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SUMMARY OF PROGRESS ON THE STUDY OF BETA TREATMENT OF URANIUM

November 1, 1959 to August 31, 1960

By R. B. Russell

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LIST OF TABLES

LIST OF FIGURES

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LIST OF FIGURES

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SUMMARY

The program to study the variables affecting the texture and grain size of uranium during beta treatment is summarized for the period November 1, 1959 to August 31, 1960.

The study of the effect of time and temperature in the beta phase on the growth index (G3) and grain size of the final alpha product is tentatively believed to show that higher beta temperatures for short times (up to about seven minutes) tend to promote slightly more negative growth indices and that higher beta temperatures give rise to somewhat finer grain sizes. More data are needed to justify these conclusions.

Results of studies of both Jominy end-quenched bars and several full-sized rods and tubes quenched by total immersion showed that large thermal gradients promoted negative growth indices and produced grains somewhat elongated in the direction of the thermal gradient. The effects of end-cooling in full-sized pieces quenched by total immersion in cold water showed that the axial growth index is negative up to distances from the end of about half the wall thickness of tubes and about half the radial dimension of rods. The grain refinement penetrates to a lesser distance from the ends.

In the radial direction the growth index for these same pieces is largely negative to a distance below the outer diameter of about midwall in two tubes studied. In the case of one tube which was studied more completely, the growth index became negative again as the inner diameter was approached. A water-quenched rod was found to have a negative growth index down to a distance from the surface of about midradius.

I. INTRODUCTION

The purpose of this program has been to study the effect of the variables in the beta treatment of uranium on the resulting grain size and texture of the final alpha product. The ultimate objective is to provide data on the effect of these variables in a practical form for use in the beta treatment of uranium at production sites. Such information should enable the heat treater to select a procedure that will result in predictable texture and grain size for a given geometry.

A range of variables encompassing those encountered in production is being studied. A detailed understanding of the effects is being obtained by determining cooling rates and thermal gradients on full sections during beta treatment and by studying the texture and grain size of the final product. These studies are being supplemented by Jominy end-quench experiments on uranium bars. Jominy tests serve as a guide to the interpretation of results and the design of experiments on heat treatment of full sections.

II. SCOPE OF PROGRAM

A. Variables

For convenience, the scope of the program, in terms of the pertinent variables and the range of initial interest, is outlined below.

1. Chemical Composition
   a. Typical dingot composition
   b. Typical ingot composition
2. Prior Condition
   a. Alpha rolled
   b. Alpha extruded
   (Note: both of these prior conditions may be modified to include the alpha recrystallized state if it appears that the presence of cold work has an effect on the final beta treated metal.)

3. Size (See Table I)

   a. Rods: from 0.5- to 2-inch diameter

   b. Tubes: from 1- to 4-inch OD with wall thickness from 0.25- to 1-inch

4. Heat Treatment

   a. Beta temperature

   b. Time in beta phase

   c. Cooling rate from beta phase

      (1) Continuous

      (2) Interrupted

   d. Thermal gradients

      (1) Axial direction

      (2) Radial direction

   e. Number of beta treatments

5. Post Beta Treatment

   Alpha phase anneal

B. Post Heat Treatment Examination

Both tubes and rods are being subjected to the same type of post heat treatment examination. This includes determination of grain size, texture, and dimensional changes.

1. Grain Size

Grain size will be determined by matching 3X photomacrographs with the UAT (ultrasonic attenuation tester) standard 3X charts when they become available. For the recrystallization experiments, determination of grain size will be at higher magnification wherever possible.

2. Texture

Texture is being determined from x-ray diffraction data and is being characterized by a number termed "growth index." Since there is no commonly accepted expression for growth index and since each index in current use has some drawbacks, the x-ray data which will be accumulated by automation will be enough to compute the older growth index GI, as well as the newer indices G_2 and G_3 both of which were discussed at the last meeting of the x-ray group at NLO.[1] For this progress report, data only for G_2, G_3, and in some cases, for the index[2] $\bar{K}$ have been gathered. Calculation of G_2 requires the use of an electronic high-speed computer and has not been carried out. In several cases the texture has been described by an entire crystallographic (inverse) pole figure based on x-ray intensities from about 40 diffraction planes.

3. Dimensional Changes

Length, wall thickness, and diameter are being measured before and after heat treatment.

III. PROGRESS REPORT OF WORK TO AUGUST 31, 1960

A. Visits to Other Sites

In order to become familiar with the heat-treatment procedures in use at production sites, visits were made early in the program. On December 14, 1959, Boltax visited SRP and SRL.[3] During January 4 to 8, 1960, Russell visited HAPO, MCW, and NLO.[4] On these trips methods of determining texture and grain size were also discussed intensively.

B. Material

The metal needed to cover the range of sizes desired by FEDC is shown in Table I together with the sizes and amounts actually received to date. No dingot metal has been received.

C. Heat-treating Salt

Work with Nu-Sal in both small (4 in. x 6 in. x 9-1/2 in. deep) Inconel cast pots and welded plain carbon steel pots has shown such severe penetration of the molten salt through the pot walls that the resistance-heating elements burned out in about 20 hours of service. The heavy black glossy scale and stalactites forming on the outside of the pot have been found by x-ray diffraction to be composed of NaCl, KCl, Fe_2O_3 and Fe_3O_4. To prevent such salt diffusion, a beryllia pot has been made for use in a small furnace. It was considered unwise to replace the Houghton LH980 ($NaCl$-KCl-$BaCl_2$) contained in a large (18 in. diameter x 22 in. deep) Inconel pot by Nu-Sal because of the danger of similar furnace element burnouts. With the concurrence of V. Montenyohl (SRP)*, LH980 is being used instead of Nu-Sal for further work. LH980 has the further advantage of a lower vapor pressure (less smoking) at the same temperature and a larger useful working range (600-900°C) than Nu-Sal (700-900°C).

D. Equipment

A Brush Instruments Ultralinear Oscillograph with a high-grain dc amplifier (Model RD 5615 00/S-2963) was installed on February 8, 1960. The maximum capacity of this instrument is eight channels. For present purposes, only six have been installed.

A Norelco high angle goniometer with scintillation counter and pulse height analyzer was installed on March 7, 1960. Automation equipment for this unit will be installed about October 15, 1960 by Mr. L. H. Cook, who has been responsible for its construction at SRL. A part of this unit, the x-ray program controller, has been constructed at NMI.

E. Reports

A progress report[5] describing the work from November 1, 1959 to April 1, 1960 has been issued.

F. Experimental Work

1. Collection of Thermal Data

For convenience, symbols representing time, temperatures, and cooling rates have been devised. They are translated in Table II and shown schematically in Fig. 1 (RA-1720).

---

* FEDC Technical Administrator of this NMI Heat Treatment Program.

A thorough knowledge of cooling rates and thermal gradients is essential to an understanding of the grain size and texture of the final product. Problems with thermocouples have required expenditure of considerable time and effort. Grounded Inconel-clad thermocouples* were chosen originally because of their inherently fast response to temperature changes. Early experiments using grounded thermocouples inserted into uranium heated in an argon atmosphere were mostly successful because the thermocouples were to a large extent electrically insulated from each other by the uranium oxide which formed on the thermocouple well surface. Since the thermocouples were only intermittently insulated, however, modulations of the thermal emf occurred (because of ground loops), especially when the thermocouples were moved slightly during the quenching procedure. When grounded thermocouples were tried in molten salt, parallel paths to ground through the salt into the Brush high-gain amplifier (one input is grounded) caused almost all of the signal to be lost. It was therefore decided to use only ungrounded Inconel-clad couples.**

The depth of the thermocouple holes is very important because there is obviously some minimum depth required to give a cooling rate which will not be appreciably increased by the conducting effect of the mass of the thermocouple extending above the mouth of the well. This effect is usually termed "loading". To establish a minimum well depth, two 3-inch OD by 2-1/2-inch ID tubes and one 1-1/2-inch OD by 3/8-inch ID tube, all 8 inches long, were drilled to make 1/16-inch wells in different orientations and depths. The tubes were then beta treated by quenching into 12°C water from 710 to 730°C molten salt (Houghton LH980). The results are shown in Table III. It is clear from Table III that a thermocouple well depth of 1/16 inch is unsatisfactory, but that depths of about 1/4 inch are borderline and 1/2 inch or deeper should be adequate. A. Guay[6] believes that depths of about 3/4 inch should be adequate. He has found that uranium

---

* Thermo Electric Company, Inc., Saddle Brook, New Jersey.

** Manufactured by Aero Research Instrument Company, Inc., 315 N. Aberdeen Street, Chicago 7, Illinois.

oxide usually penetrates past the 0.001- to 0.002-inch clearance between the thermocouple and the well surface to a depth of about 0.4 inch, and that this thermally insulating oxide tends to interfere with the observation of the true* cooling rate, unless the well depth is greater than about 0.4-inch.

2. Effect of Time at Beta Temperature on the Growth Index G_3 and Grain Size

A study of the effect of time at beta temperature was carried out on 1-inch diameter by 1/4-inch thick discs cut from 1-7/32-inch rod rolled from NLO Ingot No. 89155. Inconel-clad Chromel-Alumel thermocouples 1/16 inch in diameter were inserted radially into 1/16-inch ID wells which extended to the geometrical center of the discs. The discs were heat treated at temperatures ranging from 690 to 755°C for times of 1-3/4 to 64 minutes at temperature, then quenched to room temperature in mineral oil. Times at the beta temperature were considered adequate to transform all of the alpha phase to beta.[7]

A preliminary comparison of discs cooled in air and mineral-oil quenched from 730°C molten salt had shown that the oil-quenched 3X grain size was somehwat finer and more representative of the 3X grain size found in water-quenched 1-7/32 inch diameter rods, 3-inch OD by 2-1/2-inch ID tubes and 1-1/2-inch OD by 3/8-inch ID tubes. This study also showed that a steep texture gradient existed perpendicular to the flat faces of the discs, where the severity of texture is shown by the growth index G_3.

---

* i.e., the cooling rate given by an ungrounded thermocouple whose metal cladding is in ideal thermal contact with the well bottom.

Air Cool to Room Temperature		Oil Quench to Room Temperature	
Distance Below Surface (in.)	G_3	Distance Below Surface (in.)	G_3
0.001	- 0.26	0.001	- 0.22
0.041	+ 0.043	0.039	- 0.048
0.064	+ 0.079	0.067	- 0.017
0.088	+ 0.072	0.090	- 0.048
0.094 (one side of 1/16-inch diameter thermocouple wall)			

It is clear from the results listed above that only a mild texture (small value of G_3) exists at a depth of about 0.065-inch; consequently, all discs were ground on one side to a depth of 0.065-inch before x-ray examination was made.

Results and Discussion - The conditions, thermal data, and G_3 results of the study of the effect of time at beta temperature are shown in Table IV and Fig. 2 (RA-1751). The thermal data for an average disc were:

$t_D + t_{A_r}$
4.3 secs

$t_{\beta/\alpha}$
0.68 secs

A_r (°C)
637

R_β, °C/sec
89

R_α, °C/sec
73

Examination of the G_3 results illustrated in Fig. 2 show that, for the times at temperatures actually run, very little, if any, effect is observed on G_3. It will be seen, however, that in an area bounded by times of 1-3/4 to about 7 minutes at temperatures of 730 to 760°C the G_3 is somewhat more negative than in the other areas of the time-temperature plot. More data will be accumulated to see if this is a true effect. The sample representing 1-3/4 minutes at 757°C was x-ray scanned 11 times for reproducibility purposes (see Appendix, page 70) so that its G_3 value is well established.

Examination of the relative grain size was carried out by shuffling the 3X photomacrographs twice and sorting twice. Without the availability of HAPO 3X standard charts it is impossible to say whether differences observed in grain size are to be considered significant, but it is tentatively concluded that the finer grain sizes are generally associated with the higher beta temperatures. This problem will be examined further.

3. <u>Effect of Cooling Rate from the Beta Phase (Jominy End-Quench Tests)</u>

To evaluate the effect of thermal gradients and cooling rates on the grain size, texture, and dimensional changes of beta-treated metal, Jominy end-quench tests were made. Gardner and Riches,[8, 9] Lewis,[10] Kornfeld,[11] and Starbuck and Kloepper[12, 13] have all made use of Jominy end-quench tests to demonstrate the relation between cooling rate and various properties of uranium. In this program, Jominy tests have proved very valuable in determining the effect of temperature gradients on texture and grain size.

Eight 1-inch diameter by 3- to 6-inch long Jominy bars were cut from 1-7/32-inch diameter ingot (NLO Ingot No. 89155) rolled at NLO. The as-rolled texture is shown by the crystallographic (inverse) pole figure in Fig. 3 (RA-1539). Two Jominy bars were also prepared from cast ingot stock at NMI. The bars were drilled to make 1/16-inch ID radial thermocouple wells at various positions along the axis. Inconel-clad 1/16-inch diameter grounded Chromel-Alumel thermocouples were used to record the temperature at the well bottoms. Surface temperature was recorded by bare Chromel-Alumel thermocouples spot welded to the surface. The bars were then wrapped with two layers of 1/2-inch wide asbestos tape to secure the thermocouples as well as to reduce the radial thermal gradients in the bar. Beta treatment was 15 minutes at 730°C in a protective atmosphere provided by dried tank argon. After the bars were end quenched in 10 to 12°C water they were sectioned transversely for texture and grain size evaluation. Dimensional changes in several of the bars were measured.

a. Effect on Texture and Grain Size

The change in growth index G_3 with distance from the water-quenched end is shown in Table V together with measured cooling rates in the beta phase (R_β). The variation of G_3 and $\overline{K}$ up to 1/4-inch from the quenched end of bar J3 are shown in Figs. 4a and 4b (RA-1735 and 1736). The change in G_3 and grain size with distance from the quenched end of Jominy bar J3 is demonstrated by a series of crystallographic pole figures and 3X photomacrographs, Figs. 5a to 5i, from which it is clear that, for the maximum cooling rates of about 100°C/sec. obtained, the principal texture formed is one in which the 100 poles are parallel to the thermal gradient. The principal texture progresses from 100 to 10ℓ to 001 to approximately random as the distance increases up to about 1/4-inch from the water-quenched end. Starbuck and Kloepper[12,13] report nearly identical results from a Jominy bar end quenched at a higher (about 300°C/sec.) rate from a molten salt bath. These investigators report a somewhat different texture series: 100 to 11ℓ to 001 to $0k\ell$ in the same direction and interval. The growth indices associated with textures found in this investigation obviously must increase from negative values with increasing distance from the quenched end, as is found. Kornfeld's[11] similar observations are thus confirmed.

The x-ray data of Jominy Bar J7 was obtained from the flat surface of a true hemi-cylinder. The pole figure represents all radial textures from the quenched end to 1/4 inch from the quenched end. This 010-150 texture can be seen in Fig. 6 (RA-1731).

The grain size in all the Jominy bars is observed to be fine for the first 1/10 inch from the water-quenched end; beyond that the grains were coarser yet fairly constant for the remainder of the bar length. Grain sizes will be given later as UAT 3X Hanford standard grain sizes, when the standard charts become available.

b. <u>Discussion of Jominy Textures</u>

Both thermal parameters, temperature gradient and cooling rate, change during an end-quench Jominy test. In a valuable experiment Chiswik and Lloyd(14) maintained constant slow (4^{o}C/hr. and 0.5^{o}C/hr.) cooling rates throughout a rod of uranium during $\beta \longrightarrow \alpha$ transformation in a temperature gradient of as much as 138^{o}C/in. They found:

1. The largest thermal gradient produced the most subgraining in the final α crystals.
2. The largest thermal gradient produced grains the most elongated in the direction of the thermal gradient.
3. The larger thermal gradients produced grains which were generally oriented with their 001 poles (or c-directions) parallel to the temperature gradient.
4. There appeared to be no correlation between grain size and orientation.

With respect to their findings: in our Jominy work subgraining was not studied; grain elongation has already been reported by Lewis(10) in Jominy bars; in our work, grain elongation was also found near the quenched surfaces of rods and tubes quenched by total immersion (see III.E.4.a and Figs. 9, 10, 11); preferred orientation with the c-axis parallel to the thermal gradient was found only for moderate gradients, but strong gradients induced 100 texture (a-axis) in the gradient direction; the correlation between grain size and orientation could not be determined because of the simultaneous variation of cooling rate and thermal gradient in a Jominy bar.

c. Dimensional Changes

Dimensional changes in four cold-water-quenched Jominy bars are shown in Figs. 7a to 7d (RA-1737, 1738, 1739, 1740). It will be seen that these all show about a 0.040-inch contraction in length and an expansion in diameter of about 0.007-inch at the quenched end localized within a distance of an inch or two from the quenched end. To determine whether the distortion was due to a concentration of uranium oxide at the quenched end, Bar J-10, Fig. 7d (RA-1740), was etched in nitric acid to remove the oxide and re-measured. The measurements show that, although the presence of oxide alters the distortion curve, the diameter expansion of the quenched end persists and is undoubtedly a real effect. To prevent most oxidation, Jominy bars will be quenched from molten salt. Dimensions of these bars will be measured.

It is possible that this distortion found after beta treatment is due to the fact that the texture at the quenched end is different from the nearly random texture of the more slowly cooled remainder of the bar. Preliminary estimates of thermal contraction in the axial direction indicate that the thermal contraction coefficient from 662 to 0°C would have increased as a result of a change from an initial hko texture (see Fig. 3, RA-1539) to an approximately random texture after beta treatment. In the radial direction the prior texture was not determined but if textural changes are largely responsible for beta treatment distortion, then an observed expansion in diameter after beta treatment suggests that the original rolled radial principal texture is near 100.

Lehr,[15] on the other hand, believes that the permanent contraction in length parallel to the temperature gradient in a rod cooled slowly through the beta-to-alpha transformation is due to the relative inability of the alpha, forming in the transverse plane of transformation, to adapt itself to the required smaller transformed cross section. The result is that, to conserve the alpha volume, which is more plastic than the beta, the metal must contract in a direction perpendicular to this transverse plane, i.e., in a direction parallel to the temperature gradient.

4. Total Immersion Quenches on Full-Size Pieces

a. End Effects

The FEDC had requested studies of full-size pieces of effectively infinite length, i.e., lengths sufficient to exclude ends that were cooled in both radial and axial directions. McDonell[16], at SRO, believes that the end effects may persist up to a distance of four wall thicknesses (4w) from the end of the beta-treated tube. However, Guay,[6] of HAPO, has calculated that the end effect may be only one-half wall thickness (w/2) from the quenched end. In an effort to determine the extent of this end effect, texture studies were made near the ends of quenched 1-7/32 inch diameter rod, 1-1/2 inch OD by 3/8-inch ID, and 3-1/8 inch OD by 2-1/2-inch ID tubes.

Table VI and Fig. 8 (RA-1749) show the axial texture results near the ends. It will be observed that G_3 approaches zero at distances of nearly w/2 for the tubes and r/2 for the rod. Figs. 9, 10, and 11 (RF-7607, 7650, 7591) illustrate the grain size on longitudinally cut end sections of the 1-7/32-inch diameter rod, 1-1/2-inch OD x 3/8-inch ID tube, and 3-1/8-inch OD by 2-1/2-inch ID tube, respectively. From these photomacrographs it is seen that, near the quenched ends, there is a rim of refined grains whose shape is somewhat elongated perpendicular to these surfaces. The rim extends to about 0.07-inch (about w/9), 0.1-inch (about w/6), and 0.03-inch (about r/10) in the rod, the 1-1/2-inch OD and 3-1/8-inch OD tubes, respectively. Evidently the texture end effect, which persists up to a distance of about w/2, extends further than the visible grain size end effect. Guay's calculations that the end effect is about w/2 long seems to have been confirmed by texture results in the case of a cold water quench.

b. Cooling Rates Observed in Various Media

(1) In 3-inch OD by 2-1/2-inch ID tube (wall thickness 0.25 inch)

In order to determine the cooling rates in various media, a 3.00-inch OD by 2.50-inch ID by 8-inch long tube was successively quenched from molten salt (Houghton LH980) into the following media: 28°C air, 28°C oil, 55°C water, and 11°C water. Only ungrounded Inconel-clad thermocouples were used, and their relative positions, orientations, and well depths are shown for tube M3 in Table II. All well depths (0.5 - 1.5 in.) are considered adequate. The cooling rates are given in Table VII. In neither water quench could the A_r's be observed, nor could a cooling rate in alpha uranium (R_α) be reliably reported. For 55°C water, the R_α was sometimes greater, sometimes less than R_β. The cooling curve was more irregular than in other media. In both ranges, the cooling rates in water covered a much broader range of values than in either oil or air. Paetschke[17] has reported similar results on Mark VII-ASC core blanks (1.120-inch OD, 0.297-inch wall). It is noted that only in the case of the 11°C water quench is there any significantly higher cooling rate in the beta phase (R_β) on the outside and inside than in the center of the tube wall.

(2) <u>In 1-1/2-inch OD by 3/8-inch ID Tube (Wall Thickness 0.56-inch)</u>

Cooling rates in cold water at midwall position of tube H2 have already been given in Table III. Thermal data from adequate well depths indicate a cooling rate, R_β, of 82°C/sec. At midwall on the cold water quenched 3.0-inch OD tube (0.250-inch wall), the cooling rate was more than twice as great, 204°C/sec. (Table VII).

(3) <u>Other Sections</u>

Cooling rates on other sizes will be obtained later, to obtain an empirical relation between cooling rate and wall thickness in different media.

c. <u>Texture Gradients</u>

(1) <u>Radial</u>

From Jominy end-quench results it could be expected that there would be similar texture gradients near the surfaces of uranium sections which had been quenched from the beta phase. The negative G_3 values near the ends of 8-inch long cold-water-quenched sections (Fig. 8) have already been noted.

Samples for radial texture studies were prepared in narrow sectors (whose cut sides were parallel to the tube or rod radii). Sectors of 10^{o} width were cut near midlength from the 3-1/8-inch OD by 2-1/2-inch ID tube, and sectors of 20^{o} width from the 1-1/2-inch OD by 3/8-inch ID tube and 1-7/32-inch rod, so that the flat x-ray sample composite surface was not everywhere perpendicular to the radius, but was in error by a maximum of 5^{o} for the 3-1/8-inch OD tube and 10^{o} for the 1-1/2-inch OD tube and 1-7/32-inch rod. Radial G_3 values were obtained across a wall thickness of the 3-1/8-inch OD tube and radial pole figures near the OD and ID surfaces (Fig. 12). Radial G_3 and radial pole figures were determined for the 1-1/2-inch OD tube to midwall (Fig. 13) and for the 1-7/32-inch diameter rod down to about half radius (Fig. 14). The G_3 change with radial position is given in Table VI and Fig. 15. The similarity between these radial pole figures and the Jominy axial pole figures makes it clear that the same textures are being observed in the direction of the thermal gradient. We can, therefore, expect that the surfaces of uranium sections that have been cooled rapidly enough to have substantial 100 and/or 10ℓ radial components will tend to have a negative irradiation growth. The minimum cooling rate to avoid these undesirable components in different sizes will have to be determined later in these studies.

(2) Textures in Other Directions

Aside from a study of axial textures in Jominy bars and near the ends quenched by total immersion, no systematic study of axial textures has been carried out. It is planned, however, to study the variation of axial textures around the circumference to determine whether there is any effect of prior texture variations around the circumference on the final beta-treated texture.

IV. FUTURE WORK

Heat treatment of the sizes already received will be exhaustively investigated to give data on cooling rate, grain size, texture in different directions, warpage, effect of interrupted quench, recrystallization, and multiple beta treatment. It is expected that accumulation of data will proceed smoothly, especially after the automation equipment has been installed.

V. TABLES AND FIGURES

TABLE I

Size Ranges to be Studied for the Beta Heat Treatment Program for FEDC

Size (in.)			Type	Sizes Received (in.) (ingot)			Type	Wt (lb)
OD	Wall Thickness	Desired Length		OD	Wall Thickness	Length		
4	1	480	Tube					
3	1	480	Tube					
				3	1.5	36	Rod	176
2	1	240	Rod	1.88	0.94	116	Rod	250
4	1/2	480	Tube					
3	1/2	480	Tube					
2	1/2	480	Tube	1.5	0.55	500	Tube	553
1	1/2	240	Rod	1.22	0.61	360	Rod	290
4	1/4	480	Tube					
3	1/4	480	Tube	3.13	0.31	490	Tube	1100
2	1/4	480	Tube					
1	1/4	480	Tube					
1/2	1/4	240	Rod	{0.4 0.5	0.2 0.25	92	Rod	42

TABLE II

Symbols Representing Times, Temperatures and Cooling Rates
(See Fig. 1)

Symbol	Explanation
t_{Ac}	Time to heat to start of alpha-to-beta transformation from room temperature (A - B)
t_{β}	Time to heat to the final beta temperature from room temperature (A - C)
T_{β}	Final beta temperature
t_T	Time at final beta temperature (C - D)
t_D	Time between removal of sample from beta-treating medium and insertion into cooling medium (D - D')
t_{Ar}	Time to reach start of beta-to-alpha transformation from time sample enters cooling medium (D' - G)
t	Duration of beta-to-alpha transformation (G - J)
R_{β}	Cooling rate (^{o}C/sec) in beta phase (slope of HE)
R_{α}	Cooling rate (^{o}C/sec) in alpha phase (slope of KF)
T_Q	Initial temperature of quenching medium

TABLE III

Effect of Thermocouple Well Depth and Orientation on Apparent Cooling Rate in Two Different Size Tubes Quenched into Cold Water From Houghton LH980 Molten Salt
(All dimensions in inches)

Tube	Distance From End	Orientation	Distance From OD	Well Depth	Cooling Rate, R_β (°C/sec) *
M2(1)	4.4	Radial	0.063	0.063	1800
	2.0	Radial	0.15m**	0.15	670
	4.0	Radial	0.15m	0.15	530
	3.6	Radial	0.22	0.22	127
	0 - 0.5	Axial	0.13m	0.5	88
	0 - 1.0	Axial	0.13m	1.0	188
M3(2)	7.5 - 8.0	Axial	0.13m	0.5	160
	7.0 - 8.0	Axial	0.13m	1.0	420
	6.5 - 8.0	Axial	0.13m	1.5	178sp***
	0 - 1.5	Axial	0.05	1.5	312sp
	0 - 1.5	Axial	0.13m	1.5	142
	0 - 1.5	Axial	0.19	1.5	230sp
H2(3)	4.4	Radial	0.063	0.063	1670
	2.0	Radial	0.28m	0.28	96
	4.0	Radial	0.28m	0.28	95
	3.6	Radial	0.45	0.45	67
	0 - 0.5	Axial	0.28m	0.5	89
	0 - 1.0	Axial	0.28m	1.0	74

(1) 3-1/8-in. OD x 2-1/2-in. ID x 8-in. long (wall thickness, 0.31 in.)

(2) 3.000-in. OD x 2.500-in. ID x 8-in. long (wall thickness, 0.250 in.)

(3) 1-1/2-in. OD x 3/8-in. ID x 8-in. long (wall thickness, 0.56 in.)

* Ungrounded, Inconel-clad, 1/16-in. diameter thermocouple.

** m = thermocouple junction approximately at midwall.

*** sp = signal poor, datum unreliable.

TABLE IV

Effect of Time at Beta Temperature

(Thermal and G_3 data from 1-in. dia. x 1/4-in. thick discs quenched in room-temperature mineral oil.)

Disc No.	t_β (sec)	A_c (°C)	T_β (°C)	t_β (min)	t_D+t_{Ar} (sec)	$t_{\beta/\alpha}$ (sec)	Ar (°C)	R_β (°C/sec)	R_α (°C/sec)	Salt, Tc*	T_Q (°C)	G_3 (x 1000)
30	112	675	705	2.0	1.8	0.65	635	91.3	88.8	N,g	32	+13
31	nd	678	702	3.8	2.2	0.41	645	65.2	75.3	N,g	36	-13
310	102	678	694	4.0	b	b	b	b	b	N,g	35	-32
32	122	669	696	7.9	2.0	0.25	645	53.1	90.5	N,g	28	- 9
33	97	680	699sp	15.9	2.6	0.95	645	57.7	63.5	N,g	29	+15
34	117	680	696	32.	3.2	0.63	645	57.1	69.0	N,g	31	-16
35	S	S	693	64.	2.6	0.78	642	56.0	51.8	N,g	32	+ 9
40	S	S	712sp	19.	2.0	0.50	632sp	82.sp	79.sp	N,g	30	+ 6
41	nd	S	728	4.0	3.6	0.70	642	88.4	95.5	N,g	31	-28
42	S	S	728	8.0	3.3	0.42	629	92.5	83.5	N,g	32	- 3
430	98	S	687	16.0	S	S	S	S	S	N,g	nd	-14
430	98	S	687	16.0	S	S	S	S	S	N,g	nd	-14
44												
45												
50	63	668	733**	1.8	2.9	0.58	633	110.	79.4	L,u	26	-61(3)***
51	70	673	733**	3.7	2.7	0.87	629	130.	71.0	L,u	26	-43
52	nd	S	733sp**	8.0	2.8	0.83	626	101.	86.8	L,u	26	+ 1
53	71	678	745	15.7	3.0	0.87	636	107.	79.5	L,u	26	-49
54	nd	680	741	32.	2.9	0.64	640	120.	72.4	L,u	26	-21
55	57	678	744	64.	3.4	1.03	635	73.1	58.9	L,u	26	- 5
60	nd	680	757	1.7	9.7	0.60	639	109.	71.7	L,u	24	-86(11)
61	nd	673	752	3.9	9.9	0.63	628	109.	70.8	L,u	24	-22
62	nd	678	752	7.5	5.0	0.68	647	103.	61.6	L,u	22	-18
620	nd	678	752	7.5	b	b	b	b	b	L,g	22	-43
63	nd	677	752	14.8	9.7	1.07	630	97.7	49.2	L,u	24	+11
65	nd	673	749	64.	10.3	0.51	630	69.2	61.8	L,u	24	+16
Average		676	---	--	4.3	0.68	637	89	73	---	--	-16

(Cont'd.)

TABLE IV (Cont'd.)

* Salt = molten salt: N = Nu-Sal, L = Houghton LH980.
Tc = thermocouple: g = grounded, u = ungrounded.

** Nos. 50 to 55 inclusive were heat treated together.

*** Numbers in parentheses refer to number of runs.

Note: The following are explanations of symbols used.

nd = no data available.

b = thermocouple broke.

sp = signal poor, datum unreliable.

S = signal bad, no data.

TABLE V

The Change of Axial Growth Indices G_3 and $\overline{K}$ with Distance from the Cold-Water-Quenched End of Several 1-in. Dia. x 5-in. Long Jominy Bars*

No.	Distance From Quenched End (in)	Cooling Rate, R_{β} (^{o}C/sec)	G_3	$\overline{K}$ x 10^6 x ppm Burn-up	Principal Texture hkl Pole	Remarks (Water Temp., 11 - 13^{o}C)
J2	0.002	--	-0.38	--	100 S	As cast prior texture, solid fountain quenched
	0.250	--	+0.11	--	102,133**	
J3	0.002	--	-0.34	-173	100 S	Rolled prior texture (see Fig. 3), solid fountain quenched
	0.010	--	-0.33	-188	100-001	
	0.022	--	-0.32	-195	100-001	
	0.033	--	-0.33	-190	100-001	
	0.047	--	-0.27	-166	100,102	
	0.125 TC	51 (99)***	---	--	--	
	0.183	--	-0.06	- 64	100,001 W	
	0.204	--	-0.08	- 49	100,001 W	
	0.225	--	-0.04	- 21	100,001 W	
	0.239	--	-0.03	- 38	100,001 W	
	0.250	--	+0.01	- 10	001 W	
	0.262	--	+0.03	+ 36	001 R	
	0.272	--	+0.01	+ 6	001 R	
	0.341	--	+0.13	--	--	
	2.56	--	-0.06	--	--	
	2.63 TC	59 (nd)	---	--	--	
J5	0.002	--	-0.28	--	100 M	Solid fountain quenched, rolled prior texture
	0.013	--	-0.36	--	100-001	
	0.025	--	-0.33	--	100-001	
	0.125 TC	83 (98)	---	--	--	
	0.259	--	+0.01	--	001 S	
	2.51 TC	63 (66)	0.000	--	100 W	
J6	0.002	--	-0.23	--	100-001 B	End quenched twice by solid fountain, rolled prior texture
	0.021	--	-0.25	--	101-102 B	
	0.238	--	+0.03	--	001 S	
	0.259	--	+0.01	--	001 S	
	2.50 TC	54 (57)	---	--	--	
J8	0.002	--	-0.35	--	100 S	Rolled prior texture, spray quenched
	0.017	--	-0.35	--	100-101 S	
	0.035	--	-0.29	--	100-102 M	
	0.125 TC	82 (nd)	---	--	--	
	0.208	--	-0.003	--	001 W	
	0.244	--	-0.02	--	001 W	
	0.278	--	+0.001	--	001 W	
	0.340	--	+0.05	+ 9	001 R	
	0.351	--	+0.01	--	100,001 R	

(Cont'd.)

TABLE V (Cont'd.)

* Except for J2, all bars prepared from 1-7/32-in. dia. rods rolled at NLO (Ingot No. 89155).

** Believed that 133 inherited from prior as-cast condition.

*** Numbers in parentheses are cooling rates at surface.

Note: The following are explanations of symbols used.

S = strong.

TC = thermocouple location.

W = weak.

R = essentially random.

nd = no data

M = moderate

B = broad.

TABLE VI

The Change in Axial and Radial Growth Index (G_3) in a Rod and Two Tubes Water Quenched by Total Immersion

No.	Size (in)			Axial Direction		Radial Direction		Sector Width
	OD	ID	Wall or Radius	Distance From End (in)	G_3	Distance From OD (in)	G_3	
M1	3-1/8	2-1/2	0.31	0.005	-0.290	0.042	-0.175	10° sector
				0.026	-0.230	0.042	-0.159	
				0.050	-0.146	0.067	-0.094	
				0.068	-0.145	0.092	-0.047	
				0.093	-0.098	0.117	-0.055	
				0.143	+0.004	0.142	0.000	
				0.221	+0.071	0.167	+0.044	
				1.00	+0.066	0.192	+0.016	
				0.005*	-0.288	0.217	-0.027	
				0.060*	-0.080	0.243	-0.054	
				0.104*	0.000	0.287	-0.201	
				0.129*	+0.064	0.308	-0.338	
H1	1-1/2	3/8	0.56	0.002	-0.294	0.007	-0.340	20° sector
				0.025	-0.326	0.055	-0.277	
				0.050	-0.205	0.116	-0.165	
				0.075	-0.192	0.155	-0.089	
				0.100	-0.155	0.205	-0.053	
				0.125	-0.099	0.255	-0.022	
				0.150	-0.048	---	---	
				0.480	+0.045	---	---	
				0.500	+0.066	---	---	
K1	1-7/32	0	0.61	0.002	-0.287	0.010	-0.299	20° sector
				0.025	-0.270	0.070	-0.208	
				0.051	-0.245	0.108	-0.143	
				0.075	-0.197	0.158	-0.067	
				0.125	-0.106	0.208	-0.029	
				0.150	-0.079	0.258	+0.027	
				0.175	-0.072	---	---	
				0.200	-0.022	---	---	
				0.491	+0.117	---	---	
				0.500	+0.090	---	---	

* Distance from opposite end.

TABLE VII

Thermal Data from 3.00-in. OD x 2.50-in. ID x 8.00-in. Long Tube (M3) Cooled From Molten Salt (LH980) Successively into 28°C Air, 28°C Oil, 55°C Water and 11°C Water (Average Conditions: t_{Ac} = 61 secs; Ac = 668°C; t_β = 2.5 min; T_β = 710°C; t_D = 4.6 secs)

Quenching Medium	Distance from OD (in)	Cooling Rate R_β			Cooling Rate R_α			t_{Ar} (sec)	$t_{\beta/\alpha}$ (sec)
			Average	Range		Average	Range		
28°C Air	0.05	--	26.7	23.2-29.8	--	19.3	18.6-20.2	26.0	20.3
	0.13	23.2			18.6				
		29.8			19.7				
		26.9			20.2				
	0.19	26.9			18.6				
28°C Oil (agitated)	0.05	50.8 sp*	56.0	50.5-68.0	37.8 sp	39.8	32.7-45.0	1.3	1.1
	0.13	50.5			32.7				
		50.9			44.8				
		52.5			34.0				
		68 sp			45 sp				
	0.19	63.4			44.2				
55°C Water (agitated)	0.05	73.9 sp	76.3	69.3-87.4	--	--	44-218	1.2	1.1 ***
	0.13	69.3			--				
		87.4							
		124.2**							
		72.1 sp							
	0.19	78.9			--				
11°C Water (agitated)	0.05	312 sp	204	141.5-312	--	--	--	--	--
	0.13	141.5			--				
		160							
		420**							
		178 sp							
	0.19	230 sp			--				

* sp = signal poor, datum unreliable.

** This thermocouple gave unusually high R_β's in both water quenches. It has been excluded from the average.

*** Estimate.

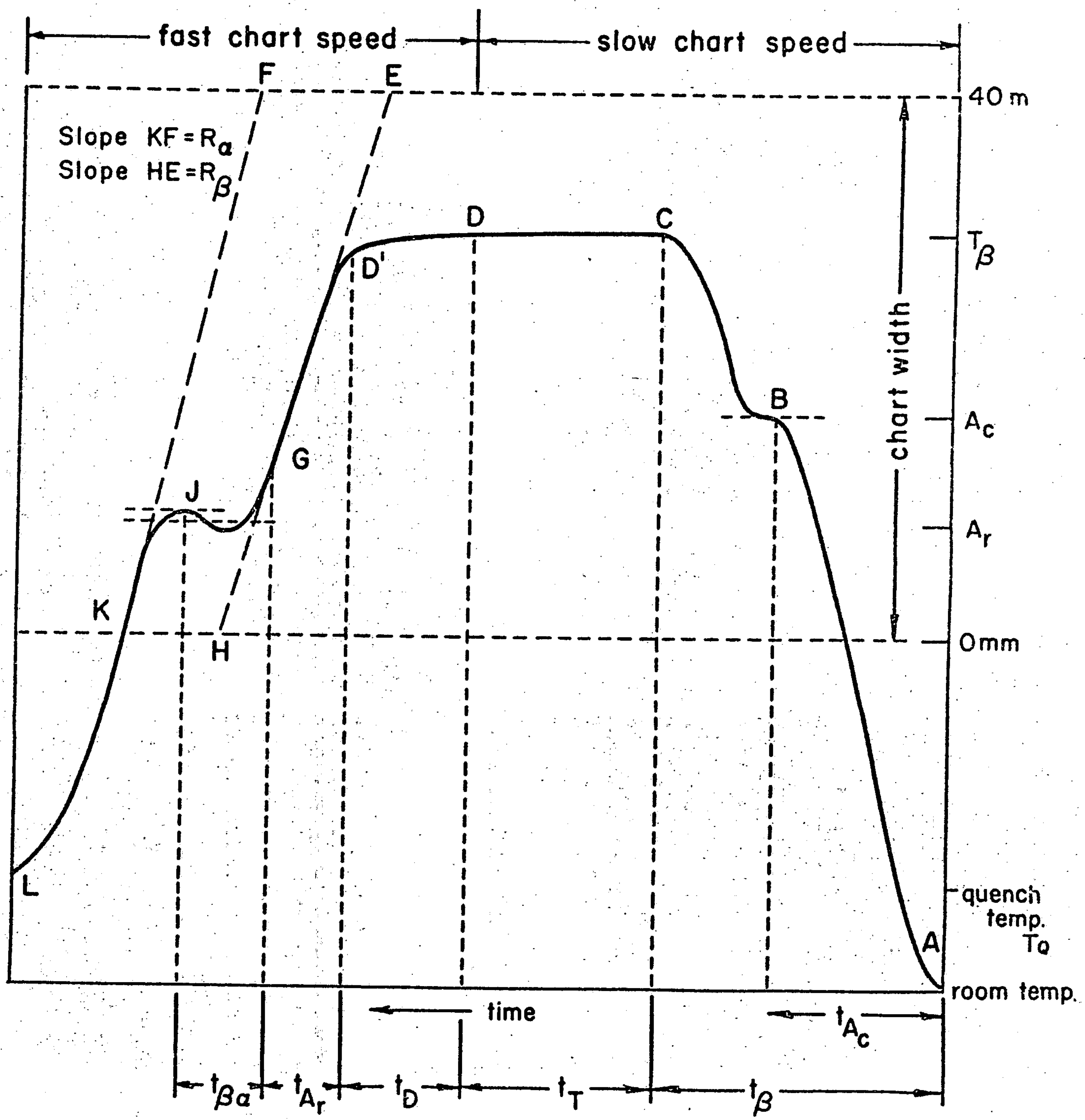

Fig. 1 - Schematic of time-temperature relations during beta treatment. (See Table II for description of symbols used in this Figure.) Note: time increases from right to left to correspond with appearance of oscillograph chart.
Drawing No. RA-1720

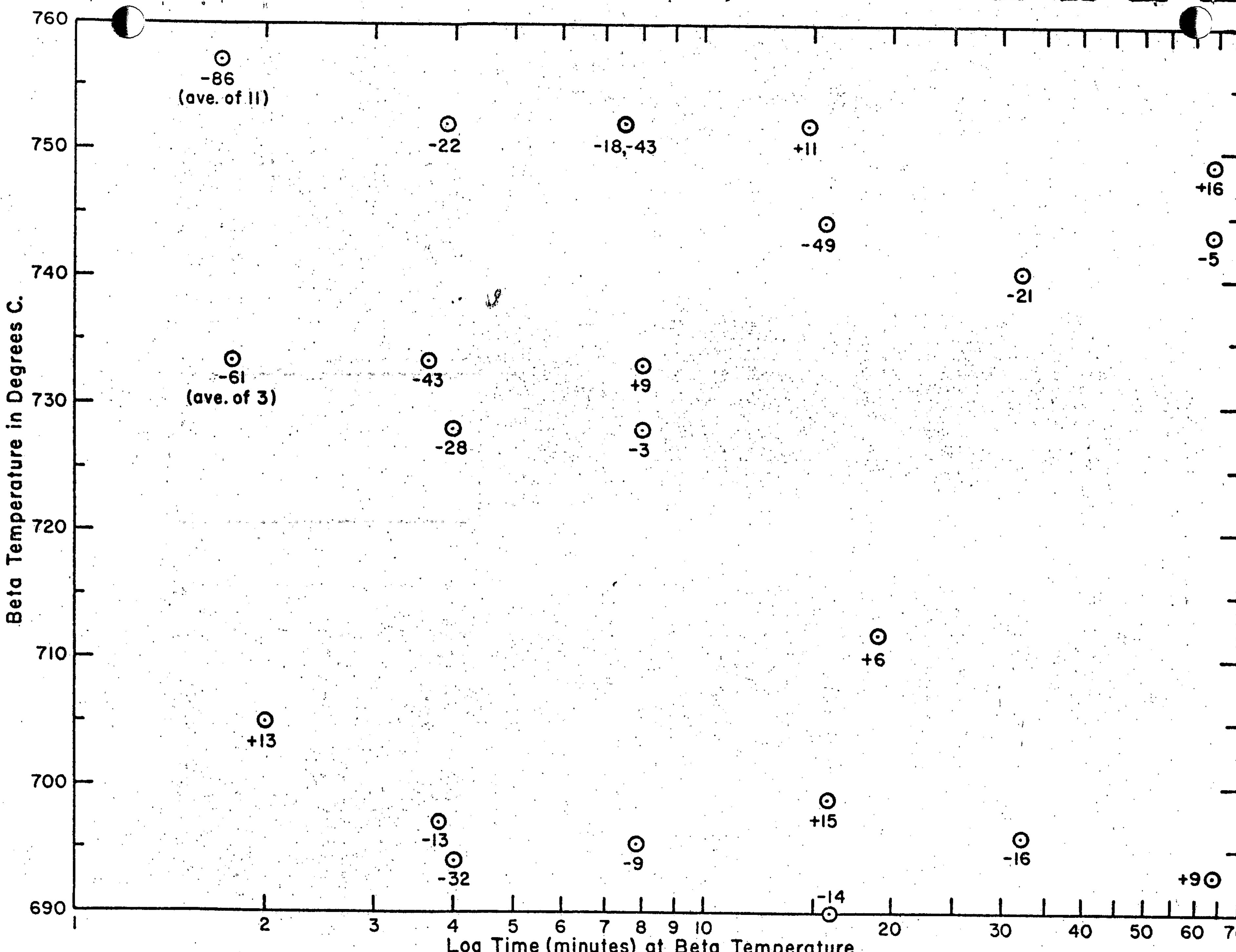

Fig. 2 - Effect of time and temperature in the beta phase on growth index, G_3, of 1-in. diameter x 1/4-in. discs (all G_3 values are X 1000). Drawing No. RA-1751

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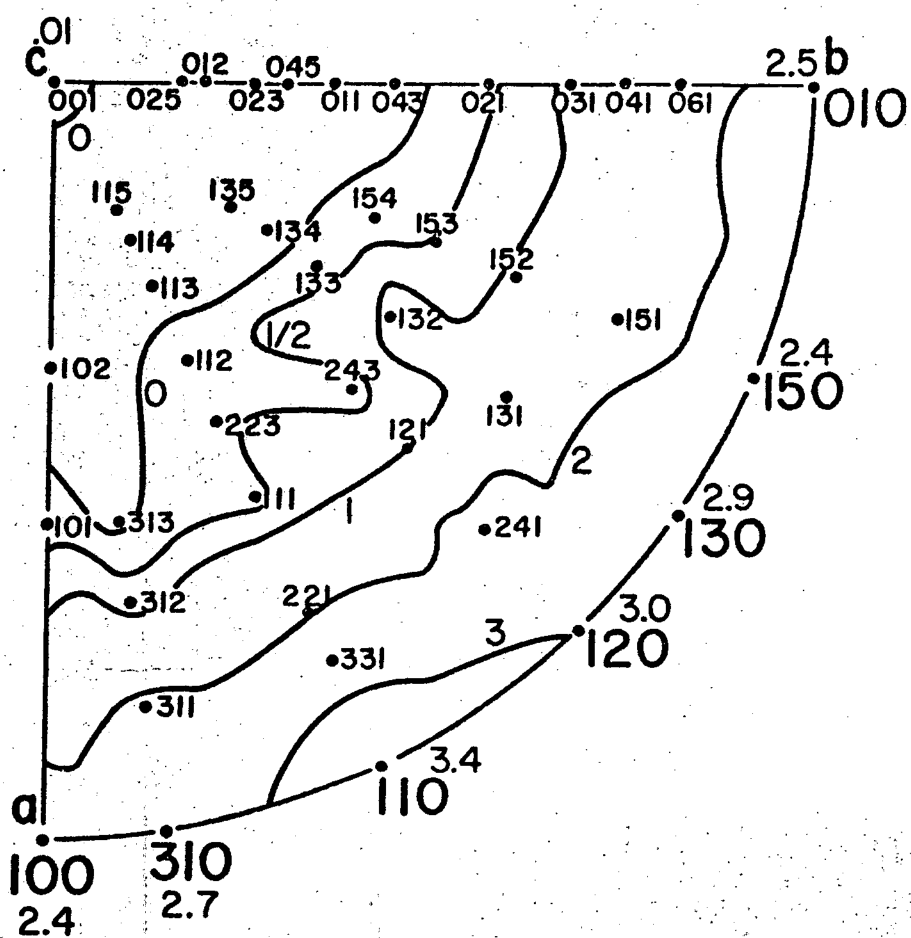


Fig. 3 - Crystallographic (inverse) pole figure for the axial direction of as-rolled 1-7/32 inch diameter rod (NLO Ingot No. 89155) G_3 = + 0.028 K = - 14.
Drawing No. RA-1539

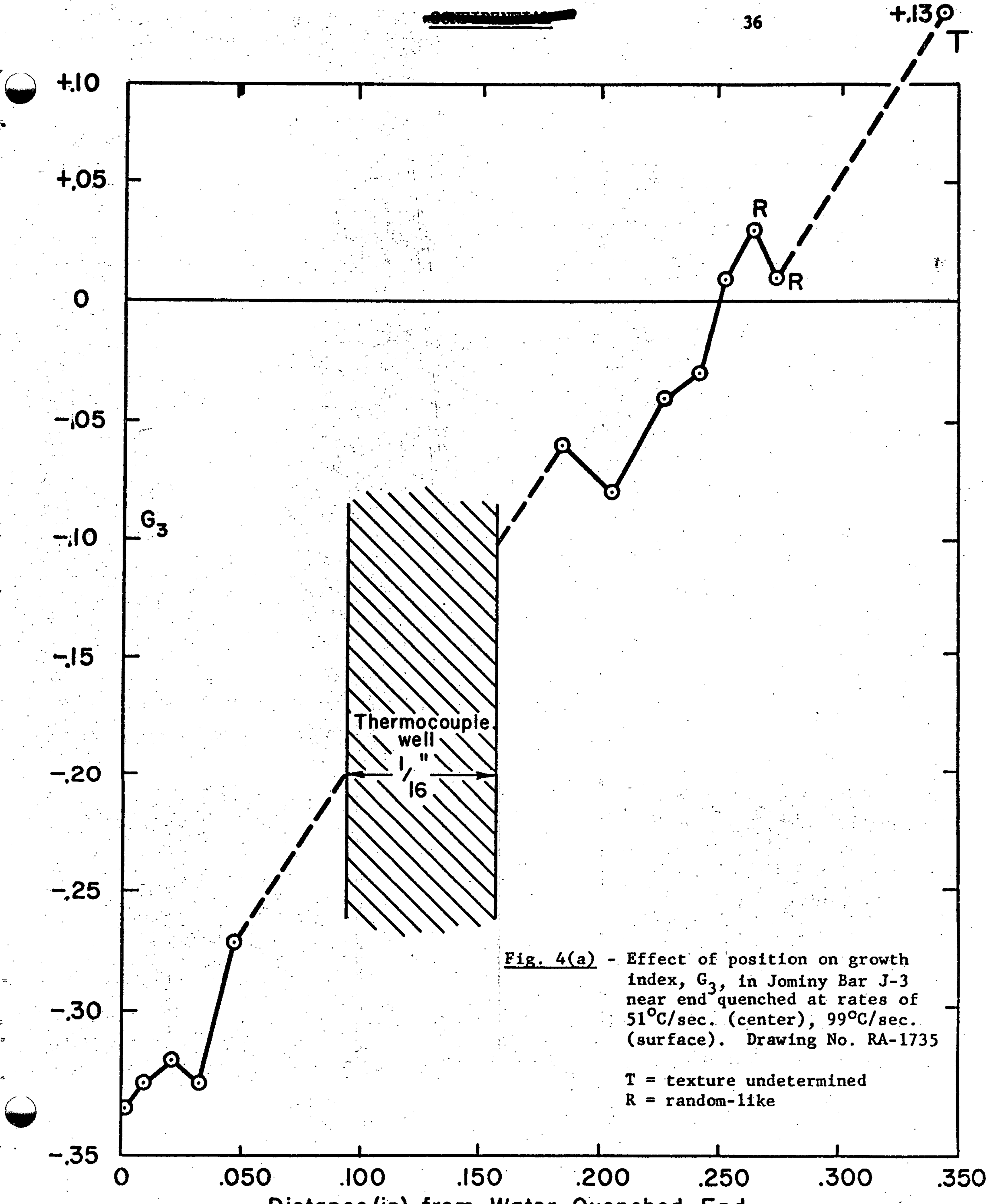


Fig. 4(a) - Effect of position on growth index, G_3, in Jominy Bar J-3 near end quenched at rates of 51°C/sec. (center), 99°C/sec. (surface). Drawing No. RA-1735

T = texture undetermined
R = random-like

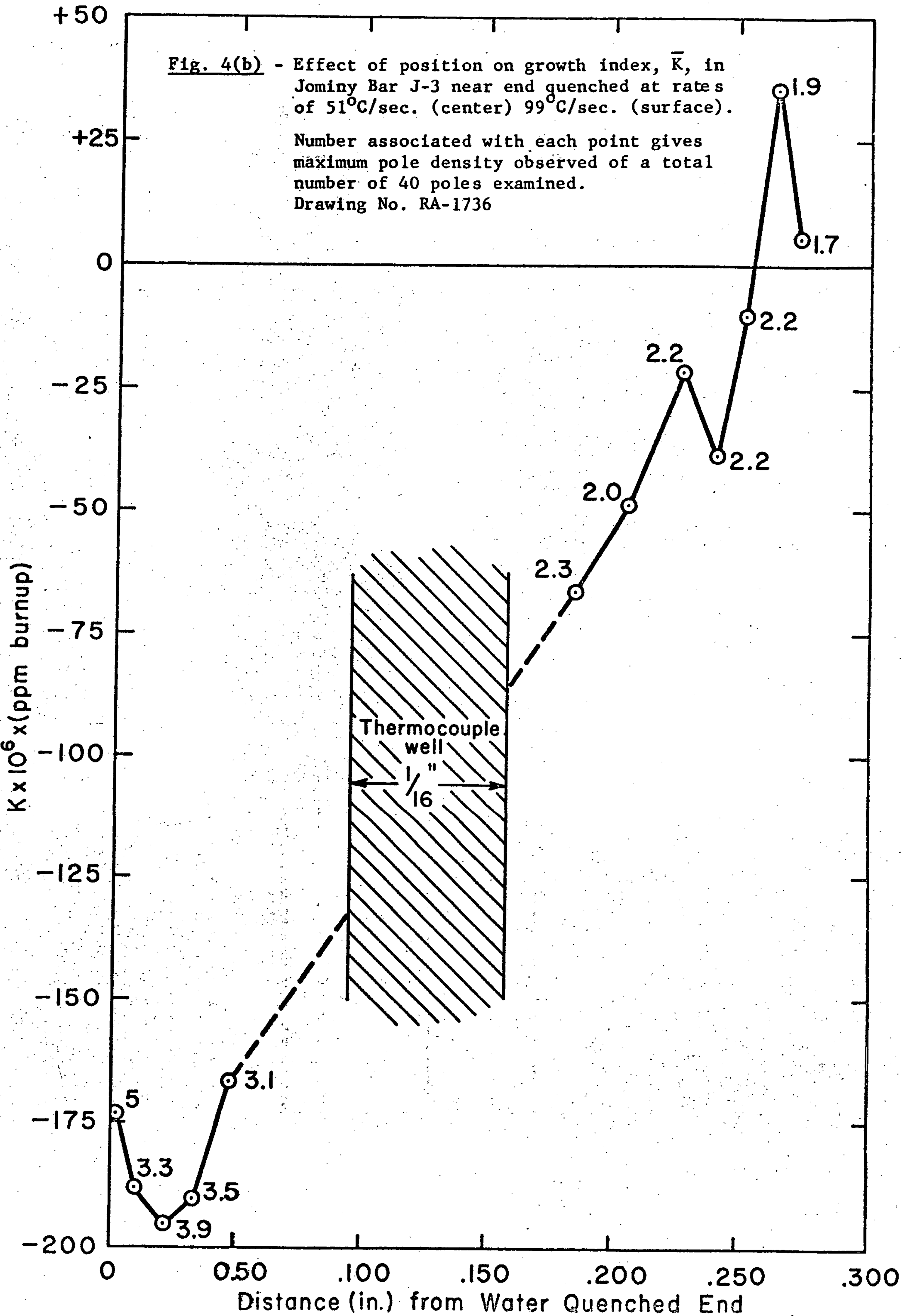
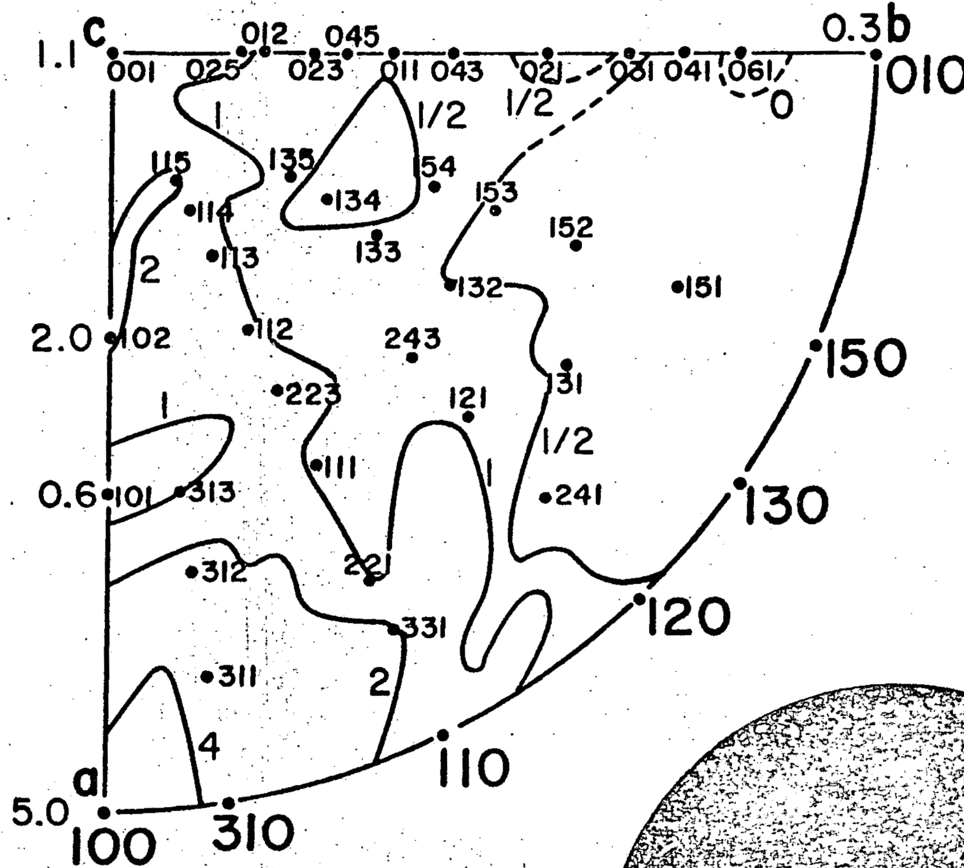
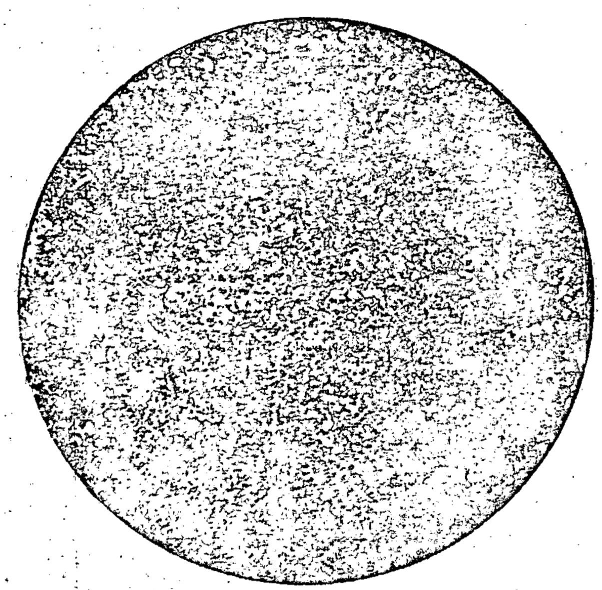


Fig. 4(b) - Effect of position on growth index, $\bar{K}$, in Jominy Bar J-3 near end quenched at rates of 51°C/sec. (center) 99°C/sec. (surface).

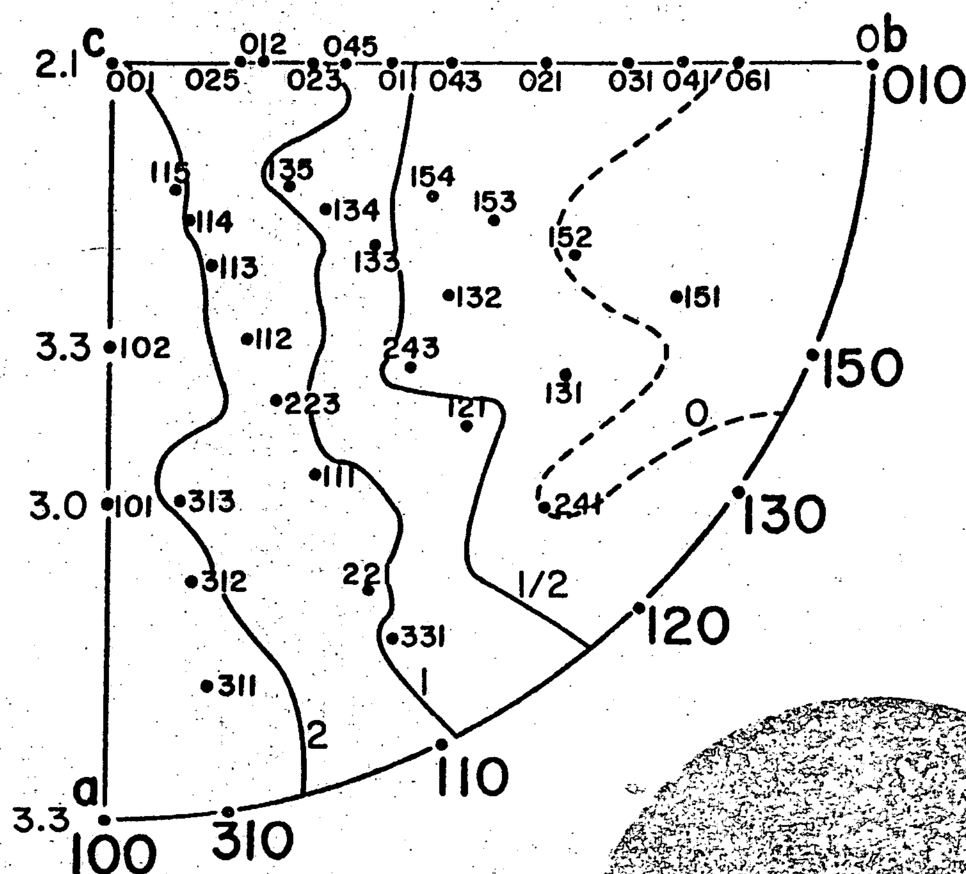
Number associated with each point gives maximum pole density observed of a total number of 40 poles examined.
Drawing No. RA-1736

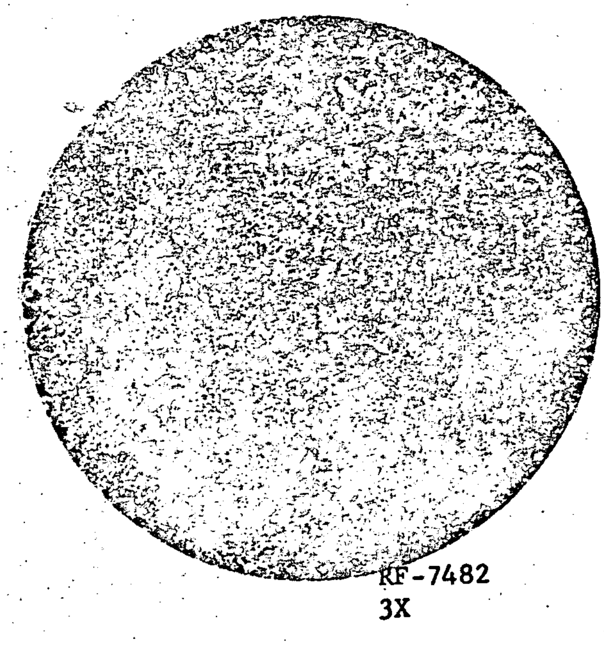




RF-7468
3X

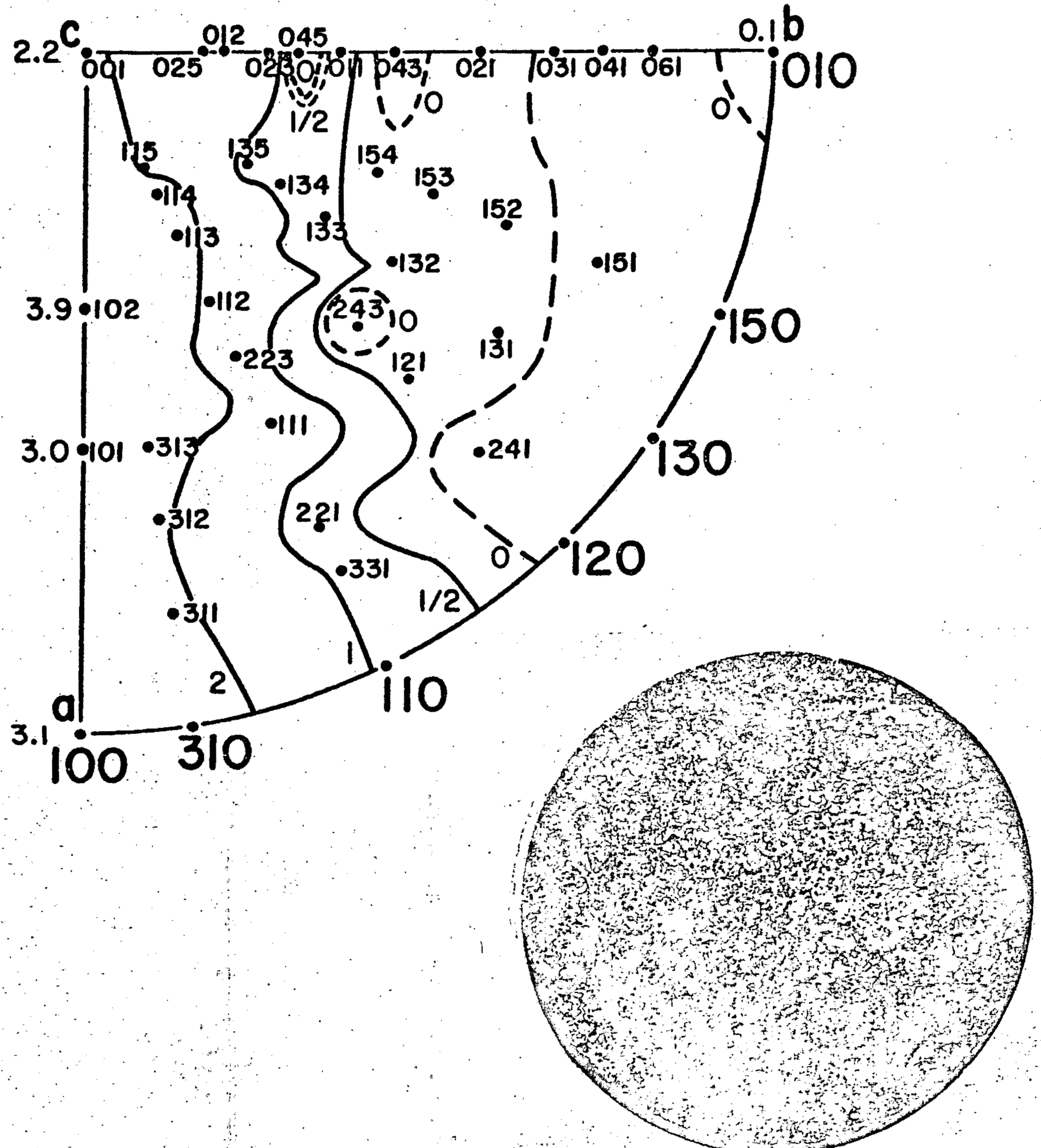
Fig. 5(a) - Crystallographic (inverse) pole figures for the axial direction of Jominy Bar J-3 at increasing distances from the water-quenched end at 0.002 in. from end.
G_3 = -0.34, $\bar{K}$ = -173
Drawing No. RA-1556





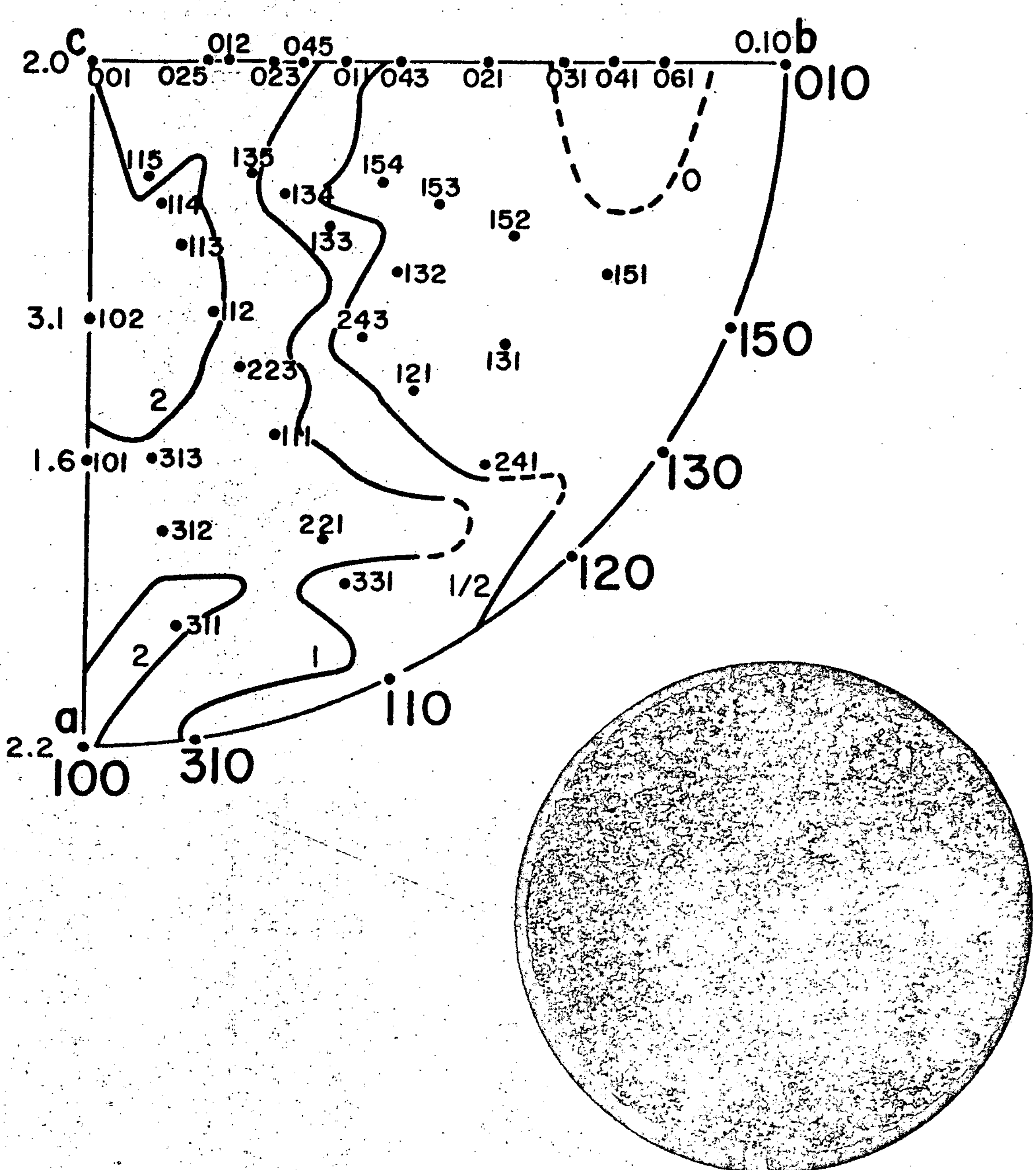
RF-7482
3X

Fig. 5(b) - At 0.010 in. from end,
$G_3 = -0.33$
$\overline{K} = -188$
Drawing No. RA-1557



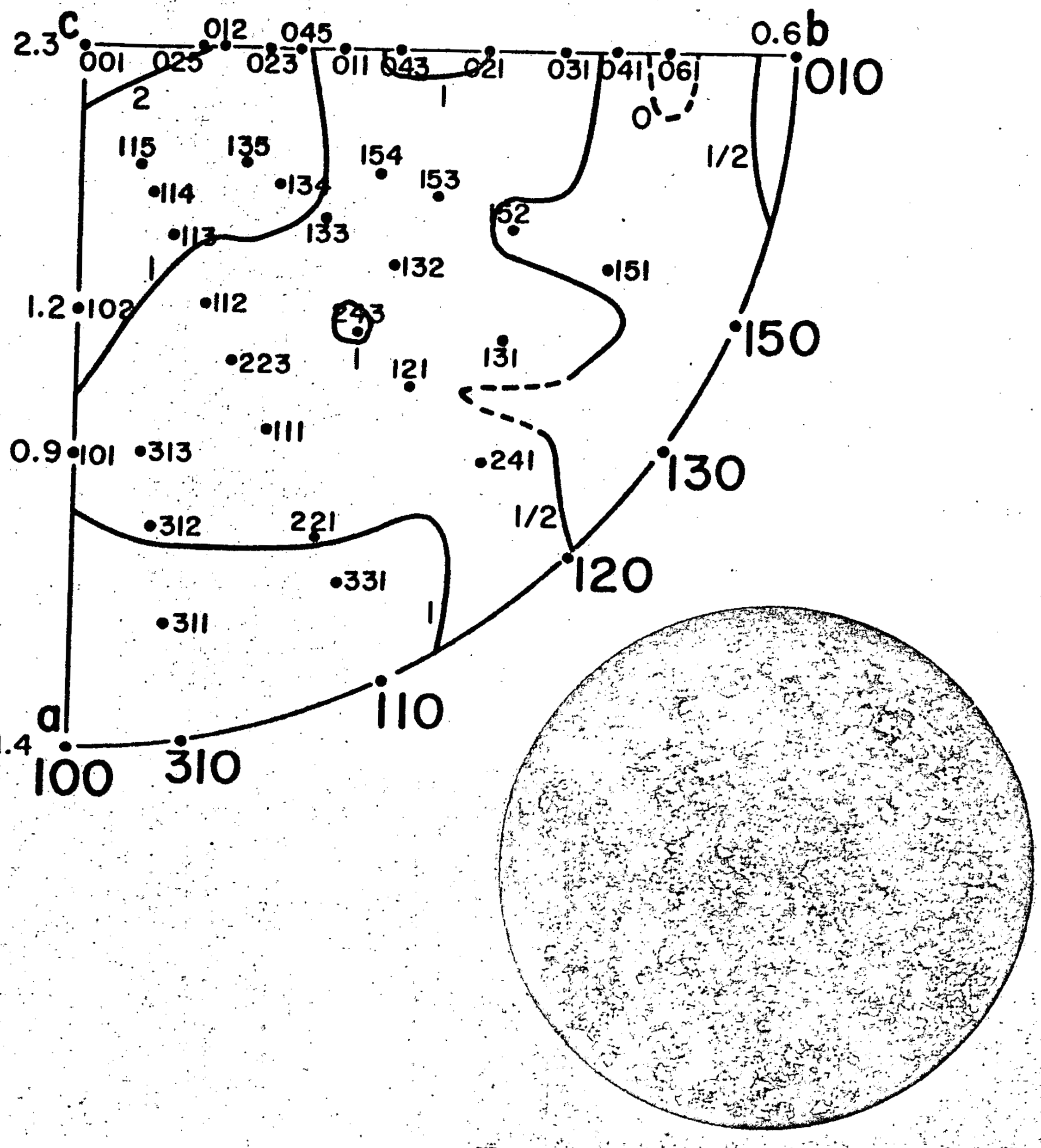
RF-7484
3X

Fig. 5(c) - At 0.022 in. from end,
G_3 = -0.32
$\overline{K}$ = -195
Drawing No. RA-1575



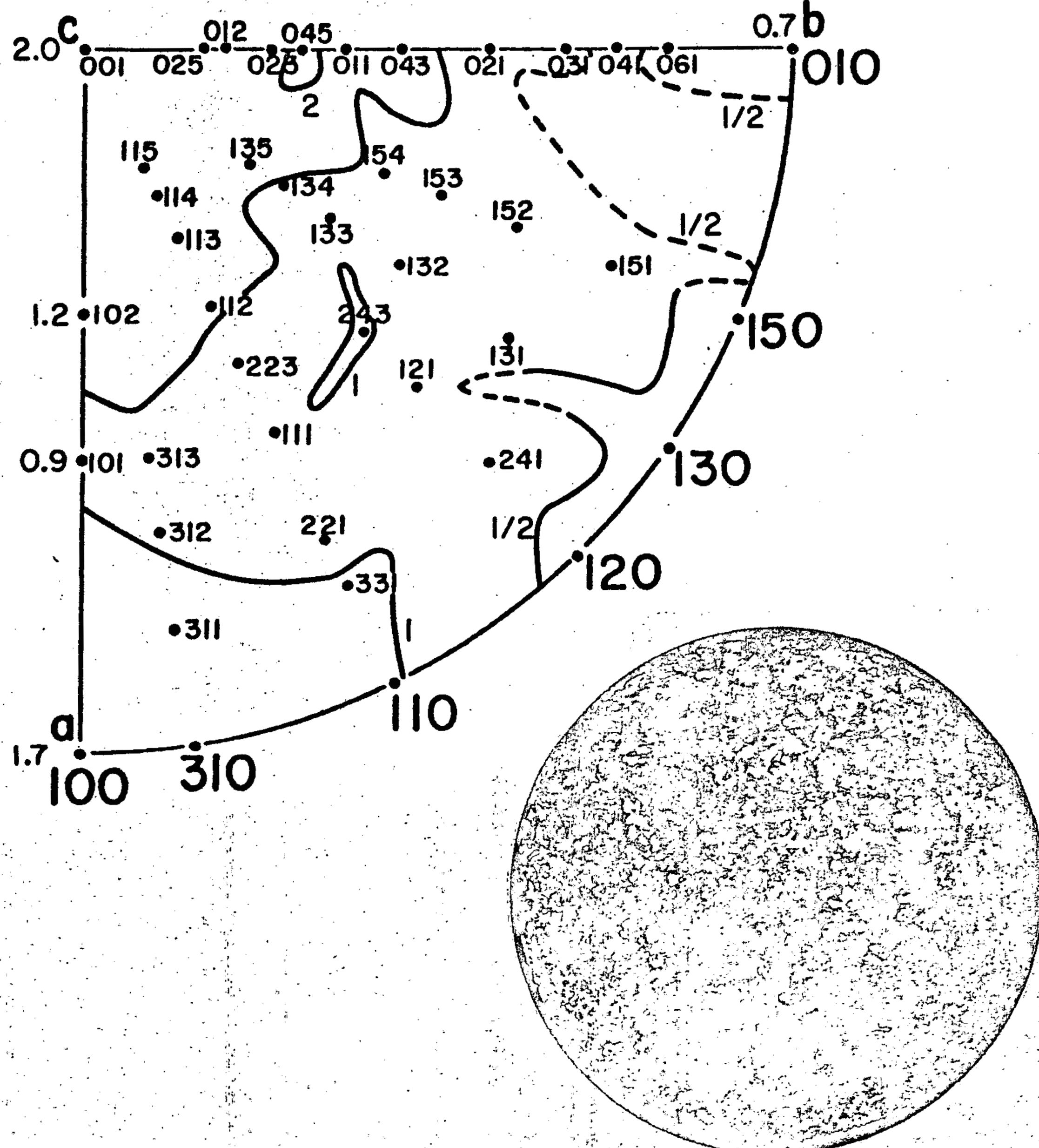
RF-7552
3X

Fig. 5(d) - At 0.047 in. from end,
G_3 = -0.27
$\overline{K}$ = -166
Drawing No. RA-1597



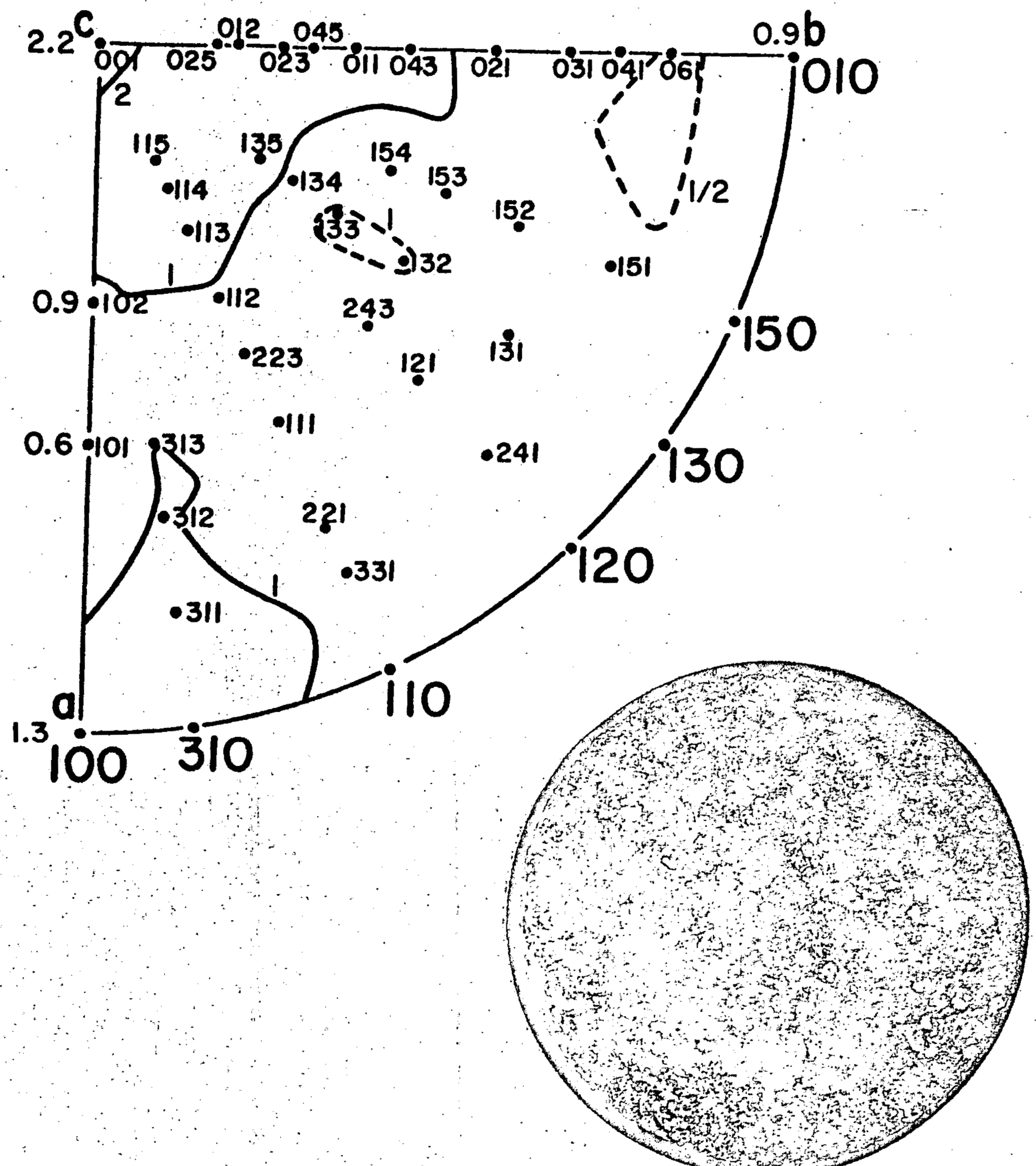
RF-7556
3X

Fig. 5(e) - At 0.183 in. from end,
$G_3 = -0.06$
$\overline{K} = -64$
Drawing No. RA-1614



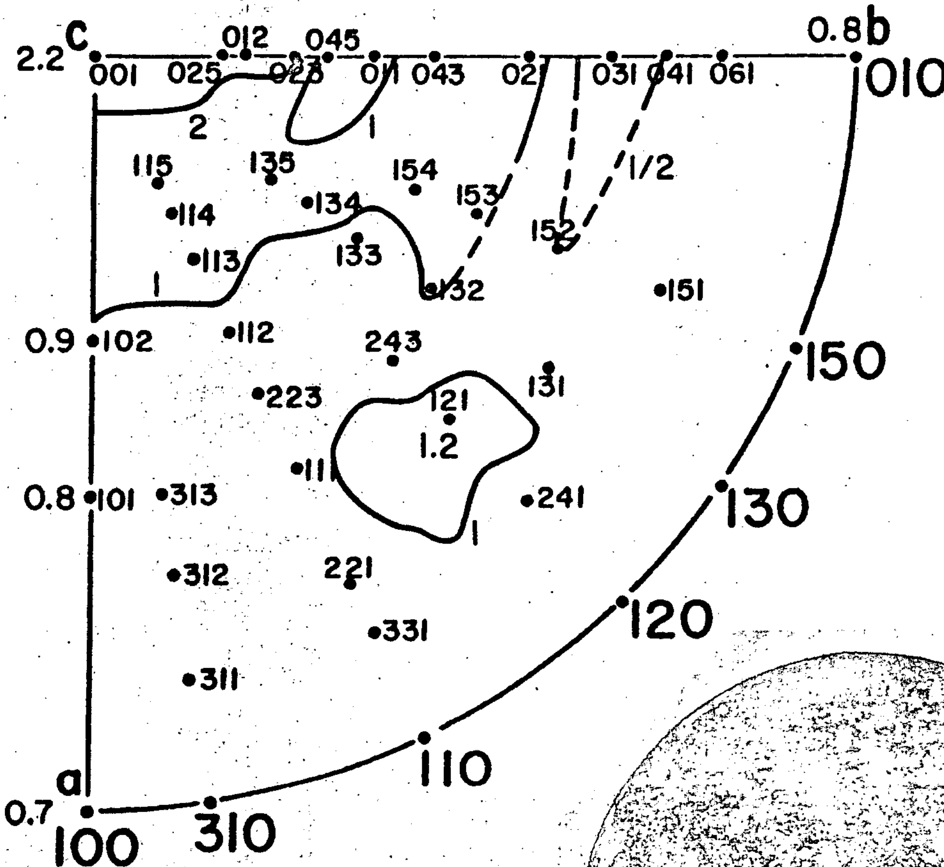
RF-7555
3X

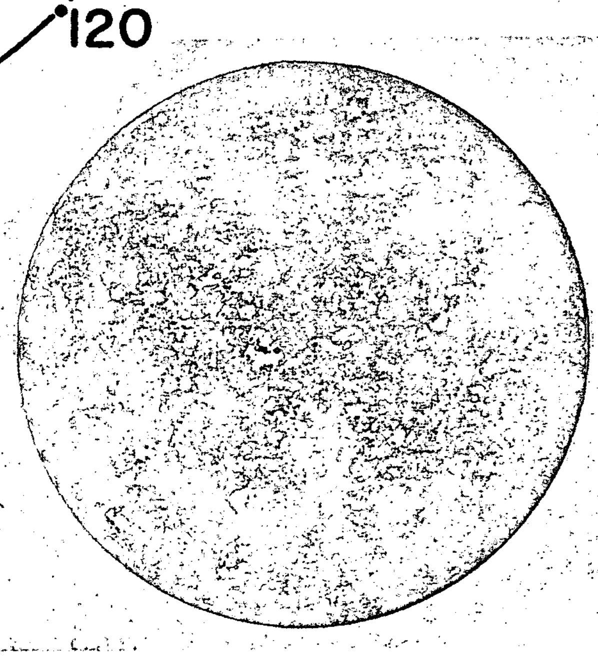
Fig. 5(f) - At 0.204 in. from end,
$G_3 = -0.08$
$\overline{K} = -49$
Drawing No. RA-1613



RF-7554
3X

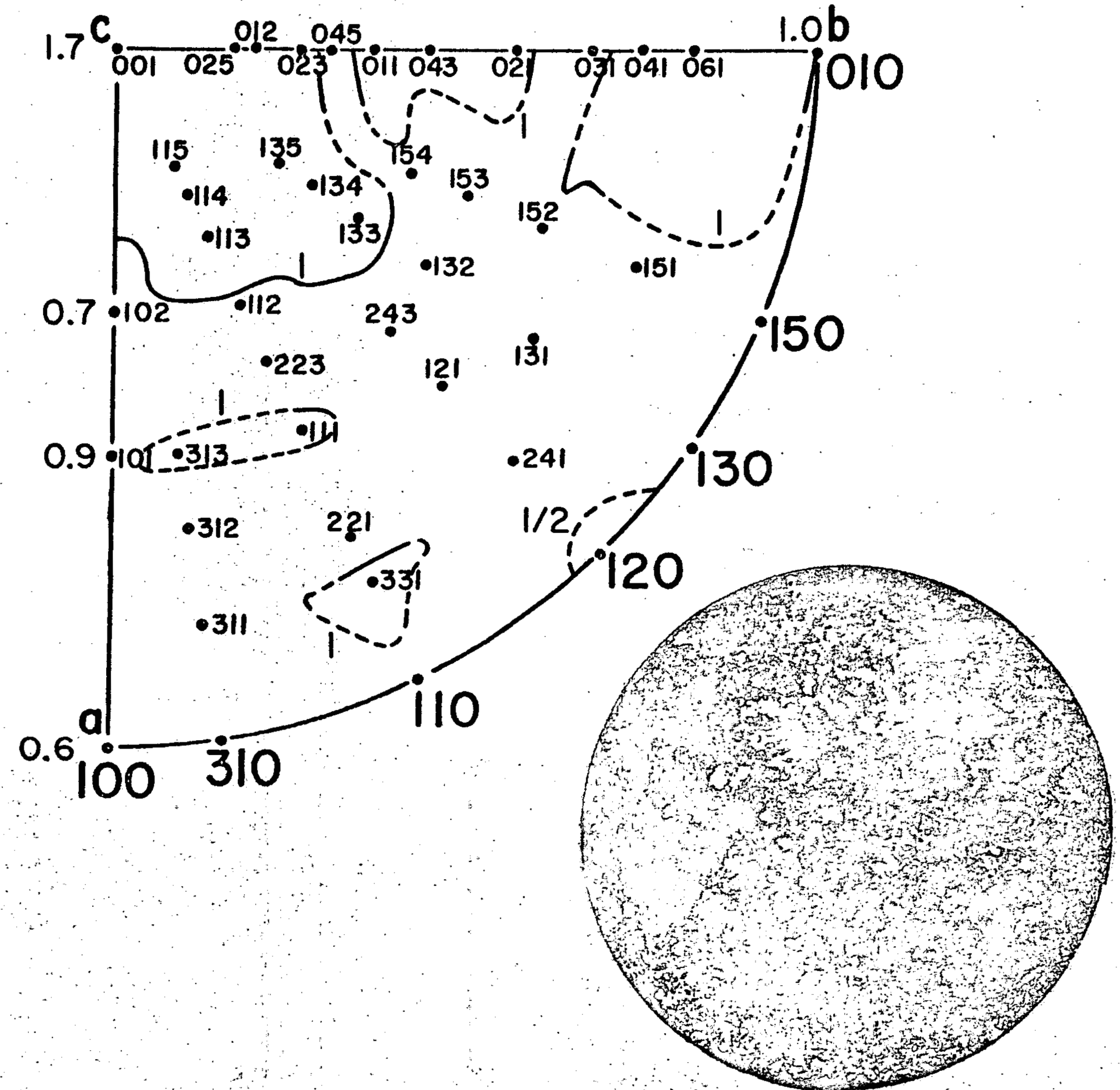
Fig. 5(g) - At 0.225 in. from end,
G_3 = -0.04
$\overline{K}$ = -21
Drawing No. RA-1599





RF-7485
3X

Fig. 5(h) - At 0.250 in. from end,
G_3 = +0.01
$\overline{K}$ = -10
Drawing No. RA-1578



RF-7467
3X

Fig. 5(i) - At 0.272 in. from end,
G_3 = +0.01
$\overline{K}$ = +6
Drawing No. RA-1558

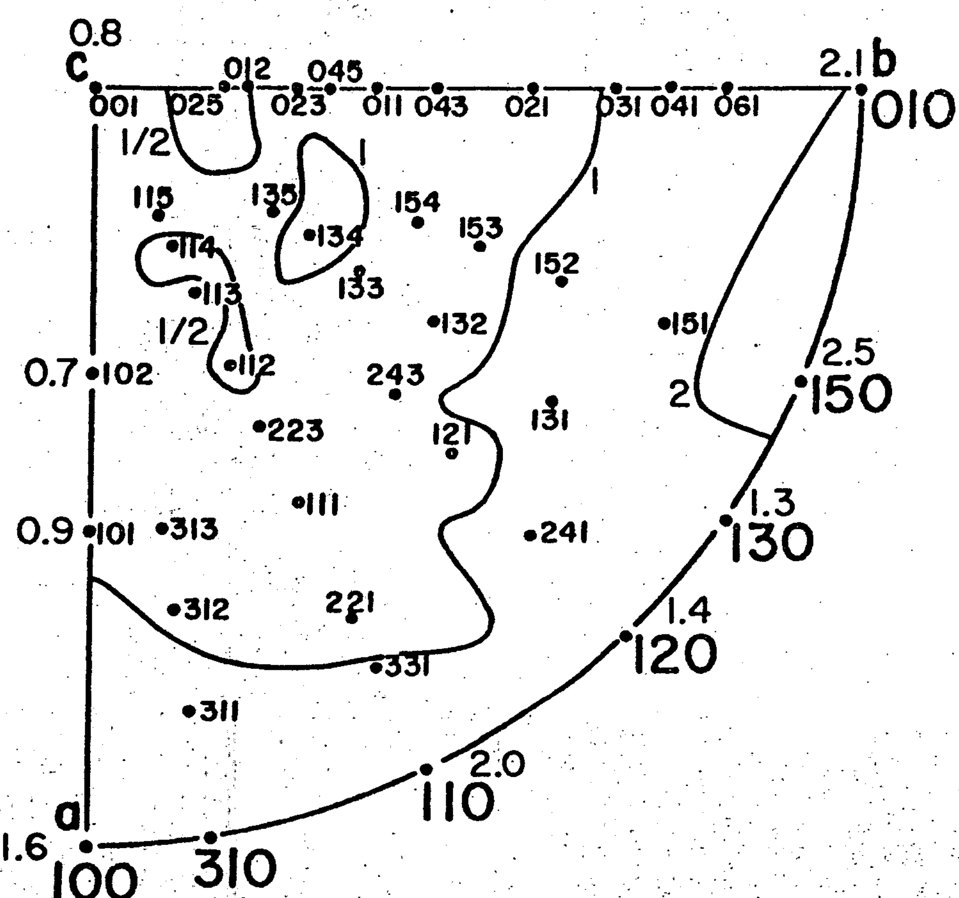
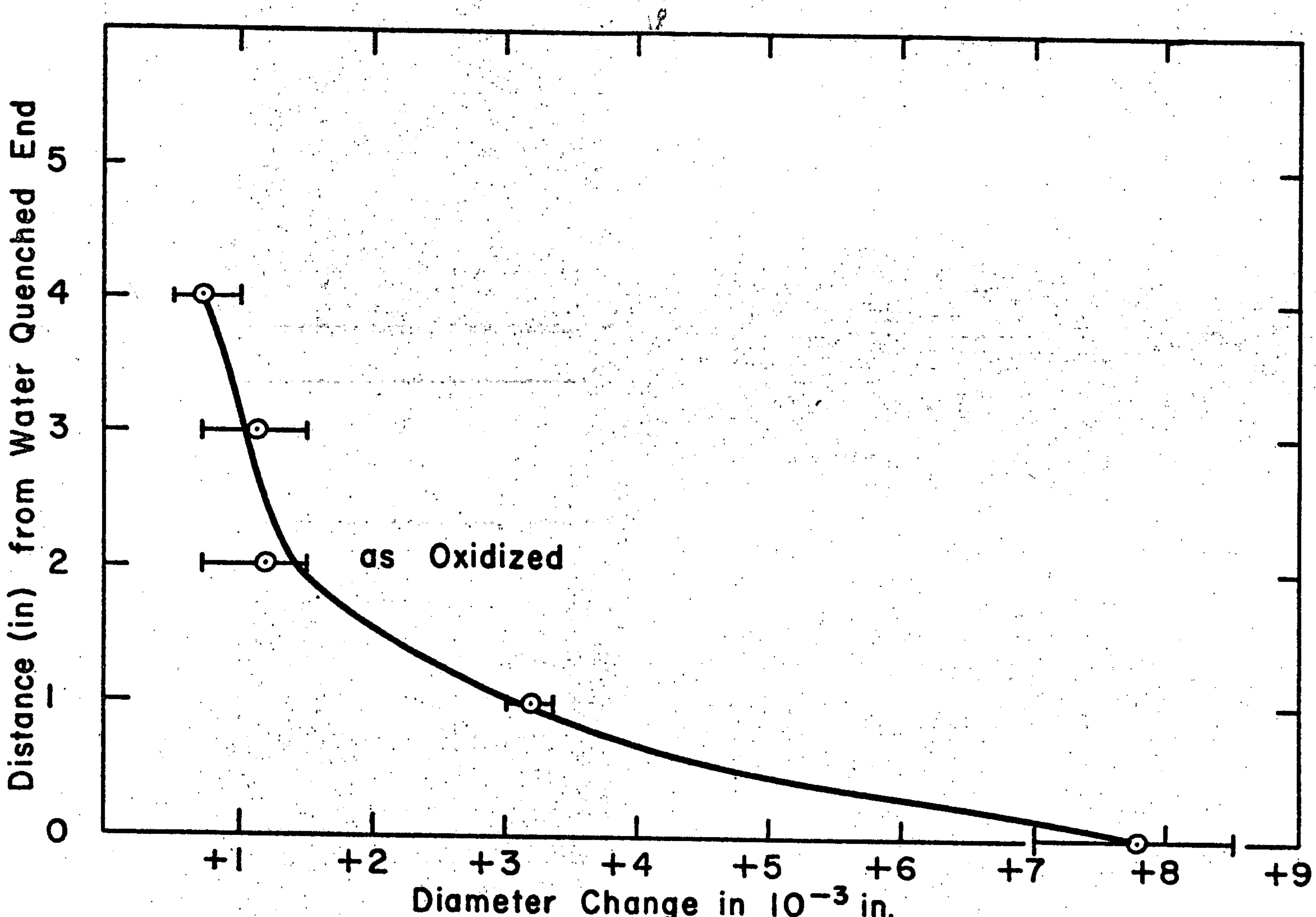


Fig. 6 - Crystallographic pole figure for a direction perpendicular to the temperature gradient in Jominy Bar J-7 over a distance of 0-1/4 inch from the quenched end.
$G_3 = +0.066 \quad \bar{K} = +18$
Drawing No. RA-1731



<u>Fig. 7(a)</u> - Distortion in J-3 showing change in diameter with length.

$\Delta L = -0.045$ in.

$L_o = 5.000$ in.

Drawing No. RA-1737

[redacted]

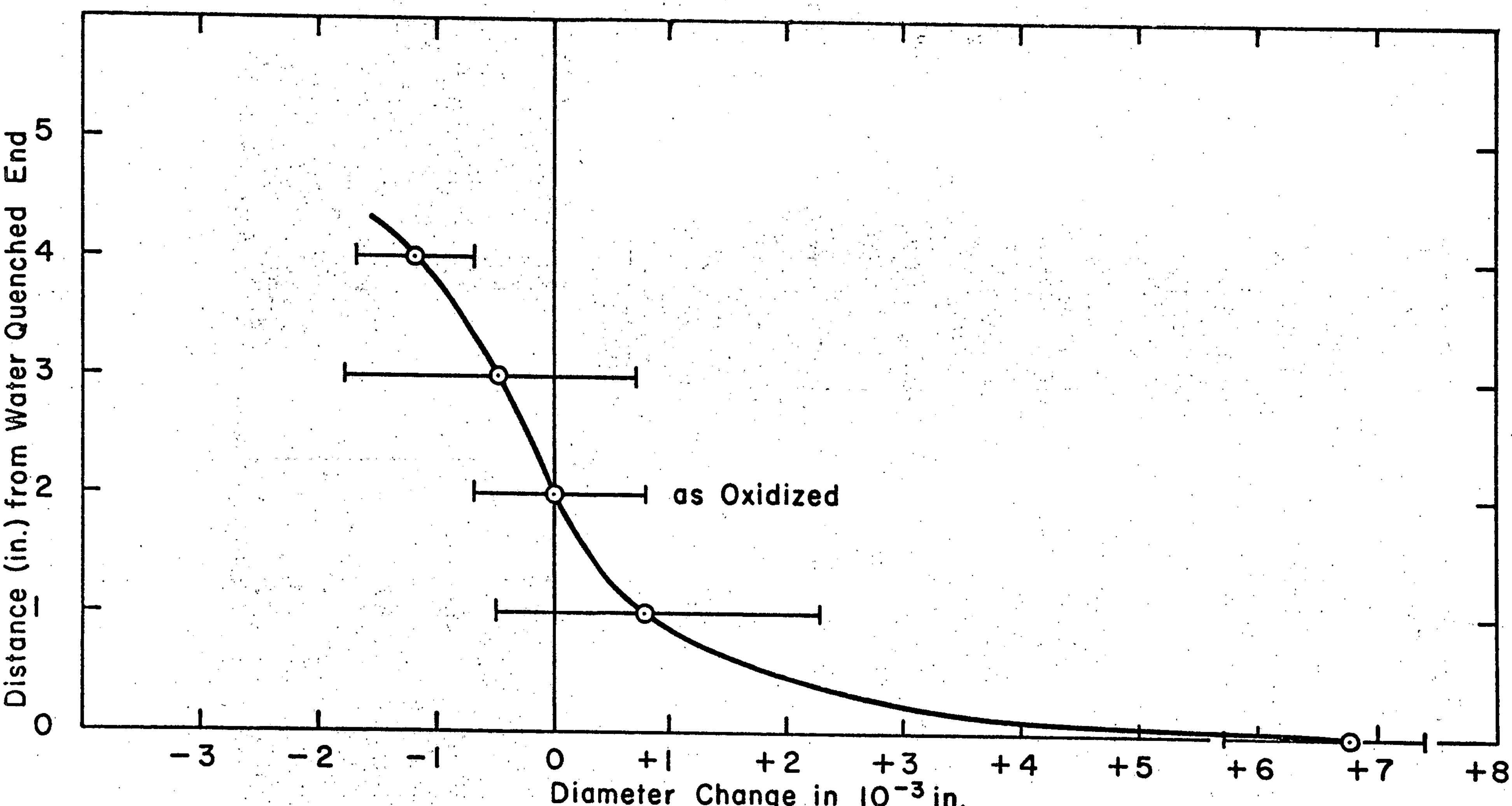


Fig. 7(b) - Distortion in J-5 showing change in diameter with length.

ΔL = -0.043 in.
L_o = 5.000 in.
Drawing No. RA-1738

[redacted]

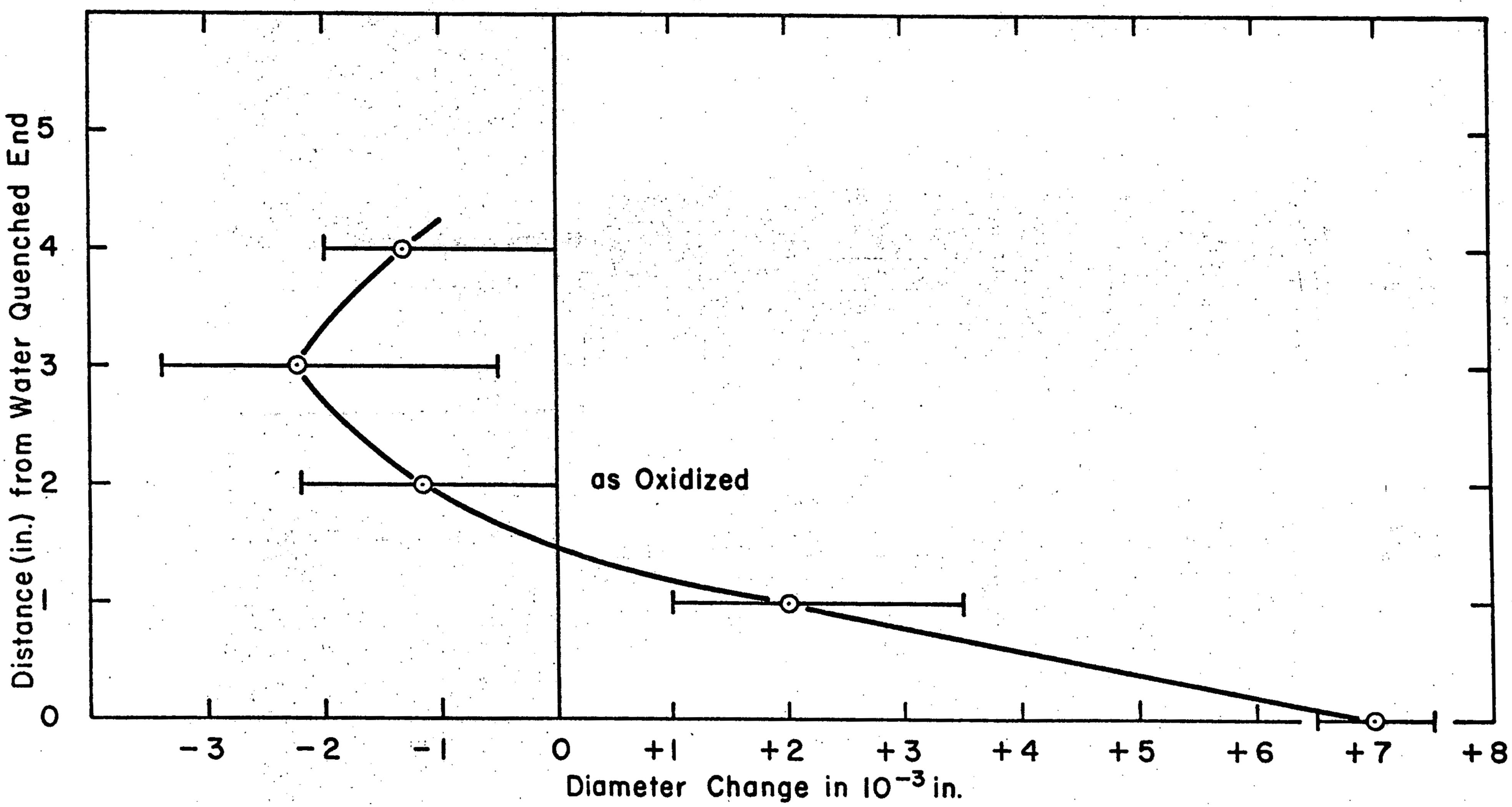


Fig. 7(c) - Distortion in J-8 showing change in diameter with length.

$\Delta L = -0.047$ in.
$L_o = 5.000$ in.
Drawing No. RA-1739

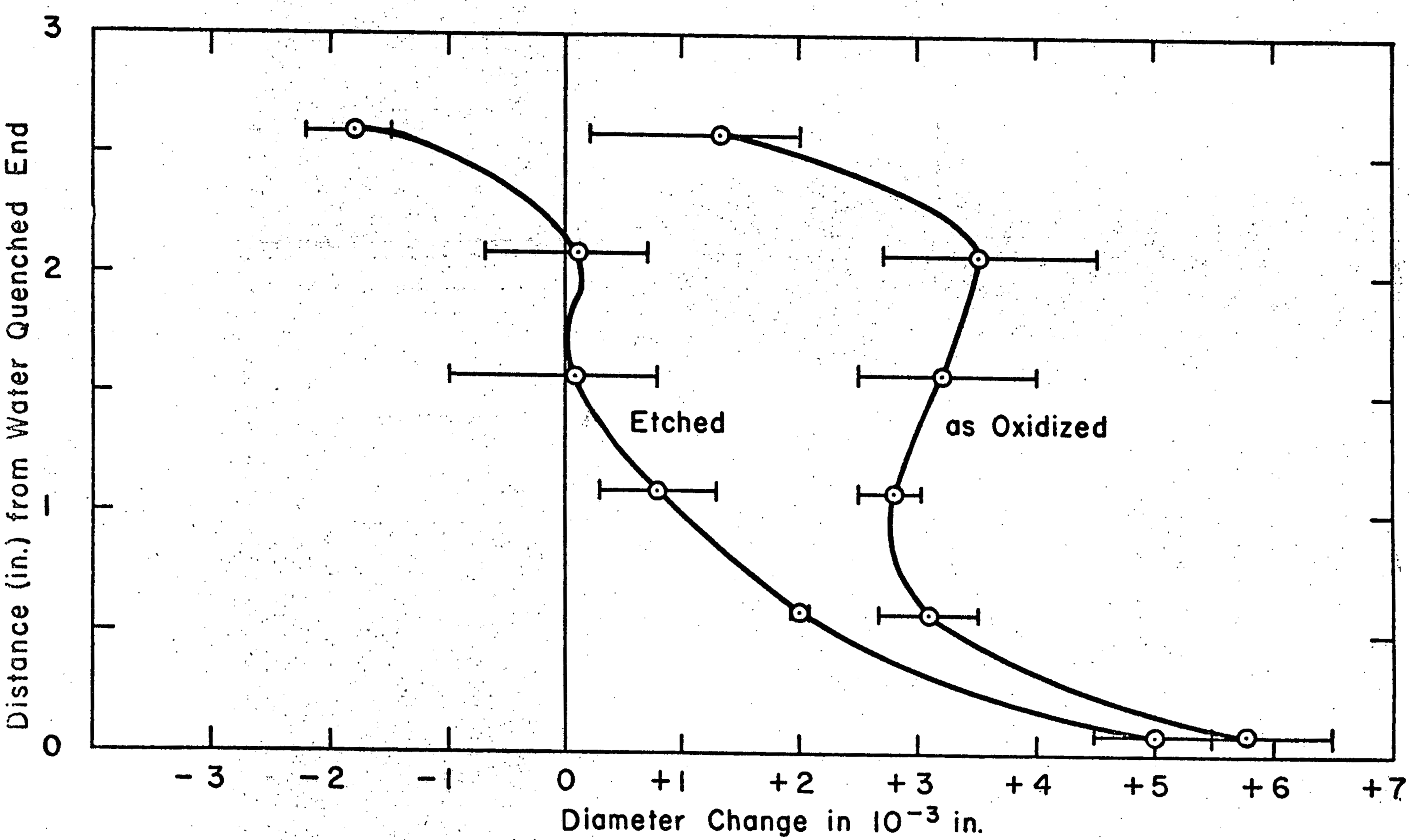


Fig. 7(d) - Distortion in J-10 showing change in diameter with length. Drawing No. RA-1740

ΔL = -.0225 in. (oxidized)
= -.0295 in. (etched)

L_o = 3.00 in.

[redacted]

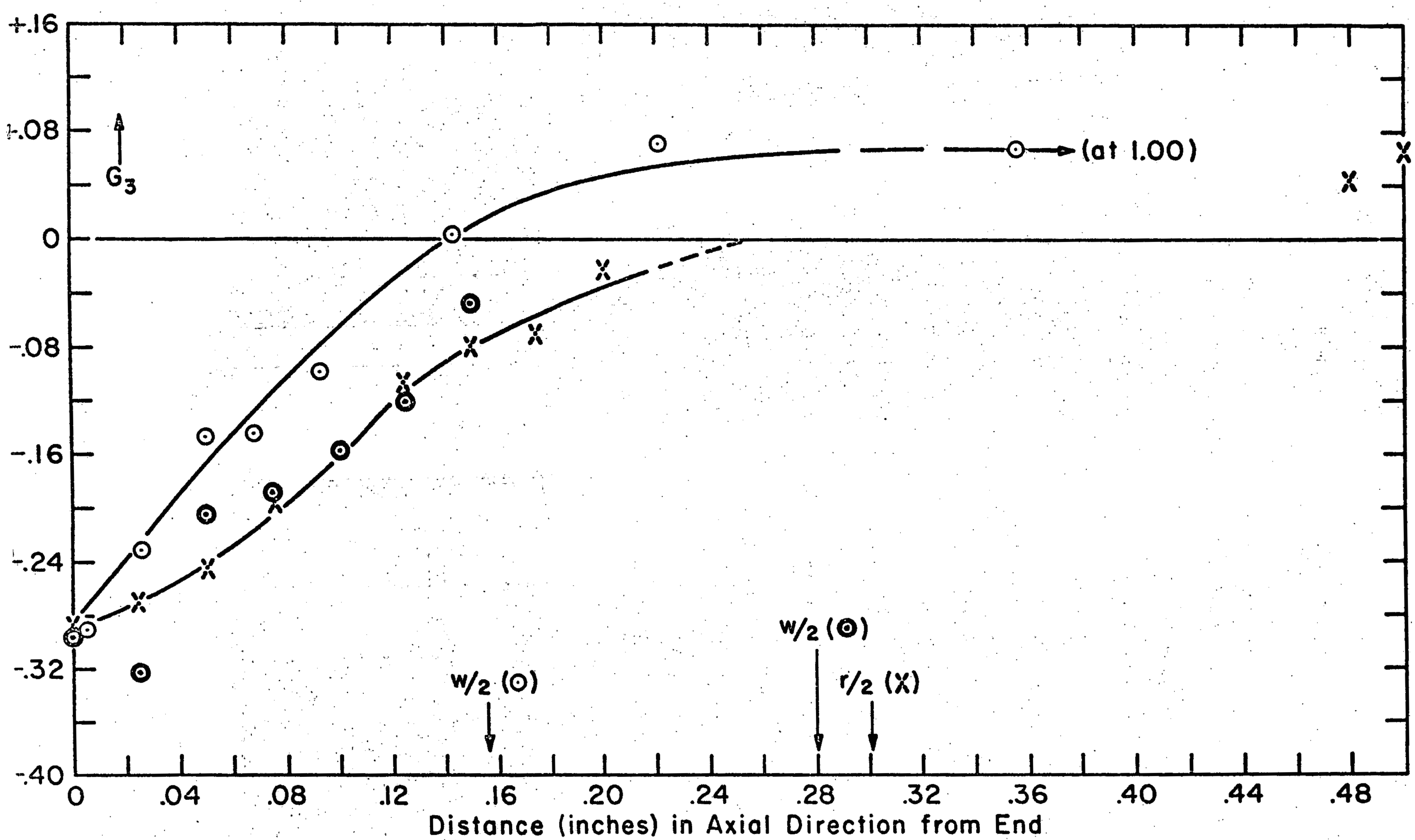
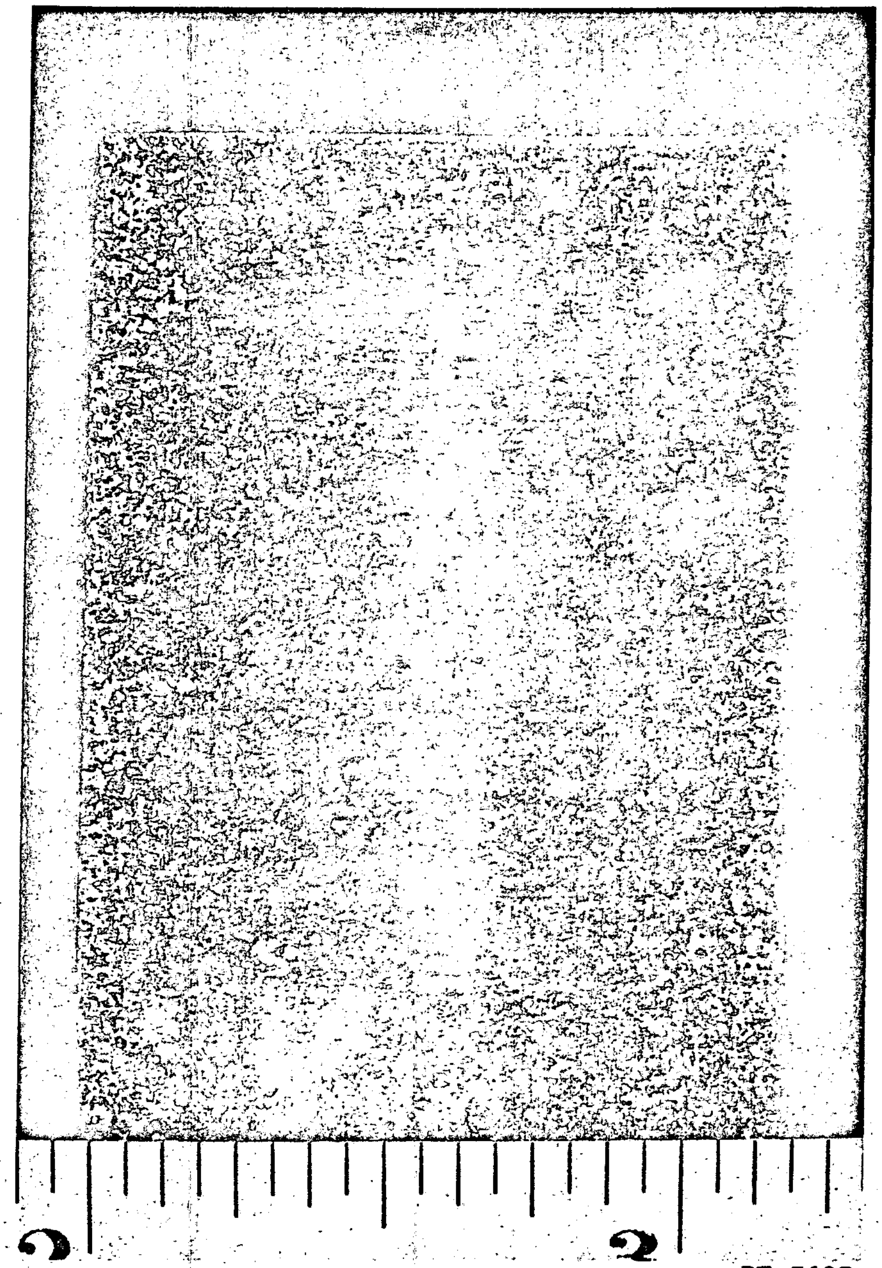


Fig. 8 - Change of axial growth index, G_3, with distance from end of three 8-in. long cylinders after cold water quenching from molten salt (LH980) at 730°C - 15 min.

⊙ 3-1/8-in. OD x 2-1/2 in. ID tube (M_1)
◉ 1-1/2-in. OD x 3/8-in. ID tube (H_1)
X 1-7/32 in. diameter rod (K_1)



3X RF-7607

<u>Fig. 9</u> - Photomacrograph of end section of 1-7/32-in. diameter rod quenched into cold water by total immersion. (HCl - HNO_3 etch)

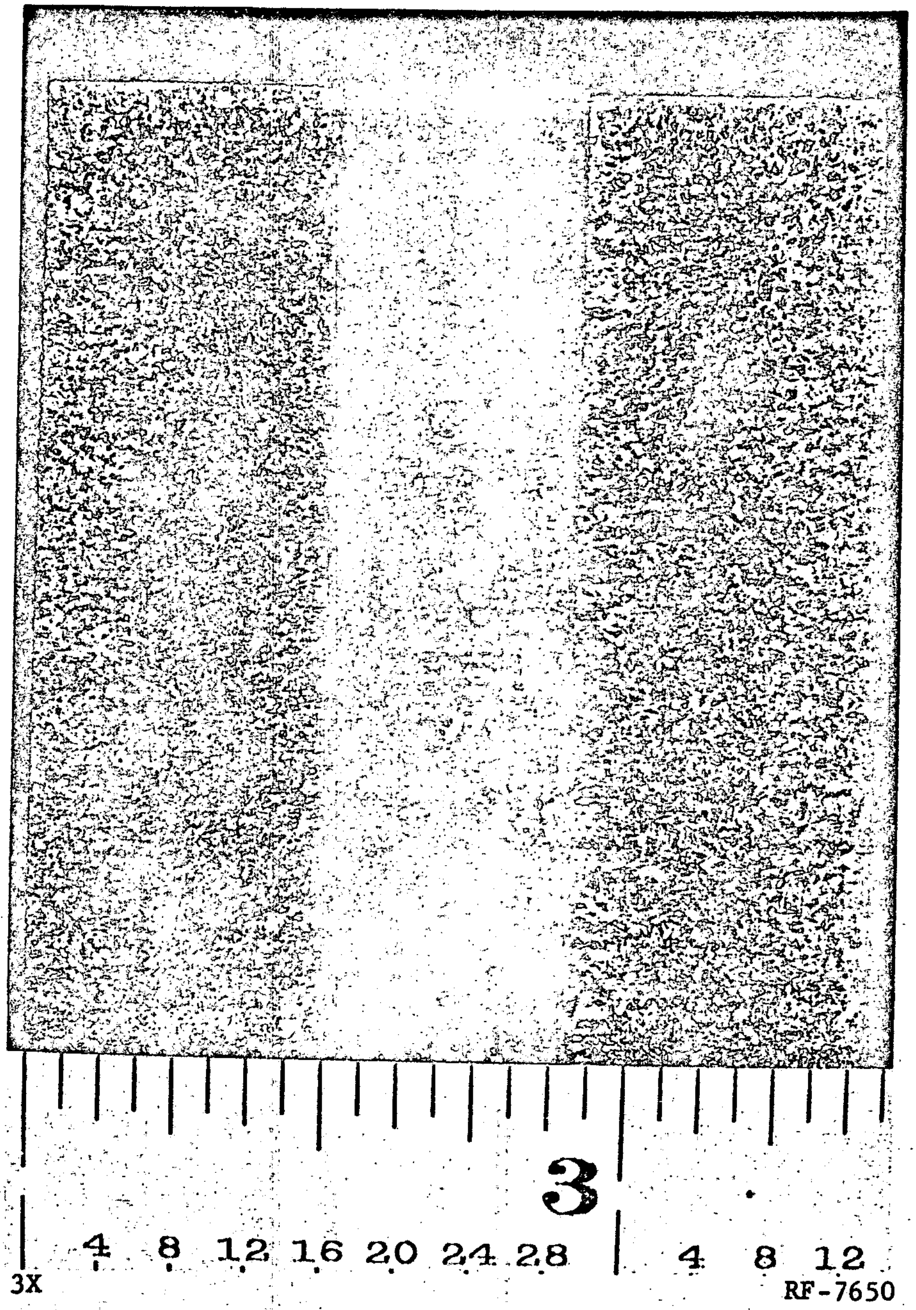
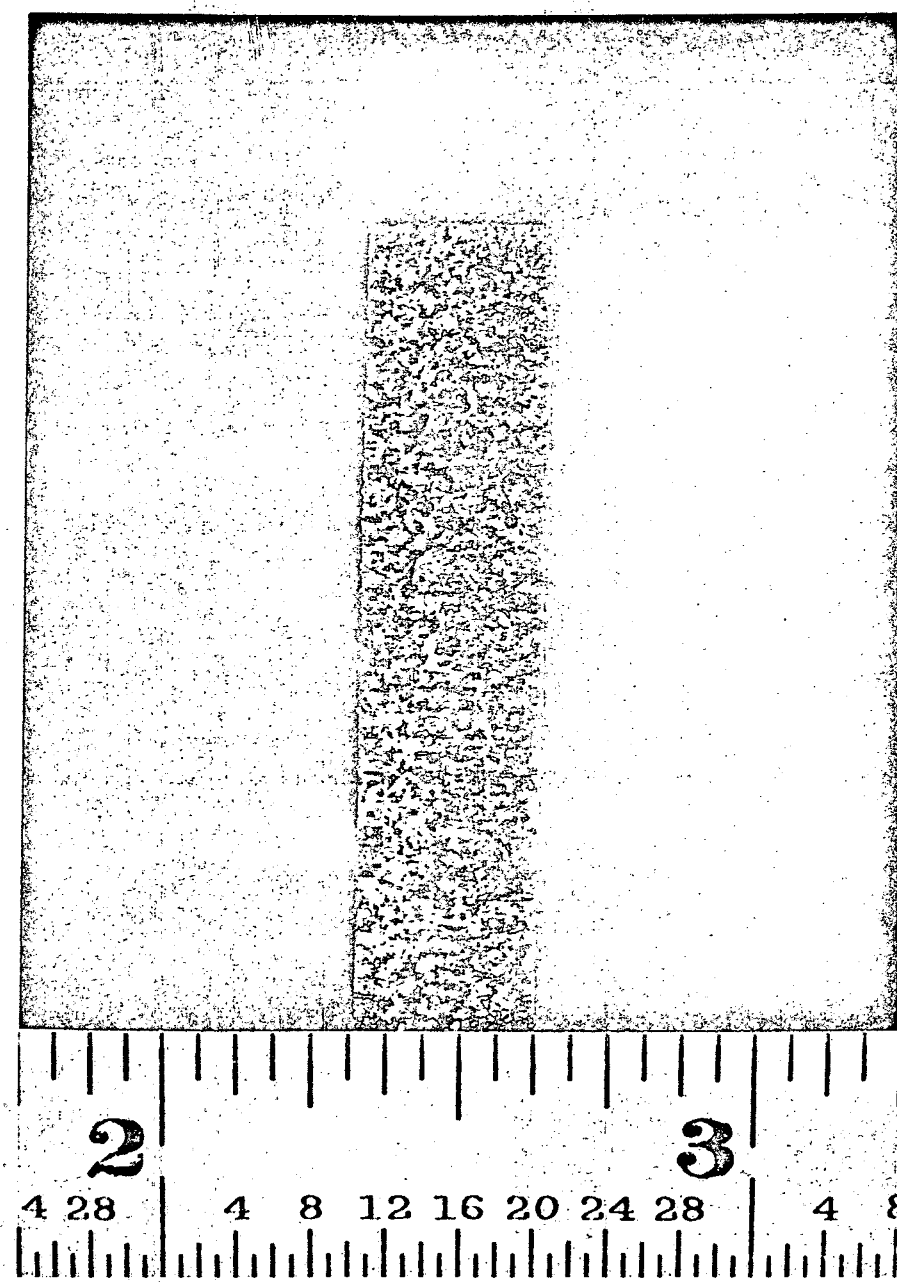


Fig. 10 - Photomacrograph of end section of 1-1/2 in. OD x 3/8 in. ID tube quenched into cold water by total immersion.



3X RF-7591

Fig. 11 - Photomacrograph of end section of 3-1/8 in. OD x 2-1/2 in. ID tube quenched into cold water by total immersion.

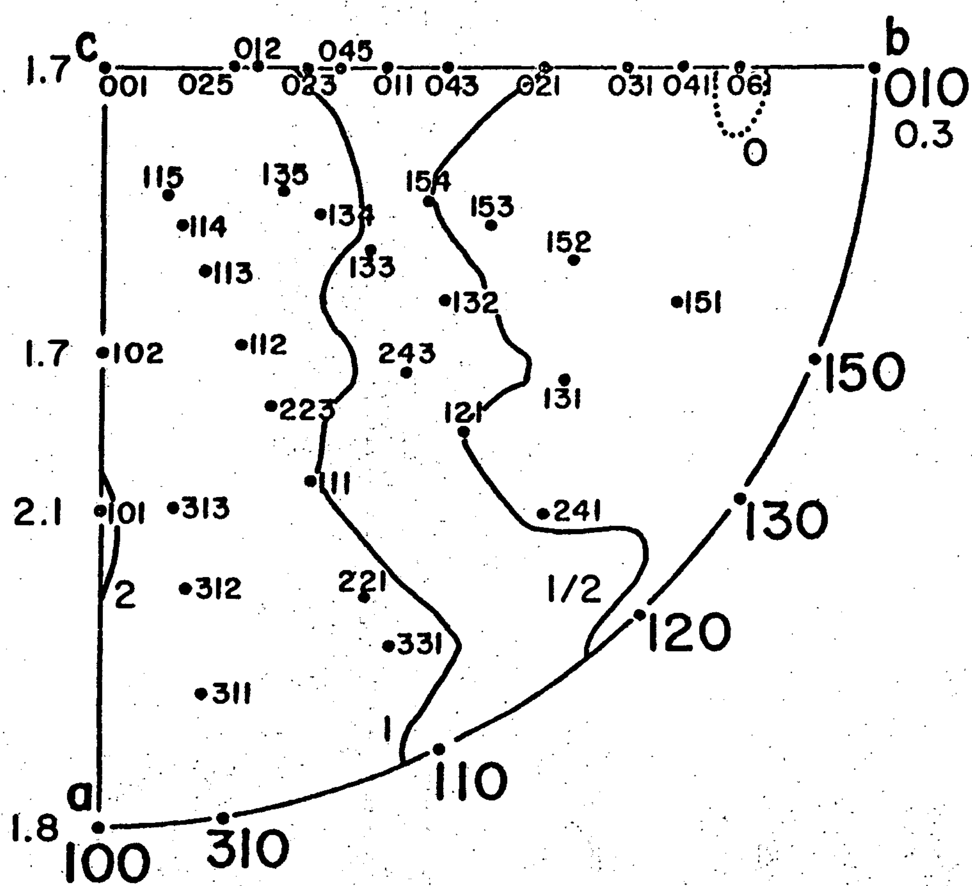


Fig. 12(a) - Crystallographic (inverse) pole figure for the radial direction in a 3-1/8 in. OD x 2-1/2 in. ID x 8 in. long tube (M1), 3.5-4.5 in. from end. Data from 10^{o} sector.

At 0.042 in. from OD. G_3 = -0.18
Drawing No. RA-1748

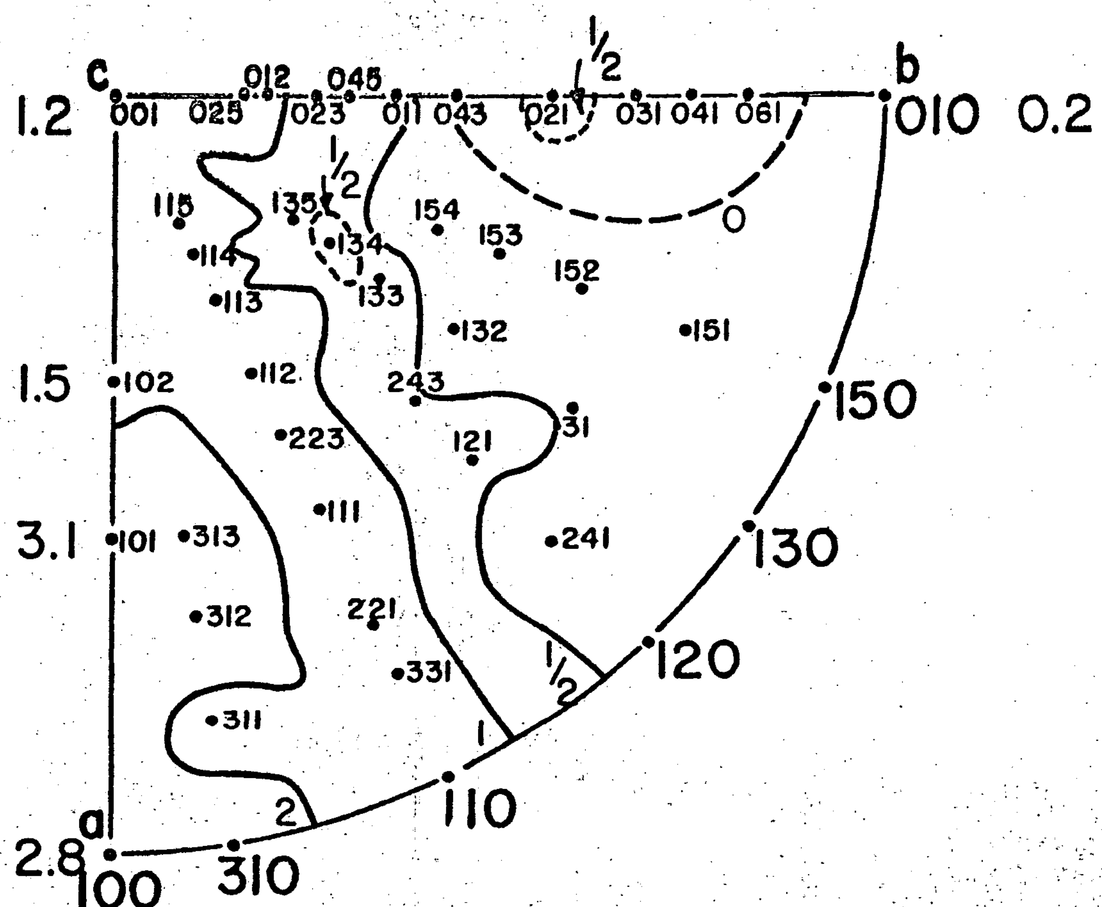


Fig. 12(b) - At 0.308 in. from OD (near ID); G_3 = -0.34
Drawing No. RA-1730

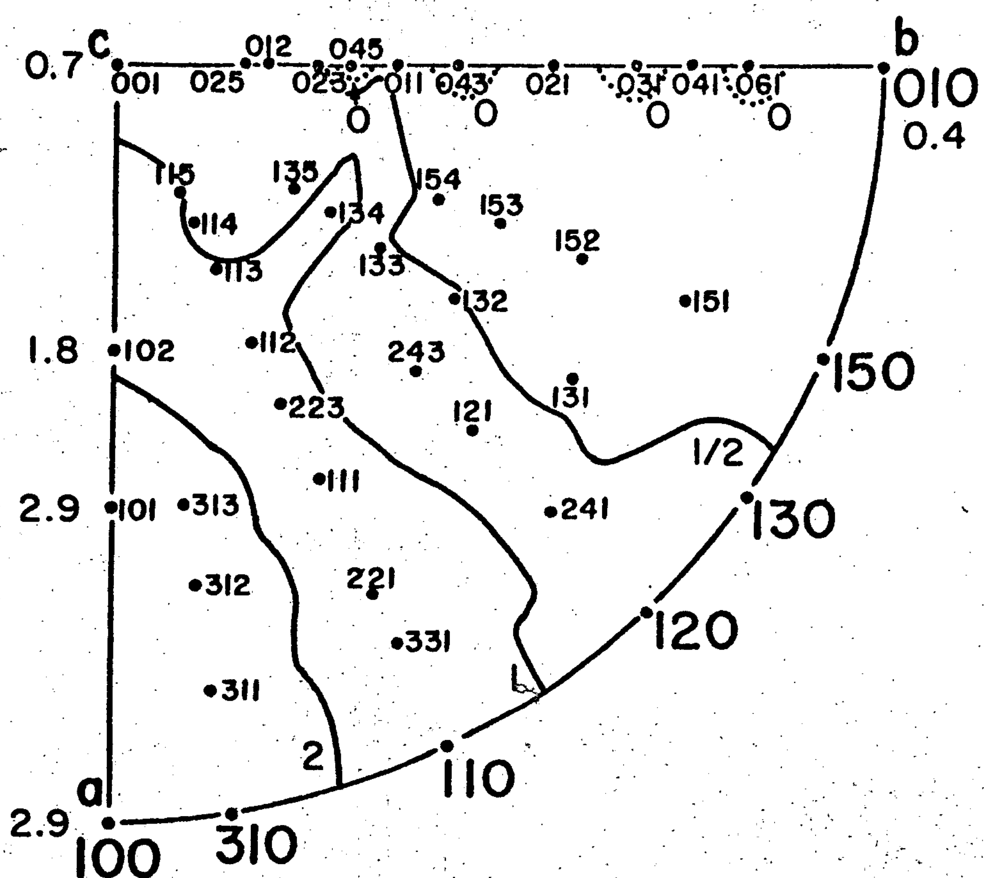


Fig. 13(a) - Crystallographic (inverse) pole figure for the radial direction in a 1-1/2 in. OD x 3/8 in. ID x 8 in. long tube (H1), 1.8-3.8 in. from end. Tube was beta quenched by total immersion in cold water.

At 0.007 from OD; G_3 = -0.34
Drawing No. RA-1741

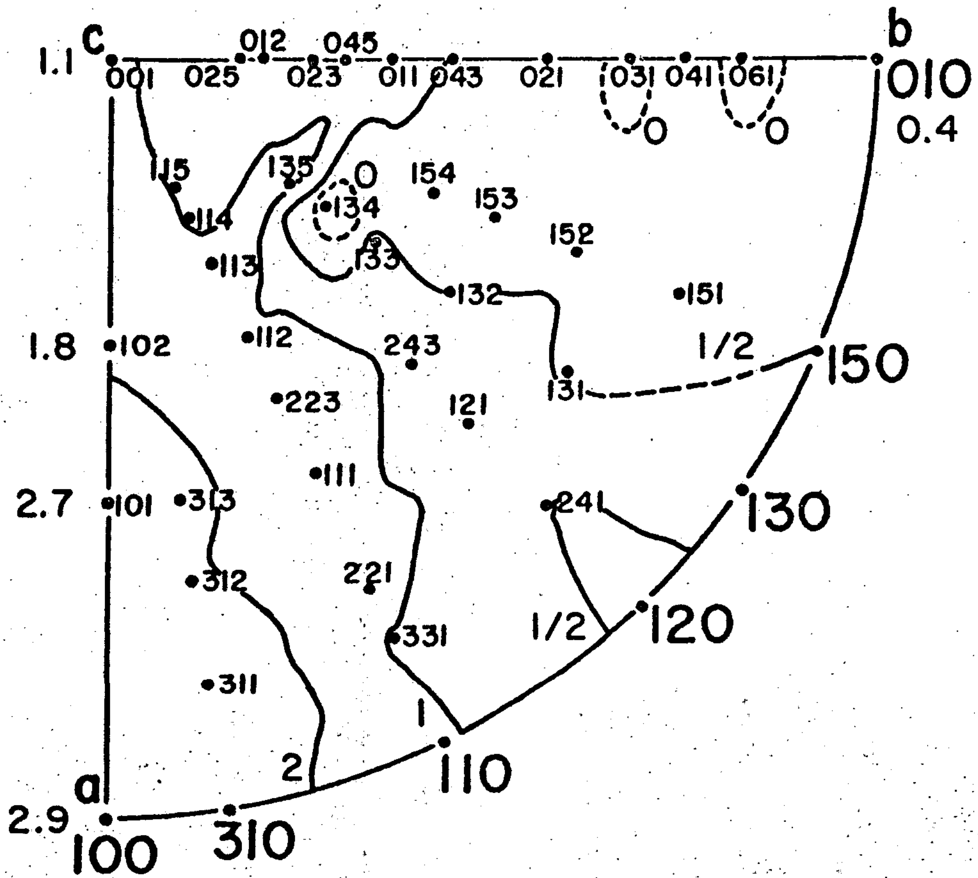


Fig. 13(b) - At 0.055 in. from OD; G_3 = -0.28
Drawing No. RA-1742

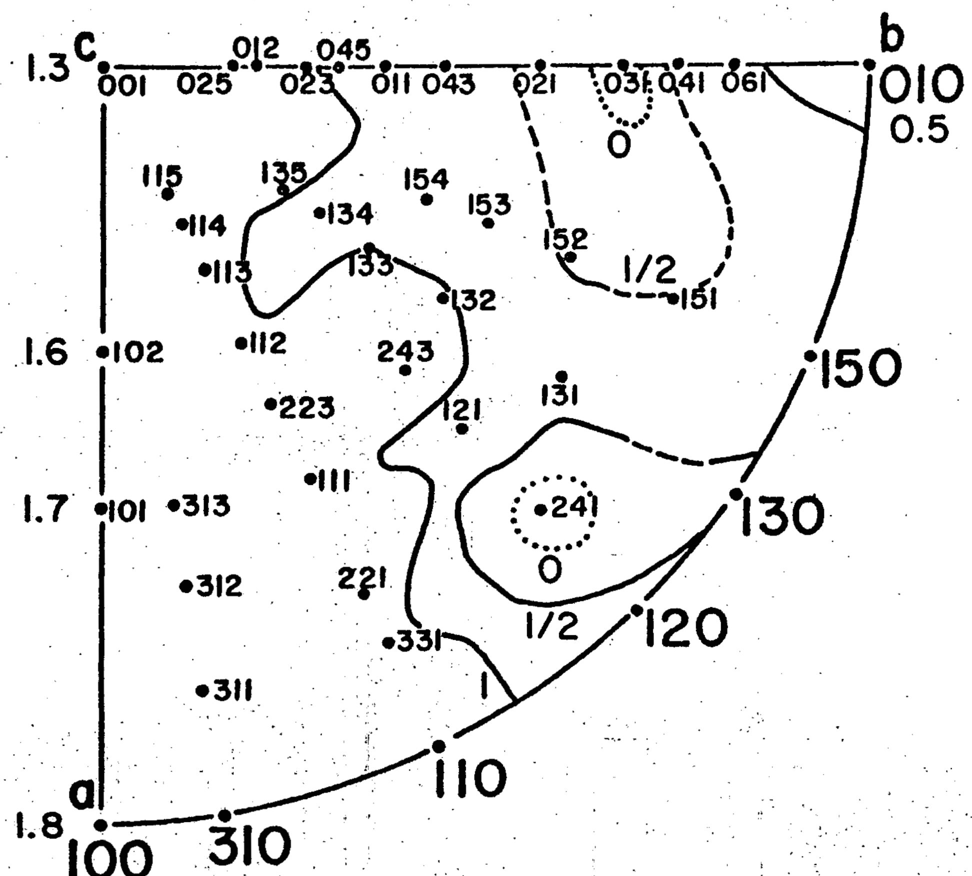


Fig. 13(c) - At 0.116 in. from OD; G_3 = -0.17
Drawing No. RA-1743

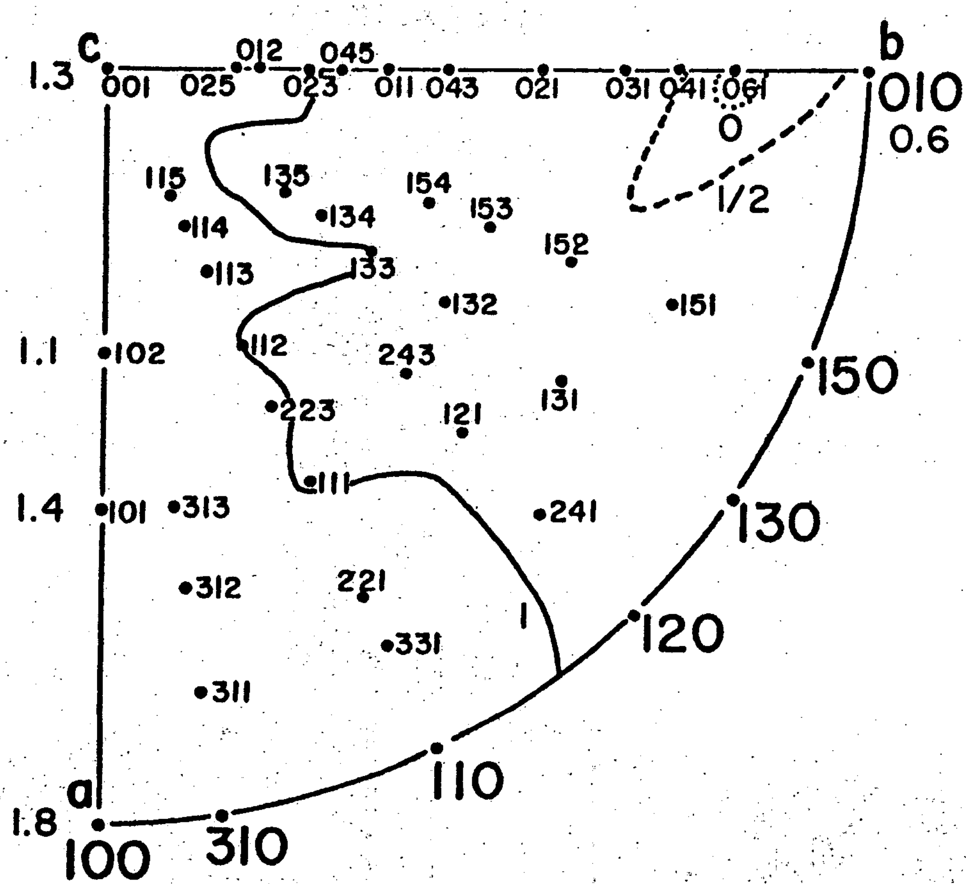


Fig. 13(d) - At 0.155 in. from OD; G_3 = -0.09
Drawing No. RA-1744

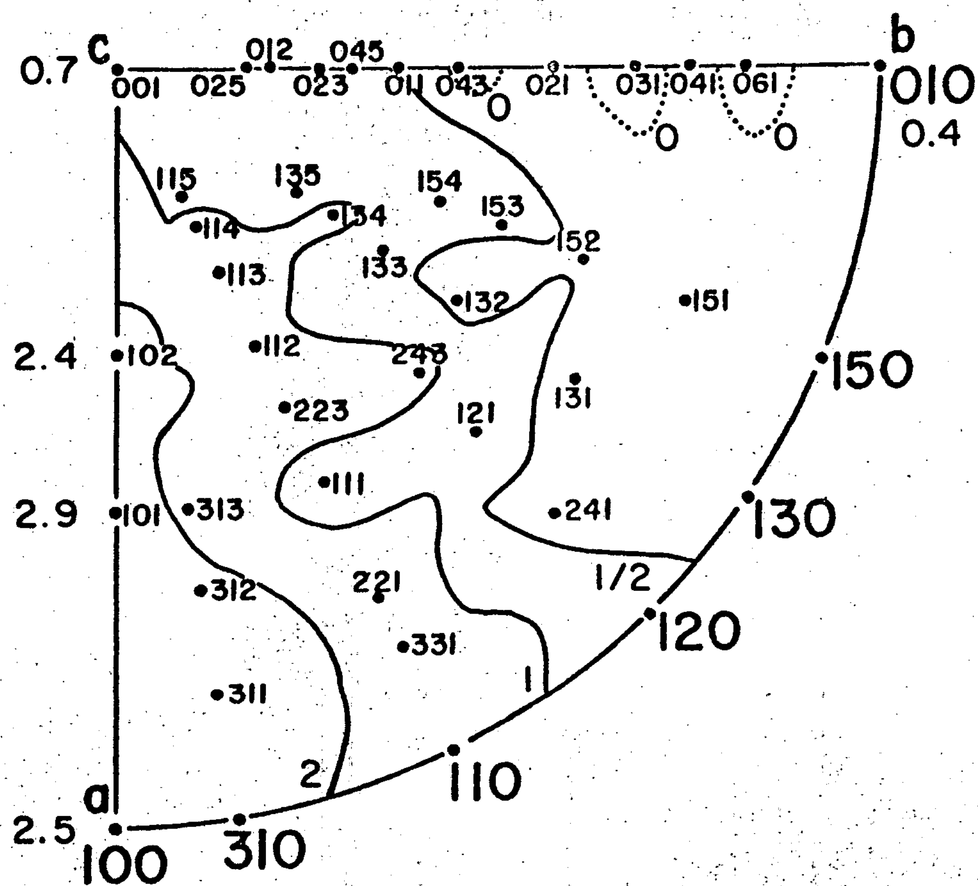


Fig. 14(a) - Crystallographic (inverse) pole figure for the radial direction in a 1-7/32 in. diameter x 8 in. rod (K1), 1.8-3.3 in. from end. Rod was beta quenched by total immersion in cold water.

At 0.010 in. from OD; G_3 = -0.30
Drawing No. RA-1745

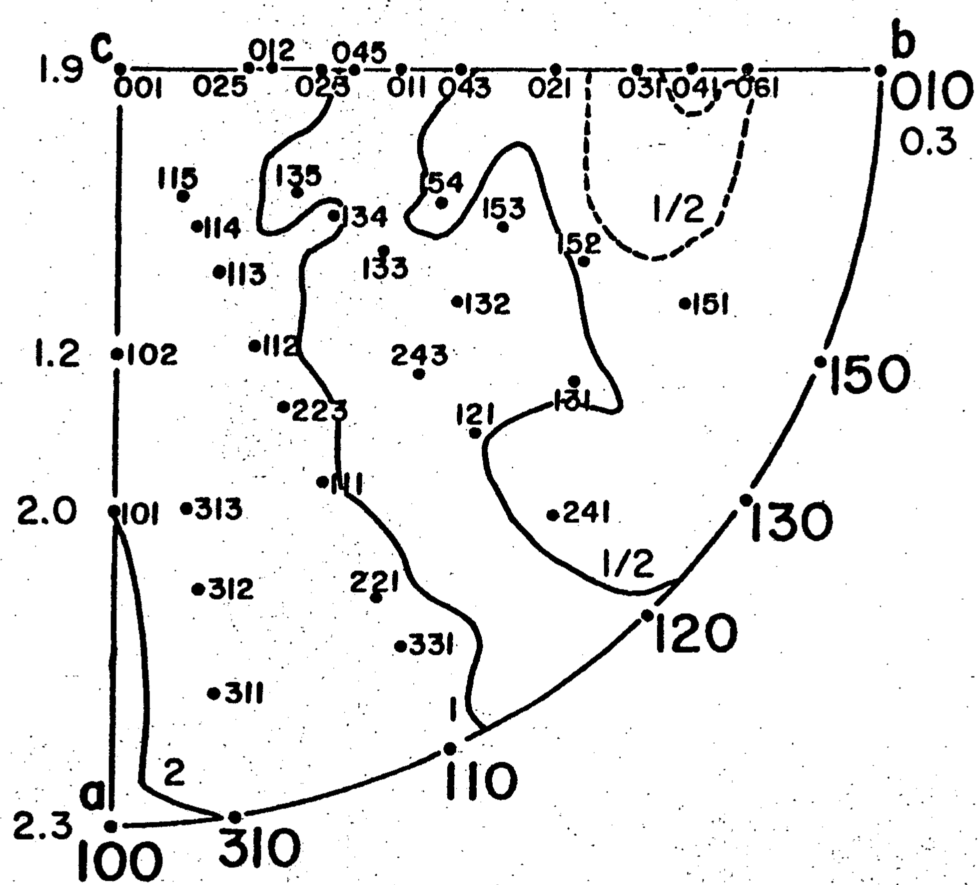


Fig. 14(b) - At 0.070 in. from OD; G_3 = -0.21
Drawing No. RA-1746

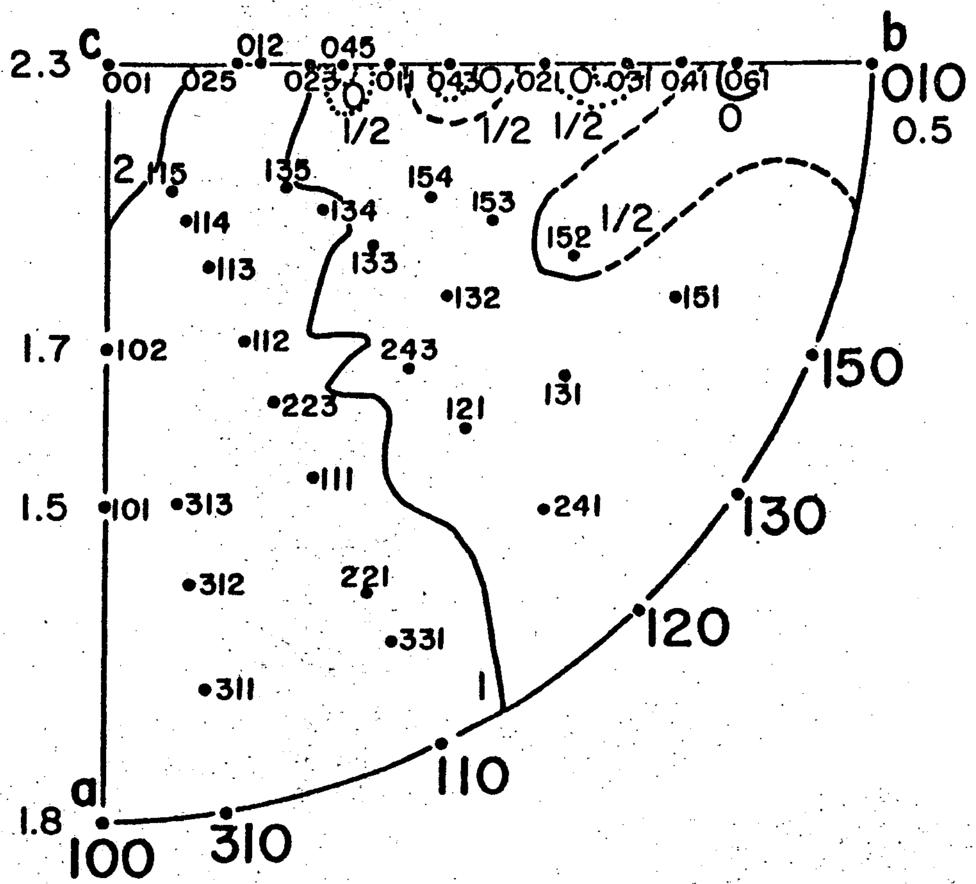


Fig. 14(c) - At 0.108 in. from OD; G_3 = -0.14
Drawing No. RA-1747

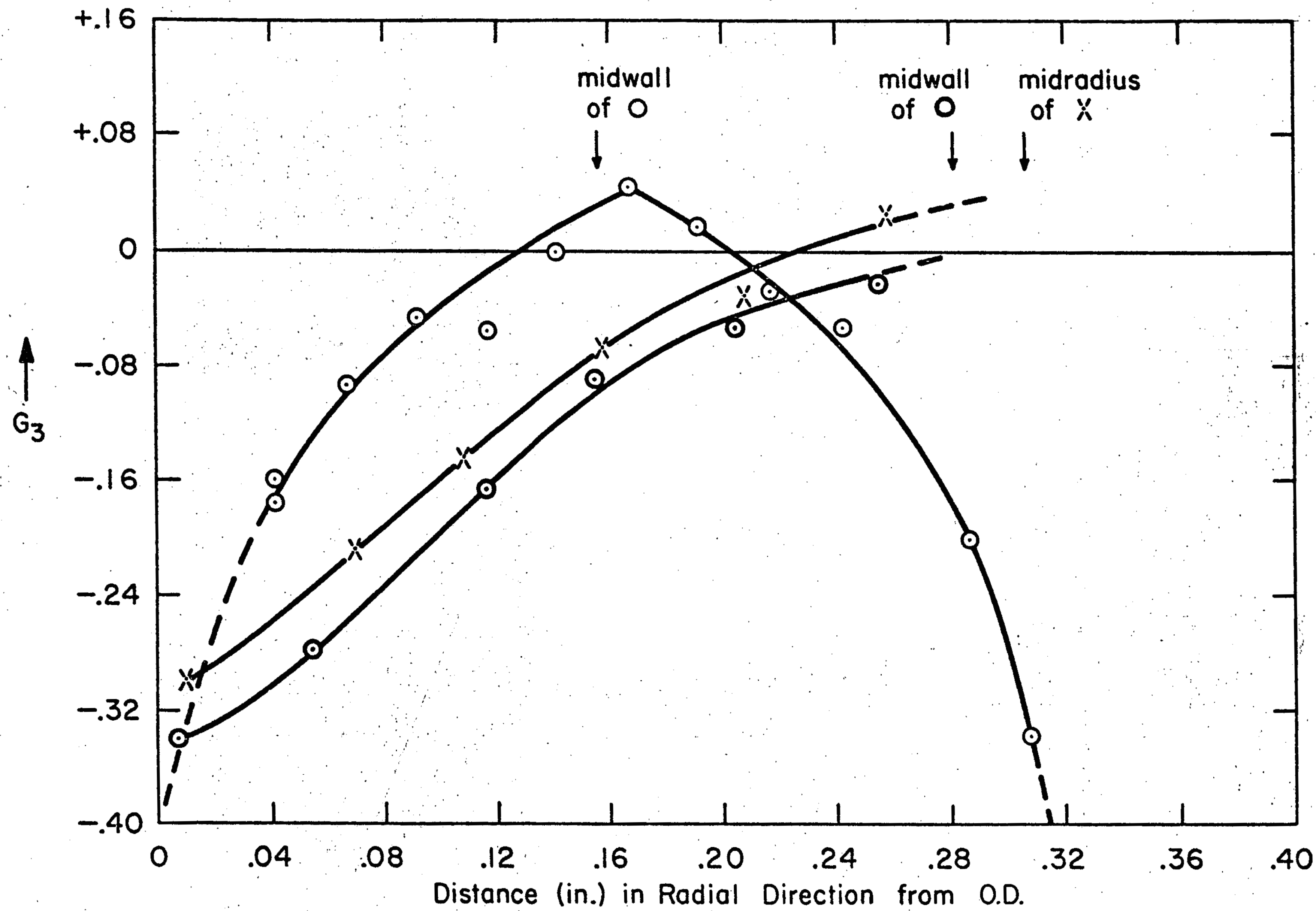


Fig. 15 - Change of radial growth index, G_3, through wall thickness of three 8-in. long cylinders after cold water quenching from molten salt (LH980) at 730°C - 15 min. Drawing No. RA-1750

⊙ 3-1/8 in. OD x 2-1/2 in. ID tube at 3.5 - 4.5 in. from end (M_1)
◎ 1-1/2 in. OD x 3/8-in. ID tube at 1.8 - 3.8 in. from end (H_1)
X 1-7/32 in. diameter rod at 1.8 - 3.3 in. from end (K_1)

VI. REFERENCES

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APPENDIX

A. Calculation of G_3

A sample G_3 calculation sheet is shown in Table A-I. The values of the standard random intensities, I_o, are those recommended by the X-Ray Preferred Orientation Group.*

B. Reproducibility of G_3

A disc beta treated at 1-3/4 minutes at 757°C, quenched into 28°C oil, was x-ray diffraction scanned 11 times and calculated to determine the reproducibility of G_3. The results are shown in Table A-II. The mean G_3 value was found to be -0.086, while the error not exceeded in 99.7 percent of the cases was ± 0.010. However, since the range was found to be ± 0.022, it is fair to say that, for one run, G_3 should be known to within ± 0.02. No attempt was made to determine the effect of the magnitude of G_3 on statistical errors.

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* P. R. Morris, ed. "Papers Presented at the X-ray Preferred Orientation Meeting Held at National Lead Company of Ohio, November 9 and 10, 1959", NLCO-804.

TABLE A-I

X-ray Data Sheet for G_3

hkl	1 s.f.	2 Area	3=I (1) x (2)	4 I_o	5=I/I_o 3/4	7 A_w x100	8=$A_w I/I_o$ 5 x 7	9 C	10=$CA_w I/I_o$ 8 x 9
020				6.34	*	3.18		+1	+
110				72.7		6.34		−.6184	−
021				100		6.08		+.7393	+
002				51.4		3.04		0	0
111				58.3		7.38		−.4876	−
112				48.3		5.22		−.2968	−
130				3.37		3.57		+.3616	+
131				40.0		6.84		+.3255	+
040				6.93					
023				16.8		6.59		+.2395	+
200				8.82		2.34		−1	−
113				11.6		4.77		−.1804	−
132				3.65		5.29		+.2525	+
133				15.3		7.95		+.1844	+
114				10.2		7.49		−.1169	−
150				7.43		3.72		+.7096	+
223				12.2		4.87		−.3843	−
152				12.8		6.89		+.5961	+
312				8.85		8.45		−.8294	−

+

−

Sum(10):

Ref.:
Prior:
Location:
Calc. by:
Remarks:

Sample No.:
Quench:
3X Grain Size:
Date:

$G_3 = \frac{Sum(10)}{Sum(8)} =$

Pole Fig.:

- - - - - - - - - - -

*Av of 020 and 040.

32-2-7/60-200, Revised 7/11/60

TABLE A-II

Reproducibility of Determination of G_3 for Eleven X-ray Diffraction Scans

Run No.	G_3
1	-0.071
2	-0.083
3	-0.090
4	-0.078
5	-0.114
6	-0.086
7	-0.079
8	-0.086
9	-0.073
10	-0.097
11	-0.087

Statistical values are as follows:

Mean, $\bar{G}_3 = \frac{1}{n} \sum_i (G_3)_i = -0.086$

Standard deviation, $\sigma = \left[\sum (G_3 - \bar{G}_3)^2/n\right]^{\frac{1}{2}} = 0.011$

Standard error of the mean, $\sigma_{\bar{G}_3} = \sigma/n^{\frac{1}{2}} = 0.003$

Probable error of the mean:

error not exceeded in 50% of the cases: $0.6745\ \sigma_{\bar{G}_3} = 0.002$

error not exceeded in 99.7% of the cases: $3\ \sigma_{\bar{G}_3} = 0.010$