NMI-2806

Progress on the Study of Beta Treatment of Uranium December 1, 1961 to March 31, 1962

R. B. Russell and A. K. Wolff

April 13, 1962

nuclear metals, inc.
concord, massachusetts

[redacted]

NMI-2806

Progress on the Study of Beta Treatment of Uranium

December 1, 1961 to March 31, 1962

R. B. Russell and A. K. Wolff

April 13, 1962

Contract No. AT(30-1)-1565

Nuclear Metals, Inc.
Concord, Massachusetts

A. R. Kaufmann
Technical Director

DISTRIBUTION

	Copy No.
EXTERNAL:	
Atomic Energy Commission	
F. R. Dowling, Washington, D. C.	1
W. Devine, Jr., Washington, D. C.	2
J. W. Ruch, OROO	3-4
P. J. Hagelston, SROO	5
A. T. Gifford, HOO	6
National Lead Company of Ohio	
F. L. Cuthbert	7
C. E. Polson	8
J. F. MacNeill	9
Mallinckrodt Chemical Works	
J. A. Fellows	10
R. F. Hartmann	11
N. F. Neumann	12
Hanford Atomic Products Operation	
S. H. Bush	13
J. T. Stringer	14-15
J. E. Minor	16
E. I. duPont de Nemours and Company	
Wilmington	
M. H. Wahl	17
Savannah River	
J. W. Croach	18
T. C. Evans	19
V. I. Montenyohl	20
USAEC, New York Patent Group, Upton, L. I.	
H. S. Potter	21
INTERNAL:	
Nuclear Metals, Inc.	
L. R. Aronin	22
A. R. Kaufmann	23
R. B. Russell	24
A. K. Wolff	25
Document Library	26-27

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. INTRODUCTION

This is the seventh progress report of the series beginning with NMI-2800 on the beta treatment of uranium. Progress in the study of the effect of variables is described for the period December 1, 1961, to March 31, 1962. The effects of composition (ingot versus dingot), prior delta condition, geometry, heat treatment and applied stress on cooling rate, grain size and texture are described for rods and tubes.

II. SUMMARY OF PROGRESS SINCE LAST REPORT

A summary of progress in the study of new sizes, their heat treatments, and the extent of their examination is symbolically indicated in Table I. In addition to the basic studies indicated in Table I, supplementary investigations have been made on the effect of stress and free surfaces on the texture distribution.

A. Operation of the Automation Unit

A weekly check, since October, 1961, on the G_3 value of a standard beta-treated sample indicated that the operation of the automation unit was satisfactory. The probable error for 99.7 percent of all determinations (based on a population of only 28) was ± 0.002 for an arithmetic mean G_3 of -0.063.

B. Thermal Data

Cooling rates were obtained for the quenching of 3-inch and 1.5-inch OD tubes with a 0.5-inch ID in different media, including a comparison of the rates between room temperature Houghto K and Poco No. 2 oils. As expected, the quenching rate in Houghto K is slightly greater. Thermal data were also obtained for the helium cooling of 0.7-inch diameter samples which were stressed axially during cooling from the beta phase.

The quenching of 1.5-inch OD by 0.5-inch ID ingot tubes into 400, 300 and 200°C molten salt revealed that the beta-phase cooling rate of a tube quenched in 400°C salt is very nearly the same as that of a Poco No. 2

oil quench. A 200°C salt-quenched tube showed a beta cooling rate roughly comparable to that of a 55°C water quench.

C. Grain Size

The response of ordinary ingot uranium to delta treatment, cited in the last report, was confirmed by the satisfactory grain refinement by delta treatment of an ingot tube which contained coarsened grains caused by gamma treatment.

A study of 1.5-inch OD by 0.5-inch ID as-extruded dingot tube quenched from the beta phase into different media showed that the FEDC grain size of water-quenched tube varied between A-6 and A-7, that oil quenching produced grains between C-4 and C-5, and that air-cooled tube had B-2 to B-3 grain size. No columnar grains were detected in this dingot.

D. Growth Index (G_3)

The suppression of severe radial texture penetration in ingot quenched in Poco No. 2 oil was again found to be lacking in the case of dingot uranium, but both ingot and dingot are similar in exhibiting severe radial texture penetration after water quenches. In general, both ingot and dingot have the same range of radial G_3 values, but with different G_3 distributions. In the case of ingot, the distribution is more sensitive to the quenching medium than is dingot, with the result that in ingot the G_3 curves are more widely separated.

Nearly identical radial G_3 distributions were found in 1.5-inch OD by 0.5-inch ID dingot bars quenched into 55°C Poco No. 2 and Houghto K oils. A 21°C Poco No. 2 oil quench gave moderately less texture penetration than either of the two other oil quenches.

A comparison between dingot tubes of different sizes (3- and 1.5-inch OD's), but the same ID (0.5 inch), showed that the radial G_3 distribution about the midwall was more asymmetrical for the smaller tube.

The relation between radial G_3 and its associated grain size was studied in the case of two beta treated (water-quenched) 1.2-inch diameter ingot bars, one of which had been coarsened (grain size 1.5 to 4) by a delta agglomeration anneal of the delta components before beta treatment.

The delta-treated bar had slightly less radial texture penetration than the bar without this treatment.

The relation between radial merit (indicating the depth of radial texture penetration in beta-treated metal) of ingot and its cooling rate in the beta phase (R_β) has been summarized by a graph. A study of the relation indicates anew that there is a significant, but not necessarily simple, relation between the beta-phase cooling rate and the radial merit of the final alpha. The plot shows that the probability of a shallow radial texture penetration increases greatly at the OD when R_β is in the neighborhood of $60^{o}C/sec$, a rate measured between 0.05 and 0.08 inch below the OD.

The effect of axial stress applied during different stages of cooling from the beta to alpha phase is shown to have results that are not easily interpreted. Tentative data indicate that application of stress has a more pronounced effect during the beta-to-alpha transformation, rather than exclusively in the beta or alpha phases.

The effect of the presence of a "free" surface parallel to and 1/16 inch from the end of a 1.8-inch diameter water-quenched ingot bar was rather startling. The distribution of G_3 parallel to the thermal gradient increased sharply towards zero very near the interior free surface, while the G_3 near the exterior free surface was characteristically a large negative value. It is not known whether the effect is a question of lower stress or thermal insulation at the interior free surface. The study will be continued.

III. PROGRESS DURING PERIOD DECEMBER 1, 1961, THROUGH MARCH 31, 1962

A. Reports

A progress report (NMI-2805) describing work from August 1 to November 30, 1961, was issued on December 29, 1961.

B. Material

No new dingot or ingot was received during this report period. The complete inventory received may be found in Table A-1 of the last report, NMI-2805.

C. Experimental

1. Tactical Summary of Work to Date

A summary of the work to date, listing basic sizes and cooling media used, is presented in Table I.

2. Operation of the Automation Unit

The over-all operation of the automated x-ray diffraction unit is checked weekly by running a standard beta-treated sample (D-60) which had previously been coated with about 1500Å of SiO. This sample has been run at least once a week since October 11, 1961, and a total of 28 G_3 values have been accumulated. The individual values and the cumulative average are shown in Fig. 1. The fluctuations of the cumulative average are expected to diminish steadily and become increasingly insensitive to electronic, electrical and mechanical defects in the equipment; however, the individual deviations of G_3 from the arithmetic mean should be relatively sensitive as indications of trouble in the unit. To date, only nine G_3 values out of 28, or about one-third, have exceeded the standard deviation for a single determination. The statistical data are as follows:

Arithmetic mean: $\bar{G}_3 = \dfrac{\Sigma\, G_3}{N} = -0.0626$

Range: $\pm(G_{3\,max} - G_{3\,min})/2 = \pm 0.008$

Standard deviation for single determination: $\sigma = \left[\dfrac{\Sigma\,(G_3 - \bar{G}_3)^2}{N}\right]^{1/2} = \pm 0.004$

Probable error for 99.7% of determinations: $3\,\sigma_{\bar{G}_3} = \pm 0.002$

3. Thermal Data

Cooling rates were determined for all new quenches of 1.5-inch diameter by 0.5-inch wall ingot tube and 3.0-inch diameter by

TABLE I

Summary of Experimental Progress on Beta Treatment of Uranium Rods and Tubes

Designation	Dimensions (in)		Chemistry	Cooling Medium	Data Obtained			
	OD	Wall			Thermal	Distortion	Grain Size	Growth Index
C	1.8	Rod	Ingot	Air	/	/	/	/
				R.T. Oil	/	/	/X	/X
				R.T. Oil (a)	/	/	/X	/X
				R.T. Oil (b)	/	/	/X	/X
				Warm Water	/	/	/X	/X
				Cold Water	/	/	/X	/
DC	1.8	Rod	Dingot	Air	/	/	/X	/X
				R.T. Oil	/	/	/X	/X
				Warm Water	/	/	/X	/X
				Cold Water	/	/	/X	/X
				R.T. Brine	/	/	/X	/X
K	1.2	Rod	Ingot	Air	/	/	/X	/X
				R.T. Oil	/	/	/X	/X
				Warm Water	/	/	/X	/X
				Cold Water	/	/	/X	/X
E	4.0	0.5	Ingot	Air	/	/	/X	/X
				R.T. Oil	/	/	/X	/X
				Warm Water	/	/	/	/
				Cold Water	/	/	/	/
DG	3.0	0.5	Dingot	Air	*	/	/	*
				R.T. Oil	*	/	/	*
				Warm Water	*	/	/	*
H	1.5	0.5	Ingot	Air	/	/	/	/
				Warm Oil	*	/	/	*
				R.T. Oil	/	/	/	/
				R.T. Oil (a)	*	/	/	/
				R.T. Oil (b)	*	/	/	/
				Warm Water	*	/	/	/
				Cold Water	/	/	/X	/X
				400°C Salt	*	/	/	/
				300°C Salt	*	/	/	/
				200°C Salt	*	/	/	/
DH	1.5	0.5	Dingot	Air	n	*	*	*
				Warm Oil	n	*	*	-
				R.T. Oil (c)	*	*	*	-
				Warm Water	n	*	*	*
				Cold Water	n	*	*	*
				Air		-		

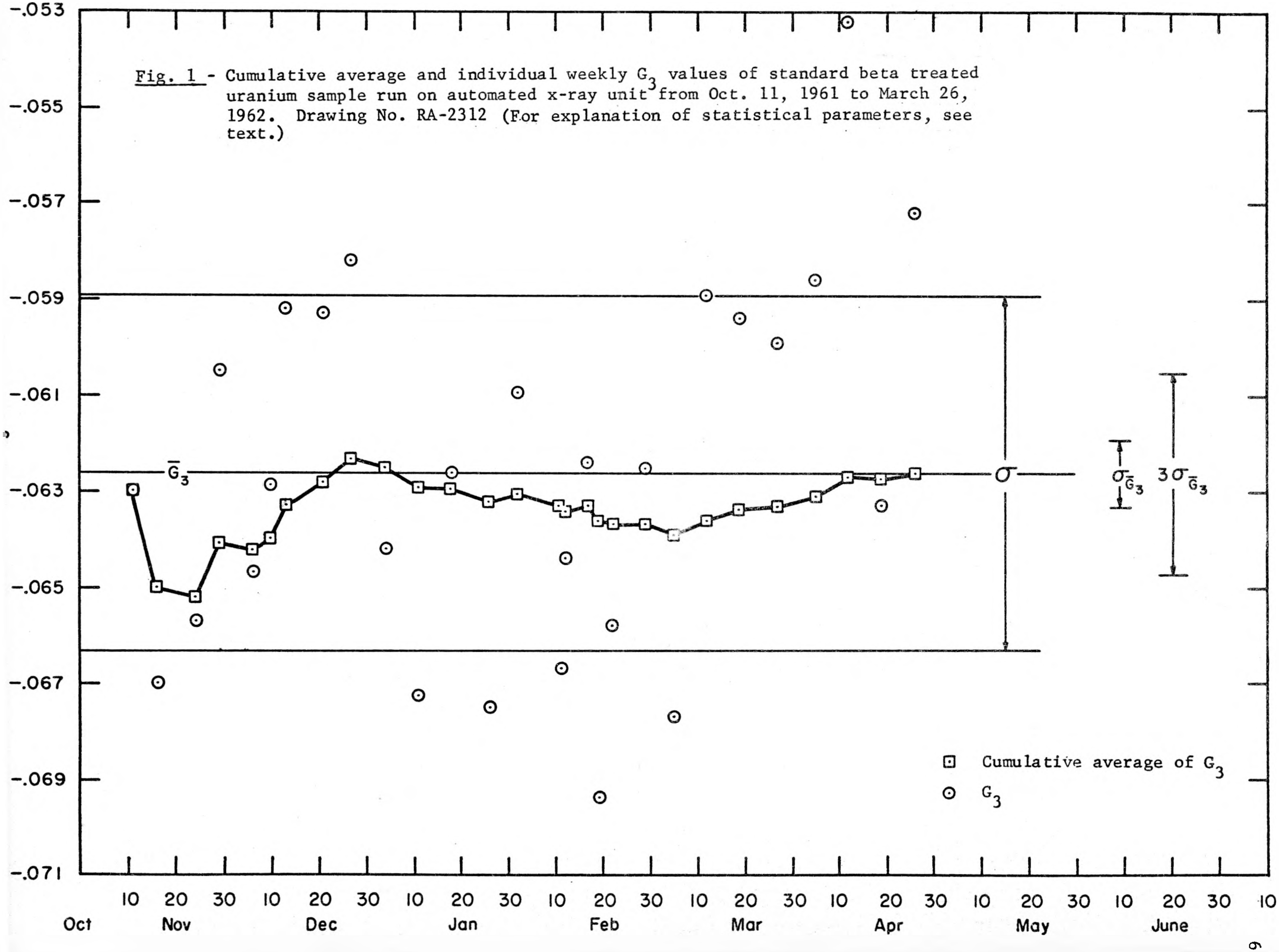


Fig. 1 - Cumulative average and individual weekly G_3 values of standard beta treated uranium sample run on automated x-ray unit from Oct. 11, 1961 to March 26, 1962. Drawing No. RA-2312 (For explanation of statistical parameters, see text.)

0.5-inch wall dingot tube. In view of the similarity of cooling rate between ingot and dingot tube of the same size[1], thermal studies of 1.5-inch diameter by 0.5-inch wall dingot tube were limited to samples quenched in room-temperature Poco No. 2 and Houghto K oils. In addition to these, cooling rates were obtained for a representative ingot tensile sample during helium quenching (sample W-T) for studies which will be outlined in a subsequent section. All cooling rates are presented in Table II. The cooling rates for air, oil, and water are comparable to those previously reported in these media for other samples.

The 1.5-inch diameter by 0.5-inch wall (size H) dingot tube quenched in Houghto K yielded cooling rates slightly faster than those observed for room-temperature Poco No. 2, as was expected. The molten salt quenches of size H ingot showed that a 400°C salt quench is very nearly equivalent (in terms of R_β and R_α) to a quench into room-temperature Poco No.2 oil (the cooling rates for the latter media, obtained earlier in the program, are presented in Table II for comparison). Quenching in 300 and 200°C salts yielded progressively faster cooling rates with the 200°C salt quench being roughly comparable to a 55°C water quench. The significance of these comparisons will be discussed in the section dealing with the effect of quenching medium on growth index.

4. Grain Size[2] and Shape

a. Delta Treatment of Ingot Tube. The sensitivity of one ingot rod (containing 115 ppm Fe and 50 ppm Si) to delta treatment was demonstrated in the last progress report.[3] This sensitivity has recently been re-demonstrated in the case of a zirconium-clad (0.005-inch thick) ingot tube of approximately the same dimensions as size H on which end closures had been made by brazing in the gamma uranium region. The resulting coarse grains at the end of the core were adequately refined (by two or three FEDC grain size numbers) by a later delta treatment.

- - - - - - - - - - -

(1) NMI-2805, p. 39.
(2) Uranium Grain Size Standards Kit, Revision No. 1 (December 7, 1961).
(3) NMI-2805, pp. 20-21.

TABLE II

Cooling Rates of Uranium Rods and Tubes Quenched from the Beta Phase into Various Media

Sample No.[a]	OD (in.)	Wall (in.)	Heat Treatment	OD		Midwall		ID	
				R_β	R_α	R_β	R_α	R_β	R_α
H-5	1.5	0.5	13°C water quench	197	---	109-70	110	72	202
H-6	1.5	0.5	55°C water quench	126	158	72-60	88	52	90
H-9	1.5	0.5	25°C Poco No. 2, 68-sec. air delay	2.2	84	2.0	52-39	2.0	52
H-8	1.5	0.5	25°C Poco No. 2, 36-sec. air delay	2.0	63	2.0	27	2.0	37
H-4	1.5	0.5	25°C Poco No. 2, 1.7-sec. air delay	72	39	54	57	36	46
H-7	1.5	0.5	55°C Poco No. 2 quench	--	--	39	45	44	70
H-10	1.5	0.5	400°C molten salt quench	68	15	35-31	22-18	27	21
H-11	1.5	0.5	300°C molten salt quench	87	--	53-52	37-35	27	40
H-12	1.5	0.5	200°C molten salt quench	125	--	54-47	49-35	49	41
DH-3	1.5	0.5	21°C Poco No. 2 oil quench	37	24	35	29	26	34
DH-4	1.5	0.5	21°C Houghto K oil quench	35	47	27	39	48	56
DG-1	3.0	0.5	55°C water quench	428	--	72	--	121	292
DG-2	3.0	0.5	21°C Poco No. 2 oil quench	49	--	53	73	76	--
DG-3	3.0	0.5	30°C air cool	2.2	1.4	2.4	1.4	2.3	1.5
W-T	0.7	---	Helium quenched after beta treatment under argon	1.2	1.4	1.1[b]	1.2[b]	1.2[c]	1.2[c]

(a) Any letter preceded by "D" is dingot. All others are ingot.
(b) Mid-radius measurement.
(c) Center measurement.

b. <u>Dingot Tubes Quenched in Different Media</u>. Six dingot tubes (size DH: 1.5-inch OD by 0.5-inch ID by 4 inches long) were beta treated and cooled in six different media that produced the following grain sizes:

No.	Cooling Medium	FEDC Grain Size	Depth (in.) from OD	FEDC Grain Size of Remaining Depth
DH-1	30°C air	B-3	0 - 0.25	B-2
DH-5	55°C Poco No. 2	C-4.5	0 - 0.21*	C-4
DH-3	21°C Poco No. 2	C-4.5	0 - 0.21*	C-4
DH-4	21°C Houghto K	C-5	**	C-5
DH-2	55°C water	A-7	0 - 0.29	A-6
DH-6	13°C water	A-6.5	0 - 0.21	A-6

* Indistinct grain size transition zone.

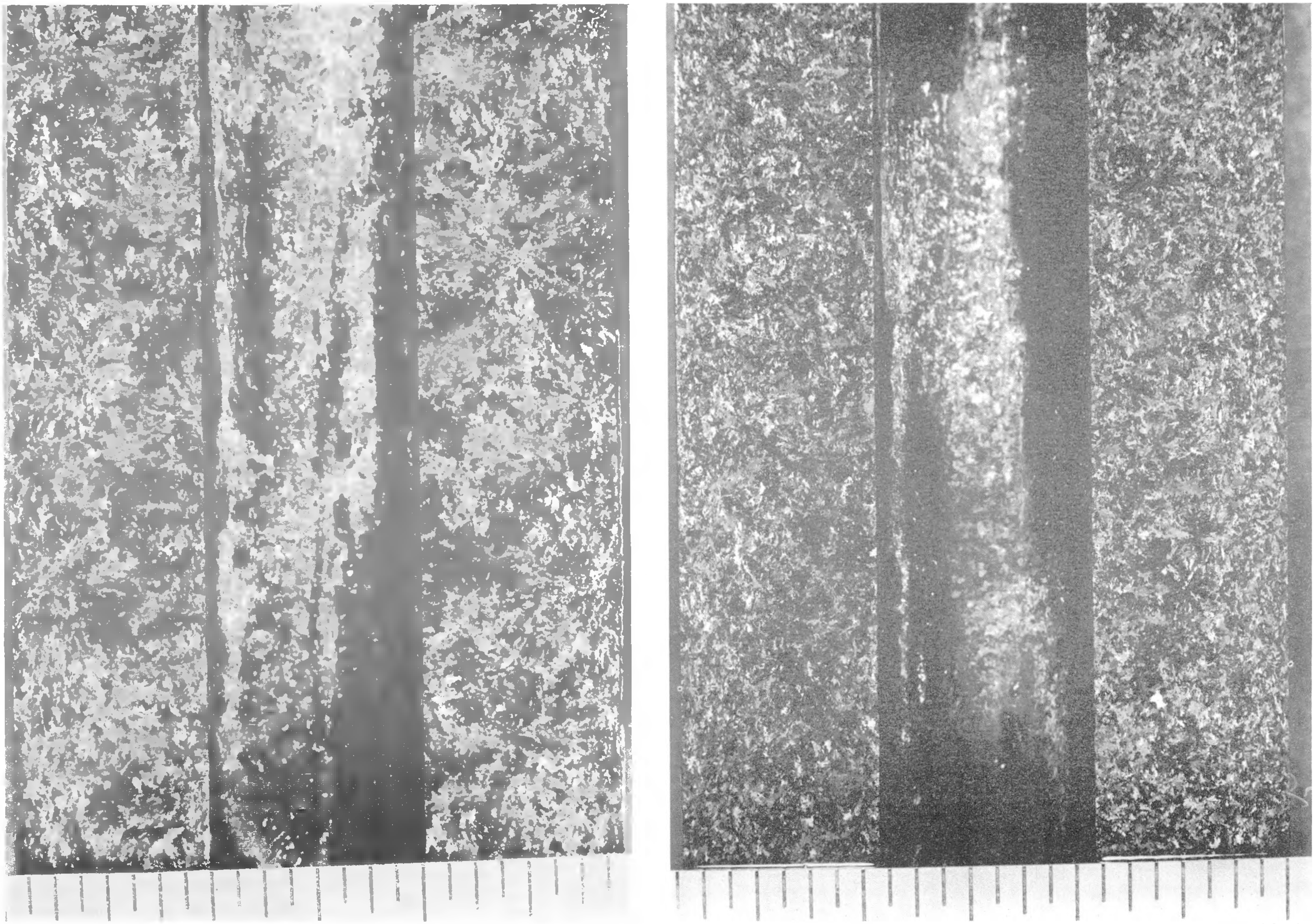
** Zone not visible

Photomacrographs (3X) are shown in Fig. 2. It is noted that the 55 and 13°C water-quenched tubes had the finest grains, the air-cooled tube the coarsest, and the oil-quenched tubes were intermediate in grain size. In the air-cooled and water-quenched tubes, a finer-grained zone extending to about 1/4 inch (about a depth of a radius) from the OD was observed. In the oil-quenched tubes, the zone was more difficult to see. The cause of these zones of differentiation between the fine and coarser grains in dingot is unknown. The zone was not observed in ingot of the same size. The grain size in both dingot and ingot is virtually the same, except for quenches into room-temperature or 55°C Poco No. 2 oil, where ingot, with a grain size of A-5.5to -6,[4] is finer than dingot, with grain size C-4.5 to -5. It should be emphasized that probably neither dingot nor ingot of this size had had equivalent delta histories; accordingly, the difference in grain sizes of the oil-quenched tubes is somewhat less significant.

No columnar grains were noted at OD or ID, but earlier studies have

- - - - - - - - - - - -

(4) NMI-2805, pp. 68-69.

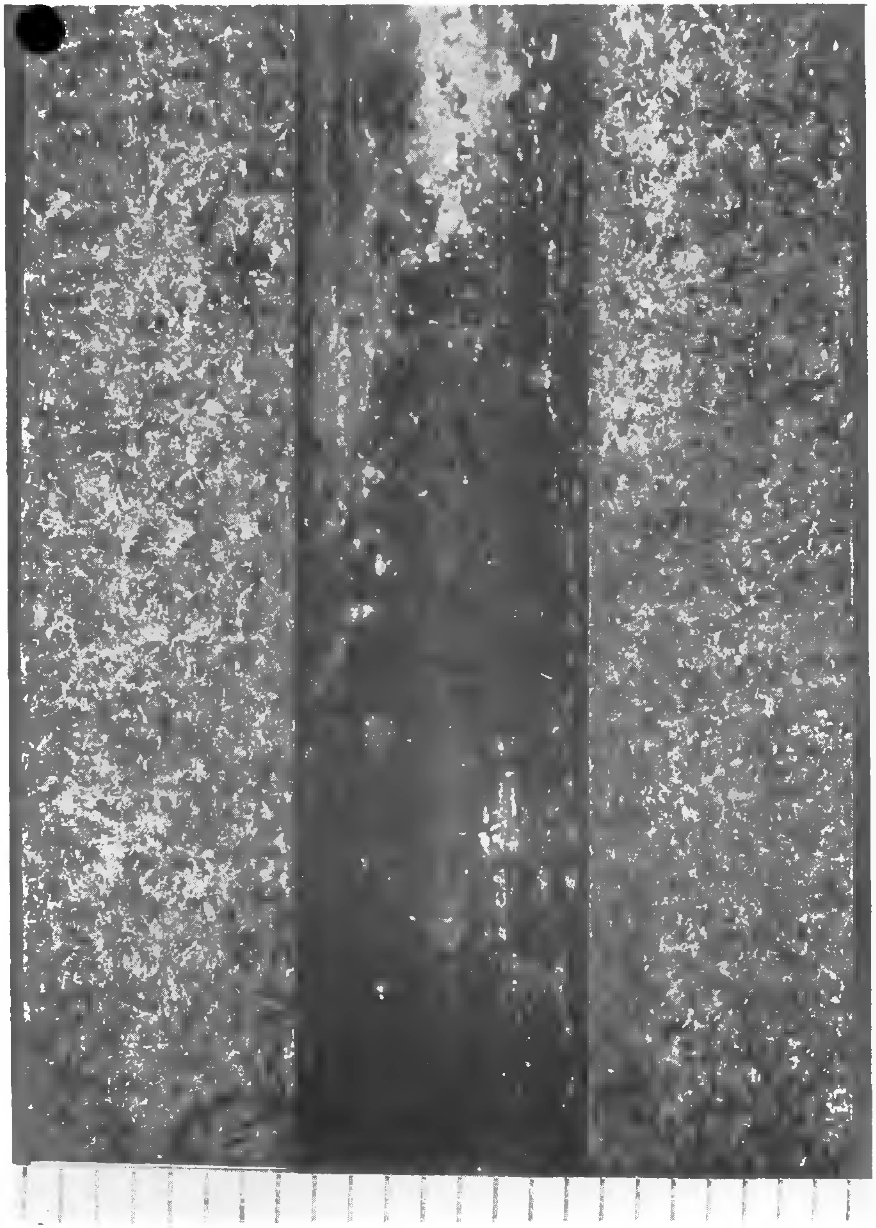


RF-8419 RF-8454

Fig. 2

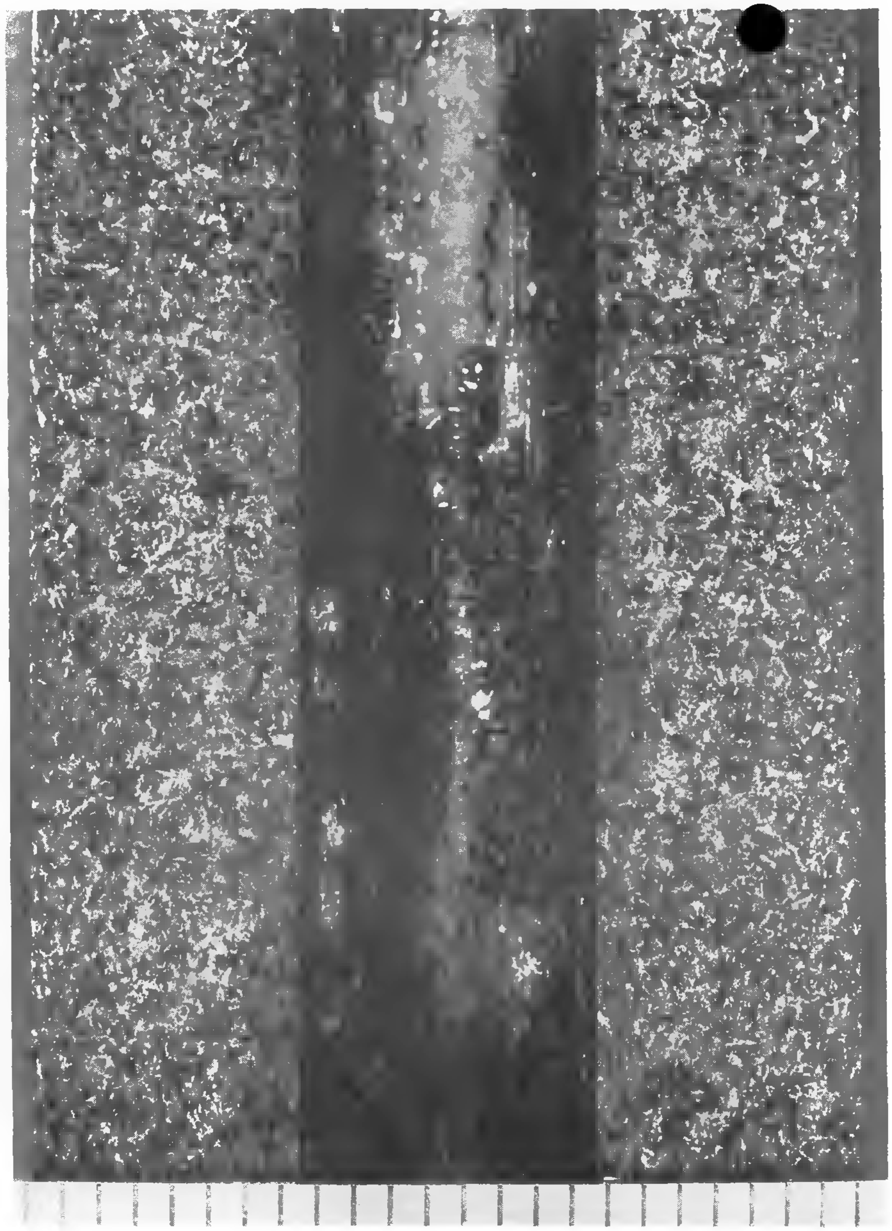
(a) Photomacrograph showing etched hemicylindrical surface of center two inches of 1.5-inch OD x 0.5-inch OD x 4-inch long dingot tube air cooled from beta phase. (DH-1). FEDC (Rev. 1) grain size: B-3 (OD), B-2 (ID). $HCl-HNO_3$ etch. 3X

(b) Same as (a), except quenched in 55^{o}C Poco No. 2 Oil. (DH-5). Grain size: C-4.5 (OD), C-4 (ID).



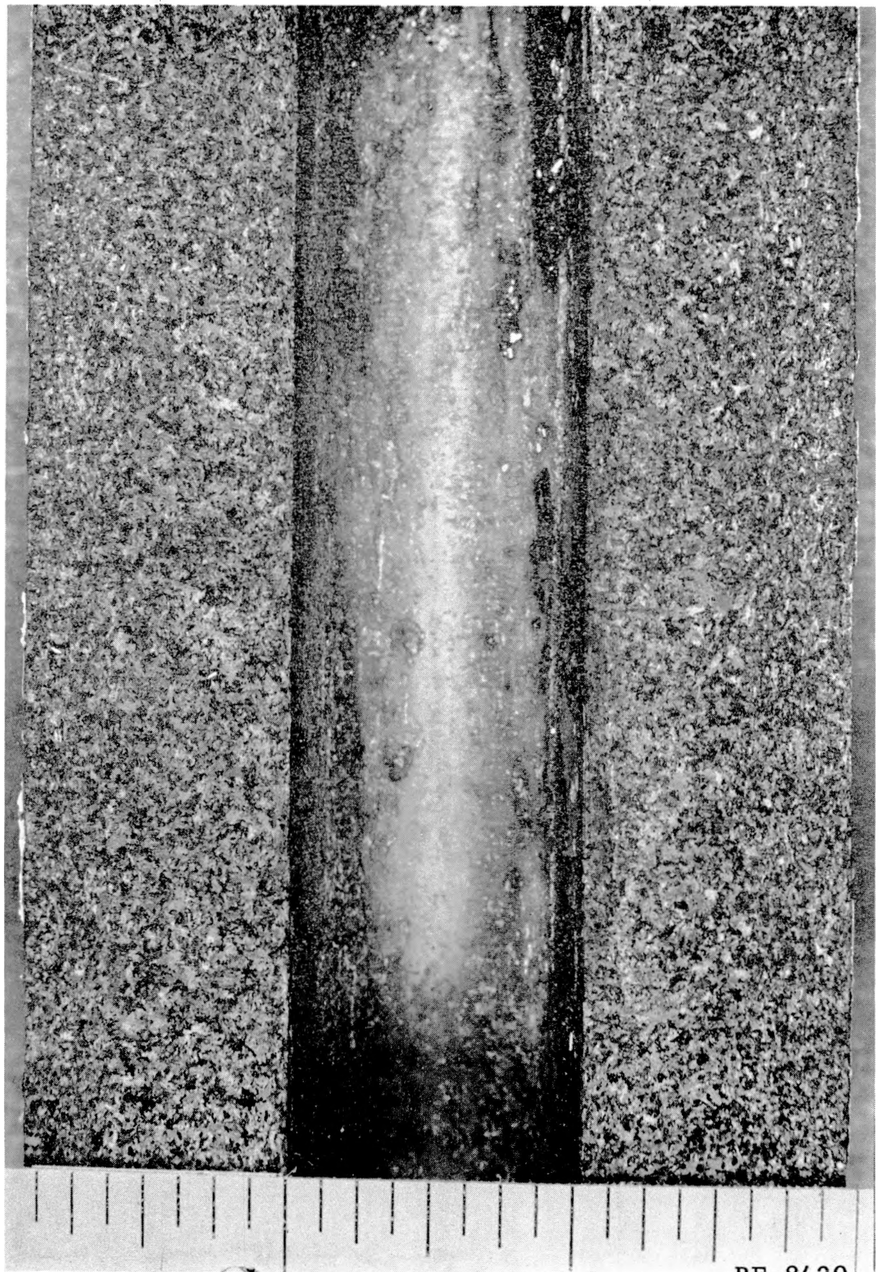
RF-8452

(c) Same as (a), except quenched in 21°C Poco No. 2 Oil. (DH-3). Grain size: C-4.5 (OD), C-4 (ID)

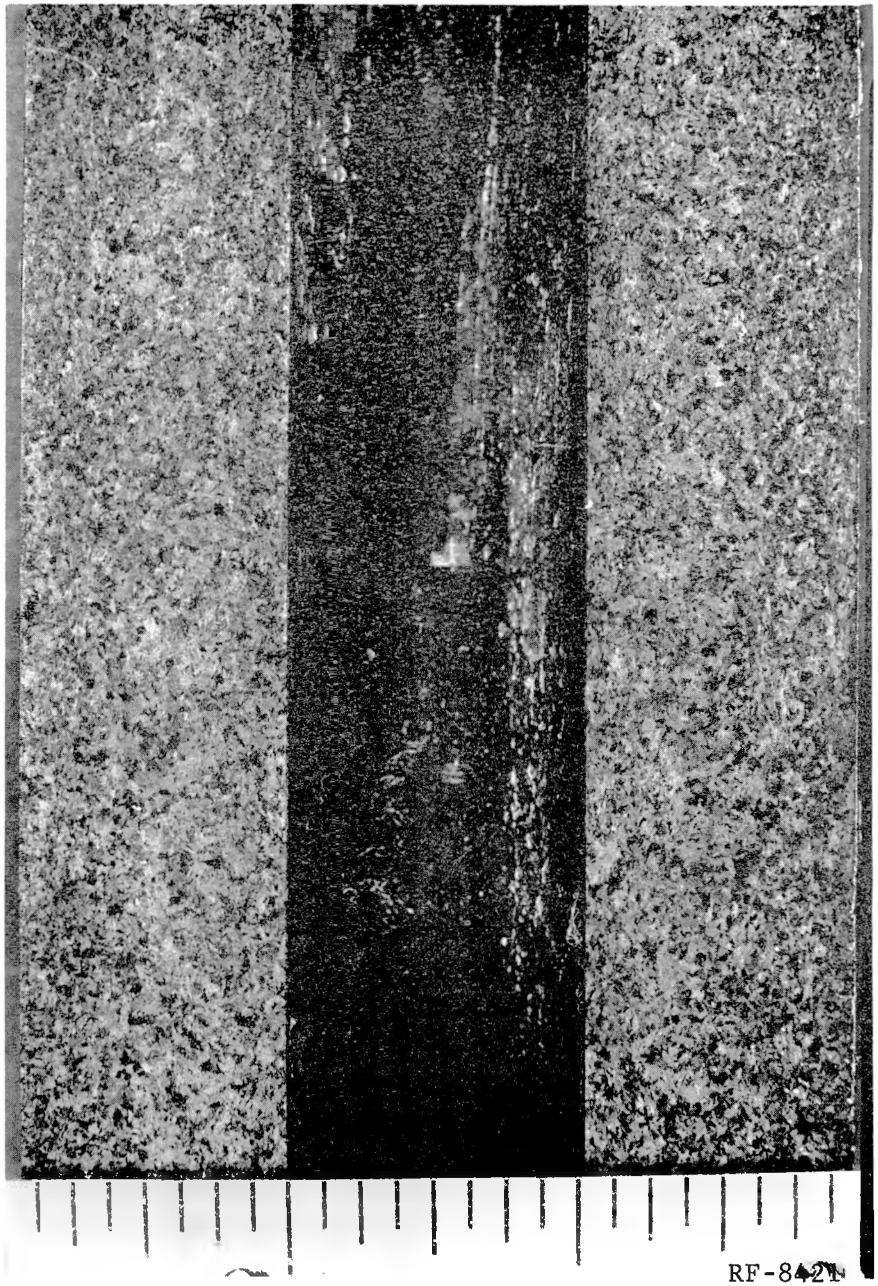


RF-8453

(d) Same as (a), except quenched in 21°C Houghto K Oil. (DH-4). Grain size: C-5 throughout



(e). Same as (a), except quenched in 55°C water. (DH-2). Grain size: A-7 (OD), A-6 (ID)



(f) Same as (a), except quenched in 13°C water. (DH-6). Grain size: A-6.5 (OD), A-6 (ID) shown that some columnarization may occur in dingot[5], and that factors other than composition are undoubtedly effective.[6]

5. Distortion in Dingot (Size DH)

Distortion studies of 1.5-inch OD by 0.5-inch wall dingot tubes are being evaluated. However, a qualitative examination of the samples indicates that the trends observed for this size (i.e. increasing severity of distortion with increasing quench rate) are virtually identical to those noted in previous studies. A quantitative description of the dimensional changes will be completed during the next report period.

6. Growth Index (G_3)

a. Effect of Composition (in one size: 1.5-inch OD by 0.5-inch ID by 4 inches long). The effect of composition may be observed by comparing Figs. 3 and 4 which show the variation of radial G_3 with radial depth in both ingot and dingot of the same size. (Radial merits are compared in Table III). Five cooling media (air, room-temperature and 55°C Poco No. 2 oil, 55°C water and 12°C water) are common to the heat treatment of both compositions. (In addition, results are shown for dingot in Houghto K oil and ingot in hot salt). The following comparisons between compositions may be made:

(1) The radial G_3 distribution caused by the 55 and 12°C water quenches is practically the same in both compositions. The radial merit values (OD) are 0.8 for ingot and 1.0 for dingot.

(2) In the air-cooled samples, the ingot OD region has a slightly more positive G_3 than dingot, but near the ingot ID, G_3 is much more negative than in dingot. The result is that the distribution of radial G_3 is much more symmetrical about the midwall in ingot than dingot.

(3) In the room-temperature and 55°C Poco No. 2 oil-quenched tube, the radial G_3 distributions from OD to midwall are very similar, except that from the OD to a radial depth of about 0.065 inch the ingot has a much shallower negative G_3 penetration

- - - - - - - - - - - -

(5) NMI-2805, p. 22.
(6) NMI-2805, p. 23.

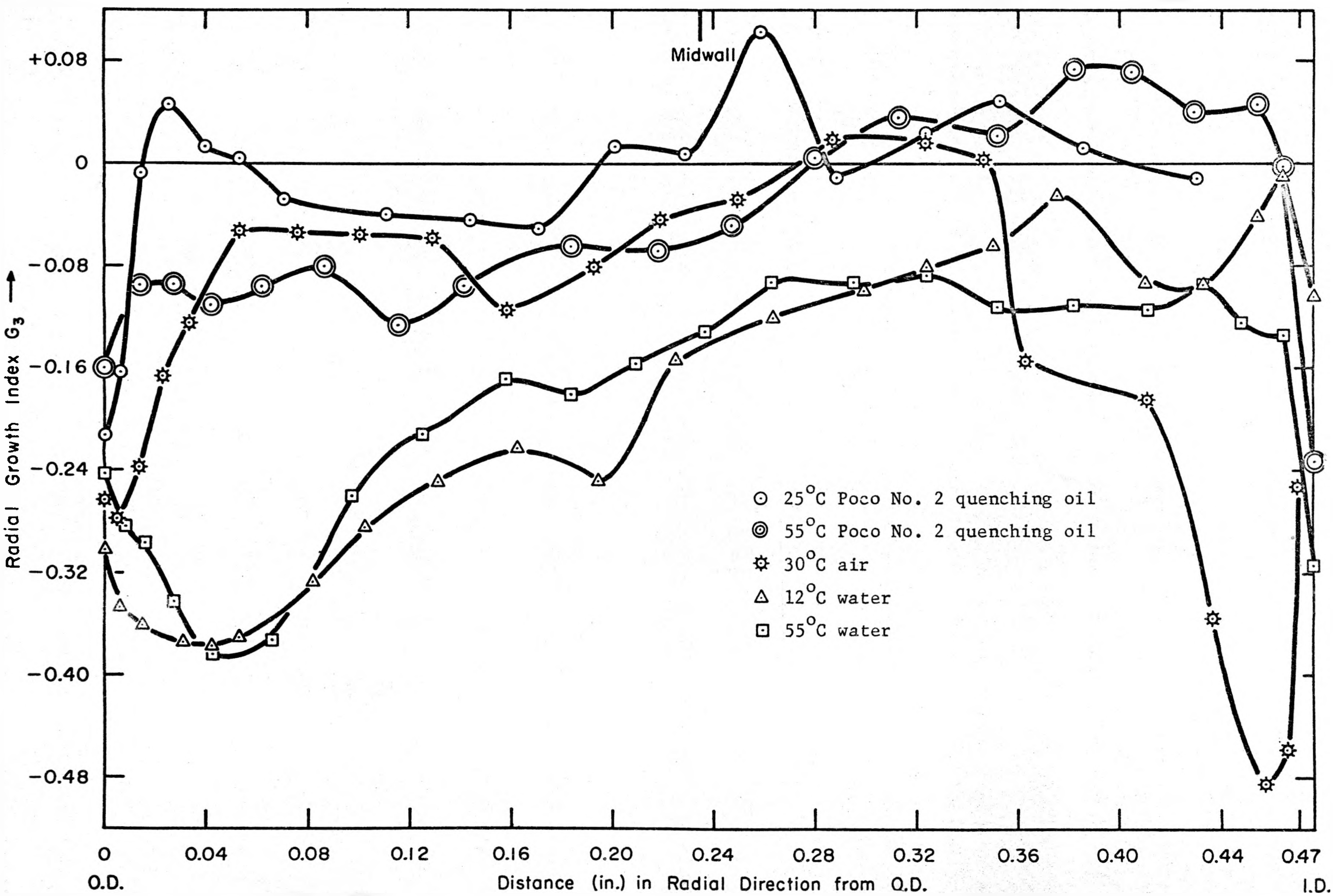


Fig. 3 - Distribution of radial G_3 with radial depth below the OD in 20° sectors from the center 2 inches of quenched 1.5 inch OD x 0.5 ID x 4.0 inch long ingot tube (size H) quenched in different media from the beta phase. Drawing No. RA-2192.

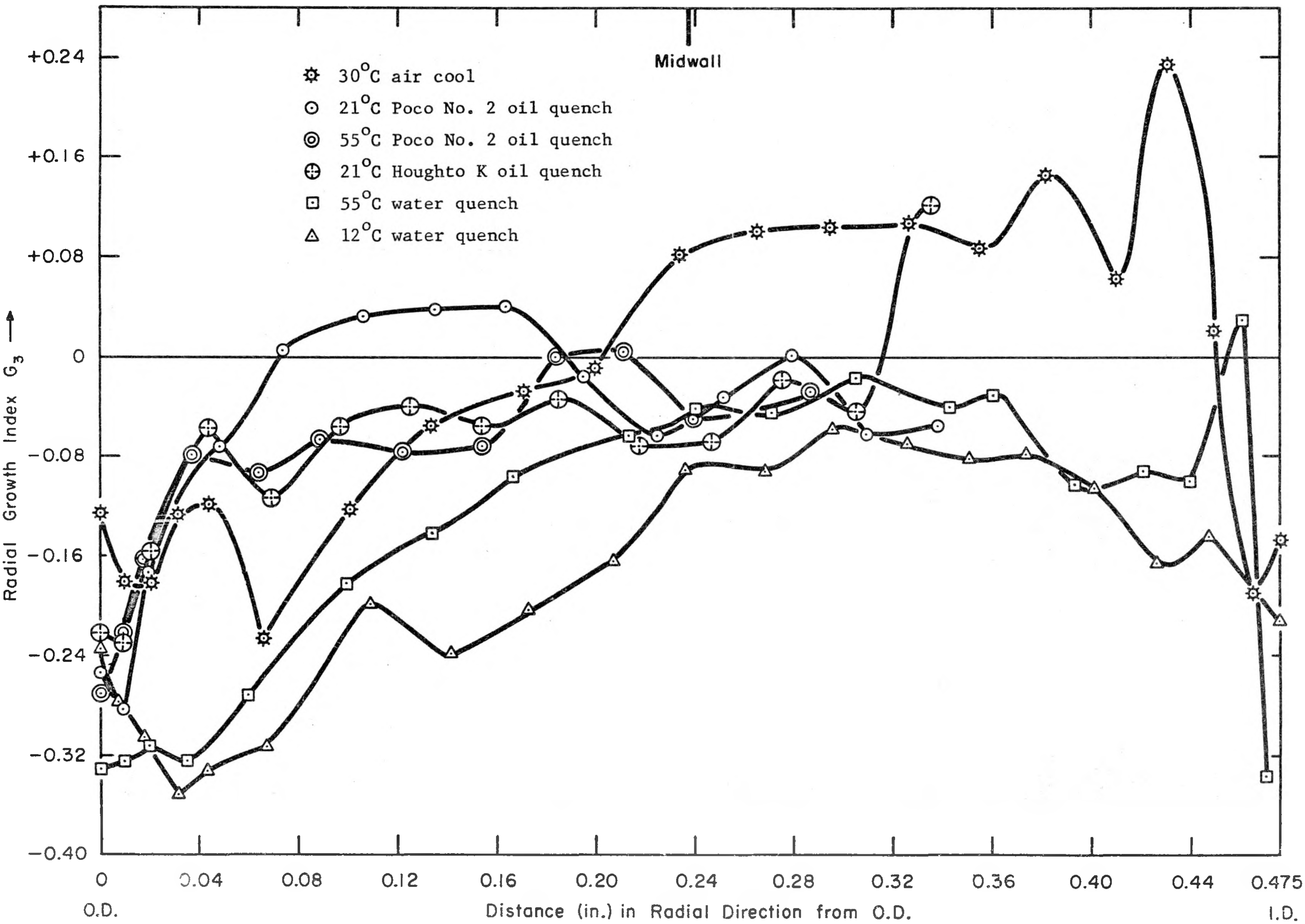


Fig. 4 - Same as Fig. 3, except for dingot tube. Drawing No. RA-2311.

TABLE III

Radial Merits of Dingot and Ingot Tube of Same Size (1.5-inch OD by 0.5-inch ID) Beta Treated at 725°C and Cooled in Different Media

Cooling Medium	Dingot	Ingot
30°C air	3.1	6.0
Air delay 68 sec., 25°C Poco No. 2 oil	---	4.5
Air delay 36 sec., 25°C Poco No. 2 oil	---	7.6
25°C Poco No. 2 oil	6.4	26
55°C Poco No. 2 oil	7-3	20
21°C Houghto K oil	7-3	---
400°C salt - 10 min., water	---	240-120
300°C salt - 10 min., water	---	7.4
200°C salt - 10 min., water	---	3.0
55°C water	1.4	0.9
12°C water	1.0	0.8

than dingot. This difference has been found to be characteristic of all sizes of ingot and dingot previously studied. The radial merit values for the OD region are 26 to 20 for ingot and 7 to 3 for dingot. (G_3 data for the ID region in dingot are incomplete.)

(4) Except for the air-cooled tubes, the over-all range of radial G_3 is about the same for ingot as dingot. The principal distinction between ingot and dingot in this size (H) is that the difference in radial texture penetration between the more slowly cooled ingot (air and oil cooling) on the one hand, and the rapidly cooled ingot (cold and hot water) on the other, is appreciably greater than for the same range of cooling rates in dingot. In other words, ingot beta-treated texture seems more sensitive to differences in cooling media than dingot.

b. Effect of Prior Delta Treatment. It has been shown that ingot containing only 115 ppm Fe and 50 ppm Si will definitely respond to delta treatment.[7] In order to find whether delta treatment affected the radial distribution of radial G_3, two 1.2-inch diameter ingot bars were examined. Both bars were cut from the same as-rolled length, but one bar had received a 645°C, 8 hour delta agglomeration before beta treatment, while the other had been simply beta treated. The delta-treated bar had a 1.5 to 4 FEDC grain size, while the bar having had the simple beta treatment had a 6 to 7 grain size. The radial G_3 distribution to a depth of about r/2 may be seen in Fig. 5. The agglomerated bar shows slightly less radial texture penetration (radial merit 3.2) than the ordinary beta-treated bar (radial merit 2.5).

It is interesting to note that, although the depth of OD columnarization previously reported[7] for these two bars differs considerably, this difference is not reflected in shape of either G_3 distribution curve, since the two curves are practically parallel. This confirms earlier observations that columnarization is not a reliable clue to the depth of texture penetration.

- - - - - - - - - - - -

(7) NMI-2805, pp. 20, 33.

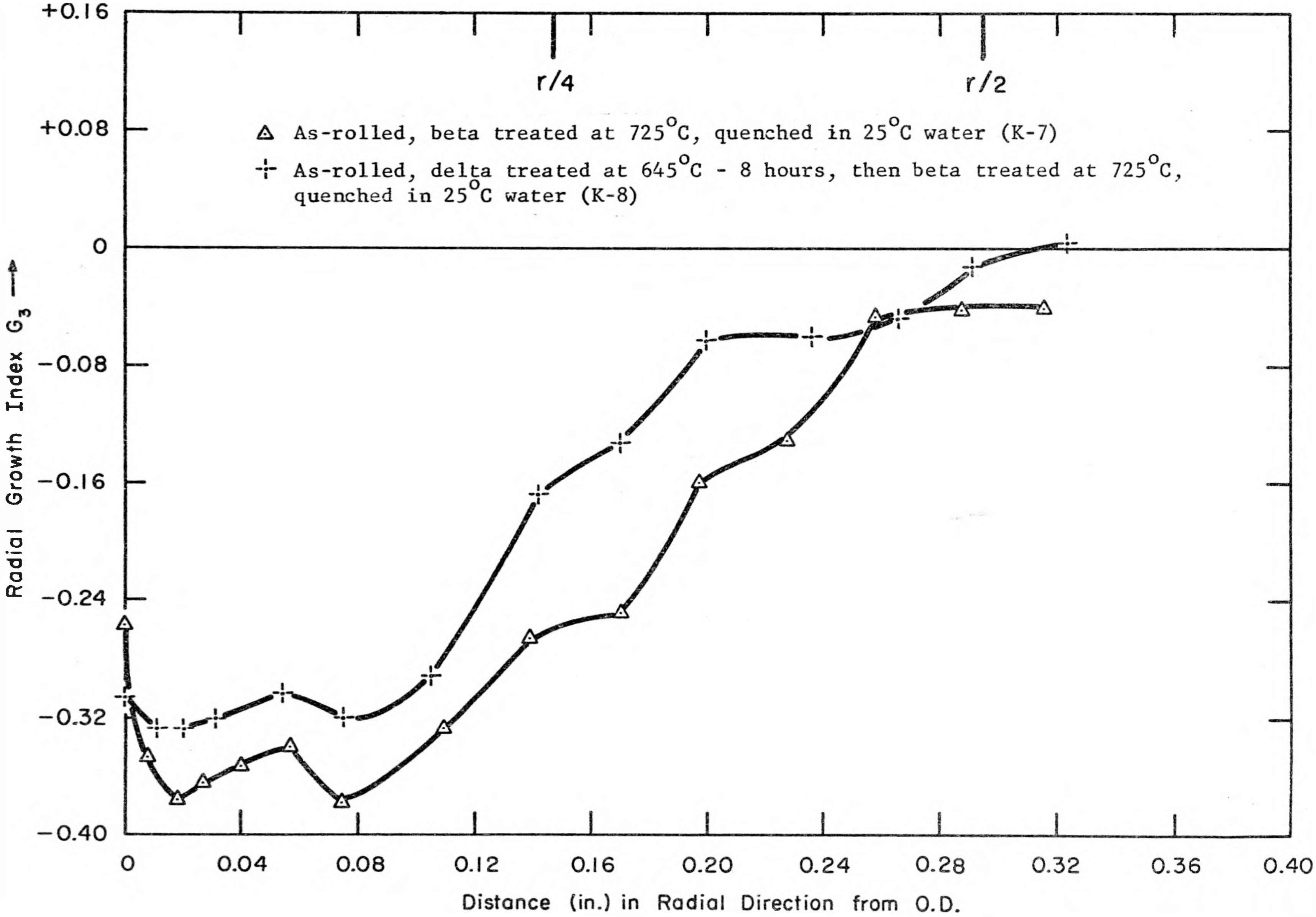


Fig. 5 - Distribution of radial G_3 with radial depth below the OD in 20° sectors from the center 2 inches of 1.2-inch diameter x 4-inch long ingot rod (size K) as affected by delta-treated grain size. Drawing No. RA-2308.

c. Effect of Geometry (comparison of 0.5-inch walled dingot tubes with 3-inch and 1.5-inch OD). The effect of composition on radial texture distribution has been described for dingot and ingot 1.5-inch OD by 0.5-inch ID tube (0.475-inch wall) (size H). The effect of size may be shown for dingot tube with nearly the same wall thickness, but twice the OD and four times the ID in the case of a 3.0-inch OD by 2.0-inch ID tube (0.498-inch wall) of the same 4.0-inch length (size DG). The radial G_3 distribution is shown in Fig. 6 for three cooling media: 30°C air, 21°C Poco No. 2 oil, and 55°C water. The values of radial merit for this larger tube are as follows:

Dingot No.	Cooling Medium	Radial Merit
DG-3	30°C air	Very low (G_3 never > -0.10)
DG-2	21°C Poco No. 2	2.5
DG-1	55°C water	1.2

The radial G_3 distribution in the smaller dingot tube has already been given in Fig. 4. Comparison of the two plots shows the following differences for dingot with respect to cooling in these three media:

(1) The total range of texture in both sizes is nearly the same, but the OD and ID surface G_3's are more negative in the larger tube.

(2) The G_3 distribution in the larger dingot tube is more symmetrical about the midwall than in the smaller tube, for all coolings.

(3) In the air-cooled larger dingot tube, the radial G_3 is always negative (range -0.11 to -0.38) even at midwall, so that no radial merit rating can be given. On the other hand, in the smaller air-cooled tube, the radial G_3 varies from -0.23 to +0.23, being essentially negative in the OD region and positive in the ID region. In the OD region, the radial merit is about 3.

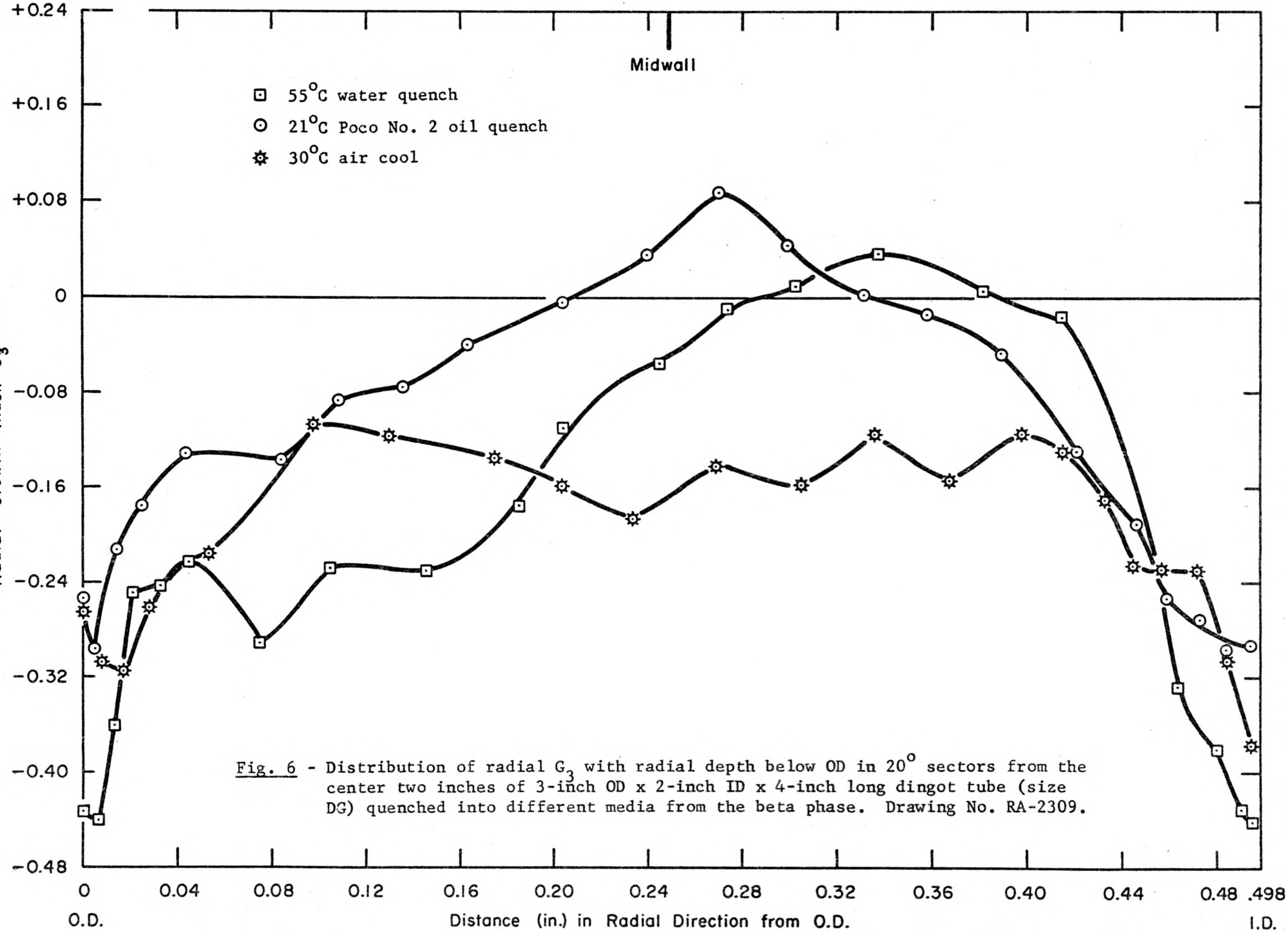


Fig. 6 - Distribution of radial G_3 with radial depth below OD in 20° sectors from the center two inches of 3-inch OD x 2-inch ID x 4-inch long dingot tube (size DG) quenched into different media from the beta phase. Drawing No. RA-2309.

(4) In the case of the room-temperature Poco No. 2 quench, there is more texture (negative radial G_3) penetration in the larger tube, as evidenced by the radial merit of 2.5 as compared to a merit of 6.4 in the smaller dingot tube.

(5) The 55°C water quench produces in both sizes nearly equal radial merits, 1.2 in the larger and 1.4 in the smaller, although the OD and ID surface G_3's are more negative and the range greater in the larger tube. The G_3 range in the larger and smaller tubes is -0.44 to +0.04 and -0.35 to -0.06, respectively.

From the foregoing observations, it would appear that the restricting effect of the smaller hole has been important in reducing negative radial G_3 penetration at the ID. This effect is most startling in the instance of air cooling, which produces in the smaller tube not only less negative radial G_3, but also extreme asymmetry in G_3 distribution. (When the G_3 data have been computed for 21 and 55°C Poco No. 2 and 21°C Houghto K oil quenches in the smaller tube, it will be interesting to see whether these oil quenches produce the same G_3 asymmetry as the air cool does.)

In order to improve understanding of the effect of cooling rates of dingot, an auxiliary experiment similar to that already performed for ingot[8] is being conducted on the effect of isothermal transformation temperature on the surface G_3 of 0.030-inch thick discs. Results will be available in the near future.

d. The Question of an Optimum Cooling Rate in Ingot and Dingot. It is apparent on the basis of previous studies that pronounced improvements in the growth index at the surface of ingot tubes and rods can be obtained by quenching in room-temperature Poco No. 2 quenching oil. It has been further demonstrated that a different medium that induces the same cooling rate (in this case 400°C molten salt) will result in nearly identical improvements in growth index. Another study has shown that the surface texture of thin ingot discs goes through a minimum with varying isothermal trans-

- - - - - - - - - - -

(8) NMI-2804, p. 30.

formation temperature. It may therefore be presumed that an optimum cooling rate and/or transformation temperature exists which yields the minimum radial texture penetration. Radial merit[9] has been used to evaluate quantitatively the ability of a given quench medium to limit undesirable radial texture. Radial merit of ingot is shown as a function of cooling rate in the beta phase in Fig. 7. It is seen that radial merit is maximized for intermediate R_β in continuous cooling through complete transformation. However, it should not be assumed that R_β is a critical parameter to attain maximum merit, since R_β is nearly proportional to R_α and the plot would have shown a maximum radial merit at some intermediate R_α. The scatter of points in the plot is due probably to R_β measurements taken at 0.05- to 0.08-inch depths below the surface in samples of much different geometries.

The upper pair of arrows in Fig. 7 refer to 1.8-inch diameter bars; the lower pair to 1.5-inch OD by 0.5-inch ID tubes. Both sets were air delayed 30 or 60 seconds before an oil quench. The four points shown by the arrows are anomalous for two reasons. In a plot of merit vs R_β the upper pair are anomalously high compared with the trend of the other points but, in a plot vs R_α, the lower pair would be too low. Secondly, it is anomalous that comparable air delays should reveal such large differences in radial merit between two different sizes, since previous work[9] showed that, in any case, in continuous cooling, radial merit is not so sensitive to size. The air delay results indicate that the determination of texture after beta treatment cannot be readily rationalized in terms of a single variable. On the other hand, these air delay studies lend further weight to the evidence that the transformation range is that portion of the cooling curve where the G_3 gradient is determined, and that the exact conditions during transformation are extremely critical.

From the limited data available for dingot (see Table III), it does not appear that radial merit changes significantly with quench rate. If an optimum cooling rate exists for dingot, it has apparently been shifted for the higher purity dingot to values that have not yet been obtained with

- - - - - - - - - - -

(9) NMI-2804, p. 28.

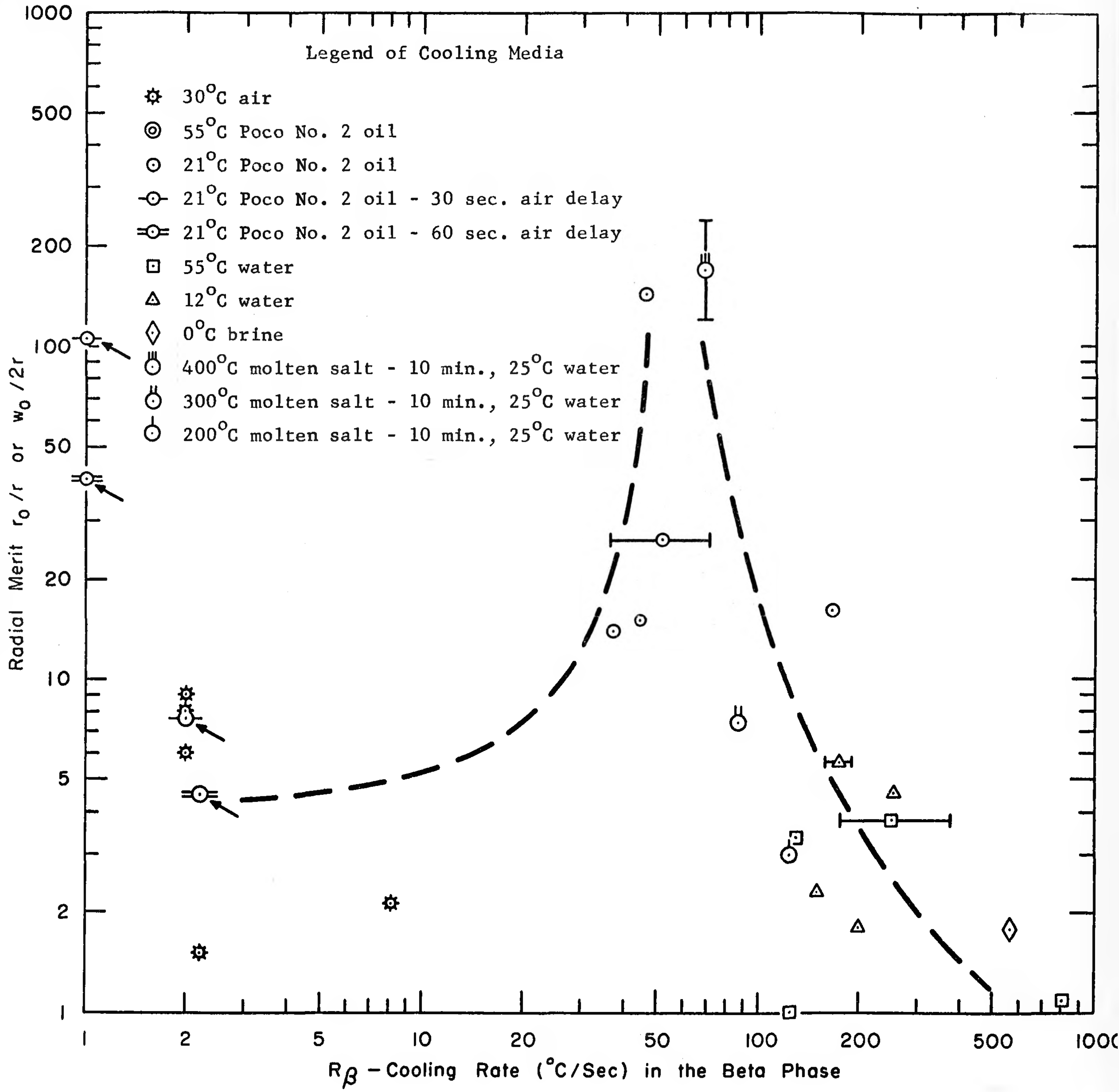


Fig. 7 - Relation between cooling rate in beta phase (R_β) and radial merit in ingot rod and tube quenched in different media (cooling rate at 0.05 to 0.08 inch below OD). Arrows refer to points of special interest discussed in the text. Drawing No. RA-2322.

the media presently employed. This suggests a set of critical transformation conditions, perhaps a specific region of the time-temperature-transformation curve. It may be possible that what is needed is the attainment of a cooling rate sufficiently slow to produce a diffusion-controlled transformation, yet fast enough to induce a critical thermal or geometrical stress during transformation.

Whatever the case, the marked differences in behavior between dingot and ingot emphasize the need for a detailed study of the effect of composition, particularly as it relates to induced texture and transformation kinetics.

e. Comparison Between Effects of Quenching Dingot Tube in Poco No. 2 and Houghto K. Figure 4 includes the three radial G_3 distribution curves of dingot quenched in 21°C Poco No. 2, 21°C Houghto K, and 55°C Poco No. 2. Although Houghto K evidently permits a slightly more rapid cooling rate than Poco No. 2, the G_3 distribution curves associated with 55°C Poco No. 2 and 21°C Houghto K are almost identical for the depths investigated to date (all three oil curves are incomplete). The texture penetration induced by 21°C Poco No. 2 is somewhat less than the penetration found after quenching in hot Poco No. 2 or room-temperature Houghto K, although the radial merit figures do not reflect this difference. A range of 7 to 3 was given for the latter quenches because of the decrease in both G_3 distribution curves after an initial increase past a G_3 of -0.10 near the OD. The comparative effects of Houghto K and Poco No. 2 in ingot will be reported in the next progress report.

f. Completion of Air Delay Experiment in 1.5-inch OD by 0.5-inch ID Ingot Tube (Size H). The study of the effect of air delays of 1.7, 36 and 68 seconds before quenching from the beta into Poco No. 2 oil was nearly completed for the last report.[10] The data for the 36-second air delay are now complete for the ID region, and the final results are given in Fig. 8. The 36-second air-delayed tube, from an OD depth of about 0.08 inch through to the ID, displays a radial texture penetration almost identical with that of the 1.7-second delay. It should be

- - - - - - - - - - - -

(10) NMI-2805, p. 27.

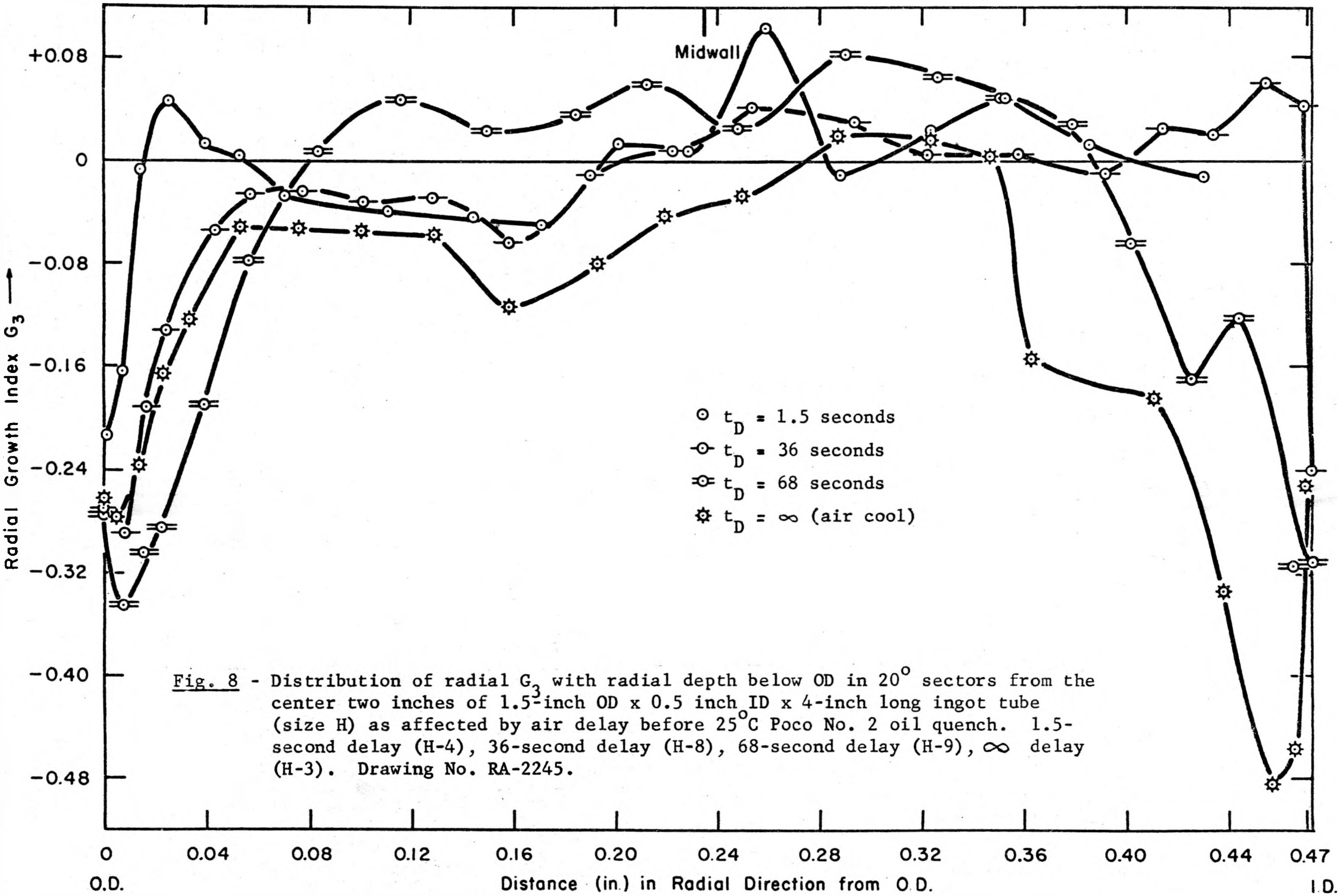


Fig. 8 - Distribution of radial G_3 with radial depth below OD in 20° sectors from the center two inches of 1.5-inch OD x 0.5 inch ID x 4-inch long ingot tube (size H) as affected by air delay before 25°C Poco No. 2 oil quench. 1.5-second delay (H-4), 36-second delay (H-8), 68-second delay (H-9), ∞ delay (H-3). Drawing No. RA-2245.

remembered that the 36- and 68-second air delays were for the start and end of beta-alpha transformation, respectively, 0.08 inch below the OD. It is perhaps not surprising, therefore, that the rapid oil quench and the 36-second delay before oil quenching should produce in the body of the tube, but not at the surface, similar texture penetrations, and that the 68-second air delay should produce texture penetration intermediate between the short delays and the complete air cool.

g. Radial Merit for Tubes and Rods. Figures 9 and 10 illustrate the cumulative radial merit values of ingot and dingot to date. It is clear that the radial merit of ingot is somewhat higher than dingot for the types of coolings and sizes studied, particularly for oil-quenched samples.

h. The Effect of Externally Applied Stress on Ingot. The effect on radial G_3 of bending during cooling from the beta phase was described previously.[11] The pronounced increase noted in OD radial G_3 indicated need for further careful study on the effect of applied stress during beta treatment. Tensile samples of a 0.7-inch gage diameter were machined from 1.2-inch diameter ingot rod. The design is shown in Fig. 11. The gage diameter was small enough to meet tensile design requirements, yet large enough to serve as radial G_3 x-ray sectors. A nichrome-wound three-zone tube furnace (with temperatures controlled to $\pm 10^{\circ}C$ over the length of the sample) was set up on a Tinius-Olsen mechanical drive tensile machine. An argon atmosphere was successfully used to minimize oxidation. In a specially designed heavy-duty tensile fixture, the samples were held for 10 to 15 minutes at $725 \pm 10^{\circ}C$, then quenched in helium, which yielded beta-phase cooling rates comparable to those obtained for an air cool (1 to $2^{\circ}C$/sec). Strain up to 5 percent was applied and held during various stages of the cooling.

Prior to these tests, the cooling curves were carefully determined by means of a similar (except for thermowells) dummy sample (W-T). The dummy sample was heat treated as outlined above, but without applied stress

- - - - - - - - - - - -

(11) NMI-2805, p. 29.

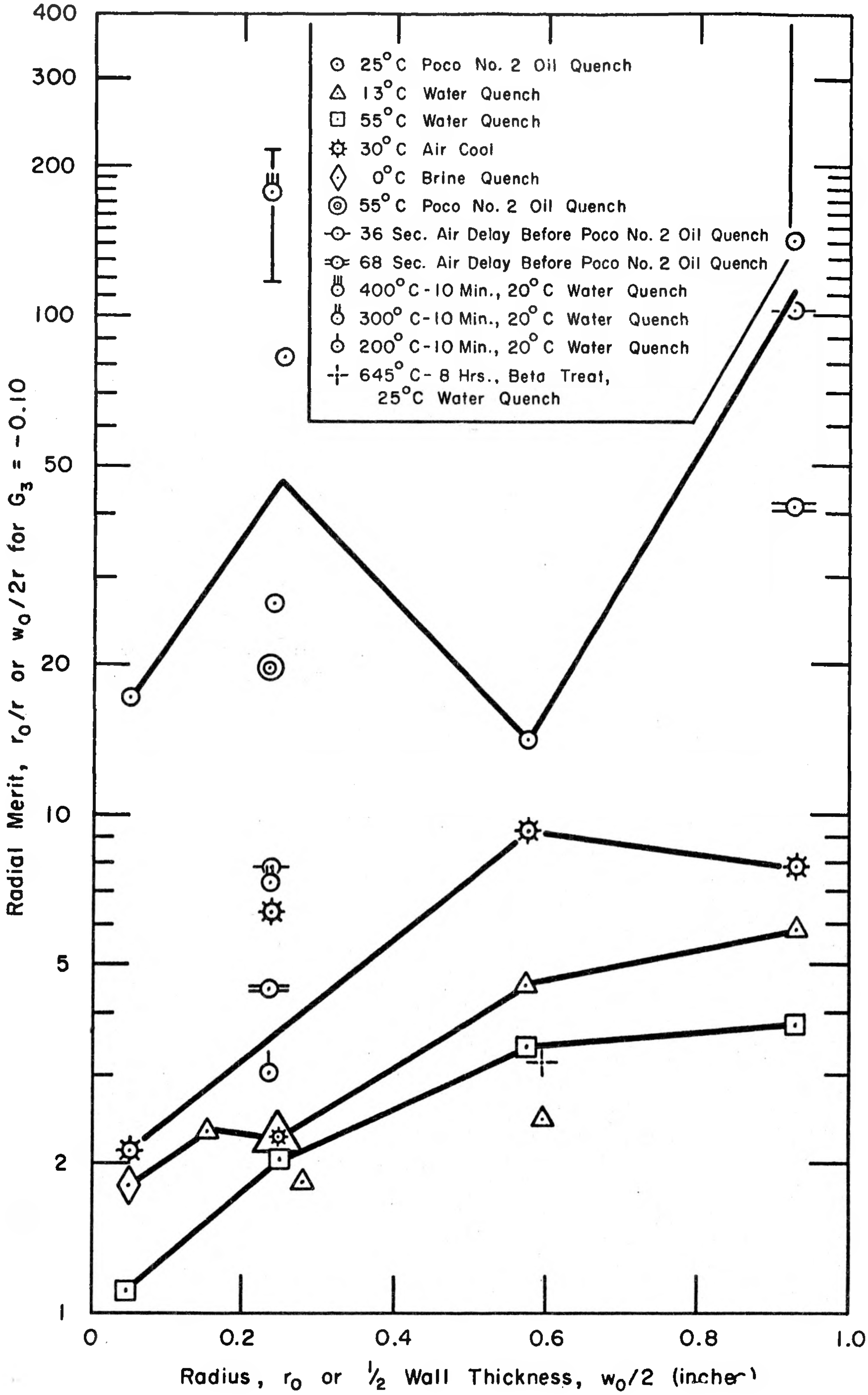


Fig. 9 - Effect of heat treatment of ingot tube and rod on radial merit, r_o/r or $w_o/2r$, where r is depth below OD where G_3 first reaches -0.1, and where r_o and w_o are rod radius and tube wall thickness, respectively.

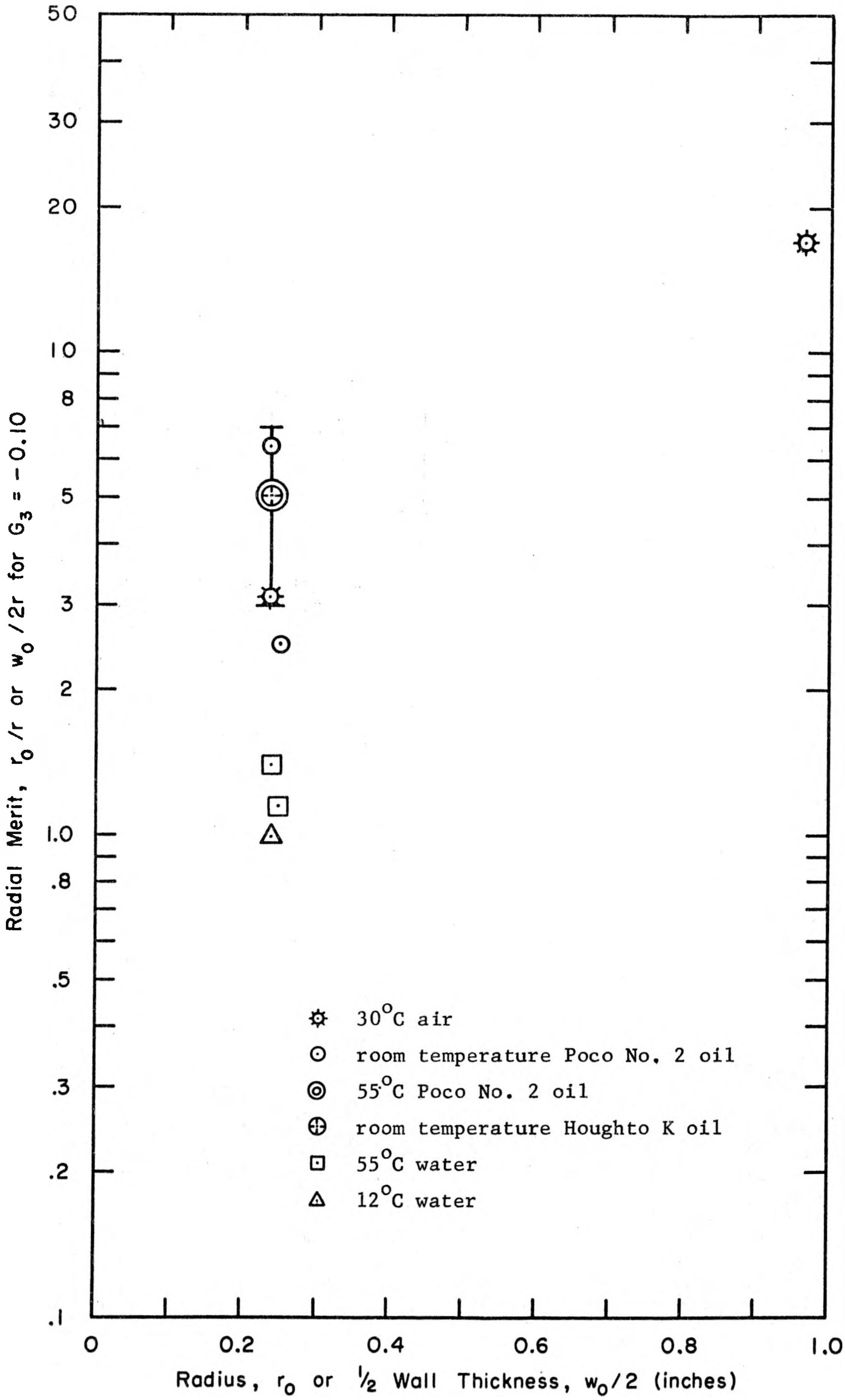


Fig. 10 - Same as Fig. 9, except for dingot uranium. Drawing No. RA-2320.

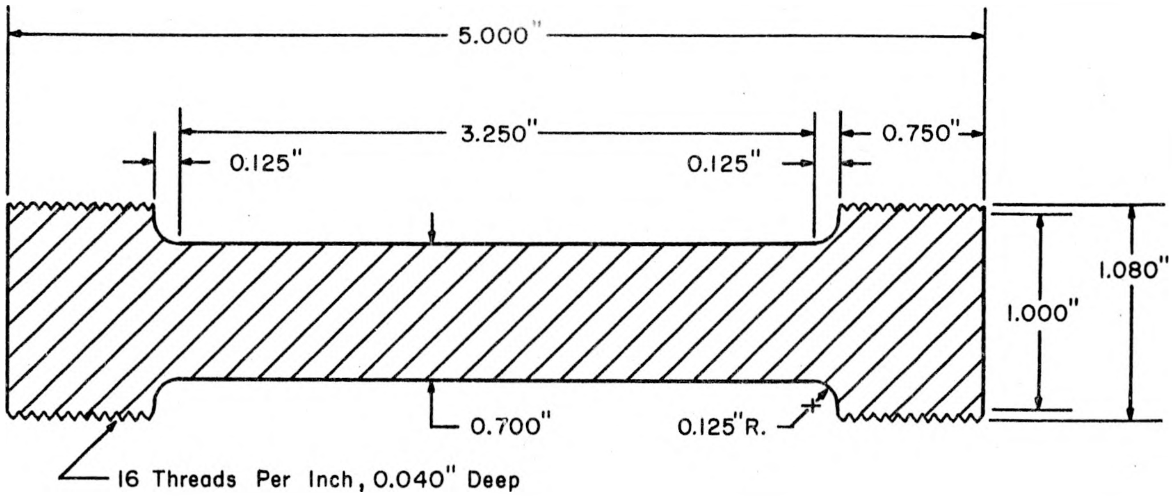


Fig. 11 - Design of 0.70-inch diameter x 3.25-inch gage length tensile sample (size W) for study of effect of applied stress on texture distribution after cooling from the beta phase to room temperature. Drawing No. RA-2183

during cooling, and the cooling curves at the various depths were automatically recorded on a Brush recorder. The cooling rates are listed in Table II. The actual tensile samples, which did not, of course, contain thermocouples, were subsequently stressed at appropriate times according to information obtained from the dummy sample.

Radial sectors (30^{o} wide) and transverse discs (1/4 inch thick) were cut from all stressed samples for radial and axial G_3 determinations, respectively.

Values of G_3 as a function of radial depth are presented in Fig. 12 along with the axial growth indices. Sample W-1 was air cooled from molten salt and stressed at room temperature, while samples W-2, W-3 and W-4 were helium cooled and stressed during cooling. Sample W-2 was stressed while cooling through the beta phase but fractured before reaching the transformation temperature. Sample W-3 was kept under stress while being cooled through the beta phase, the beta-to-alpha transformation, and the high alpha region. Sample W-4 was stressed in the beta phase, unloaded during transformation, and then restressed in the high alpha. It was hoped that by this procedure the effect of loading in each region might be isolated without having to load during transformation, which occurs in a relatively short time and where it was felt that excessively fast straining might result in premature fracture.

A study of Fig. 12 shows that the most anomalous G_3 distribution occurs in sample W-3. Since sample W-4 was stressed in both the alpha and beta fields and does not exhibit these changes, it may be presumed that the difference in behavior between W-3 and W-4 is due to the stressing of W-3 during transformation. W-3 has the following unusual characteristics:

(1) An unusually small negative value of G_3 at the surface (-0.07) which rapidly becomes more negative in to a depth of about 0.040 inch.

(2) A drop-off to a large negative value (-0.39) near 0.160 inch, or almost mid-radius, where most samples exhibit values near zero or slightly positive. The radial texture here is 100-101.

(3) A large positive axial G_3 (+0.24) compared to values near zero for all other samples. This over-all axial texture is 001-010.

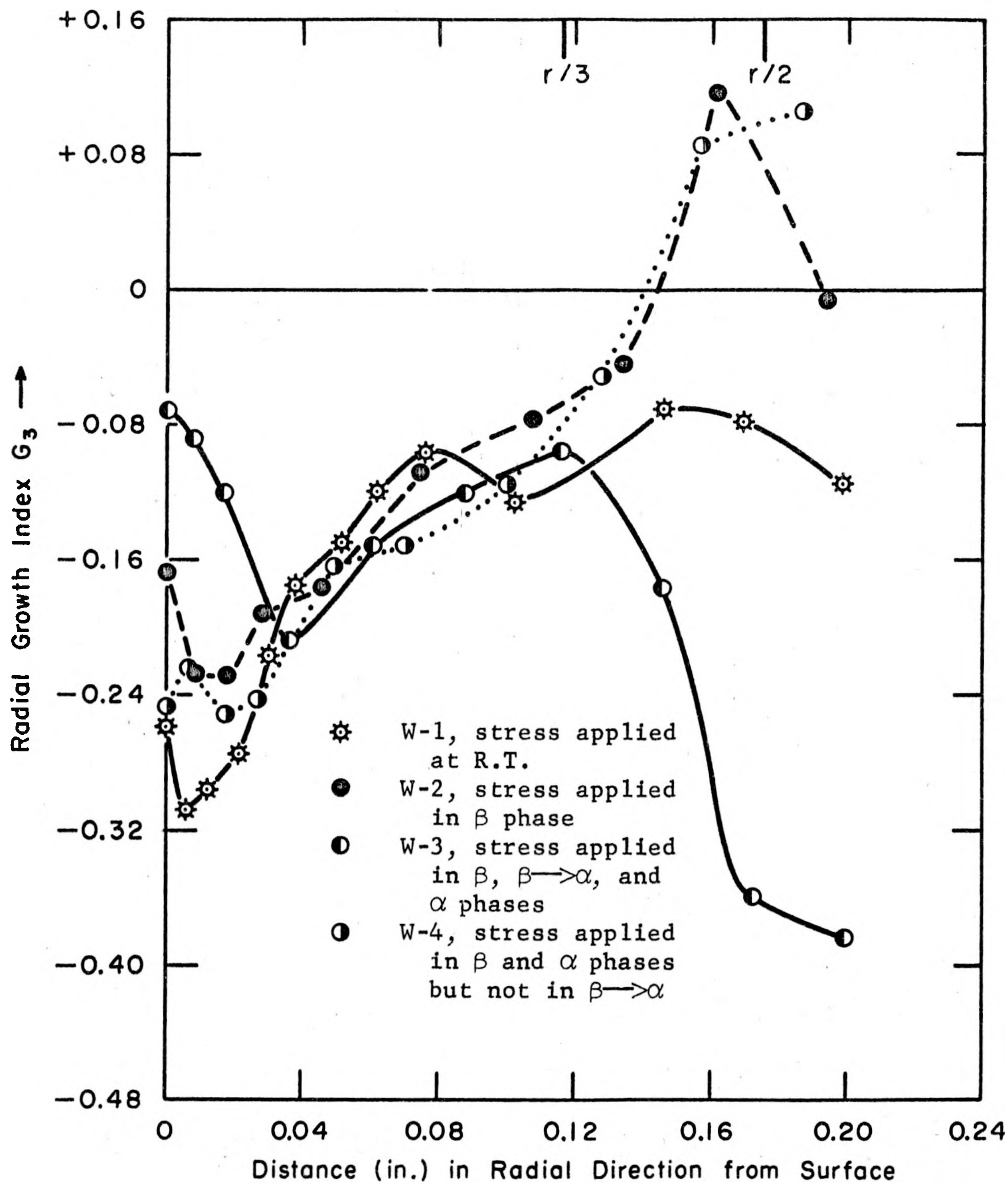


Fig. 12 - Distribution of radial G_3 with radial depth below OD in 1-1/4 inch long x 30^o wide sectors cut from middle of 0.70-inch dia. x 3.25-inch gage length tensile samples, axially strained during 1 to 2^oC/sec cool from beta phase. Drawing No. RA-2321.

Axial G_3: W-1 (+0.063)
W-2 (-0.029)
W-3 (+0.242)
W-4 (+0.001)

The other three samples have fairly similar G_3 profiles except near mid-radius, where it appears that stressing in the beta phase results in more positive values of G_3. Additional stressing in the high alpha phase does not result in any significant change in growth index, as evidenced by the close similarity between samples W-2 and W-4. It is interesting to note that the differences observed in texture near the sample surfaces appear to be due to "hooks" of varying magnitudes and depths of penetration. At radii greater than the depth of penetration of the "hook", however, it is surprising that all the samples show virtually identical G_3 profiles to a depth of about 0.120 inch (about r/3) beyond which deviations in G_3 behavior again occur. Because of the small sample size, it was not possible to obtain G_3 values for greater depths.

The pronounced improvement in radial texture gradient observed for three-point binding [(12)] could not be obtained for any of the conditions of uniaxial tension applied. It can only be concluded that the greater complexity of both the bend stresses and thermal condition in the bend sample, and possibly differences in magnitude of stress, resulted in a texture formation that cannot be duplicated using the simpler controlled procedures of tensile testing.

i. <u>Effect of Internal Free Surface on Ingot</u>. The observation that the most pronounced changes in G_3 are almost always noted at or near the cooling surface has led to speculation that the unique stress and thermal conditions arising from the presence of a free surface may be responsible for the induction of a negative G_3 texture parallel to the thermal gradient. Accordingly, a jointed, four-component sample was machined as shown in Fig. 13 from 1.8-inch diameter ingot rod, beta treated and quenched in $13^{o}C$ water. The ideal interface between the various sections should act as free surfaces unrestrained by, yet not insulated from, the surrounding metal. In practice, it is realized, the present sample falls somewhat short of both of these goals, since there

- - - - - - - - - - -

(12) NMI-2805, p. 29.

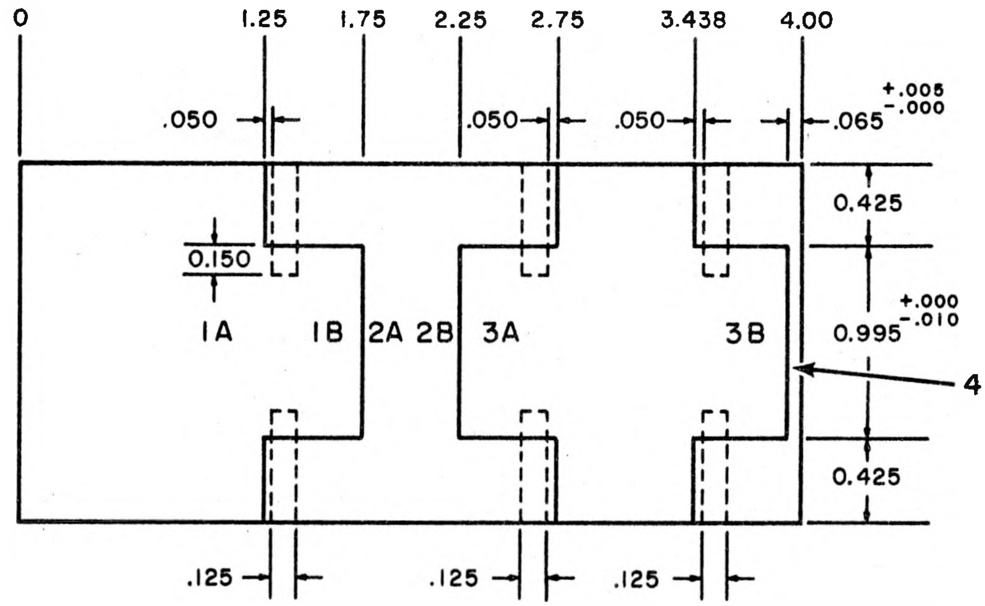


Fig. 13 - Design of jointed 1.845-inch diameter x 4.000-inch long ingot bar (C-11) to study effect of "free surface" in and out of thermal gradient. Drawing No. RA-2262.

will undoubtedly be some geometrical restraints in some directions, and since thermal discontinuities will exist at the interfaces because of oxidation and the lack of metallic bonding. However, it is hoped that the sample offers a close enough approximation to the desired conditions to yield useful information.

Growth indices are being determined across internal surfaces,

(a) in a large thermal gradient (e.g. across interface 4-3B [axial]), (This determination has been completed).

(b) a moderate thermal gradient (e.g. across interface 1B-2A [radial]), and

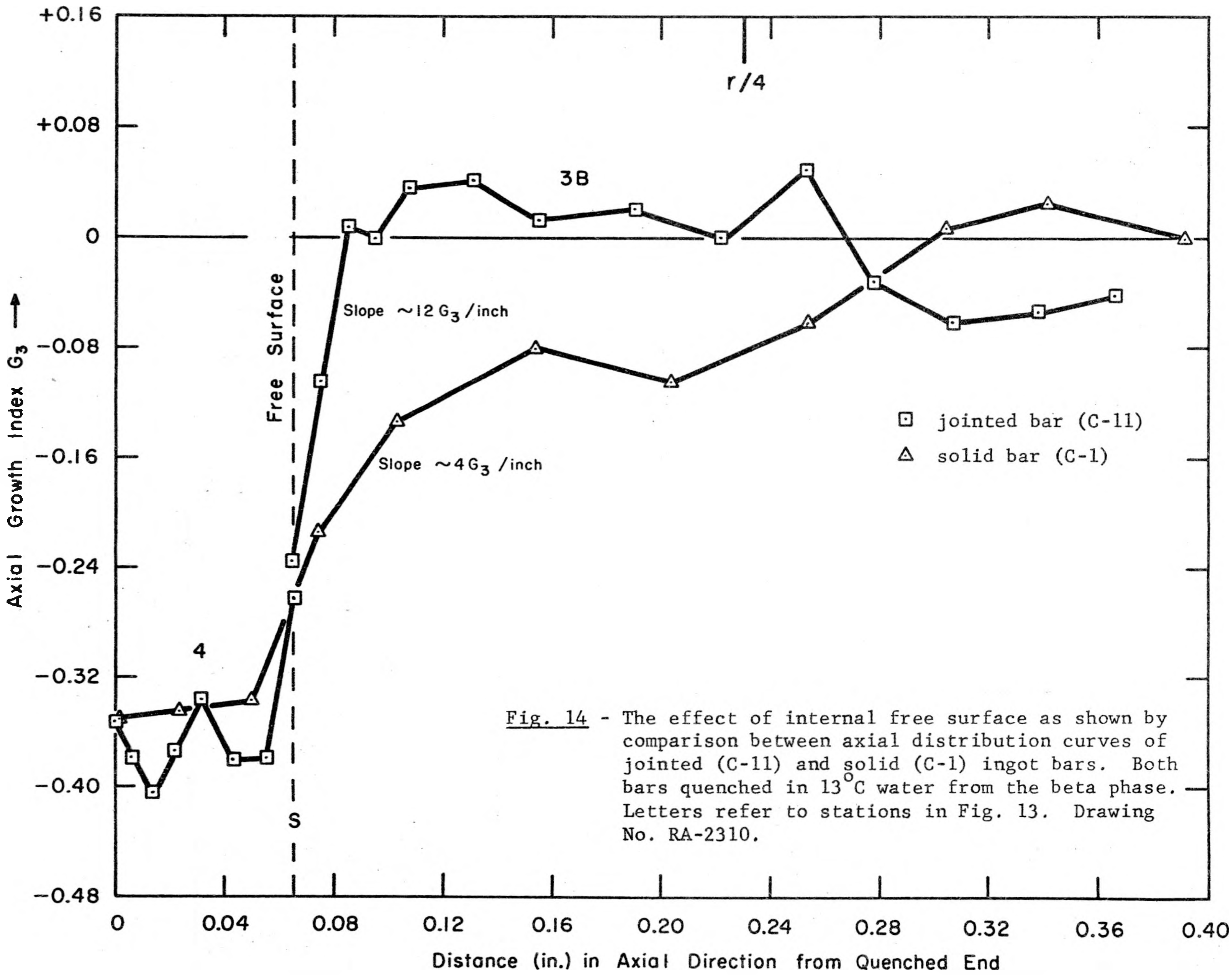
(c) for little or no thermal gradient (e.g. across interface 2B-3A [axial]).

To date, axial G_3 has been determined only across interface 4-3B. Axial G_3, taken from an earlier report,(13) is also given for a bar without internal joints. A comparison between the two curves (Fig. 14) shows that the axial G_3 for both is -0.40 to -0.34 to about 0.050 to 0.055 inch from the quenched end, and both increase beyond this distance, the G_3 associated with the joint rising much more steeply. The slope of the curve is about 12 G_3/inch at the joint, whereas at the same depth in the solid bar the slope is about 4 G_3/inch. The rapid increase of G_3 at the joint or interior free surface is a phenomenon not previously observed, but it should be emphasized that the behavior of G_3 at neither external nor internal free surfaces has yet been explained. There is, of course, a remarkable similarity between the sharp increase in G_3 observed at room-temperature Poco No. 2 oil-quenched ingot external surfaces and at this interior surface.* The conditions of stress and temperature are not known for these surfaces, but the possibility exists that the special conditions necessary for the elimination of severe texture penetration are present in both the exterior free surface of the oil-quenched bar and the interior free surface of the jointed bar. In particular, it is possible that, for one thing, a critical cooling rate is attained at the interior

- - - - - - - - - - -

(13) NMI-2802, p. 26.

* The slope of radial G_3 in oil-quenched 1.5-inch OD by 0.5-inch ID ingot tube, Fig. 3 is about 14 G_3/inch, which compares favorably with a value of about 12 G_3/ inch in the jointed bar.



<u>Fig. 14</u> - The effect of internal free surface as shown by comparison between axial distribution curves of jointed (C-11) and solid (C-1) ingot bars. Both bars quenched in 13°C water from the beta phase. Letters refer to stations in Fig. 13. Drawing No. RA-2310.

joint by virtue of the lowered conductivity across the joint, even though the outside of the jointed bar had been water quenched. During the next report period, more data will be accumulated for the regions of a moderate and nearly vanishing thermal gradient. In order to determine the effect of a double interface between the quenching medium and the underlying uranium on the G_3 distribution, both dingot and ingot bars that have a few thousandths of an inch of electroplated nickel over part of each bar will be beta treated and quenched into cold water. In several cases being studied in other investigations at NMI, the severe radial negative G_3 penetration characteristic of quenched bare metal was conspicuously absent in those instances where ingot was clad.

IV. WORK IN PROGRESS AND PLANNED

Comparison studies between ingot and dingot are being continued. The effect of size and cooling rate for both compositions is under study. More emphasis will be placed on finding the optimum cooling rate for ingot (by varying the cooling capacity of oils) and determining whether the response of dingot to changes in cooling rate really is much less sensative than ingot. These questions should be clarified by the response of thin dingot discs quenched by hot salt.

More work will be conducted on the effect of applied stress on texture distribution of beta-treated metal. The effect of the "free surface" will be explored further; in view of the results so far, it may be profitable to look at the radial texture distribution under some claddings having different degrees of metallurgical bonding. It may be possible to reduce the extent of texture penetration of both dingot and ingot in different media.