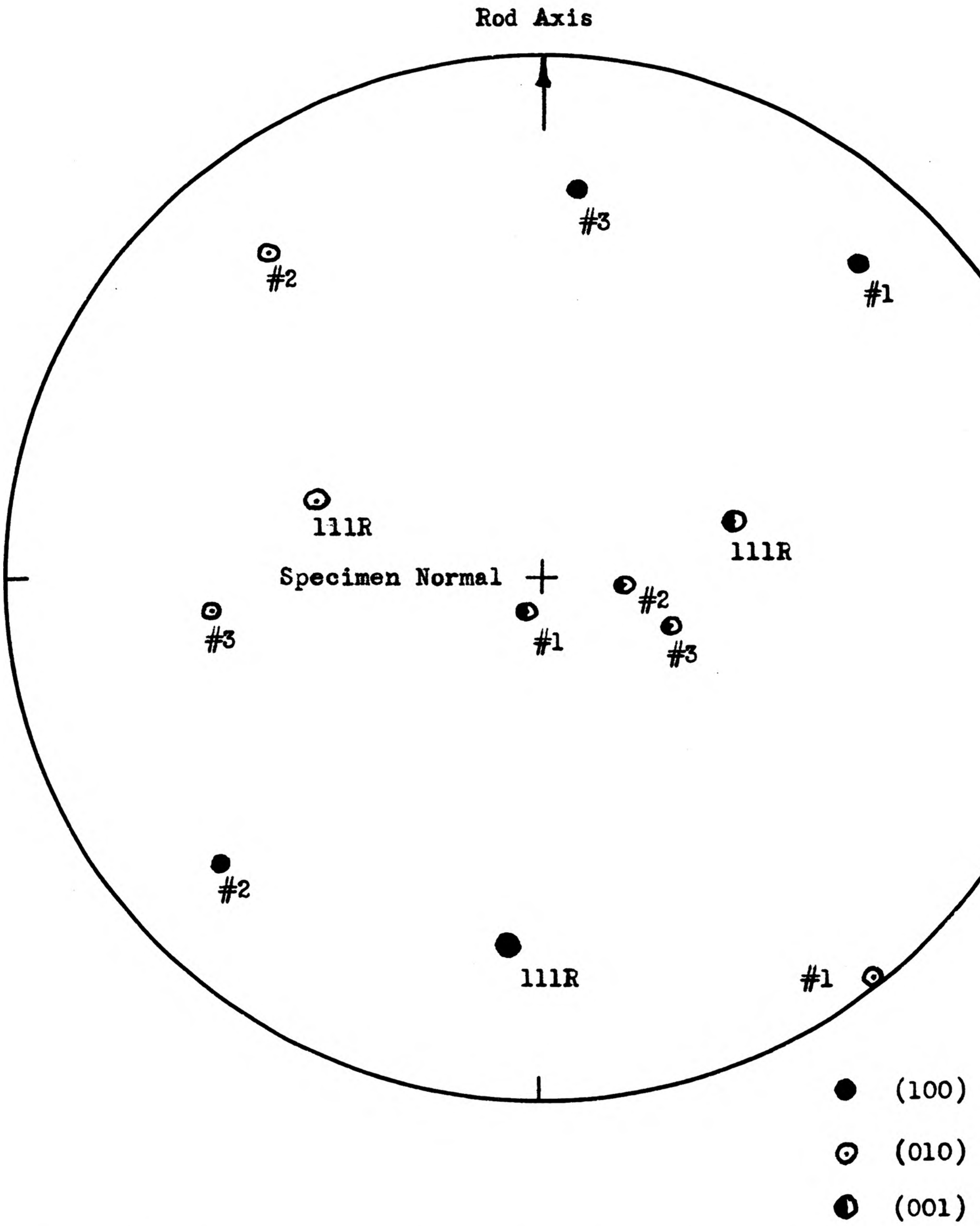

Figure 22. Stereographic Projection of Specimen 111R Showing Locations of the Three Principal Poles of the Original and Recrystallized Grains with Reference to Rod Axis and Specimen Normal.

VITA

Albert Eugene Bolon was born July 19, 1939, at Montgomery, Alabama. He attended the public school system of Rolla, Missouri and was graduated from Rolla High School in 1957. The following fall he entered the Missouri School of Mines and Metallurgy. The summer of 1960 he was employed at the United States Naval Ordnance Laboratory at Silver Spring, Maryland. He received a Bachelor of Science Degree in Physics in June, 1961. As a Student Aide he worked in the Physical Metallurgy Group at the Argonne National Laboratory during the summer of 1961. He entered graduate school at the Missouri School of Mines and Metallurgy with an United States Atomic Energy Commission Special Fellowship in Nuclear Science and Engineering in September, 1961.