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SUBJECT: RESEARCH AND DEVELOPMENT ON PLASTIC DEFORMATION AND DIRECTIONAL PROPERTIES OF TITANIUM

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RAD No. ORDTB-1-12045-2
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WAL REPORT NO. 401/78-12

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THE NEW JERSEY ZINC COMPANY (OF PA.)
RESEARCH DEPARTMENT
PALMERTON, PENNSYLVANIA

AUGUST 15, 1952

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WAL Report No. 401/78-12

RESEARCH AND DEVELOPMENT ON PLASTIC DEFORMATION AND DIRECTIONAL PROPERTIES OF TITANIUM

By E. A. Anderson* and D. C. Jillson*

ABSTRACT

Alpha titanium crystals of sufficient size for plastic deformation studies were produced by repeated cycling in vacuo between four hours at 1200°C. and three to five days at 850°C. Deformation studies showed that slip can occur in alpha titanium at a critical resolved shear stress of about 5000 g./mm.^{2} on the $\{10\bar{1}0\}$ planes or about 11,000 g./mm.^{2} on the (0002) plane, in both cases in the close-packed direction. Twinning can take place on the $\{11\bar{2}1\}$, $\{11\bar{2}2\}$, and $\{10\bar{1}2\}$ planes.

The recyrstallization temperature of cold rolled iodide titanium is about 475°C. for conditions of fairly slow heating and one hour at temperature. The recrystallization temperature is virtually independent of amount of cold reduction between 30 and 96.5 per cent. Grain size is independent of per cent reduction in this range and dependent upon temperature. The recrystallization curves show unexplained anomalies.

Cold rolling tends to produce a $\{11\bar{2}4\}\langle 10\bar{1}0\rangle$ texture while subsequent annealing tends to produce a $\{20\bar{2}5\}\langle 11\bar{2}0\rangle$ texture. Both require the largest cold reduction or the highest alpha temperature anneal to be developed sharply. Lower reductions or annealing temperatures result in mixtures of the two textures. Recrystallization without extensive grain growth results in a mixture of the two textures. Increase in temperature results in growth of the grains of the $\{20\bar{2}5\}\langle 11\bar{2}0\rangle$ texture at the expense of the others.

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THE NEW JERSEY ZINC COMPANY (OF PA.)

FINAL TECHNICAL REPORT

RESEARCH AND DEVELOPMENT ON PLASTIC DEFORMATION AND DIRECTIONAL PROPERTIES OF TITANIUM

August 15, 1952

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Report No. 401/78-12 RAD No. ORDTB-1-12045-2

O.O. Project No. TB4-15

Priority Designation: DA-10

Title of Project: Research and Development on Plastic Deformation and Directional Properties of Titanium

Objects:

To develop methods for preparing large grains or single crystals of alpha (hexagonal) titanium.

To determine the modes of deformation operative in such crystals at or near room temperature.

To determine the orientation textures produced in polycrystalline alpha titanium under a variety of rolling and annealing procedures.

To rationalize these textures from the information derived in this work.

To correlate directional properties (physical and mechanical) with the textures.

Summary:

A method was developed with which a satisfactory number of large grains of alpha titanium were produced. Deformation studies disclosed two mechanisms of slip and three of twinning.

All rolling under this project was carried out in one direction only (no cross rolling) and at approximately room temperature. Two principal textures were found: {11$\bar{2}$4} <10$\bar{1}$0> and {20$\bar{2}$5} <11$\bar{2}$0>. These differ essentialy only in that in the former the close-packed direction in the basal plane is transverse to the direction of rolling whereas it is longitudinal in the latter. The {11$\bar{2}$4} <10$\bar{1}$0> texture was developed most sharply in material cold rolled extensively (96.5 per cent reduction). The {20$\bar{2}$5} <11$\bar{2}$0> texture was developed sharply when heavily cold rolled metal was annealed just below the alpha-beta transformation temperature and was still further intensified by annealing at 900°C. Under most, if not all, conditions investigated both textures co-existed in various ratios of intensity.

Recrystallization studies previously started by this Company are included in this report. Anomalies were found in the properties of specimens annealed to moderately coarse grain sizes.

Efforts to rationalize the rolling and annealing textures were unsuccessful. The problem was complicated by the numerous permissible modes of deformation and, in the case of the annealed texture, by the absence of basic information on the grain growth mechanisms involved.

The available time did not permit an exploration of directional properties as a function of texture.

Conclusions:

1. Large grains of random orientation suitable for deformation studies can be produced in pure titanium by repetitions of the heating cycle of four hours at 1200°C. and three to five days at 850°C.

2. If the specimens are sealed in glass the environment should be a good vacuum. In the presence of argon the titanium reacts with the SiO_2 of the glass and becomes contaminated.

3. Cycling in vacuo results in roughening of the specimen surface due to sublimation of titanium. Contamination by oxygen occurs but to a lesser extent than in argon.

4. At room temperature titanium can deform by slip on the {0002} (basal) plane and the {10$\bar{1}$0} (prismatic) plane, in both cases in the close-packed <11$\bar{2}$0> direction.

5. The critical resolved shear stress for basal slip is about 11,000 g./mm.^{2} and for prismatic slip about 5000 g./mm.^{2}.

6. Under tension or compression stress, basal slip is inhibited by grain boundary or grip restraints. {11$\bar{2}$1} twining in tension and {11$\bar{2}$2} twinning in compression take place instead. Basal slip is unlikely to be an important mode of deformation in fine grained polycrystalline titanium.

7. While the evidence as to the identities of the slip planes and slip directions is conclusive, the slip lines usually are not simple in nature.

8. When deformed extensively by slip, titanium crystals behave similarly to those of other metals.

9. Twinning can occur in titanium on the {10$\bar{1}$2}, {11$\bar{2}$2} and {11$\bar{2}$1} planes.

10. {10$\bar{1}$2} twins resemble the {10$\bar{1}$2} twins in zinc in that they are lenticular in shape and appear to grow in size with increase in stress.

11. {11$\bar{2}$1} twins are thin, have sharply defined parallel sides and traverse the entire cross-section of the specimen. They do not appear to grow as the stress is increased.

12. {11$\bar{2}$2} twins are intermediate in appearance between the {10$\bar{1}$2} and {11$\bar{2}$1} twins.

13. For angles between the basal plane and the tension axis of about 40° or less prismatic slip occurs; between about 40° and 60° basal slip occurs but is readily replaced by {11$\bar{2}$1} twinning under restraint conditions; above 60° {11$\bar{2}$1} and {10$\bar{1}$2} twinning predominate with little basal slip; at 75° {10$\bar{1}$2} twinning apparently is the only mechanism.

14. It is not regarded as established that no other deformation mechanisms are possible in alpha titanium at room temperature.

15. Two principal textures were found. These may be characterized as $\{11\bar{2}4\}\ \langle 10\bar{1}0\rangle$ and $\{20\bar{2}5\}\ \langle 11\bar{2}0\rangle$. The essential difference between them is that the close-packed direction in the basal plane lies transverse to the rolling direction in the former and longitudinal in the latter.

16. Each of the principal textures is accompanied by subordinate textures positioned in all of the $\{10\bar{1}1\}$ twin positions of each of the peaks in the principal orientations. It is not concluded that a $\{10\bar{1}1\}$ twinning mechanism is involved.

17. The $\{11\bar{2}4\}\ \langle 10\bar{1}0\rangle$ texture is found in its most sharply developed form in titanium which has been cold rolled in one direction to a very large reduction in thickness (e.g., 96.5 per cent).

18. The $\{20\bar{2}5\}\ \langle 11\bar{2}0\rangle$ texture is most sharply developed in heavily cold rolled titanium annealed at high temperatures (e.g., 825 or 900°C.).

19. Under most, if not all, conditions of strip rolling and annealing, grains having both orientation textures are present with one or the other texture predominating.

20. Under some cold strip-rolling conditions a superficial layer of grains in a distorted $\{20\bar{2}5\}\ \langle 11\bar{2}0\rangle$ texture can develop over the normal $\{11\bar{2}4\}\ \langle 10\bar{1}0\rangle$ cold rolled texture in the substrate.

21. The recrystallization of heavily cold rolled titanium (96.5 per cent reduction) without extensive grain growth results in no substantial change from the cold rolled texture.

22. Annealing at successively higher temperatures to produce grain growth results in a transition with rising temperature from the $\{11\bar{2}4\}\ \langle 10\bar{1}0\rangle$ texture predominating to (at 800°C. and higher) the $\{20\bar{2}5\}\ \langle 11\bar{2}0\rangle$ texture predominating. There is no sudden change in texture at any temperature.

23. The mechanism involved in this change in orientation must be the selective absorption of grains of the one orientation by those of the other during grain growth. The basic cause of this selectivity in growth is not known.

24. Annealing in the beta region does not change the texture produced in the highest alpha temperature anneals ($\{20\bar{2}5\}\langle 11\bar{2}0\rangle$).

25. The recrystallization temperature (circa 475°C.) of cold rolled iodide titanium, selected as the temperature required to reduce the hardness half-way to the minimum value, differs by only 25°C. for cold reductions varying from 30 to 96.5 per cent.

26. The recrystallization temperature is higher by about 75°C. for cold reductions of the order of 15 per cent.

27. The recrystallization curves show unexplained anomalies. These are particularly pronounced in the hardness data.

28. Specimens cold rolled 30 per cent and more show similar grain shapes and grain sizes for equal anneals.

29. Annealing at 600°C. of titanium cold rolled 15 per cent results in a coarser, more equiaxed structure than is found in more heavily cold worked metal annealed at this same temperature. This grain size difference tends to disappear as the annealing temperature is increased.

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INTRODUCTION

It was the purpose of the work carried out under this contract to develop knowledge of the basic factors which lie behind the ease or difficulty with which titanium can be fabricated. Without such basic knowledge the development of commercial fabrication processes can proceed only with uncertainty.

The scope of the work originally planned is set forth in the following objectives:

1. Development of methods for producing large grains or single crystals of alpha (hexagonal) titanium.
2. Determination of modes of deformation operative in such single crystals at or near room temperature.
3. Determination of the rolling textures produced in polycrystalline alpha titanium under a relatively large number of fabrication procedures.
4. Rationalization of these textures from the information obtained in this work.
5. Correlation of directional properties (physical and mechanical) with rolling textures.

In addition, studies of the relation of recrystallization temperature to amount of cold reduction, previously started by this company, were completed as a part of the contract.

I. PRODUCTION OF SINGLE CRYSTALS OR LARGE GRAINS OF PURE TITANIUM

Introduction

No reference could be found in the literature to successful methods of producing large grains or single crystals of titanium. Production from the melt by slow solidification from one end was not feasible in the absence of a suitable mold material. Of necessity, the methods investigated involved heat treatments of solid titanium. The objective in this phase of the work was the obtaining of specimens with which to study the deformation processes and not a study of crystal growth itself.

Single Crystal Growth Experiments

Five possible procedures were considered:

1. Light straining followed by slow heating into the high alpha-temperature range (e.g. 825°C.).
2. Heavy cold reductions followed by long time annealing in the high alpha-temperature range.
3. The production of a strain gradient by stressing a tapered specimen in tension followed by slowly heating into the high alpha-temperature range and holding at the top temperature.
4. The use of a moving temperature gradient such that the beta-alpha transformation temperature was moved slowly along the specimen.
5. Cycling back and forth through the alpha-beta transformation temperature.

The first of these procedures to yield specimens suitable for deformation studies was the cycling method (No. 5). Since it was not the objective of this work to study the crystal growth process in detail, the work on the other methods was abandoned without further investigation. It cannot be concluded from these brief studies that the production of large grains or single crystals cannot be accomplished by one or more of these other procedures.

The Cycling Method

While a number of variations of the cycling procedure were tried, the method which gave the best yield of useful specimens was the following:

1. The specimens were sealed individually in Vycor glass tubes in a good vacuum (1 micron or less).
2. The specimens were heated in a 1200°C. furnace for 4 hours.
3. They were then transferred fairly quickly to an 850°C. furnace where they were held for 3 to 5 days.
4. Operations 2 and 3 were repeated several times.

Under these conditions a few specimens out of any group processed together would develop suitably large grains after three, or usually more, cycles. In some specimens more than one suitable grain was found.

Specimens were identified by TiXl numbers with subscript letters to identify particular grains. A list of the useful specimens with the approximate lengths of the useful grains is set forth in Table 1. The orientations as determined by standard Laue back-reflection X-ray procedures are plotted stereographically in Figure 1. A fairly random distribution of orientations was obtained.

Not much is known about the internal perfection of the crystals. Visual inspection of the Laue spots on the back-reflection photograms revealed no recognizable evidence of imperfections but fine-structure imperfections might have been present and not detected by this simple test. In two cases two or more back-reflection patterns were obtained at different points on the same grain. In such cases the orientations found were in complete agreement within experimental error. It was judged that the crystals were reasonably perfect and suitable for use in deformation studies.

Procedure

Titanium Used

All of the work on single crystal growth was carried out on specimens from a single fabricated ingot of iodide-process titanium initially weighing 5 pounds 3-1/4 ounces.

The analyses of the four lots of iodide titanium and of the final ingot are assembled in Table 2.

Melting

Melting was done in a tungsten electrode-water cooled copper hearth furnace in an atmosphere of ungettered 99.96 grade tank argon. The furnace was pumped down to a pressure of 10 microns or less and flushed twice with argon before establishing the final atmosphere. A slow stream of argon was allowed to flow through the arc chamber during the melting campaign. Satisfactory freedom from leaks was not assumed if the leak rate under vacuum exceeded 5 microns per minute.

The furnace produces a 3" diameter ingot weighing about one pound per inch of height.

Forging

Before forging, the ingot was machined on all surfaces to remove surface irregularities. Forging was accomplished in a steam hammer on open dies using a starting temperature of 950-1000°C. (1750-1850°F.). Preheating time was 51 minutes. Forging to the final size of 7/8" x 2-3/4" x 12-1/2" was accomplished in three minutes with no reheating.

After cooling, the bar was sand-blasted to remove loose scale and was machined on all surfaces to remove subscales. The machined bar had the dimensions 3/4" x 2-1/2" x

12-1/2". Identity of the original ingot top and bottom was preserved.

The ingot hardness was R_A 26 (R_B 39), which changed only moderately to R_A 31 (R_B 47) as the result of forging and machining.

Rolling

The forged bar was cut into four 3" long segments, each of which was rolled separately as need for more specimens developed.

Segment 1, cut from the portion of the bar corresponding to the original ingot bottom, was started in 3" diameter rolls using reductions per pass of 0.015"-0.020". By the time the bar thickness had been reduced from 0.752" to 0.411" the piece had curled badly. It was straightened in a hydraulic press. Rolling was then transferred to an 18" diameter commercial mill where, due to a misunderstanding, reduction from 0.411" to 0.233" was carried out in a single pass.

Segment 2 was cut adjacent to Segment 1 and was rolled in the 18" mill in 15 passes from 0.752" to 0.250". The final strip was reasonably flat. All rolling of Segments 1 and 2 was in one direction only.

Segment 3, cut adjacent to Segment 2, was rolled in

the 18" mill, taking 16 passes to reduce the thickness from 0.752" to 0.275". At this point the slab was turned 90 degrees and cross-rolled to 0.248" in four additional passes. It was hoped, at the time, that the cross-rolling would better insure obtaining random orientations in the large grains by producing a different starting texture.

Segment 4, corresponding to the top of the original ingot, was rolled in the same manner as Segment 3.

Specimen Preparation

The standard starting specimen was 1/4" x 1/4" x 2". In a few cases 4" bars were prepared. The specimens were milled on all four principal surfaces. They were hand polished on all surfaces on 280-mesh and 400-mesh emery papers, subjected to a 3-minute etch in 50Hf-50 glycerine solution and dried in alcohol and ether before sealing them into the Vycor tubes.

Long Vycor glass tubes of 9 mm. inside diameter were necked down at suitable intervals to accommodate a number of individual specimens. With the specimens in place the tubes were evacuated while warming the glass to outgas the surfaces. With the vacuum pump still working, the neck at the far end was sealed off, the process continuing until the section nearest the pump was sealed. The average individual tube volume

was 6.5 cc. The specimens weighed 7.5 grams each at the start.

Cycling in Vacuum

The only useful specimens were those which were cycled in vacuo. It was noted, however, that the surfaces became roughened and the cross-section area diminished as a result of this treatment. This probably was the result of sublimation of titanium with subsequent reaction between the metal vapor and the silica of the tube. This process had the advantage, however, that the higher rate of sublimation at 1200°C. frequently resulted in a clear revelation of the beta grain size and shape.

The standard practice was to cycle several times, following which the tubes were broken and the specimens removed. Polishing with 280-mesh and 400-mesh emery papers on one surface followed by a suitable etch made it possible to determine whether suitably large grains were developed. If not, the specimens were resealed in tubes and the cycling continued.

Specimens deemed suitable for use were polished on emery paper on all four sides. Following this they were metallographically polished on four wheels. Etching to aid in the removal of distorted metal was resorted to between wheels. A very short etch was used, where necessary, after the final polish to reveal the grain boundaries.

Argon Environment Tests

The surface roughening and cross-section loss resulting from vacuum etching were troublesome. The vacuum procedure involved the further impediment of requiring that the Vycor tubes be sealed in vacuo in an Inconel tube to avoid collapse due to atmospheric pressure at the upper temperatures.

To overcome these difficulties the apparently safe procedure was adopted of admitting 1/5 atmosphere of ungettered 99.96 grade tank argon to the tubes before sealing them off. At temperature this would produce about one atmosphere of pressure, which would eliminate the danger of tube collapse and reduce the sublimation of the titanium. The specimens used at this time were those machined from Segment 3 which had had about 10 per cent cross-rolling as the final rolling step.

With the exception of a few specimens previously cycled in vacuo, not one useful specimen was obtained when the argon atmosphere was used. Examination of the specimens revealed indications of contamination in high hardness and the presence of uncoalesced alpha lamellae. Oxygen, nitrogen and hydrogen analyses revealed the following:

Specimen		Analysis - Per Cent O2	H2	N2	Hardness R_A	DPH*
Ti182	(a)	0.027	0.0076	0.003	--	--
TiX1-3d	(b)	0.058	0.015	--	26-33	115-127
Run 23-1	(c)	0.15	0.011	--	42-45	--
Run 23-2	(c)	0.13	0.011	--	44-50	--
Run 24-1	(c)	0.17	0.008	--	45.5-47.5	--
Run 24-2	(c)	0.11	0.010	--	39-41	--
Run 22-1	(c)	0.30	0.028	0.002	--	--
Run 22-2	(c)	0.29	0.019	0.002	--	--
TiX1-15	(d)	0.16	0.013	--	45.5-47	170-180
TiX1-15	(e)	0.42	0.007	0.002	54-55	218-235

*Microhardness values.
(a) Original bar.
(b) Annealed in vacuum.
(c) Annealed in argon.
(d) Annealed in argon - top end.
(e) Annealed in argon - bottom end.

The evidence of oxygen contamination is, of course, striking. Simple calculation shows that the amounts of oxygen involved could not have come from the argon. Indeed, the admission of 1/4 atmosphere of air wouldnot have sufficed. It seems evident that the major source of the oxygen must be the glass tube. Evidence to this effect is shown below:

Specimen	Treatment*	O_2 - Per Cent
1	Ti-138** as forged	0.04
2	Cycled - no contact with glass	0.036
3	Cycled - no contact with glass	0.036
4	Cycled - no contact with glass	0.035
5	Cycled - immersed in glass beads	0.91
6	Cycled - immersed in glass beads	1.14
7	Cycled - immersed in glass beads	1.18
8	Cycled - lying against glass tube	0.068
9	Cycled - lying against glass tube	0.067

*One cycle 4 hours at 1200°C. - two days at 850°C. in 1/4 atmosphere of argon.
**Similar iodide titanium ingot.

It is probable that the oxygen contents of specimens 8 and 9 would have increased progressively had additional temperature cycles been added.

Three variables were included in the argon cycling experiments, any one or all of which might have contributed to the failure to obtain large grains. These were (1) the observed oxygen contamination, (2) the specimens were cut from strip rolled from a different portion of a possibly segregated ingot and (3) the strip was cross-rolled. Item (2) seems unlikely as a cause since the hardness tests on the ingot and on the forged bar failed to reveal significant differences from end to end. Item (3) is difficult to evaluate. While it is true that subsequent to the argon cycling tests specimens from Segment 4, similarly rolled, were cycled in vacuo for a relatively small number of cycles without great success, the holding time at 850°C. for these experiments was only 20 hours, which probably was too short.

It is the writers' opinion that the oxygen contamination was the most important impediment to grain growth. The visual evidence indicated that the coalescence of the alpha lamellae from the decomposition of the beta was very slow in those specimens which were contaminated. As will be discussed below, complete alpha coalescence seems essential to the

growth of large grains.

It is difficult to understand why the reaction of the titanium with SiO_2 should result in substantial contamination in the presence of argon and lesser contamination in the presence of a vacuum. It may be, however, that the slow sublimation of titanium in vacuo removed surface contamination about as rapidly as it formed and before substantial diffusion into the body of the specimen could occur.

Discussion

While it was not the purpose of this work to study the mechanism of single crystal growth, a few observations of possible value were made during the experiments. As noted above, the vacuum cycling procedure afforded a means for identifying the size, shape and location of beta grains. It was notable that after the 850°C. treatment for three to five days the alpha lamellae, formed as the beta decomposed on cooling, had coalesced into a single orientation occupying, as nearly as one could tell, the volume originally occupied by the beta grain. This must mean that lamellae of one of the six possible alpha orientations stemming from a single beta grain absorbed the others. This would be expected as possible under the oriented growth hypothesis which states that rapid absorption of one grain by another can take place most readily when the

difference in orientation is of the order of 30 degrees.

It seems apparent that failure to complete the absorption process at 850°C. may result in a grain refinement of the beta upon reheating to 1200°C. since each alpha lamellus orientation potentially will result in a different beta orientation. This may be the mechanism by which contamination prevents the growth of large grains but there may well be other equally plausible explanations.

The writers have no good evidence as to whether the growth of large grains takes place in the alpha state or in the beta state. The fact that vacuum etched beta grains of large size are seen may imply that the growth takes place in the beta material but this evidence is far from conclusive.

II. THE RECRYSTALLIZATION OF ALPHA TITANIUM

Introduction

It is important to the study of any new metal or alloy that information be sought on the response to cold working and the relation of the recrystallization characteristics to the degree of cold working. Such studies should be made initially on the purest obtainable metal in order that the effects of impurities and intentional alloyages may be evaluated in other work. The work reported here was started and largely carried out by this Company at their own expense. Completion

of the recrystallization work and the studies of rolling and annealing textures was done under this contract with the permission of the contract supervisor.

General Approach

The plan of the work involved rolling to a single final gauge by four final cold reductions, all of the material being obtained from a single forged ingot of iodide process titanium. To do this a series of rolling schedules was set up which permitted preceding the final reductions by at least one 50 per cent cold reduction and 600°C. anneal. The actual schedules are shown schematically below:

I. 0.400"* - 0.184" (54%) - 600°C. anneal - 0.092" (50%) - 600°C. anneal - 0.046" (50%) - 600°C. anneal - 0.023" (50%) - 600°C. anneal - 0.020" (15%).

II. 0.400"* - 0.236" (41%) - 600°C. anneal - 0.118" (50%) - 600°C. anneal - 0.059" (50%) - 600°C. anneal - 0.0295" (50%) - 600°C. anneal - 0.020" (30%).

III. 0.400"* - 0.170" (58%) - 600°C. anneal - 0.075" (56%) - 600°C. anneal - 0.0375" (50%) - 600°C. anneal - 0.020" (45%).

IV. 0.400"* - 0.200" (50%) - 600°C. anneal - 0.100" (50%) - 600°C. anneal - 0.050" (50%) - 600°C. anneal - 0.020" (60%).

*Approximate thickness of forged bar.

The final 0.020" strip was machined into tension specimens which were annealed at temperatures ranging from

room to 871°C. Tensile strength, elongation, Rockwell A and B hardness and microstructure were used as criteria of change. Material was segregated at every step of the processing for possible use in the studies of texture.

Procedure

Titanium Used

A total charge weight of 2619.5 grams (5.77 pounds) of iodide titanium was supplied, consisting of approximately equal parts of iodide titanium lots IT-123, 142, 146, 147 and 163. The analyses listed in Table 3 were obtained on the turnings developed in machining the ingot (Ti-116).

Melting

The ingot, identified as Ti-116, was prepared in the same arc furnace and under the same operating conditions as previously described for ingot Ti-182. It was the first large size ingot made in this laboratory. At the very end of the run a leak developed in the rubber bellows around the electrode, resulting in some contamination of the top of the ingot.

The ingot hardness after machining varied along the bar in the manner shown as follows:

Position	Hardness R_A	Hardness R_B
Top surface	42	57
Side near top	33-1/2	--
Side - 1/4" from top	29-1/2	--
Side - 1" from top	28-1/2	--
Side - 1-1/2" from top	26-1/2	--
Side - 2" from top	25-1/2	--
Side - 2-1/2" from top	24	--
Side - 3" from top	25-1/2	--
Side - 3-1/2" from top	25-1/2	--
Bottom surface*	20	25

*4-1/2" from top.

Since sufficient material for the projected studies could be obtained from the strip from 4/5 of the bar length, the upper 1/5 was not included in the main experiments. It was carried through the rolling and annealing procedure, however, to afford a comparison with the uncontaminated metal from the bottom of the ingot. The spread in side hardness from R_A 25-1/2 to R_A 28-1/2 in the accepted length was not considered intolerable.

Forging

The ingot was heated for one hour at 950-960°C. (1740-1760°F.) after a half-hour preheat from room temperature. The final forged bar was 0.6" x 2-1/2" x length. It was not necessary to reheat to complete the forging. The final stages of forging were probably carried out below the transformation point.

After sand blasting to remove loose scale, the bar was straightened in a hydraulic press and machined to remove at least 0.06" from all principal surfaces. Micro-studies had indicated a maximum sub-scale depth of 0.05". The final machined bar dimensions were 0.42" x 2-3/8" x 17". The identity of the original ingot top and bottom was preserved.

The machined ingot was annealed in 99.96 grade tank argon with the additional protection of piles of clean titanium chips in the steel-tube chamber used. Three and one-half hours were required to reach an ingot temperature of 825°C. which was then maintained for one hour before removing the tube from the furnace. The surface of the annealed bar was bright. Hardness values obtained at intervals along the bar follow:

Position	Hardness R_A	Hardness R_B
Top*	34	52
1" from top	33	55
4" from top	33	47
8" from top	29	42
9" from top	28	--
12" from top	24-1/2	37
15" from top	27	36
16" from top	26	37
17" from top**	24	37

*Corresponds to top of original ingot
**Corresponds to bottom of original ingot

Microexaminations had shown some structure differences between the two ends of the forged bar. The differences

involved essentially variations in degree of coalescence of the lamellar, basket-weave, decomposed beta structure. The softer, lower end of the ingot displayed a greater tendency to form small equiaxed grains. After annealing, however, both areas were equiaxed and roughly of the same order of grain size.

Rolling

The general plan was to roll the bar in one piece until the thickness (0.236") representing the first special schedule was reached. At this point the top and bottom fifths were cut off, the bottom to be continued separately under Schedule II to a final cold reduction of 30 per cent. The top fifth, representing the contaminated area, was to be rolled in the same way for comparison.

The remainder of the bar was then rolled, without anneal, to 0.200" where a segment adjacent to the top piece was removed for rolling under Schedule IV to a final 60 per cent reduction. Similarly, segment 3 was removed at 0.184" for Schedule I (15 per cent final reduction). The last remaining segment of the bar (adjacent to segment II-B bottom) was then continued to 0.170", without anneal, at which point rolling under Schedule III (45 per cent final reduction) was started.

This first stage of the rolling was carried out on 12" diameter rolls using reductions of the order of 0.02" per pass. Under these conditions the metal quickly became too hot to handle without gloves. After each 10 per cent of reduction the bar was cooled in water to 24°C. and tested for Rockwell A and B hardness in each of the segment areas. These curves all had the same shape, which is typified by the Schedule III data in Figure 2. All work hardening data for this and the later stages of rolling are compiled in Table 4.

The individual segments were annealed in argon in an Inconel muffle for one hour, at temperature, at 600°C. followed by slow cooling to room temperature in the tube after removal from the furnace. Rolling according to the special schedules outlined above was then continued on 3" diameter rolls, taking room temperature Rockwell A hardness readings at 10 per cent reduction intervals in some, but not all, cases. All anneals were at 600°C. under the conditions described immediately above. In all cases the metal rolled well, showing no signs of edge or surface cracking. The work hardening curves were similar in shape and scale to that shown in Figure 2. Micro-studies indicated that the anneals given resulted in a reasonably uniform grain size of 0.035 mm.

Occasional streaks of coarser grains noted in the earlier stages of rolling no doubt were eliminated or reduced in magnitude by the time the final rollings were started.

Specimens and Final Anneals

From each of the final strips there were machined 50 small longitudinal tension test pieces to the dimensions shown in Figure 3. At every stage in the rolling, including the final processing, portions of the bar or strip were set aside to provide for texture studies where such were later desired.

The temperatures selected for the final annealings were room, 300, 400, 500, 600, 700, 800 and 900°C. Later, when the available data had shown need for them, anneals at 450 and 550°C. were added. Due to controller trouble, the anneal intended to be at 900°C. actually was at 871°C.

All of the specimens for any one anneal were treated together. The Inconel muffle was used with an argon atmosphere. In each case the time was set arbitrarily as one hour at temperature. While attempts were made to control the heat-up time on a comparable basis, the conditions did vary from one temperature to another. In general, the heat-up time was of the order of one hour.

Testing

The annealed specimens were tested for tensile strength, elongation and Rockwell A and B hardness, the latter readings being taken on the grip ends of the tension specimens before they were pulled. The tension tests were carried out in a 20,000 pound capacity hydraulic testing machine using Templin self-aligning grips and a free head speed of 1/4" per minute. Elongation was measured by dividers after abutting the broken specimen ends.

Data

The data obtained are assembled in Table 5. Analyses made on certain of the broken specimens are to be found in Table 3. The data of Table 5 have been plotted in Figures 4 through 8.

With the exception of the Schedule I specimens (15 per cent cold reduction), the grain sizes of the other specimens were very similar for similar anneals. It is convenient to illustrate the general effect of annealing temperature on grain size with micrographs from a single series. These appear as Figures 9 through 14 and are for the Schedule III (45 per cent reduction) series. Four photomicrographs (Figures 15 through 18) illustrating typical structures in the Schedule I material are included.

Discussion

The objective of this work was to determine the recrystallization temperature of pure titanium for several degrees of cold reduction and one annealing time. For the purpose of this portion of the discussion, the recrystallization temperature is defined as the minimum temperature at which no distorted, cold-worked material remains. When taken arbitrarily as the temperature required to change any property to half-way between the maximum and minimum values, the temperatures listed in Table 6 are found.

Outside of the Schedule I material (15 per cent reduction), the effect of per cent reduction is rather small. From other work it is known that increasing the percentage reduction to 96.5 does not change the figures for 60 per cent reduced metal appreciably. The total spread in recrystallization temperature from 15 to 96.5 per cent reduction is only 75 to 80 degrees.

While it is very difficult to determine from microstructure alone when recrystallization is complete, it is the writers' opinion that completion, in the sense defined above, was attained at about 500°C. except for the Schedule I material. In this latter case identification of old grains in a matrix of new grains was not easy and the only certainty is

that recrystallization was complete and grain growth well advanced at 600°C.

In addition to the features normally found in recrystallization curves, the present data for titanium reveal a number of anomalies. Most marked are the rise in hardness and the decrease in elongation as the annealing temperature is increased above 600°C. Tensile strength is reasonably constant in this annealing range. Less marked but still very evident are discontinuities in all properties in the range 450 to 600°C.

From approximate grain size measurements it can be shown that the change in grain size with temperature is a smooth curve, possibly logarithmic. There is no evidence of any discontinuity in the curve. While there is further discussion of the phenomenon later in this report, the writers advance no explanation for the anomalies.

The effects of the contamination of the top of the ingot during melting were not large. The effects on recrystallization temperature were in opposite directions for the tensile and the hardness results. A very small but seemingly definite reduction in grain size was observed in the harder material. It seems apparent that contamination to the extent encountered here is incapable of altering the recrystallization behavior of titanium substantially.

III. PLASTIC DEFORMATION STUDIES

Specimens

The specimens available for this work have already been described. The orientations are shown in Figure 1.

Test Procedures

Tension tests were run at room temperature in a 20,000 pound capacity hydraulic testing machine. The specimens were gripped for a length of about 1/4 inch at each end in Templin self-aligning grips. The axiality of loading was judged visually. Gauge marks were produced by very light scratching. Loading was at the rate of 0.1 inch per minute, free head speed. In all cases the specimens were unloaded after each of various increments of loading to permit inspection for deformation markings, measurement of permanent deformation and, where desired, X-ray determination of the orientation. While no attempt was made to obtain precise stress-strain data, efforts were made to identify the stress corresponding to the first permanent deformation detectable (by comparison at 18X with a machine-ruled scale graduated in units of 0.01 inch from which thousandths could be estimated).

For the few compression tests the specimens were cut carefully and machined to produce plane, parallel ends. The test pieces were about 1/2 inch long, giving a length-to-width ratio of about 3. Compression was carried out between lubricated

polished steel blocks in the same testing machine and at the same free head speed. The same procedure was used of interrupting the loading periodically to permit examination of the specimens.

Original orientations and changes in orientations due to deformation were determined by standard Laue X-ray back-reflection techniques. A North American Phillips Unicam set-up permitted lining up the specimen surface normal to the beam to within 1/2 degree. Unfiltered copper radiation was used with a specimen-to-film distance of 1 inch and an exposure time of about 1 hour.

As the specimens deformed their originally square cross-section frequently changed to a parallelogram and the surfaces became somewhat rumpled by the deformation. Since it is important that the X-ray beam impinge on the specimen surface in a precisely normal direction, a practice was developed to accomplish this. A semi-transparent zinc sulfide mirror was affixed to the specimen surface in question, thus providing a reflecting surface for the optical set-up procedure. In those cases where the angles between two faces had departed from 90°, the actual angle could be measured by using two such mirrors in conjunction with the goniometer portion of the Unicam. The mirror was left in place during the X-ray exposure.

About the time this technique was established it was noted that the films seemed sharper and freer of extraneous darkening than before. It is not known whether this was due to the mirror or to other general technique improvements.

Experimental Work

The work on deformation is probably best presented as separate descriptions of the experiments performed on individual specimens. In what follows this practice is used, presenting the tension test data first.

Tension Tests

TiX1-6

This was the nearest to a truly single crystal specimen produced in this work. Its orientation (see Figure 1) was such that basal slip could occur with almost maximum ease if deformation by this process were possible in titanium. The specimen was 2 inches long x 0.179 inch x 0.139 inch, with a cross-section area of 0.0249 square inch.

A series of increasing tensile loads was applied; after each stressing the load was released. At the 12th load application the load reached the value of 1050 pounds, or 42,000 p.s.i. While it could not be measured within the sensitivity of the elongation measurement procedure, there was evidence of a very slight permanent set. Up to this point the only evidence

of permanent deformation had been slight clicking sounds associated with twinning in or very near the grips.

Upon further stressing to 44,000 p.s.i. (1100 pounds) a permanent elongation of 0.8 per cent in 1/2 inch was observed, which increased to 2.6 per cent at 46,000 p.s.i. (1150 pounds); 10.5 per cent at 47,000 p.s.i. (1175 pounds) and 22 per cent at 47,320 p.s.i. (1183 pounds). The load reached a maximum of 1183 pounds, dropping to 1140 pounds as the specimen elongated. The next and final stressing brought the total elongation to 28.2 per cent but the load could only be brought up to 1068 pounds, which decreased to 980 pounds as the specimen elongated.

Laue back-reflection X-ray photographs were taken after 2.6, 10.5, 22.0 and 28.2 per cent elongation. The changes in orientation, shown stereographically in Figure 19, establish a rotation consistent with slip on the {0002} plane in the most highly stressed $\langle 11\bar{2}0 \rangle$ close-packed direction. The deformation markings observed on the specimen confirm that the slip was indeed on the basal plane.

When the load of 1183 pounds was applied to the specimen, a kink developed near the bottom end of the test region. A Laue pattern was taken in the kink after this loading. An additional pattern was taken after the final stressing to 28.2 per cent total elongation.

In plotting these two special points it was necessary to assume a stress axis in the kink. The true stress axis cannot be determined easily since the direction of pull on the specimen was along the original specimen axis. It was decided, however, to use the specimen axis as the basis for estimating the rotations due to the formation of the kink. The points, marked ⊕, so derived are plotted in Figure 19. Taken at their apparent value the evidence indicates that the kink was formed by a localized $\{10\bar{1}0\}$ slip which, in the first extension in which it occurred, brought the kink orientation to the point where a second $\{10\bar{1}0\}$ plane was equally highly stressed. As a consequence, continued deformation in this area was by simultaneous (double) slip with a net rotation toward a $\langle 10\bar{1}0 \rangle$ direction.

The markings in the kink were examined carefully. The markings were confusing and did not furnish confirmation of the mechanism indicated by the X-ray data discussed above. However, they did not furnish a basis for an alternative explanation.

After each stressing in the plastic range the main portion of the specimen was examined carefully on all four surfaces for deformation markings. As shown at low magnification in Figure 23, markings appeared in the early stages of

deformation which definitely were due to basal slip. There is evidence in these micrographs of cross markings. These became more definite as the load was increased and are clearly shown at 250X in Figure 24.

It will be noted that the cross markings do not correspond directly to activity on a low index plane. The writers suggest, however, that they may be due to a very fine unresolved composite slip on the basal plane and some other low index plane having a close-packed direction in common.

Markings found near the grip restraints, which could be traced around a corner, were found to be due to $\{11\bar{2}1\}$ twinning.

The critical resolved shear stress for basal slip calculated from the 42,000 p.s.i. stress at which first indications of permanent set were noted is 13,100 g./mm.^{2}. The Rockwell A hardness of the crystal was 35.5, taken near the grip after the final extension. Analysis of the specimen* revealed an oxygen content of 0.14 per cent and a hydrogen content of 0.016 per cent. No nitrogen analysis was made since several made on other specimens of similar history revealed only 0.002 per cent present.

*As determined near the center of the crystal. Values of 0.39 and 0.18 per cent oxygen were obtained at the end and adjacent to the end, respectively.

TiX1-3A

This was a crystal about 1 inch long at one end of a specimen which also contained TiX1-3D, to be discussed later. TiX1-3A had the dimensions 0.224 inch x 0.171 inch, giving a cross-section area of 0.0383square inch. The orientation was suited to basal slip.

A series of increasing stresses was applied, unloading for specimen inspection after each. The first permanent deformation developed in crystal TiX1-3D at a stress of 15,600 p.s.i. (12th stressing). There was no evidence at this time of any permanent extension or deformation markings on TiX1-3A.

The first permanent set in TiX1-3A occurred when a load of 1300 pounds (34,000 p.s.i.) was applied. At 1473 pounds (38,700 p.s.i.) an elongation of 4.2 per cent was observed, which increased to 9.2 per cent at 1535 pounds (40,000 p.s.i.). The change in orientation (see Figure 19) due to deformation was the rotation caused by basal slip in the most highly stressed $\langle 11\bar{2}0 \rangle$ direction. Slip lines were found which when traced over a corner established that basal slip had occurred.

While the first markings were those due to basal slip, twin markings identified as $\{11\bar{2}1\}$ were seen near the grips at an early stage in the permanent deformation. These spread toward the center of the section as the load increased and were

seen in all areas after the final stressing (40,000 p.s.i.). A micrograph showing the typical appearance of the basal slip markings and the $\{11\bar{2}1\}$ twins is shown as Figure 25.

The $\{11\bar{2}1\}$ twins are thin and straight-sided. They traverse the entire cross section and do not appear to grow with increased stress; instead new $\{11\bar{2}1\}$ twins form. As the twins approach a specimen edge they frequently become branched as other $\{11\bar{2}1\}$ planes become active.

Due to special stress developments at restraints, other twinning systems became active locally. A lenticular marking identified as a $\{10\bar{1}2\}$ twin is shown in Figure 26. Markings identified as $\{11\bar{2}2\}$ twins are shown in Figure 27. These were found in TiX1-3E, a small crystal, the orientation of which was known.

The critical resolved shear stress required to initiate basal slip in TiX1-3A was determined to be 10,900 p.s.i. In the TiX1-3E area the Rockwell A hardness was about 30. The oxygen content was 0.058 per cent and the hydrogen was 0.015 per cent. In the TiX1-3A area the oxygen content was found to be 0.11 per cent. The hydrogen was 0.012 per cent.

TiX1-2

TiX1-2 was a 3/8-inch long grain in an otherwise coarsely polycrystalline specimen. Its orientation (Figure 1)

was very similar to that of TiX1-6A. The cross-section was 0.220" x 0.104", giving an area of 0.0229 square inch.

When this specimen was stressed, the principal deformation took place in the polycrystalline region. In fact, rupture in this zone occurred before very marked deformation developed in TiX1-2. The specimen was regripped and the stressing continued to a maximum load of 930 pounds (40,500 p.s.i.).

Laue back-reflection X-ray photographs were taken at various steps in the loading. While these showed strong evidence of deformation, no measurable rotation due to slip could be found. Basal slip markings were seen early in the deformation but the preponderant markings were those due to $\{11\bar{2}1\}$ twinning. Some $\{10\bar{1}2\}$ twinning developed near the restraints. These markings, all of which were clearly identified by angle measurements on two surfaces, are shown in Figure 28.

TiX1-2 was a preliminary specimen made from strip at hand which had been rolled from iodide titanium ingot Ti-138. Analyses of the cold rolled strip yielded the following:

Oxygen	0.03%	Molybdenum	0.0009%
Nitrogen	0.002%	Aluminum	0.015%
Hydrogen	0.014%	Manganese	0.02%
Carbon	0.06%	Tin	0.002%
Iron	0.01%	Lead	0.0035%
Copper	0.005%	Magnesium	0.0025%

No analyses were made on TiX1-2.

TiX1-11B

TiX1-11B was a grain about 3/4 inch in length in a specimen containing other large grains. The cross-section was 0.215 inch x 0.1735 inch, with an area of 0.0373 square inch. The orientation is shown in Figure 1.

When stressed to 29,500 p.s.i. no permanent deformation resulted. At the next and final loading the stress reached 51,000 p.s.i., at which point a small (0.8 per cent) set occurred. Examination of the markings indicated a predominance of {1121} and {10$\bar{1}$2} twins, together with faint basal slip markings. At 51,000 p.s.i. stress the resolved shear stress for basal slip in the most highly stressed close-packed direction would be 13,500 p.s.i. The oxygen content determined in the TiX1-11B area was 0.17 per cent; the hydrogen was 0.004 per cent. An oxygen analysis of 0.26 per cent (hydrogen 0.012 per cent) was reported for a sample taken at one end of the 2-inch long specimen.

TiX1-19

TiX1-19 was a 1-1/2-inch long grain so oriented as to place the basal plane more nearly normal to the specimen axis than any other crystal obtained (see Figure 1). The cross-section area was 0.193 square inch. When pulled to a stress of 57,000 p.s.i., abundant twinning developed. Five sets of

{10$\bar{1}$2} twins traceable over an edge were found. At this stage of the deformation there were no detectable markings corresponding to basal slip or {11$\bar{2}$1} twinning. Attempts to reach higher stresses resulted in a maximum stress of 71,600 p.s.i., at which point abundant twinning of the {10$\bar{1}$2} type was evident and an elongation of about 14.5 per cent occurred. The specimen necked slightly.

The extensive {10$\bar{1}$2} twinning roughened the specimen surfaces so greatly that inspection for other markings was difficult. However, no markings indicative of basal slip or {11$\bar{2}$1} twinning were found. It was not possible to observe whether any slip process had operated in the {10$\bar{1}$2} twins.

TiXl-19 had a Rockwell A hardness of 55, measured at one end before testing. The oxygen content at the center of the crystal was 0.17 per cent; the hydrogen was 0.015 per cent. In many cases the oxygen content at the ends of a specimen was higher than in the middle. This might account for the relatively high hardness value.

Discussion

The five specimens just described all were oriented in such a manner as to make basal slip a possibility if no easier mechanism was available. The evidence from TiXl-6, 3A, 2 and 11 establishes beyond doubt that {0002} slip in the <11$\bar{2}$0>

direction can occur in alpha titanium under the conditions of temperature and rate of loading used here. The critical resolved shear stress required is high (10,900 to 13,100 gm./mm.^{2}).

As the length between restraints decreased (TiX1-6, 1-1/2 inch; TiX1-3A, 3/4 inch; TiX1-2, 3/8 inch) the amount of basal slip decreased and $\{11\bar{2}1\}$ twinning became important. The conclusion is drawn that basal slip is not likely to be an important mode of deformation in polycrystalline titanium of moderate grain size.

TiX1-2, 3A and 6 were not widely different in the angle chi (52-45°) between the basal plane and the specimen axis. TiX1-11B, on the other hand, had an angle chi (X) of more than 60°. This difference in orientation had the interesting consequence of adding the $\{10\bar{1}2\}$ twinning mechanism to the $\{11\bar{2}1\}$ twinning previously observed. Basal slip was barely detectable in this specimen. TiX1-19 with an angle chi of more than 75° apparently deformed solely by $\{10\bar{1}2\}$ twinning, although complete certainty of the absence of other modes was not possible.

It would appear from these results that grains in polycrystalline alpha titanium oriented to give an angle chi with the principal tension stress direction of about 45° or

higher will (1) require relatively high stresses to initiate deformation and (2) deform, initially at least, by one or more twinning mechanisms. It is not known whether any slip mechanism will subsequently operate in the twinned material.

TiX1-10A

TiX1-10A was a 1-1/8-inch long crystal in a specimen which also contained TiX1-10B (5/8 inch long). The cross-section dimensions were 0.189" x 0.138", giving a cross-section area of 0.026 square inch. The loading history is detailed below:

Extension No.	Stress p.s.i.	Elongation Per Cent in 1/2"
0	None	0
1	7670	0
2	11500	0
3	13400	0
4	15300	1
5	16100	8
6	16500	12.5
7	17000	16.2
8	20840	28.3
9	22300	37
10	22350	41.4
11	< 22350	49.3
12	< 22350	*

*Not determined - see text.

The first evidence of permanent deformation was observed at extension No. 4 (15,300 p.s.i. stress). The changes in orientation are plotted in Figure 20. The deformation markings were identified positively as due to slip on the most

highly stressed {10$\bar{1}$0} plane. The rotation shown in Figure 20 establishes that the slip direction was the close-packed direction, <11$\bar{2}$0> , in this plane.

The slip markings are shown, for two surfaces, in Figure 29 after the 6th extension. As the deformation progressed the rumpling noted in these photomicrographs became more marked. In addition, a pattern of light and dark streaks developed at a small angle to the slip markings (see Figure 30). These have been observed in zinc by the writers and in other metals by other workers. Their nature and cause is not understood.

It will be noted in Figure 20 that the rotation of the crystal relative to the stress axis brought it to the point where another {10$\bar{1}$0} plane was equally as highly stressed as that on which slip had originally taken place. When this point was reached (extension No. 11), a kink developed which formed the unusual necked-down configuration shown in sketch form in Figure 31. Simultaneous slip on the two equally stressed {10$\bar{1}$0} planes apparently resulted in a composite rotation in the <10$\bar{1}$0> direction as shown in Figure 20. The Laue back-reflection x-ray patterns were taken 1/8-1/4 inch from the kink.

The deformation was so extensive by this time that

the markings were confused. Evidence to support the above hypothesis could not be obtained from them but no alternative mechanism was indicated.

The critical resolved shear stress for prismatic slip in the close-packed direction was determined to be about 5000 g./mm.^{2}. Rockwell A hardness readings taken near the grips after the 12th extension gave values of 25 in the TiX1-10A section and 47 in the TiX1-10B section. No observations were made on TiX1-10B. The oxygen content determined at the middle of TiX1-10A was 0.098 per cent; the hydrogen was 0.011 per cent.

TiX1-3D

TiX1-3D was of irregular length (varying from 1/8 inch to 1/2 inch) in the specimen containing TiX1-3A already discussed in this report. As shown in Figure 20, deformation took place by glide in the $\langle 11\bar{2}0 \rangle$ direction. The slip plane was identified from the markings as the $\{10\bar{1}0\}$. The critical resolved shear stress for this slip was about 5000g./mm.^{2}, in good agreement with TiX1-10A. An analysis in the approximate region of TiX1-3D gave values of 0.058 per cent for oxygen and 0.015 per cent for hydrogen.

TiX1-9A and 9B

TiX1-9A was 1-5/8 inch long; 9B was 5/16 inch long.

Both were in the same specimen. The orientations are shown in Figure 1. The cross-section dimensions were 0.167 x 0.152, giving a cross-section area of 0.0254 square inch.

Both crystals deformed by $\{10\bar{1}0\}$ slip in the $\langle 11\bar{2}0\rangle$ direction (see Figure 20). The deformation markings were similar to those seen on TiX1-10A (Figures 29 and 30). TiX1-9B was somewhat more favorably oriented for $\{10\bar{1}0\}$ slip than TiX1-9A and deformation started in it at a somewhat lower load. This deformation resulted in additional stress at the boundary between the two grains, as a consequence of which slip bands were seen in this region in TiX1-9A before they became evident in the main portion of the crystal.

The critical resolved shear stresses for $\{10\bar{1}0\}$ slip in TiX1-9A and 9B were about 4500 and 5500 g./mm.^{2}, respectively. Analyses at the middle of TiX1-9A showed 0.093 per cent oxygen and 0.012 per cent hydrogen. When measured at the end of the specimen in crystal 9A, the values were 0.32 and 0.012 per cent, respectively.

TiX1-11A

TiX1-11A was an irregular grain varying from 1/8 inch to 5/8 inch in length in the specimen containing TiX1-11B discussed above. While the slip direction was not determined, the deformation markings established that $\{10\bar{1}0\}$ slip had

occurred. The critical resolved shear stress for prismatic glide was determined to be between 6700 and 8300 g./mm.^{2}. The oxygen content of the end of the specimen adjacent to TiX1-11A was found to be 0.26 per cent and the hydrogen 0.012 per cent.

TiX1-12A

TiX1-12A was a 1-1/4-inch long crystal, of dimensions 0.210 inch x 0.172 inch, having a cross-section area of 0.0361 square inch. The deformation in tension, as established by the slip markings and the crystal rotation (Figure 20), was clearly by $\{10\bar{1}0\}$ slip in the $\langle 11\bar{2}0\rangle$ direction.

TiX1-12A had a Rockwell A hardness of 54.5 measured at the specimen end. The critical resolved shear stress for prismatic glide was calculated to be 11,700 g./mm.^{2}. Analysis disclosed the presence of 0.24 per cent oxygen and 0.010 per cent hydrogen in the region where the hardness was determined. In the middle of TiX1-12A the values were 0.16 per cent oxygen and 0.008 per cent hydrogen.

Discussion

The crystals in this group (TiX1-10A, 3D, 9A, 9B, 11A and 12A) have angles between the basal plane and the specimen axis of from about 5° (TiX1-11A) to about 35° (TiX1-9A). All of of them deformed entirely by prismatic slip on the most highly stressed plane and, where the direction of rotation was determined,

in the close-packed direction. The critical resolved shear stress for prismatic glide varied from about 4500 g./mm.^{2} to to about 11,700 g./mm.^{2}, the variation possibly being connected with the degree of oxygen contamination in the individual specimens.

While some interesting areas in the orientation distribution were not covered by the existing specimens, the general situation regarding the relationship between crystal orientation and tensile deformation seems established. Crystals having angles of less than 40° between the basal plane and the stress axis deform readily by prismatic slip at relatively low specimen stresses. Between about 40° and about 60° deformation is by basal glide unless restraints are present. In the latter case $\{11\bar{2}1\}$ twinning becomes the predominant mechanism. As the angle increases above about 60°, basal slip becomes less important; $\{10\bar{1}2\}$ twinning develops; and finally at 75° $\{10\bar{1}2\}$ twinning apparently becomes the only operative mechanism. Much higher specimen stresses are required to deform the basal glide-twinning group than are needed for prismatic glide.

While it could not be established clearly from the present work, it is apparent that for specimens of equal purity deformed in the same manner a boundary must exist, somewhere near a basal plane-stress axis angle of 40°, between the two

major zones of deformation mechanism. The rotation due to basal slip in TiXl-6 could not have continued indefinitely since a consequence of this rotation is to bring the crystal orientation into the region where prismatic slip is the preferred mechanism. The behavior in the region of the kink is understandable on this basis.

The sharp change in deformation mechanism and load requirements between the two deformation regions may have important effects on the fabricating properties of metal of different orientation textures.

Compression Tests

TiXl-17

The orientation of this specimen is shown in Figure 1. The cross-section area was 0.0386 square inch. When stressed to 26,200 p.s.i. the crystal formed $\{11\bar{2}2\}$ twins which increased in size and number as the load was increased further. At 39,200 p.s.i. a compression of 2.5 per cent was noted. A photomicrograph (Figure 32) taken after a stress of 36,400 p.s.i. had been applied shows prominent $\{11\bar{2}2\}$ twins and some evidence of basal glide. All markings were observed on at least two surfaces.

The oxygen content adjacent to TiXl-17 was 0.039 per cent and the hydrogen was 0.010 per cent.

It seems evident that basal slip can occur in

compression but is not likely to be an important mode of deformation under restraint conditions. Actually the situation here differs from that noted in TiXl-3A in tension in that twinning preceded basal slip in compression and followed it in tension.

TiXl-8

The orientation of this crystal was very similar to that of TiXl-12A which was stressed in tension. The cross-section area was 0.0293 square inch. When the present specimen was compressed first permanent set (0.05 per cent) was observed after a stress of 21,200 p.s.i. was applied. Additional loadings brought the stress to a final value of 37,600 p.s.i. and a permanent set of 4.5 per cent.

The early deformation took the form of $\{10\bar{1}0\}$ slip around a bend plane near one end of the specimen. The bend plane appeared to have the indices $\{11\bar{2}0\}$. As the deformation progressed the $\{10\bar{1}0\}$ slip markings became more numerous and extended over the entire specimen length. The direction of slip was not determined but undoubtedly it was $\langle 11\bar{2}0\rangle$ in the opposite sense to tension slip.

TiXl-8 contained 0.18 per cent oxygen. The hydrogen content was 0.008 per cent. The critical resolved shear stress for prismatic slip in the close-packed direction was about

6500 g./mm.^{2}, which is in reasonable agreement with the values obtained in tension.

TiX1-1A, 1B

Like TiX1-2, the present specimen was from a preliminary lot made from strip rolled from ingot Ti-138. The crystals were about 1/4 inch in length and both were contained in a single compression specimen.

When compressed with a load of 319 pounds, both crystals showed markings positively identified as due to $\{10\bar{1}0\}$ slip. Some $\{10\bar{1}2\}$ twins were seen in TiX1-1A at the boundary between the two crystals.

The number of compression specimens tested was too small to permit conclusions. It may be stated, however, that outside of the change in type of twinning required to accommodate extension or compression of the crystal, no evidence was seen in these limited tests which would indicate any great difference between the tension and compression behavior of titanium.

The work carried out here may be summarized briefly in the statement that slip on the $\{0002\}$ and $\{10\bar{1}0\}$ planes and twinning on the $\{11\bar{2}1\}$, $\{11\bar{2}2\}$ and $\{10\bar{1}2\}$ planes were observed. The only other work along these lines known to the writers was that carried out by Sylvania Electric Products, Inc., for the United

States Air Force*. These workers confirmed the above observations excepting that they did not report {0002} slip and did report {1011} slip.

IV. ROLLING AND ANNEALING TEXTURES

Introduction

In order to obtain equal properties in all directions in a metal piece it would be necessary to provide a completely random orientation of the crystal grains of which it is composed. Under most conditions of deformation or heat treatment random orientation is not attained. On the contrary, a majority of the grains will assume a narrow range of orientations called a texture. The study of the textures developed in a new metal or alloy by various deformations and heat treatments is an important part of the evaluation of the material.

In the present work studies have been made of the textures produced in iodide quality titanium by cold strip rolling to various reductions and by annealings at various temperatures subsequent to the rolling. A few attempts at hot rolling were made.

*C. A. Dube, F. D. Rosi, B. H. Alexander and F. C. Perkins, Third Quarterly Progress Report, May 1, 1951 to August 1, 1951, U.S.A.F. Contract AF-33-038-16031.

Metal Used

With the exception of the specimens from ingot Ti-116 previously referred to in the section on recrystallization, all of the specimens were taken from strip rolled from iodide titanium ingot Ti-138. Ti-138 was a 2-1/2 pound arc melted ingot prepared from portions of several iodide titanium hairpins. The melting, machining and forging practices used were similar to those previously described for Ti-116 and Ti-182. After final machining subsequent to forging, the bar was 1-1/16 inch x 1-1/16 inch in cross-section. Analysis of strip, cold rolled from this bar, gave the following results:

Oxygen	0.03%	Molybdenum	0.0009%
Nitrogen	0.002%	Aluminum	0.015%
Hydrogen	0.014%	Manganese	0.02%
Carbon	0.06%	Tin	0.002%
Iron	0.01%	Lead	0.0035%
Copper	0.005%	Magnesium	0.0025%

Pole Figure Procedure

The conventional X-ray procedure for determining the orientations present in the surface of a sample are tedious and not well adapted to the obtaining of quantitative data. A procedure developed by Schulz*, modified in some respects for use in the present work, proved more useful since pole figures with considerable, reasonably quantitative detail could be produced quite rapidly.

*L. G. Schulz, J. App. Physics, 20, 1030, November 1949.

The essential feature of the Schulz procedure is the rotation of a 1-inch diameter flat disc of metal about a diameter at the Bragg angle θ to a thin, nearly rectangular X-ray beam impinging on the surface. The diffracted beam is recorded by a Geiger counter positioned at the proper angle (2θ) for the plane under investigation. In practice Schulz made a series of exposures, rotating the specimen about the diameter axis in steps of 5 degrees. After a traverse of 180° had been taken the specimen was turned about its own axis through some selected angle, such as 45° or 90°, and the traverse was repeated. In the present work traverses were taken at every 15°.

During a single 180° traverse every crystal in the surface whose orientation is suited to diffraction from the selected plane will report in the Geiger counter as the diametric rotation brings it into the required angular position. By taking a sufficient number of traverses about different diameters practically every crystal in the surface of the specimen is brought into its reflecting position. Consequently, the relative numbers of crystals of like orientation and their angular relationships to the surface and to the rolling direction can be established.

Three modifications were made of the basic Schulz procedure to adapt it to the work under this contract. In

one of these a synchronous motor drive was fitted to the specimen holder to provide for continuous instead of step-wise rotation about the diameter axis. Secondly, in order to provide a permanent record and to permit the calibration correction discussed immediately below, arrangements were made to convert the Geiger counter signals to relative intensity curves on a Brown Instrument Company strip chart recorder. The drum rotation of the recorder was synchronized with that of the specimen.

In the original Schulz procedure the slit dimensions defined a beam which impinged upon the exposed surface of the 1-inch diameter disc. It became expedient in the present work to overcome the low reflection intensities from the titanium samples by enlarging the beam. This was needed to achieve a sensitive differentiation between concentrations of orientations. A consequence of this procedure was the loss of part of the beam in certain positions of the disc. Since the procedure depends upon the integration of the entire beam in terms of individual crystal reflections, correction for the error due to these losses was necessary.

It was found possible to develop a series of calibration curves with which to correct the primary data. These were obtained from fine grained, randomly oriented copper

(actually compacts of fine copper powder were used). While a titanium specimen having complete orientation randomness was not attained, some specimens were sufficiently so to permit a reasonable comparison with the copper standards. Such comparisons indicated little error due to the use of copper.

All of the pole figure work was carried out by the above method. Copper radiation was used. The equipment was mounted on a North American Phillips low-angle spectrometer. The time required for a single traverse was about 20 minutes.

In practice the calibration net was placed over the chart for any given traverse and angular positions and intensities, which are a measure of the number of crystals of that orientation, were picked off and plotted on the pole figure chart. As many as 1600 points were plotted in some instances. The drawing-in of the actual pole figure was done in much the same manner as the drawing of topographic maps. The plotting and drawing of a pole figure chart required about ten hours.

All important data were plotted. In some instances, however, experienced inspection of the original recorder chart was enough to determine with sufficient certainty what type of texture existed in the specimen in question.

While any plane capable of giving reasonably strong reflections could be used, it was found by experience that the

{10$\bar{1}$1} plane gave best results. In critical cases {0002} and, sometimes, {10$\bar{1}$0} pole figures also were obtained in order to be more certain. In all, charts were obtained for 157 pole figures, not all of which were plotted.

Experimental Work

The pole figure texture work can be summed up rather simply with the statement that two principal textures were found. While these could be brought to sharp definition under appropriate conditions, most of the rolling and annealing conditions resulted in mixtures of the two textures with one or the other somewhat predominant. Actually, in such instances, there probably were a sizable number of grains present which were not oriented in either texture.

The Cold Rolled Texture

The texture produced in pure titanium by severe cold rolling in one direction is shown in Figure 33 for the {0002} plane; in Figure 34 for the {10$\bar{1}$1} plane; and in Figure 35 for the {10$\bar{1}$0} plane. The specimen involved was rolled 96.5 per cent on 3-inch diameter rolls from the forged, machined Ti-138 bar.

The outstanding characteristics of the {0002} pole figure are: (1) the relative absence of grains oriented with the basal plane parallel to the strip surface, (2) the high

concentrations of crystals tilted in the transverse direction, and (3) the existence of minor peaks in the approximate locations of {10$\bar{1}$1} twins of the principal orientations. There is no evidence, incidentally, that {10$\bar{1}$1} twinning can occur in titanium.

The {10$\bar{1}$1} pole figure confirms the indications of the basal plane figure and establishes that the <11$\bar{2}$0> close-packed direction in the basal plane is disposed transversely. The {10$\bar{1}$0} pole figure has relatively little detail but it does confirm the other two.

The Annealed Texture

When severely cold rolled pure titanium is annealed just below the transformation temperature, the only change in texture that occurs is that the <11$\bar{2}$0> close-packed direction in the basal plane is now disposed in the longitudinal or rolling direction (see Figures 36, 37 and 38). The minor peaks have assumed the {10$\bar{1}$1} twin positions of the new major peaks.

Both of these major textures have been found by other workers using conventional X-ray techniques. As far the writers are aware, however, the existence of the minor peaks has not previously been detected by those methods. The following references to previous work are at hand:

H. T. Clark, Jr., J. Metals, Sept. 1950, p. 1154-1156, Trans. A.I.M.E., 188, 1154-1156, 1950.

M. K. Yen and J. P. Nielsen, Discussion of Clark's paper, J. Metals, July 1951, p. 549-550.

M. K. Yen, WAL Report No. 401/14-3, March 20, 1950 - Interim Report to Watertown Arsenal.

C. J. McHargue, Progress Reports from May 1951 to May 1952 under Contract AF30(038)-19574 United States Air Force.

Similar results were obtained on zirconium by R. K. McGeary and B. Lustman (J. Metals, November 1951, p. 994-1002).

The cold rolled texture places a $\{11\bar{2}4\}$ plane approximately parallel to the strip surface and a $\langle 10\bar{1}0 \rangle$ direction parallel to the rolling direction. While the designation has no crystallographic significance, it is convenient to refer to this texture as a $\{11\bar{2}4\}$ $\langle 10\bar{1}0 \rangle$ texture. Similarly, the annealed texture may be referred to as a $\{20\bar{2}5\}$ $\langle 11\bar{2}0 \rangle$ texture.

From the practical standpoint it is of interest to attempt to interpret these textures in terms of directional properties. While time did not permit direct measurements in the present work, data from earlier studies here show that neither texture results in large directional differences. Actually, differences of the order of 2 to 3 per cent were observed between longitudinal and transverse tensile strength for either texture. Since both textures place $\{10\bar{1}0\}$ planes in

position for relatively easy deformation by slip in either the longitudinal or the transverse direction, these observations are not unexpected.

Surface Texture Effects

Since the penetration of the X-ray beam is relatively shallow under the conditions used in this work, the data obtained refer only to the surface exposed to the beam. As in all texture work on any material, the question arises as to whether the textures under the surface are the same or different. The experience here has been mixed. Earlier work, for example, involving 65 per cent cold rolling of hot rolled, annealed commercial purity strip indicated no differences between the surface and the subsurface layers. In the present work definite differences were observed in some instances.

The cold rolled texture shown in Figures 33-35 was duplicated in a second and a third rolling of sections of Ti-138. A fourth section rolled to the same 96.5 per cent total reduction in the same rolls gave the very different texture shown in Figures 39 and 40. The texture in this case is the annealed one with some transverse distortion which tends to form transverse ridges between pairs of peaks. Removal of 0.006 inch of surface metal by etching disclosed the perfectly normal cold rolled texture shown in Figure 41.

The metal reduced 15, 30, 45 and 60 per cent from Ti-116 for the recrystallization series showed similar effects. A distorted $\{20\bar{2}5\}\langle 11\bar{2}0\rangle$ texture similar to that in Figures 39 and 40 appeared quite strongly at the surface after 60 per cent reduction and progressively less strongly after 45 and 30 per cent. It was not observed after 15 per cent reduction.

Removal of 0.005 inch from the surface of the 60 per cent cold rolled specimen revealed that the subsurface material had the characteristic $\{11\bar{2}4\}\langle 10\bar{1}0\rangle$ cold rolled texture.

In the rolling of the Ti-138 specimen from which Figure 39 and 40 were derived, specimens were removed after 60, 80 and 90 per cent reduction as well as after the final 96.5 per cent reduction. The texture of the machined, hot-forged ingot was fairly random. After 60 per cent cold reduction a moderately strong $\{11\bar{2}4\}\langle 10\bar{1}0\rangle$ texture was observed at the surface. At 80 per cent reduction the distorted $\{20\bar{2}5\}\langle 11\bar{2}0\rangle$ texture had started to develop. This became increasingly pronounced at 90 per cent reduction and very pronounced at 96.5 per cent (Figures 39 and 40).

While no subsurface examinations were made of the three earlier specimens of 96.5 per cent reduction, it is concluded from their similarity to the subsurface structure in the fourth specimen that the surface texture was that of the main

mass of the metal. Confirmation of this conclusion is available in the fourth progress report issued by C. J. McHargue (loc.cit.). McHargue found the {1124} <10$\bar{1}$0> texture at and below the surface after 90 per cent cold reduction.

While it seems well established that a specific type of surface texture can develop during the cold rolling of titanium, no clear explanation for it has been forthcoming. There are some unproved indications, however, that development of the surface texture is facilitated by elevation of the strip temperature.

Effects of Annealing

A complete study of the effects of annealing temperature upon texture was carried out on the fourth 96.5 per cent cold reduced Ti-138 specimen after the surface texture was removed by etching away 0.006 inch of metal. The as-rolled subsurface texture was that shown in Figure 41. This specimen was annealed successively at 400, 450, 500, 550, 600, 650, 800 and 900°C. Up to 600°C. the specimen was sealed in vacuo in Pyrex glass; Vycor was used for the higher temperatures. The anneals were for one hour at temperature. The specimen was etched for one-half minute in 87H_2O-12HNO_3-1HF after each anneal. Previous tests had shown that the grain size achieved during such an annealing sequence was the same for any

temperature as that obtained in a single anneal at that temperature.

The {10$\bar{1}$1} pole figures obtained in this annealing study are presented as Figures 42 through 49. Comparison of Figure 41 with Figure 48 shows that the original fairly sharp {1124} <1010> cold rolled texture had been converted to a sharp {20$\bar{2}$5} <11$\bar{2}$0> texture by annealing at 800°C. It is of interest to trace the transition from the one texture to the other through the annealing sequence.

Annealing at 400°C. caused only a sharpening of the as-rolled texture. Evidence of definite texture change started with the 450°C. anneal, which temperature is very close to the 475°C. assumed earlier as the temperature at which all distorted grains have been recrystallized. At 600°C., where minimum hardness was observed on the recrystallization curves and where a somewhat equiaxed grain structure of modest grain size was found, the two primary textures clearly were present. As the annealing temperature was increased, the {20$\bar{2}$5} <11$\bar{2}$0> texture became increasingly predominant. There was no apparent discontinuity in the changes in texture with temperature.

It is evident from this series (1) that the {20$\bar{2}$5} <11$\bar{2}$0> texture is not a unique product of recrystallization and (2) that the process of grain growth is selective, the grains

of the {20$\bar{2}$5} <11$\bar{2}$0> orientation growing at the expense of those of the {11$\bar{2}$4} <10$\bar{1}$0> and probably other orientations.

A similar annealing series was carried out on a 96.5 per cent cold reduced specimen showing the special surface texture. The pole figures and charts for this series show that as the temperature was increased the undistorted {20$\bar{2}$5} <10$\bar{1}$0> texture (Figures 36, 37 and 38) became increasingly predominant until at about 800°C. the conversion was essentially complete. Evidently the grains in the principal {20$\bar{2}$5} <10$\bar{1}$0> orientation absorbed the others during grain growth just as they did in the subsurface material.

A detailed discussion of the fundamental implications of these observations is not germane to the primary objectives in this present work. However, the writers feel that these discoveries should add measurably to the understanding of the recrystallization and grain growth processes.

When viewed in the light of the observations on mechanisms for plastic deformation, the present information may offer a possible explanation for some of the anomalies observed in the recrystallization curves. The sharply defined {20$\bar{2}$5} <10$\bar{1}$0> annealing texture developed at 825°C. places a preponderance of the grains in orientations which do not readily permit deformation by {10$\bar{1}$0} slip under a compressive force

normal to the strip surface. High hardness values should not be unexpected when the indentation is made on this surface. At 600°C., however, in addition to the partially developed {20$\bar{2}$5}<10$\bar{1}$0> and {11$\bar{2}$4} <11$\bar{2}$0> textures there probably were many grains rather randomly oriented. Any of these which were in a position to deform by prismatic slip would do so rather easily. A lower hardness value could result.

Annealing in the Beta Range

In both of the series just described the last anneal was at 900°C. The texture which resulted is shown in Figure 49. There was no essential difference between the two specimens.

In spite of the fact that three beta orientations are possible from a single alpha grain and six alpha orientations are possible from a single beta grain, the result of the 900°C. anneal was not to randomize the alpha texture but rather to sharpen slightly the {20$\bar{2}$5} <10$\bar{1}$0> texture produced at 800°C. This may be taken to indicate a reproducible selection of particular alpha and beta orientations during the transformation. C. J. McHargue (loc.cit.) has obtained similar results. The sharpening conceivably could have been due to reaching a higher temperature in the alpha range during heating to 900°C. than had been used in the annealing studies.

Commercially Rolled Titanium

The present work was limited essentially to strip rolling at or very near room temperature. There is evidence, however, that textures other than those disclosed above can be obtained by other rolling practices. An example of this is shown in Figures 50 and 51. The material was purchased in rolled form from a commercial supplier and was reported to have been melted in carbon, hot forged, hot rolled, annealed and pickled.

The texture places the basal plane approximately in the plane of the strip with a greater deviation in the transverse direction than in the longitudinal. The $\langle 11\bar{2}0 \rangle$ direction tends to line up in the rolling direction. In a sense this is the $\{20\bar{2}5\} \langle 11\bar{2}0 \rangle$ texture with the principal peaks brought near the center of the basal plane pole figure.

Samples of this strip were cold rolled to a minimum reduction of 65 per cent. Pole figures taken at steps in this series reveal (1) that the first effect of the cold rolling was to move the crystals from the center of the basal pole figure and (2) to start developing the typical $\{11\bar{2}4\} \langle 10\bar{1}0 \rangle$ cold rolling texture.

It may be said of the present work that regardless of the starting texture or the roll diameter or any other variable, possibly excepting temperature, the effect of cold

rolling was to develop the {11$\bar{2}$4} <10$\bar{1}$0> texture. Where the degree of cold reduction was small; e.g., 30-60 per cent, this texture did not always become strongly defined but it clearly was developing. Similarly, the effect of annealing was to produce the {20$\bar{2}$5} <11$\bar{2}$0> texture. At temperatures in the range 500 to 700°C. this texture did not always become marked but it was definitely developing.

In the range of reductions and annealing temperatures to be expected in commercial practice; e.g., 30-60 per cent reduction, 600-700°C. annealing, it may be that sharp textures will not always be found. Also, it is to be expected that, in general, mixtures of the two textures together, probably, with other minor orientations will be found

Hot Rolling Tests

A few attempts were made to produce hot rolled textures. The work never progressed beyond the preliminary stages and no truly hot rolled samples were produced.

V. RATIONALIZATION OF TEXTURES

It was the intent when this work was started to attempt to rationalize the textures which were found. This was not possible. The discovery that there are five possible modes of deformation involving 24 systems, together with the evidence

of highly selective oriented grain growth, makes it impossible at this time, at least, to attempt a rationalization.

VI. DIRECTIONAL PROPERTIES

Since the textures developed in this work were such that strong directionality in properties would not be expected, the available time and money were expended on extending the single crystal deformation and the texture studies as far as possible.

ACKNOWLEDGMENTS

A research of this type carried out in an industrial research laboratory involves the close cooperation of many men. While it is difficult to single out individuals, the writers would like to make particular acknowledgment of the contributions of the following:

S. R. Dunbar in the preparation of single crystals and the microstudies.

C. B. Tennant in all phases of the X-ray work.

A. G. Lesko, III, in the plotting of the numerous pole figures.

B. A. Shippy in the determination of oxygen by the vacuum fusion method

Table 1 - Identification and Lengths of Large Titanium Grains

Crystal No.	Approximate Length - Inch	Crystal No.	Approximate Length - Inch
TiX1-1A	1/4	TiX1-11A	1/8-5/8(c)
TiX1-1B	1/4	TiX1-11B	3/4
TiX1-2	3/8	TiX1-11C	1/8-1/4(c)
TiX1-3A)		TiX1-11D	1/4-1/2(c)
TiX1-3B)(a)	1	TiX1-12A	1-1/4
TiX1-3C)		TiX1-12B	5/8
TiX1-3D	1/8-1/2(c)	TiX1-13	2/3
TiX1-3E	5/8(d)	TiX1-15A	3/8
TiX1-3F	1/4-3/8(c)	TiX1-15B	3/8
TiX1-6A)(b)	1-3/4	TiX1-15C	3/8
TiX1-6B)		TiX1-15D	3/8
TiX1-6C	1/4	TiX1-15E	3/8
TiX1-7	3/4	TiX1-16	1/2
TiX1-8	1-1/4	TiX1-17	5/8
TiX1-9A	1-5/8	TiX1-18	1-1/2(e)
TiX1-9B	5/16	TiX1-19	1-1/2
TiX1-10A	1-3/8	TiX1-20	1
TiX1-10B	5/8	TiX1-21	7/8

(a) 1 grain - 3 back reflection patterns taken at different points.

(b) 1 grain - 2 back reflection patterns taken at different points.

(c) Boundary not normal to specimen axis - grain length variable.

(d) Grain did not go completely through specimen.

(e) Grain almost went completely through specimen - surface polishing would remove thin extraneous layer on one side.

Note: Crystals having a common TiX1 number all were in the same specimen.

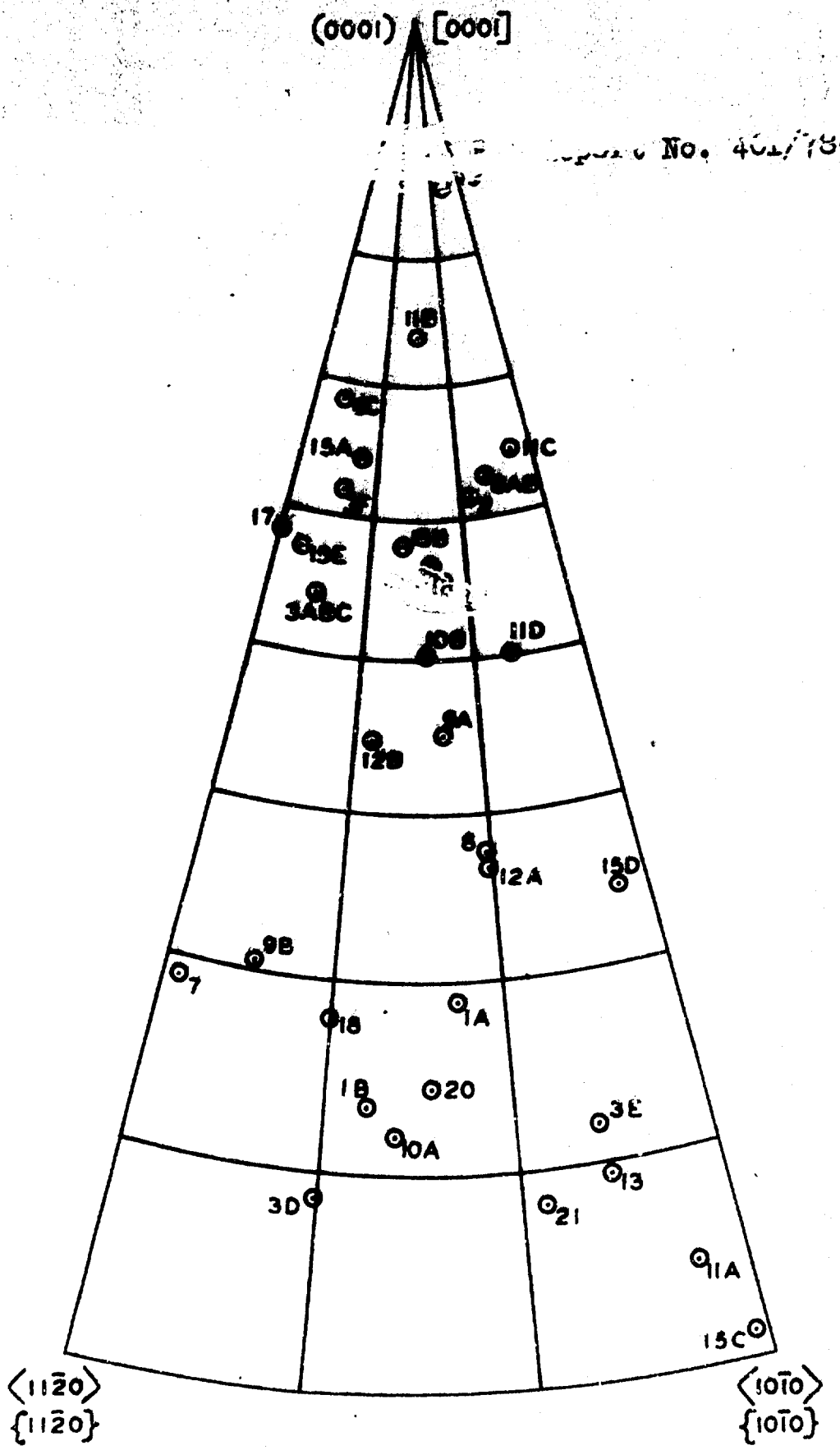


Figure 1 - Orientations of Useful Large Crystals of Titanium
(Points Indicate Stereographic Projections of Specimen Axes)

Table 2 - Analyses of Iodide Titanium and Ingot Ti-182

Sample	Analyzed Content - Per Cent												
	N_2	C	O_2	H_2	Mn	Fe	Al	Mo	Pb	Ni	Cu	Sn	Mg
IT-247	.002	(a)	(a)	(a)	.0025	.005	.01	.001	.0035	.0025	.0015	.001	.001
IT-250	.004	(a)	(a)	(a)	.0075	.007	.0085	.0007	.0065	.002	.0015	.0055	.0007
IT-251	.002	(a)	(a)	(a)	.006	.0045	.009	.001	.005	(b)	.012	.001	.0012
IT-252	.002	(a)	(a)	(a)	.035	.0055	.013	.001	.0055	(b)	.0035	.0008	.0015
Ti-182	.002	.05	.027	.0076	.02	.02	.03	(b)	.006	(b)	.015	.008	.006

(a) Not determined.

(b) Not detected.

Note: Nitrogen, on turnings from ingot, by modified Kjeldahl method.
Carbon, on turnings from ingot, by combustion.
Oxygen and Hydrogen, on piece of forged, machined bar, by vacuum fusion.
Metallic elements, on turnings from ingot, spectrographically.

Table 3 - Analyses of Ingot Ti-116 and of Specimens Processed From It

Sample	Segment	Anneal °C.	Analysis - Per Cent O2	N2	H2	C	Mn	Fe	Al	Mo	Pb	Ni	Cu	Sn	Mg	W
Ingot Top(d)	--	--	(a)	.018	(a)	.09	.047	.0092	.015	.0034	.0028	(b)	.0023	.0027	(a)	<.005
Ingot Top(c)	--	--	.20	.025	(a)	.17	.037	.0054	.013	.0019	.0029	(b)	.0025	.0030	(a)	<.005
Bottom (d)	--	--	.058	.011	(a)	.05	.028	.0055	.0076	.0006	.0030	.0020	.0037	.0034	(a)	<.005
Side(c)	--	--	(a)	.010	(a)	.06	.043	.0065	.0088	.0020	.0038	.0034	.0016	.0027	(a)	<.005
FH-276	I	RT	.050	(a)	.011	(a)										
FH-277B	II (Bot.)	RT	.032	.003	.012	.02										
FH-277T	II (Top)	RT	.046	(a)	.013	(a)										
FH-272	III	RT	.032	.002	.013	.04										
FH-273	IV	RT	.032	.0025	.016	.03	.015	.006	.015	.0025	.004	(b)	.004	.0015	.0015	(b)
FH-308	IV	400	.033	(a)	(a)	(a)										
FH-310	IV	600	.023	(a)	(a)	(a)										
FH-312	IV	806	.030	(a)	(a)	(a)										
FH-313	IV	871	.10	.002	.022	.025	.015	.009	.015	.0025	.005	(b)	.005	.0015	.0015	(b)
FH-286	I	871	.11	(a)	(a)	(a)										

Table 3 (Contd.)

(a) Not determined.

(b) Not detected.

(c) Chips from finishing cut in turning

(d) Chips from rough machining cut - outer surface of ingot.

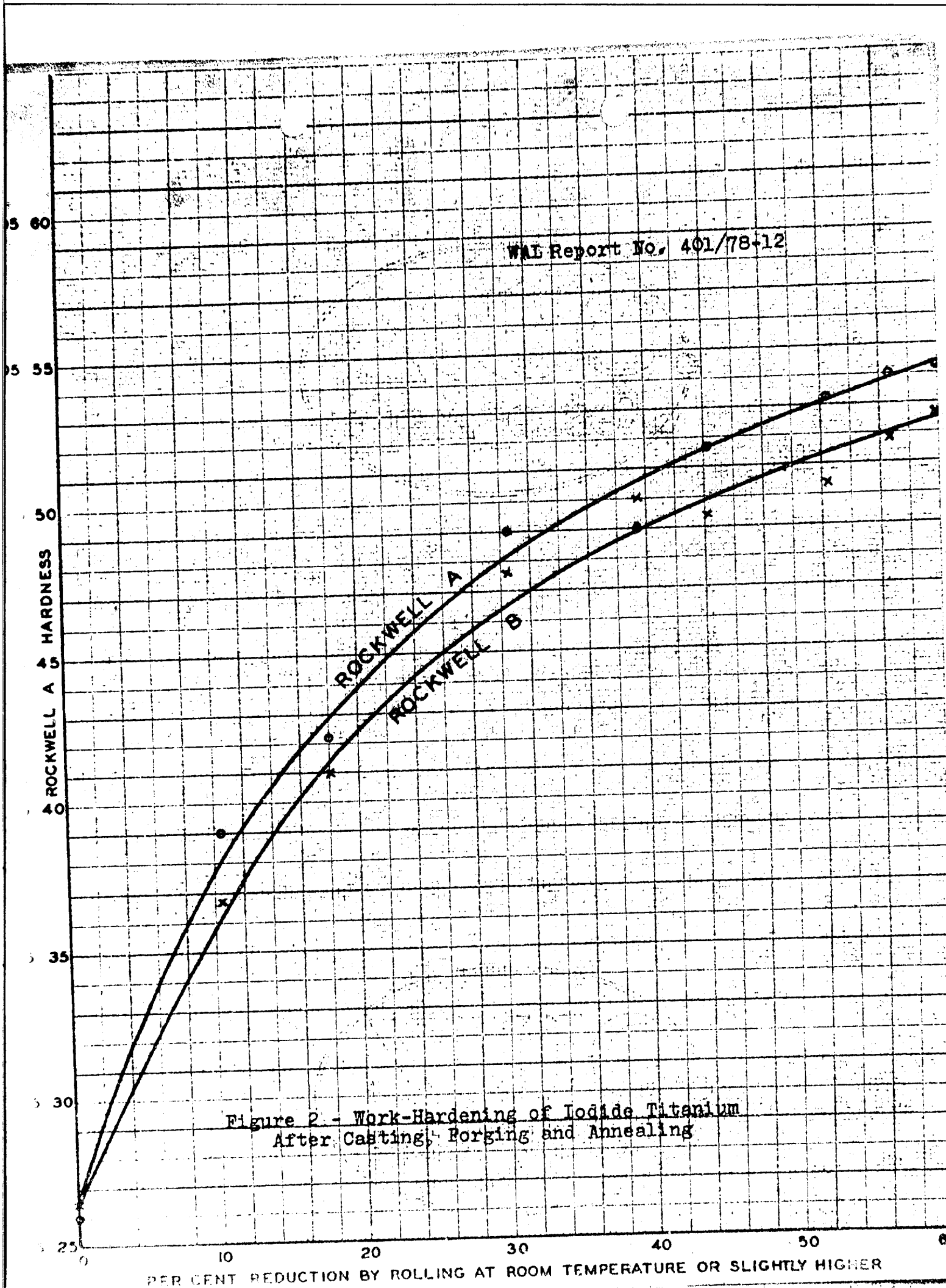


Figure 2 - Work-Hardening of Iodide Titanium After Casting, Forging and Annealing

Table 4 - Work Hardening Data - Ti-116

Pass No.	Gauge Inch	Per Cent Reduction	Schedule	Hardness - R_A I	II-B	II-T	III	IV	Hardness - R_B I	II-B	II-T	III	IV
0	.421	0		29	25.5	34.5	26	32	41.5	35	51.5	38	46
1	.392												
2	.377	10.4		40	39	37	39	41	65	55	64	58.5	65.5
3	.362												
4	.345	18.0		46	42	44	42.5	44	74.5	67.5	74.5	67	75
5	.322												
6	.293	30.5		51	48	50.5	49	50	82.5	79.5	84	81	82
7	.273												
8	.254	39.4		53	49.5	53	49	51.5	87	83	87	85	88
9	.237	44.3		54.5	50.5	52.5	52	53	89	86	89.5	84	90
10	.218												
11	.200	52.4		55	--	--	53	55	93	--	--	86	92
12	.182	56.7		56	--	--	54	--	92	--	--	88.5	--
13	.1675	60.1		--	--	--	54	--	--	--	--	90.5	
Anneal 600°C.				34	34	37	33	38	--	--	--	--	--
0	.183	0	I	34									
	.137	25		47									
	.091	50		54									
Anneal 600°C.				34									
	.063	31.5		51									
	.0455	50		55									
Anneal 600°C.				30									
	.023	50		51									
Anneal 600°C.				28									
	.0195	15*		40.5									

*Final reduction.

Table 4 (Contd.)

Pass No.	Gauge Inch	Per Cent Reduction	Schedule	Hardness - R_A I	II-B	II-T	III	IV	Hardness - RB I	II-B	II-T	III	IV
0	.236	0	II-B		34								
	.164	30			48								
	.117	50			53.5								
Anneal 600°C.					36								
	.075	36			54								
	.059	50			55								
Anneal 600°C.					35								
	.029	51			53								
Anneal 600°C.					30								
	.0205	29*			47.5								
0	.233	0	II-T			37							
	.175	25				49							
	.116	50				54							
Anneal 600°C.						36							
	.077	35				53							
	.059	50				56							
Anneal 600°C.						35.5							
	.029	51				54							
Anneal 600°C.						32							
	.020	30.5				49							

*Final reduction.

Table 4 (Contd.)

Pass No.	Gauge Inch	Per Cent Reduction	Schedule	Hardness - R_A I	II-B	II-T	III	IV	Hardness - R_B I	II-B	II-T	III	IV
0	.165	0	III				33						
	.113	31.5					48						
	.074	55					54						
Anneal 600°C.							33						
	.046	39					52						
	.037	50					53						
Anneal 600°C.							30						
	.020	46*					50						
0	.200	0	IV					38					
	.145	27.5						51					
	.100	50						55					
Anneal 600°C.								39.5					
	.067	33						53					
	.050	50						55					
Anneal 600°C.								35.5					
	.020	60*						52					

*Final reduction.

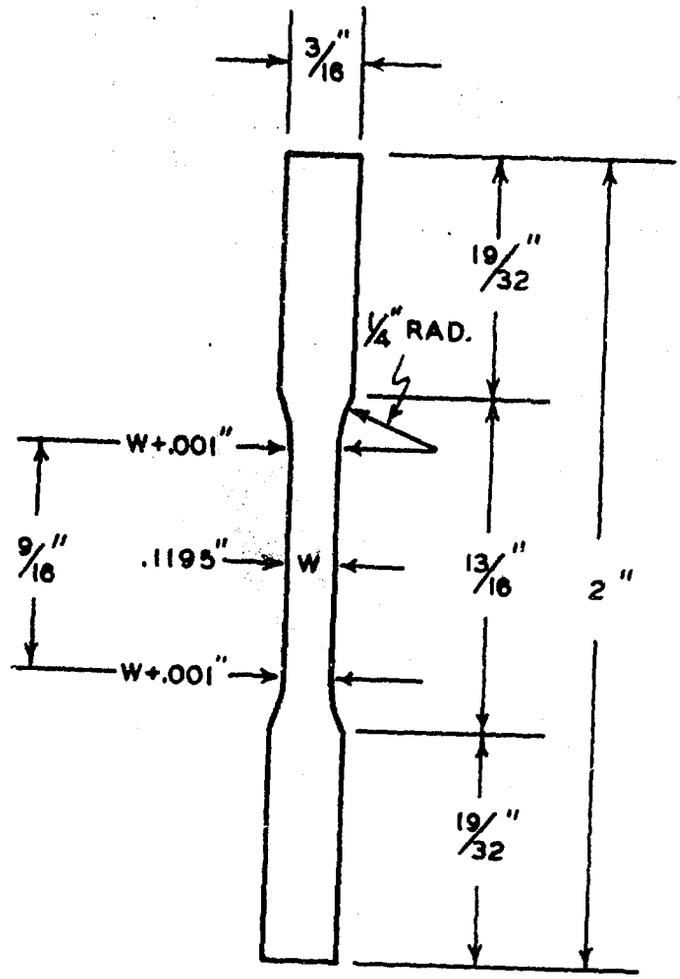


Figure 3 - Tension Test Specimen

Table 5 - Properties After Final Annealing

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness R_A	Hardness R_B
Ti-116 FH276-1	I	15	RT	63900	20	43	69
-2				56300	26	41	66
-3				57600	28	40	63
-4				62500	24	43	69
-5				59200	24	41	67
Average				59900	24	41.5	67
Ti-116 FH280-1	I	15	307	54700	28	41	66
-2				49400	26	42	69
-3				59100	28	42	67
-4				58600	28	42	70
-5				59000	24	43	69
Average				56200	27	42	68
Ti-116 FH281-1	I	15	400	55100	32	40	62
-2				52700	32	40	65
-3				53200	30	41	65
-4				52600	30	40	65
-5				54600	30	40	68
Average				53600	31	40	65
Ti-116 FH391-1	I	15	450	51400	34	39*	61**
-2				52400	34	40*	59**
-3				51300	34	40*	54**
-4				51400	36	39*	61**
-5				51600	36	40*	60**
Average				51600	35	40	59
Ti-116 FH282-1	I	15	500	51400	40	38	66
-2				50400	38	39	65
-3				50600	38	38	65
-4				51600	36	40	65
-5				51400	36	40	66
Average				51100	38	39	65

*Slight anvil mark.
**Definite anvil mark.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness R_A	Hardness R_B
Ti-116 FH395-1	I	15	550	47400	42	37**	55**
-2				47600	42	36**	59**
-3				47500	46	36**	60**
-4				47200	48	37**	60**
-5				47800	46	35**	56**
Average				47500	45	36	58
Ti-116 FH283-1	I	15	600	40600	60	29	32
-2				40600	60	29	40
-3				40800	58	30	42
-4				41000	58	30	38
-5				42300	56	30	44
Average				41100	58	30	39
Ti-116 FH284-1	I	15	705	42300	58	31	41
-2				40900	60	31	45
-3				42500	58	32	47
-4				40200	64	31	50
-5				41100	60	31	49
Average				41400	60	31	46
Ti-116 FH285-1	I	15	806	41700	50	30**	53**
-2				40200	60	28**	53**
-3				42500	52	30**	56**
-4				41000	50	31**	54**
-5				41700	48	33**	56**
Average				41400	52	30	54
Ti-116 FH286-1	I	15	871	43700	38	33	60
-2				43700	40	36	61
-3				42200	38	35	62
-4				43300	36(a)	35	59
-5				45400	36	38	62
Average				43900	38	35.5	61

*Slight anvil mark.
**Definite anvil mark.
(a) Broke outside of gauge marks - value not included in average.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness R_A	Hardness R_B
Ti-116 FH277B-1 (b)	II	30	RT	78000	14	48	79
-2				76000	14	47	78
-3				77200	14	48	80
-4				75700	16	47	79
-5				78900	16	47	80
Average				77200	15	47	79
Ti-116 FH289B-1	II	30	307	69500	24	47	77
-2				68400	20	46	77
-3				68500	24	46	75
-4				70900	20	46	78
-5				72000	22	47	78
Average				69900	22	46	77
Ti-116 FH290B-1	II	30	400	65800	26	45	75
-2				65600	24	45	75
-3				65400	26	45	75
-4				62000	26	44	74
-5				66100	28	45	76
Average				65000	26	45	75
Ti-116 FH392B-1	II	30	450	60000	28	43*	71**
-2				62100	30	44*	73**
-3				60000	30	44*	71**
-4				60800	26	41*	69**
-5				62100	28	45*	71**
Average				61000	28	43	71
Ti-116 FH291B-1	II	30	500	49100	42	38	62
-2				51700	44	38	65
-3				50200	44	37	62
-4				50800	44	38	63
-5				50000	40	38	61
Average				50400	43	38	63

*Slight anvil mark.
**Definite anvil mark.
(b) From bottom of original ingot.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness RA	Hardness RB
Ti-116 FH296B-1	II	30	550	43700	50	29**	42**
-2				46000	50	31**	49**
-3				46000	50	32**	52**
-4				43200	50	29**	47**
-5				46000	46	31**	51**
Average				45000	49	30	48
Ti-116 FH292B-1	II	30	600	42300	54	24	45
-2				42600	52	23	48
-3				39200	56	21	44
-4				43000	50	23	48
-5				43000	54	22	48
Average				42000	53	23	47
Ti-116 FH293B-1	II	30	705	40800	58	30	48
-2				39200	60	29	46
-3				42200	58	30	51
-4				40200	58	30	49
-5				41400	64	29	50
Average				40800	60	30	49
Ti-116 FH294B-1	II	30	806	41900	52	32**	53**
-2				42000	50	32**	56**
-3				39300	56	33**	55**
-4				38800	52	31**	49**
-5				41700	46	33**	58**
Average				40700	52	32	54
Ti-116 FH295B-1	II	30	871	40000	50	31	56
-2				41800	44	34	54
-3				42100	48	36	60
-4				41800	50	35	54
-5				44600	44	38	57
Average				42100	47	35	56

*Slight anvil mark.
**Definite anvil mark.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness RA	Hardness RB
Ti-116 FH272-1	III	45	RT	85200	12	49	82
-2				86000	12	47	82
-3				85600	14	48	82
-4				85900	12	50	84
-5				85600	12	49	83
Average				85700	12	49	83
Ti-116 FH298-1	III	45	307	63000	16	49	81
-2				76000	18	45	81
-3				62500	14	48	81
-4				74700	16	48	81
-5				77100	16	48	82
Average				70700	16	48	81
Ti-116 FH299-1	III	45	400	72300	22	47	80
-2				71800	22	47	80
-3				70900	22	47	80
-4				72000	22	46	80
-5				71900	20	47	79
Average				71800	22	47	80
Ti-116 FH393-1	III	45	450	61800	28	45*	73**
-2				63200	28	45*	74**
-3				64300	30	45*	74**
-4				64000	30	45*	74**
-5				64000	30	45*	74**
Average				63500	29	45	74
Ti-116 FH300-1	III	45	500	45500	48	32	49
-2				45300	50	32	50
-3				43800	48	33	50
-4				44000	46	33	48
-5				44600	46	32	51
Average				44600	48	32	50

*Slight anvil mark.
**Definite anvil mark.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness R_A	Hardness R_B
Ti-116 FH397-1	III	45	550	41500	50	29**	41**
-2				41700	54	30**	42**
-3				42000	50	30**	41**
-4				43100	52	29**	43**
-5				40700	48	28**	44**
Average				41800	51	29	42
Ti-116 FH301-1	III	45	600	39200	54	20	47
-2				39300	54	22	41
-3				39900	56	20	44
-4				39300	60	23	48
-5				38900	58	20	37
Average				39300	56	21	43
Ti-116 FH302-1	III	45	705	39100	64	30	48
-2				38800	58	29	49
-3				39100	58	29	49
-4				39600	56	29	50
-5				40200	60	28	46
Average				39400	59	29	48
Ti-116 FH303-1	III	45	806	38000	50	30**	54**
-2				36600	46	31**	56**
-3				37400	50	31**	42**
-4				37800	54	31**	43**
-5				36900	50	31**	51**
Average				37300	50	31	49
Ti-116 FH304-1	III	45	871	42300	48	34	59
-2				46100	50	36	59
-3				40000	42	35	60
-4				43500	48	35	61
-5				40400	42	35	61
Average				42500	46	35	60

*Slight anvil mark.
**Definite anvil mark.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness RA	Hardness RB
Ti-116 FH273-1	IV	60	RT	99200	14	53	88
-2				108800	12	53	90
-3				97600	10	53	90
-4				97500	14	53	90
-5				98800	12	53	89
Average				98800	12	53	89
Ti-116 FH307-1	IV	60	307	91600	16	52	88
-2				88800	16	53	89
-3				89100	16	52	86
-4				92300	20	53	89
-5				91600	20	52	88
Average				90700	18	52	88
Ti-116 FH308-1	IV	60	400	84100	20	51	85
-2				84900	16	51	86
-3				85200	16	51	85
-4				86000	20	51	85
-5				89100	22	52	86
Average				85900	19	51	85
Ti-116 FH394-1	IV	60	450	80000	28	49*	83**
-2				72800	28	47*	79**
-3				73800	26	48*	81**
-4				70900	28	47*	80**
-5				72300	28	47*	80**
Average				74000	28	48	81
Ti-116 FH309-1	IV	60	500	47700	46	36	58
-2				48800	46	35	59
-3				46900	48	36	62
-4				49200	48	36	61
-5				48800	46	36	58
Average				48300	47	36	60

*Slight anvil mark.
**Definite anvil mark.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness R_A	Hardness R_B
Ti-116 FH273-1	IV	60	RT	99200	14	53	88
-2				108800	12	53	90
-3				97600	10	53	90
-4				97500	14	53	90
-5				98800	12	53	89
Average				98800	12	53	89
Ti-116 FH307-1	IV	60	307	91600	16	52	88
-2				88800	16	53	89
-3				89100	16	52	86
-4				92300	20	53	89
-5				91600	20	52	88
Average				90700	18	52	88
Ti-116 FH308-1	IV	60	400	84100	20	51	85
-2				84900	16	51	86
-3				85200	16	51	85
-4				86000	20	51	85
-5				89100	22	52	86
Average				85900	19	51	85
Ti-116 FH394-1	IV	60	450	80000	28	49*	83**
-2				72800	28	47*	79**
-3				73800	26	48*	81**
-4				70900	28	47*	80**
-5				72300	28	47*	80**
Average				74000	28	48	81
Ti-116 FH309-1	IV	60	500	47700	46	36	58
-2				48800	46	35	59
-3				46900	48	36	62
-4				49200	48	36	61
-5				48800	46	36	58
Average				48300	47	36	60

*Slight anvil mark.
**Definite anvil mark.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness RA	Hardness RB
Ti-116 FH277T-1 (c)	II	30	RT	75600	14	50	84
-2				84200	18	50	83
-3				90500	16	51	86
-4				86800	16	50	85
-5				81800	14	50	83
Average				83800	16	50	84
Ti-116 FH289T-1	II	30	307	74600	14	48	80
-2				75800	20	47	79
-3				75700	20	48	81
-4				76600	20	49	82
-5				76800	20	49	81
Average				75900	20	48	81
Ti-116 FH290T-1	II	30	400	67000	28	45	78
-2				68300	28	46	78
-3				69100	26	46	79
-4				69800	26	46	78
-5				68400	26	46	79
Average				68500	27	46	78
Ti-116 FH392T-1	II	30	450	66500	28	47*	77**
-2				66300	28	43*	76**
-3				65800	28	46*	76**
-4				66300	26	46*	77**
-5				65000	26	46*	77**
Average				66000	27	46	77
Ti-116 FH291T-1	II	30	500	47700	34	38	64
-2				47500	38	39	66
-3				54700	36	38	63
-4				53400	40	39	62
-5				57500	34	40	69
Average				52200	36	39	65

*Slight anvil mark.
**Definite anvil mark.
(c) From top of original ingot.

Table 5 (Contd.)

Specimen No.	Schedule	Per Cent Final Reduction	Anneal °C.	Tensile Strength p.s.i.	Elongation Per Cent in 1/2"	Hardness R_A	Hardness R_B
Ti-116 FH396T-1	II	30	550	46600	46	32**	51**
-2				48800	48	35**	53**
-3				45800	50	34**	49**
-4				47500	48	35**	54**
-5				47800	46	35**	53**
Average				47300	48	34	52
Ti-116 FH292T-1	II	30	600	45800	46	34	48
-2				46700	46	34	51
-3				46300	48	34	51
-4				45800	46	33	50
-5				44600	46	32	48
Average				45800	46	33	50
Ti-116 FH293T-1	II	30	705	44200	50	32	55
-2				44200	56	33	56
-3				44500	60	32	56
-4				44300	52	36	57
-5				44300	58	34	56
Average				44300	55	33	56
Ti-116 FH294T-1	II	30	806	44600	60	34**	58**
-2				45100	60	35**	59**
-3				45600	54	37**	62**
-4				44600	54	37**	61**
-5				44100	56	36**	59**
Average				44800	57	36	60
Ti-116 FH295T-1	II	30	871	42300	50	36	60
-2				45300	48	37	62
-3				44900	56	36	62
-4				47200	54	36	65
-5				47900	46	36	64
Average				45500	51	36	63

*Slight anvil mark
**Definite anvil mark.

Note: In addition to those indicated in the Table, other specimens may have shown anvil effects. However, the notes fail to show this.

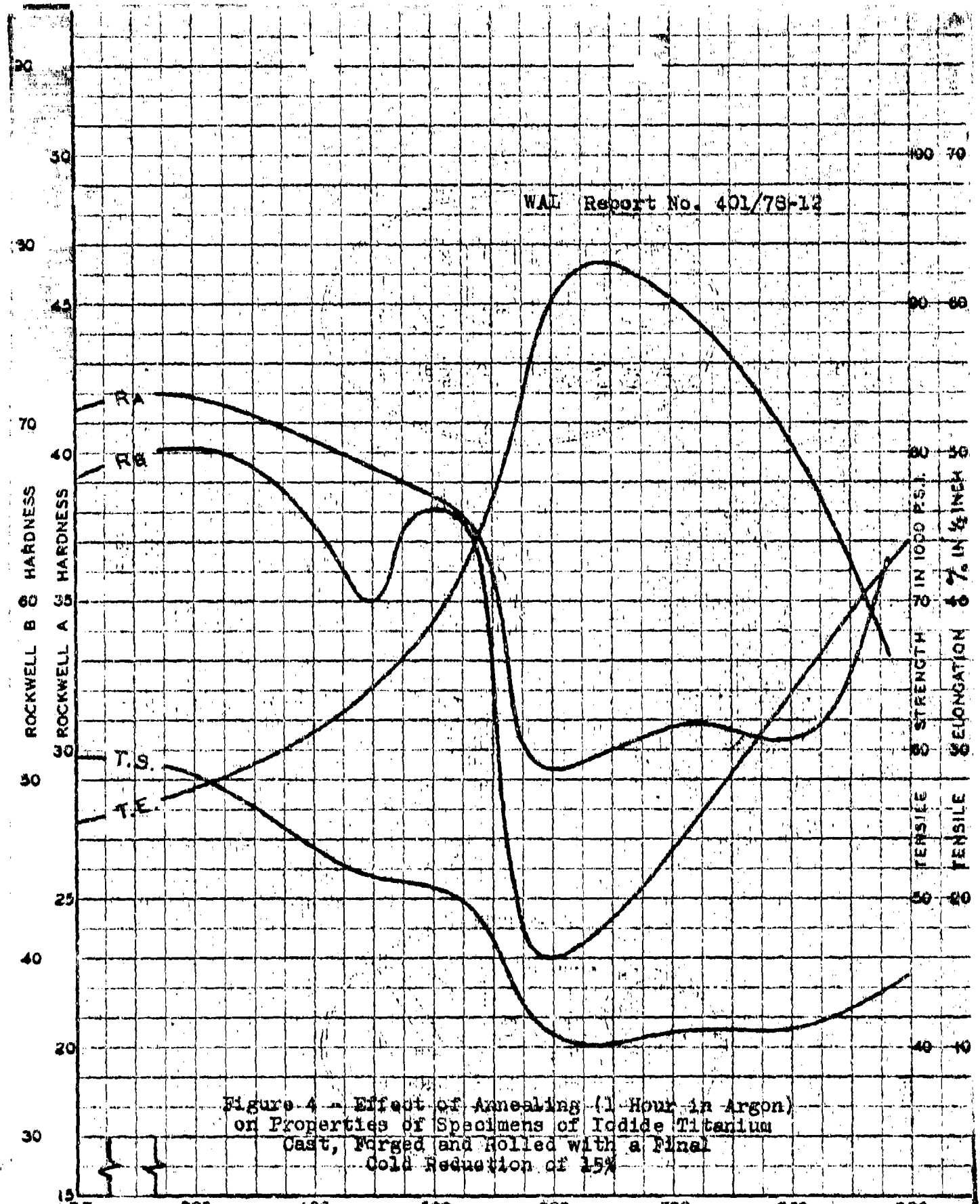


Figure 4 – Effect of Annealing (1 Hour in Argon) on Properties of Specimens of Iodide Titanium Cast, Forged and Rolled with a Final Cold Reduction of 15%

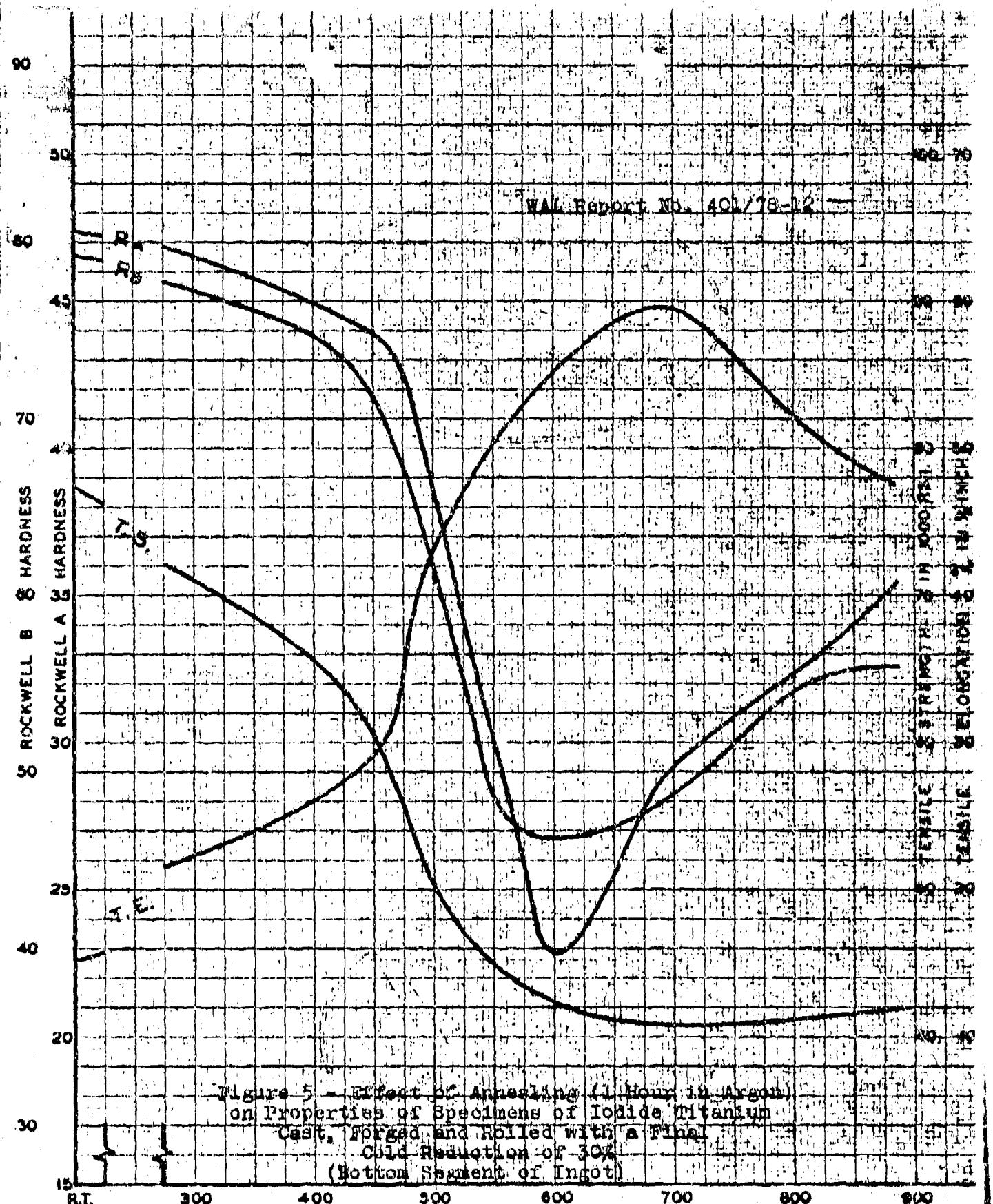


Figure 5 – Effect of Annealing (1 Hour in Argon) on Properties of Specimens of Iodide Titanium Cast, Forged and Rolled with a Final Cold Reduction of 30% (Bottom Segment of Ingot)

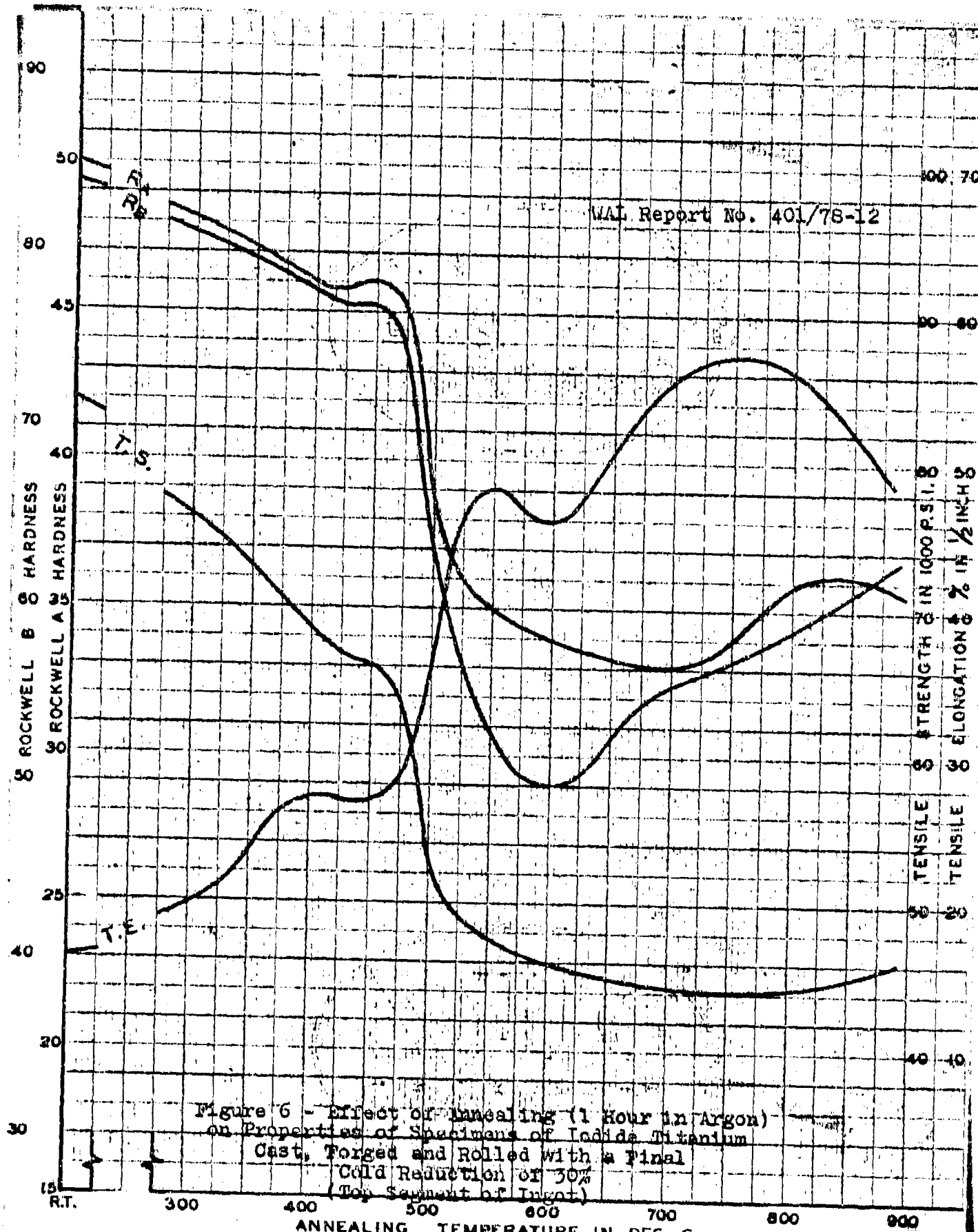


Figure 6 - Effect of Annealing (1 Hour in Argon) on Properties of Specimens of Iodide Titanium Cast, Forged and Rolled with a Final Cold Reduction of 30% (Top Segment of Ingot)

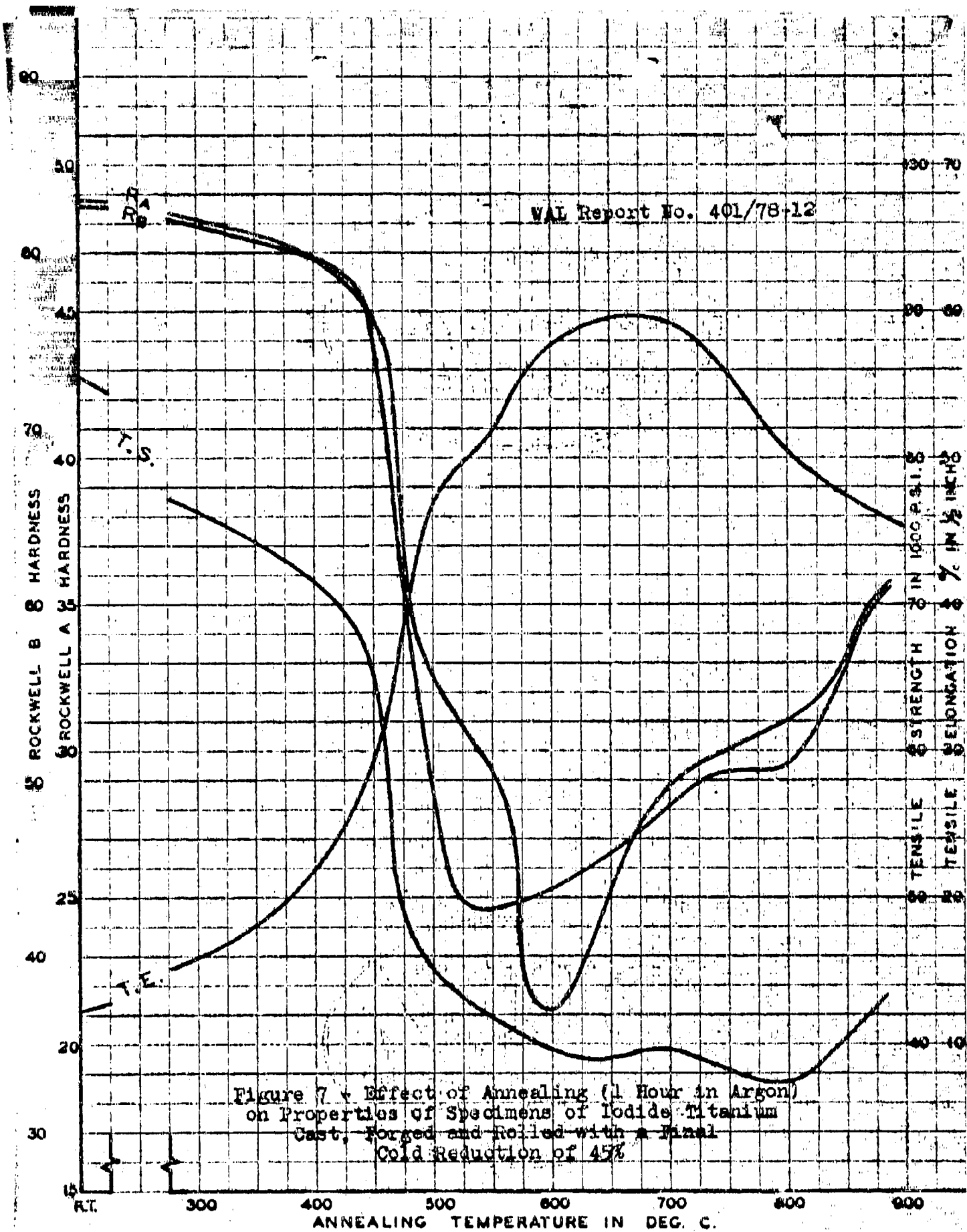


Figure 7 - Effect of Annealing (1 Hour in Argon) on Properties of Specimens of Iodide Titanium Cast, Forged and Rolled with a Final Cold Reduction of 45%

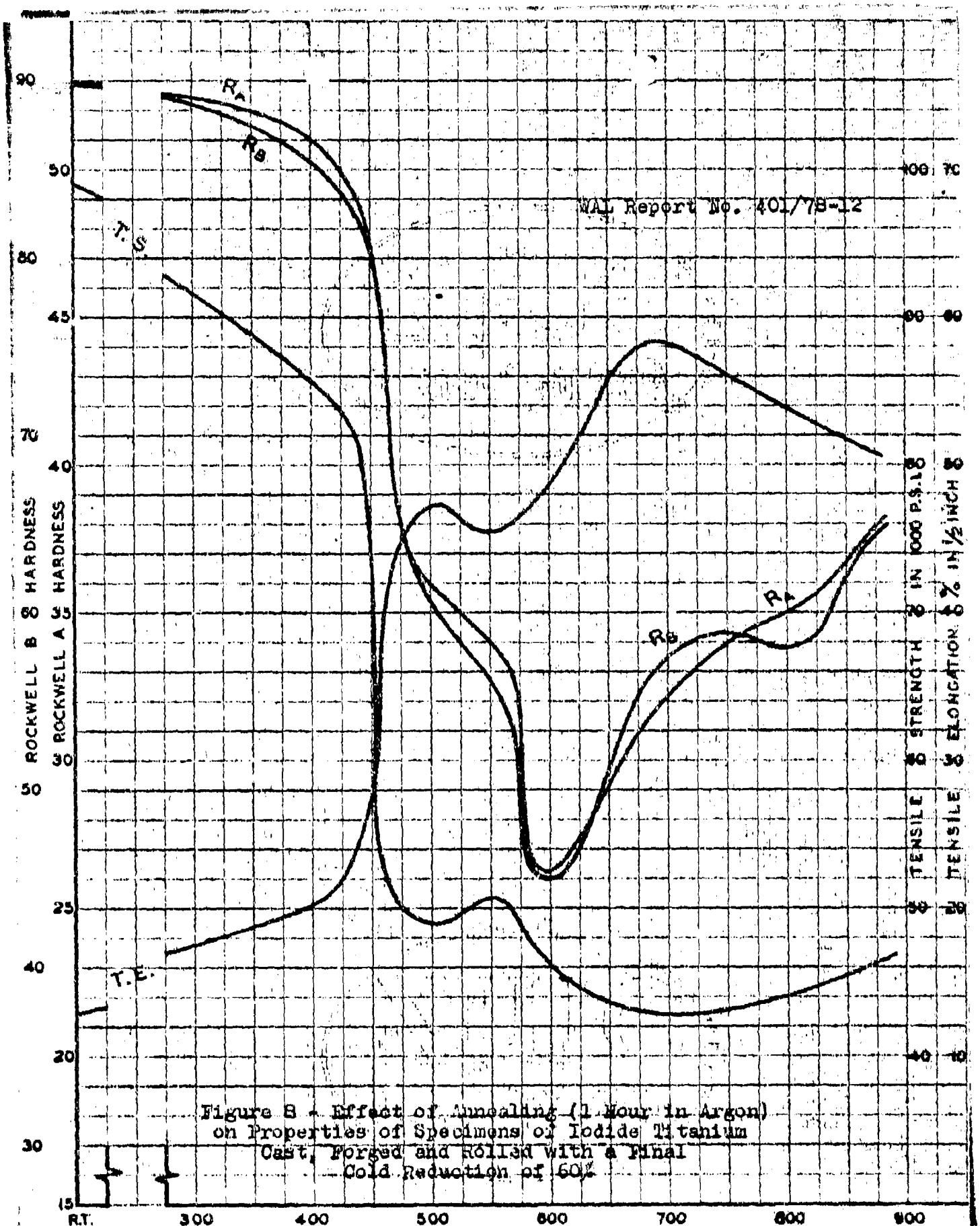


Figure 8 - Effect of Annealing (1 Hour in Argon) on Properties of Specimens of Iodide Titanium Cast, Forged and Rolled with a Final Cold Reduction of 60%

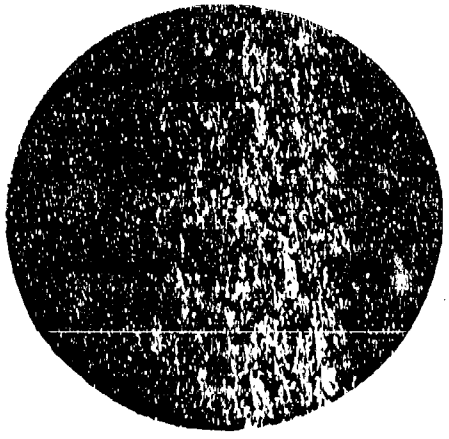


Figure 9 - Cold Rolled 45%
As Rolled.
With Grain Section.
M35543 - 100X

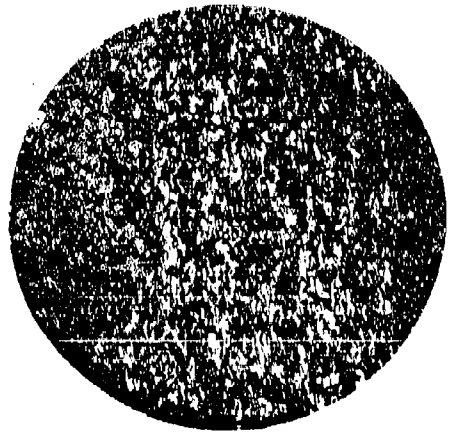


Figure 10 - Cold Rolled 45%
Annealed at 400°C.
With Grain Section.
Not Completely Recrystallized.
M35585 - 100X

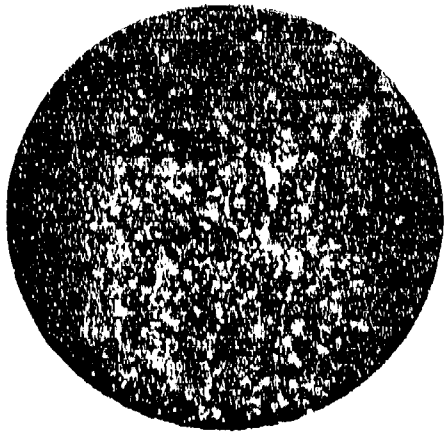


Figure 11 - Cold Rolled 45%
Annealed at 500°C.
With Grain Section.
Approx. Grain Size .012 mm.
M35590 - 100X

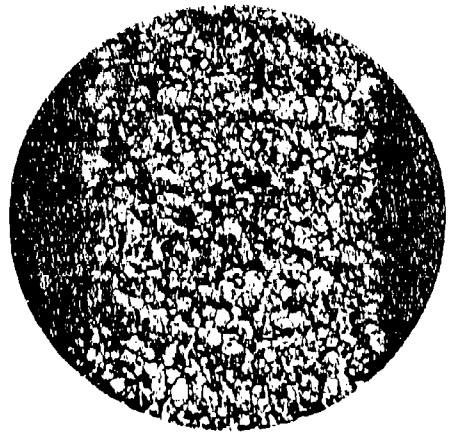


Figure 12 - Cold Rolled 45%
Annealed at 600°C.
With Grain Section.
Approx. Grain Size .022 mm.
M35595 - 100X

Schedule III

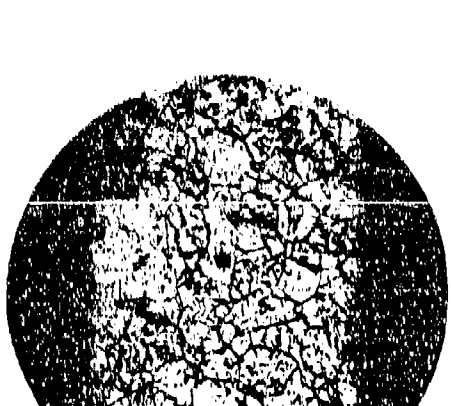


Figure 13 - Cold Rolled 45%
Annealed at 700°C.
With Grain Section.
Approx. Grain Size .060 mm.
M35629 - 100X

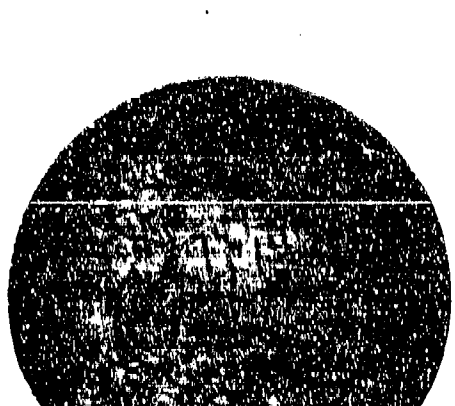


Figure 14 - Cold Rolled 45%
Annealed at 800°C.
With Grain Section.
Approx. Grain Size .120 mm.
M35600 - 100X

Schedule III (Contd.)

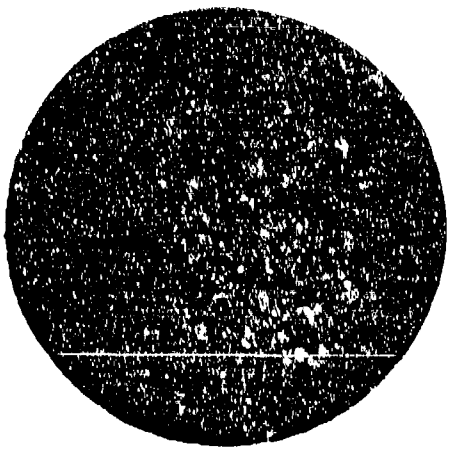


Figure 15 - Cold Rolled 15%
As Rolled.
With Grain Section.
M35541 - 100X

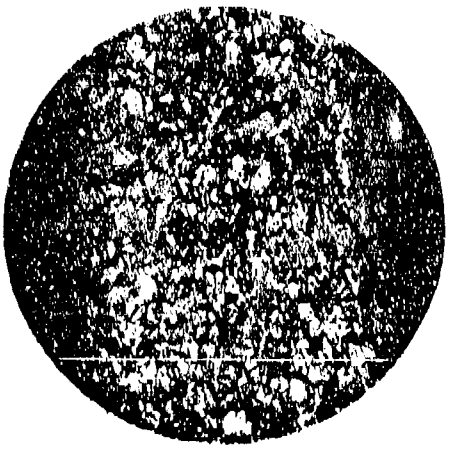


Figure 16 - Cold Rolled 15%
Annealed at 500°C.
With Grain Section.
Not Completely Recrystallized.
M35587 - 100X

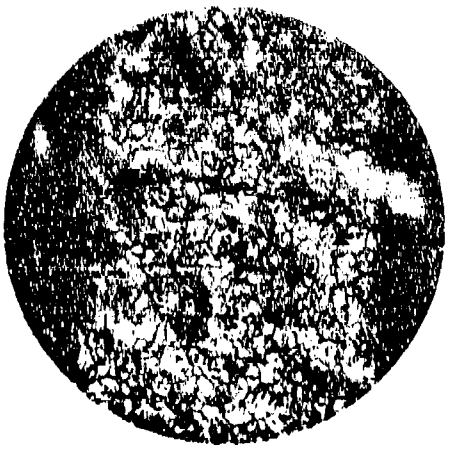


Figure 17 - Cold Rolled 15%
Annealed at 600°C.
With Grain Section.
Approx. Grain Size .036 mm.
M35592 - 100X

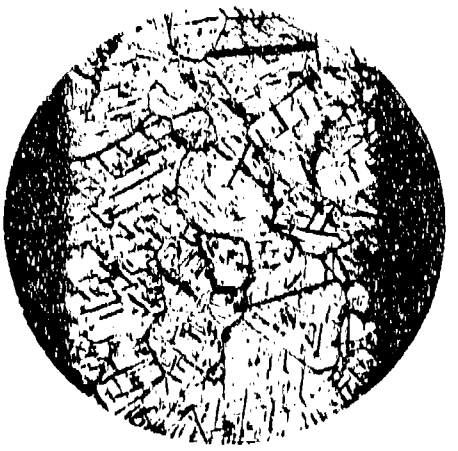


Figure 18 - Cold Rolled 15%
Annealed at 800°C.
With Grain Section.
Approx. Grain Size .090 mm.
M35897 - 100X

Schedule 1

Table 6 - Recrystallization Temperature of Titanium

	Recrystallization Temperature - °C., Based on Change in:			
	Tensile Strength	Elongation	Hardness R_A	Hardness R_B
15 Per Cent Reduction	530	535	550	550
30 Per Cent Reduction*	460	480	510	500
30 Per Cent Reduction**	470	510	490	495
45 Per Cent Reduction	460	470	480	480
60 Per Cent Reduction	450	460	475	470
96.5 Per Cent Reduction***	--	--	475	470

*Bottom of ingot.

**Top of ingot.

***Material rolled 96.5 per cent from a different forged iodide titanium ingot with no intermediate anneals.

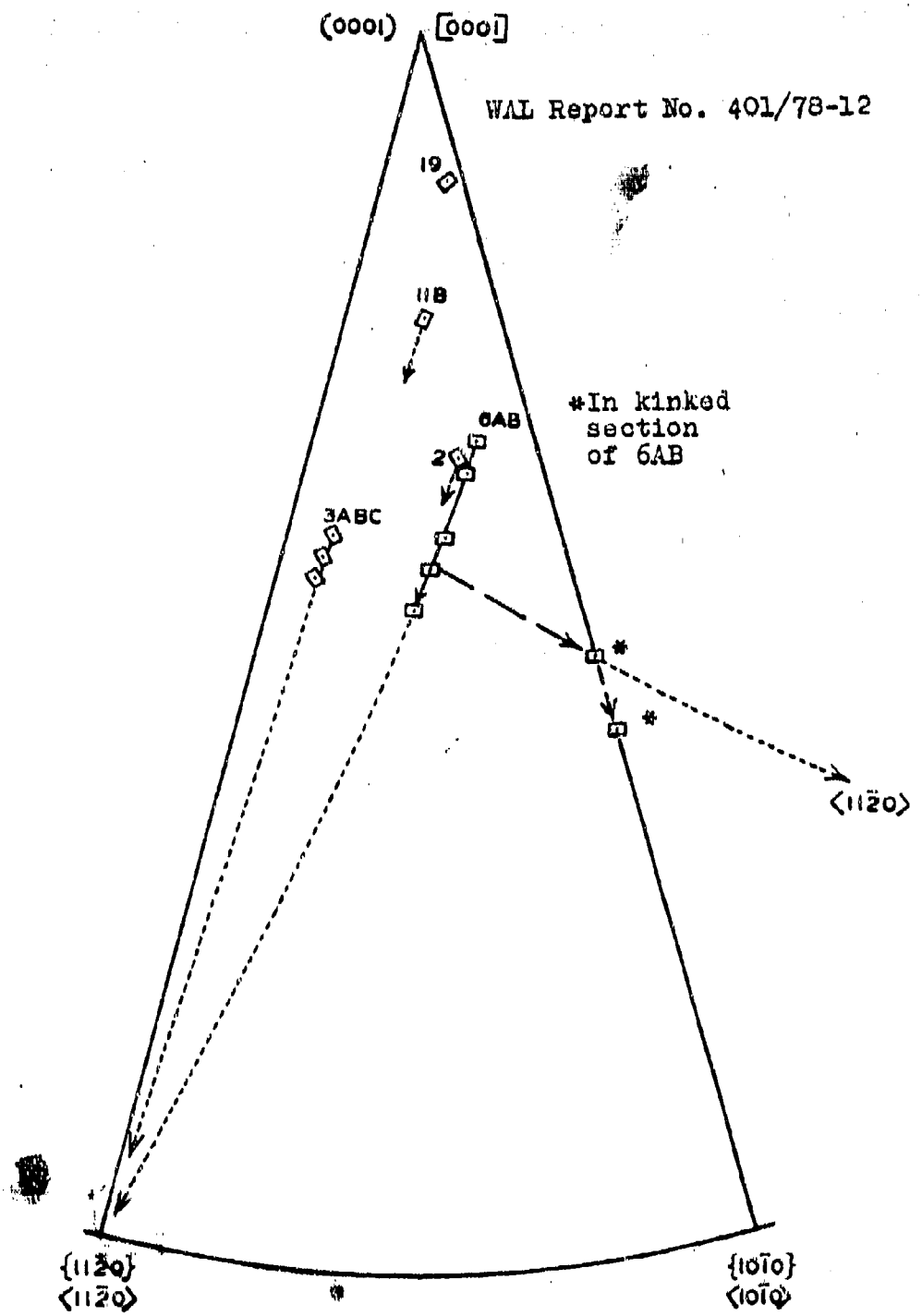


Figure 19 - Stereographic Plot Showing Rotation of Specimen Axis Relative to Crystal During Extension of Large Crystals Grown from Iodide Titanium

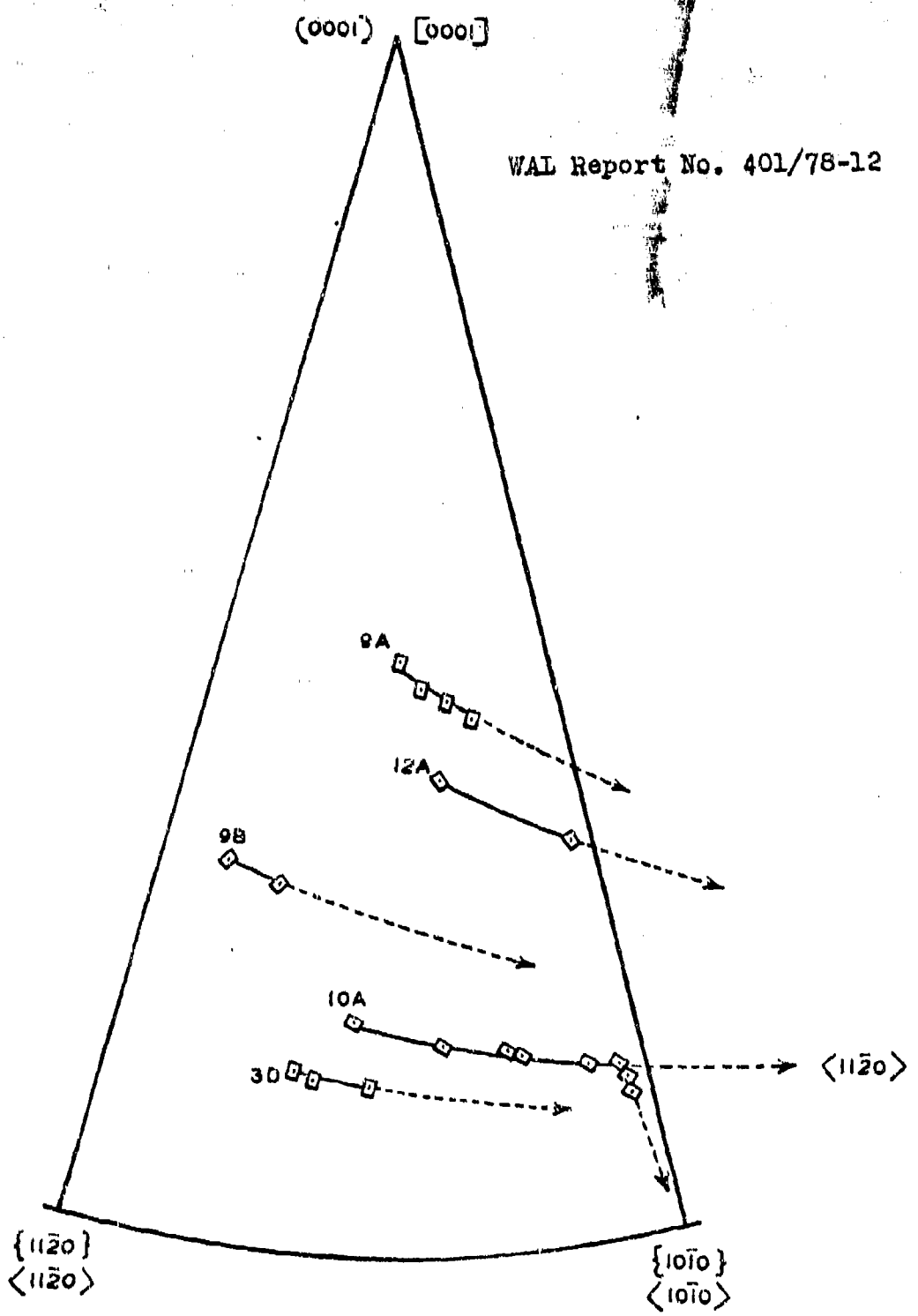


Figure 20 - Stereographic Plot Showing Rotation of Specimen Axis Relative to Crystal During Extension of Large Crystals Grown from Iodide Titanium

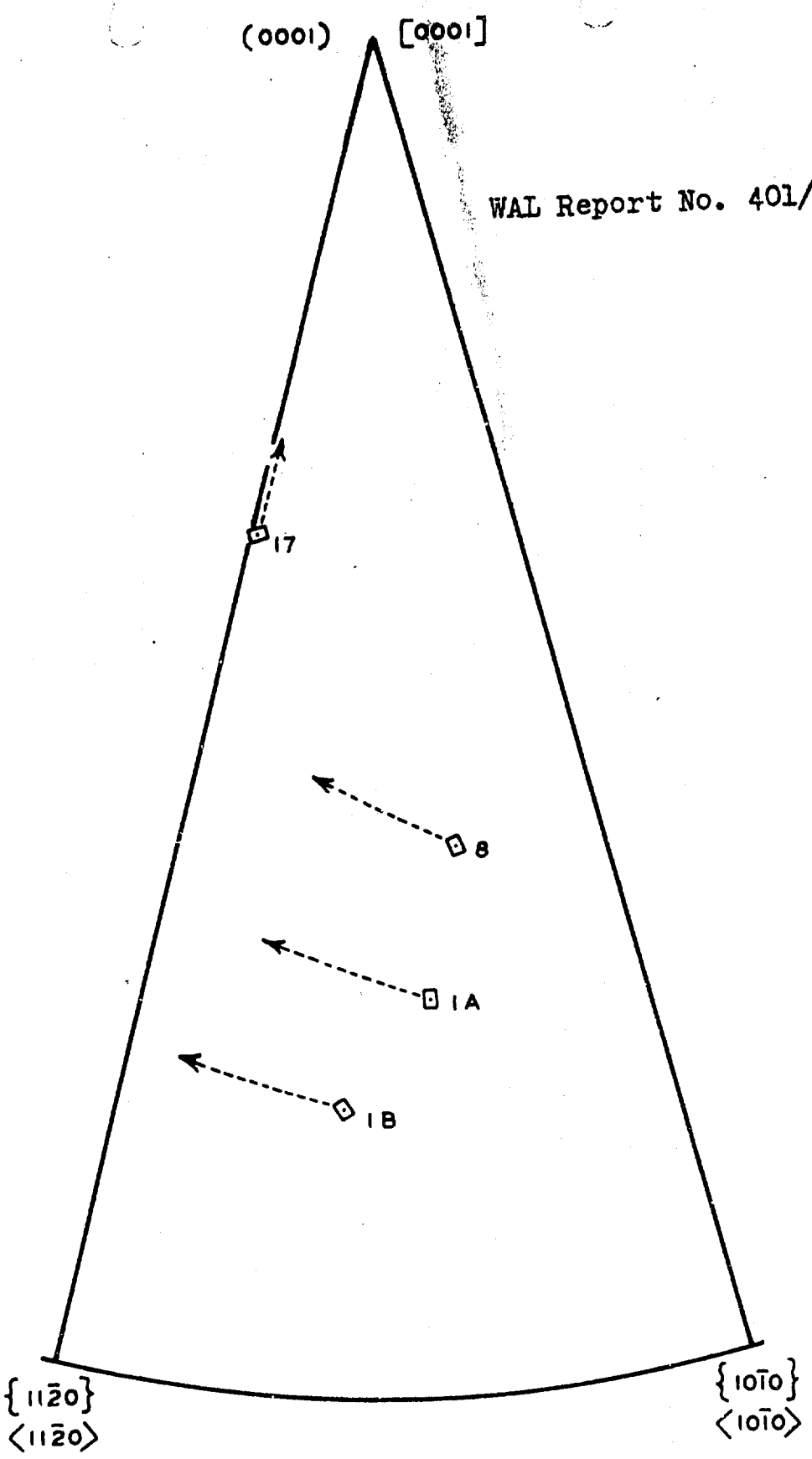


Figure 21 - Stereographic Plot Showing Rotation of Specimen Axis Relative to Crystal During Compression of Large Crystals Grown from Iodide Titanium

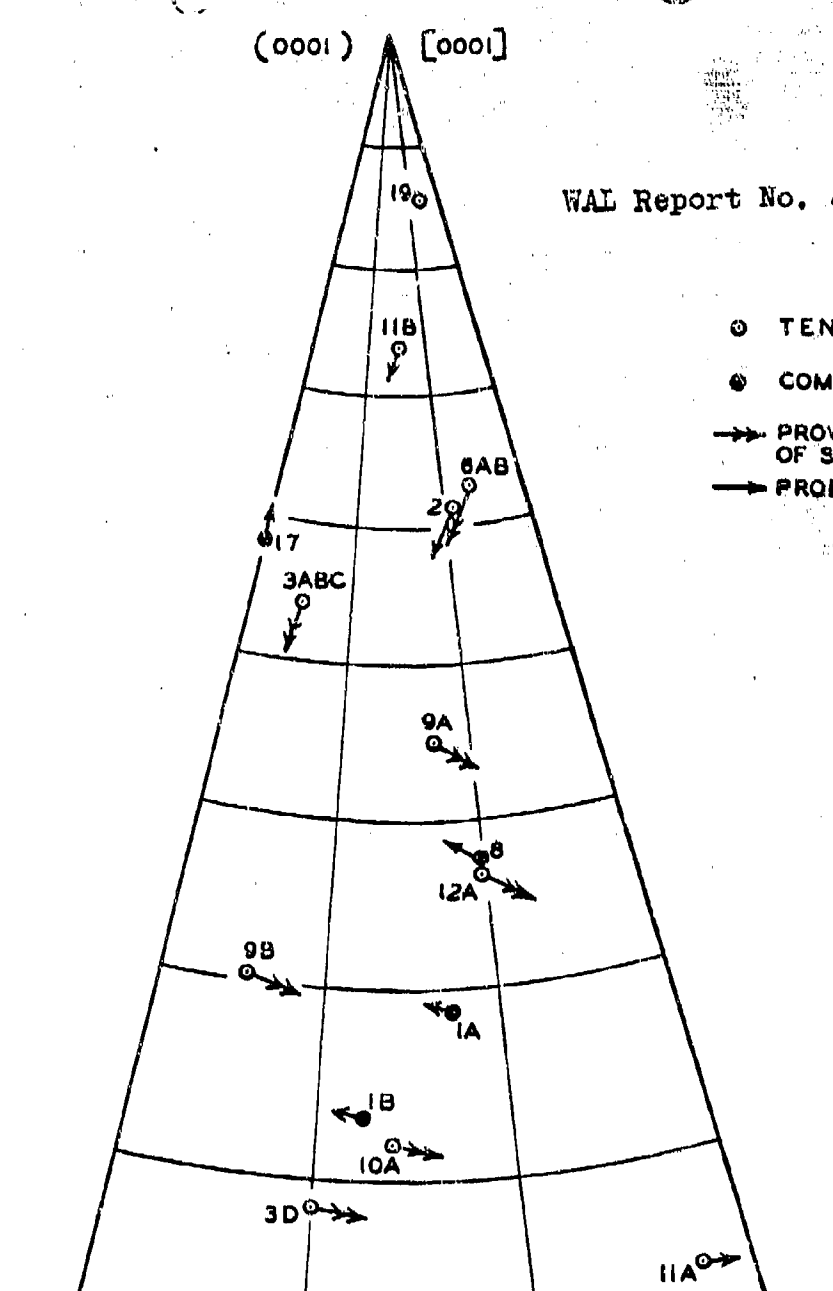


Figure 22 - Stereographic Plot Showing Direction of Initial Slip Rotation of Specimen Axis Relative to Crystal During Extension or Compression of Large Crystals Grown from Iodide Titanium

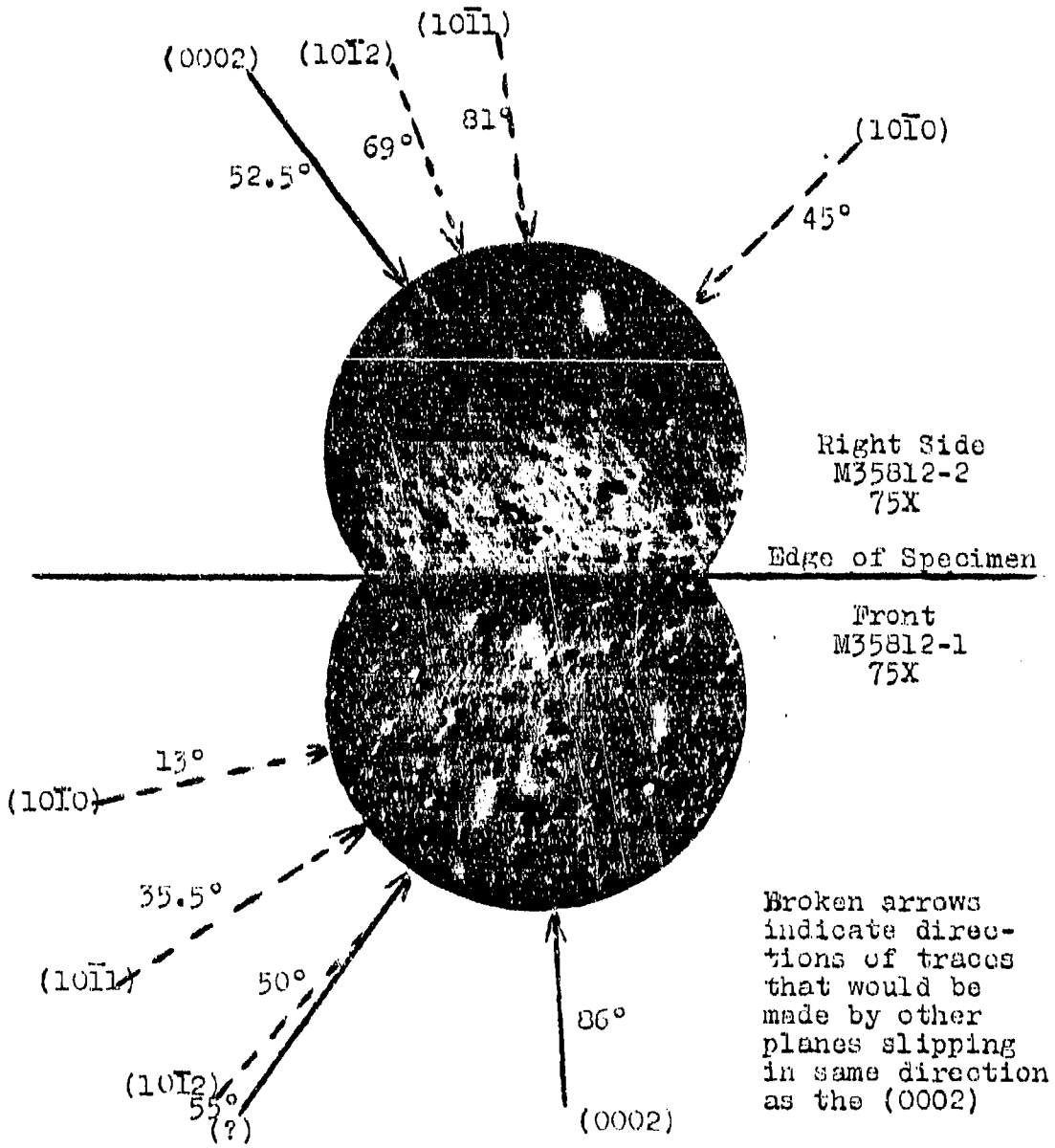


Figure 23 - Micrographs (75X) of Surfaces of TiX1-6 Pulled to 46,000 p.s.i. Stress (2.5% Elongation) Showing Deformation Markings

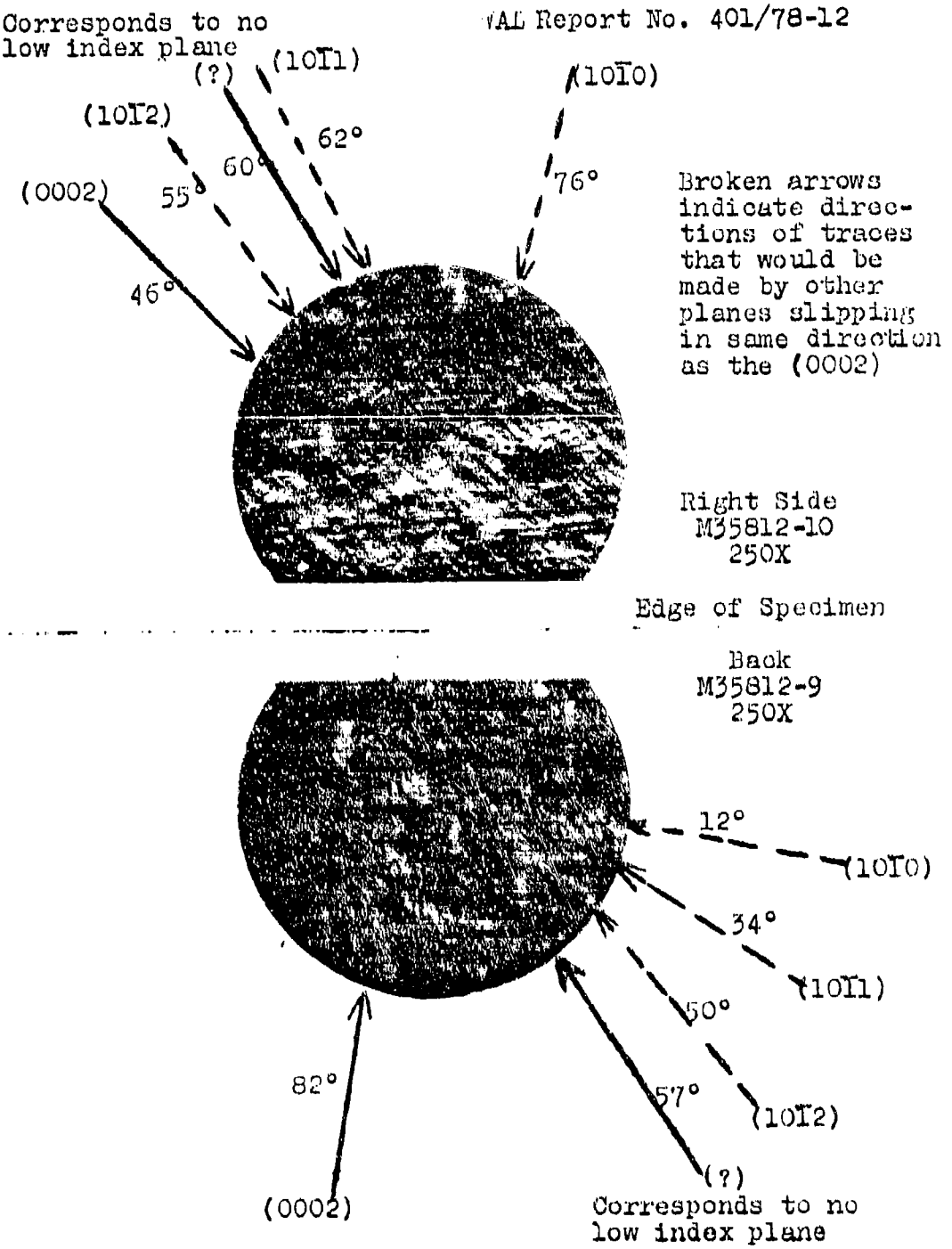


Figure 24 - Micrographs (250X) of Surfaces of TiXl-6 Pulled to 47,000 p.s.i. Stress (10% Elongation) Showing Deformation Markings

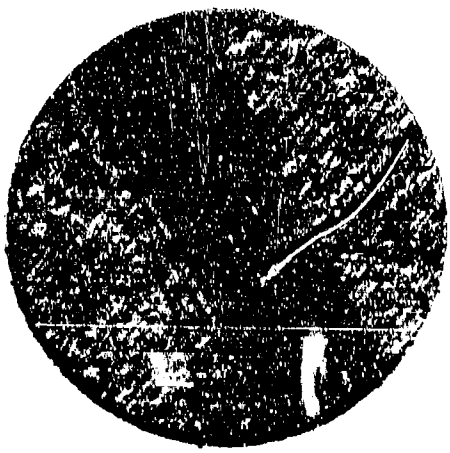


Figure 25 - TiX1-3A (0002) and (11$\bar{2}$1) Twinning. Note Slight Change in Slip Direction in Twinned Area.
M35810 #4 - 75X

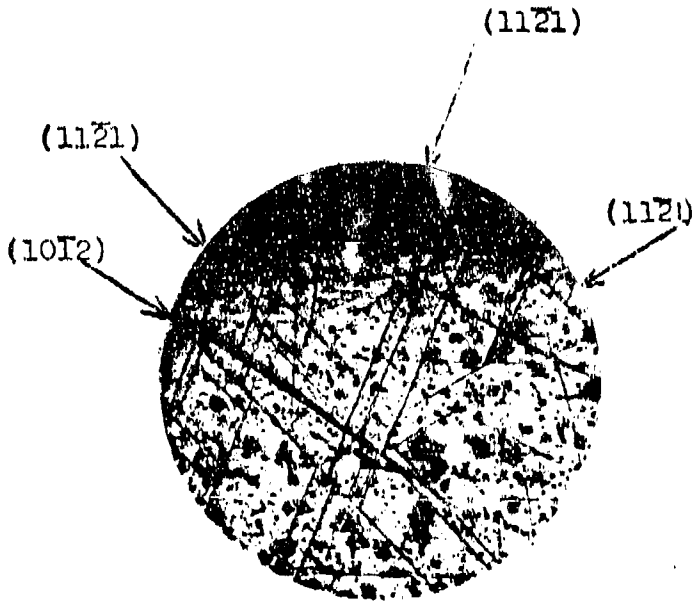


Figure 26 - TiX1-3A - Showing (10$\bar{1}$2) Twin and 3 Sets of (11$\bar{2}$1) Twins.
M35810 #2 - 75X

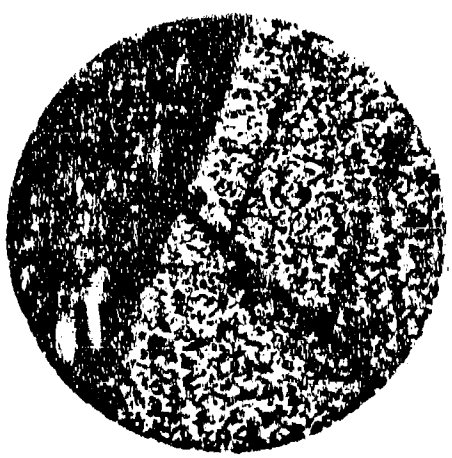


Figure 27 - TiX1-3E (Crystal at Right) - 2 Sets of (11$\bar{2}$2) Twins.
M35810 #1 - 75X
Crystal at Left is 3D Showing (10$\bar{1}$0) Slip.

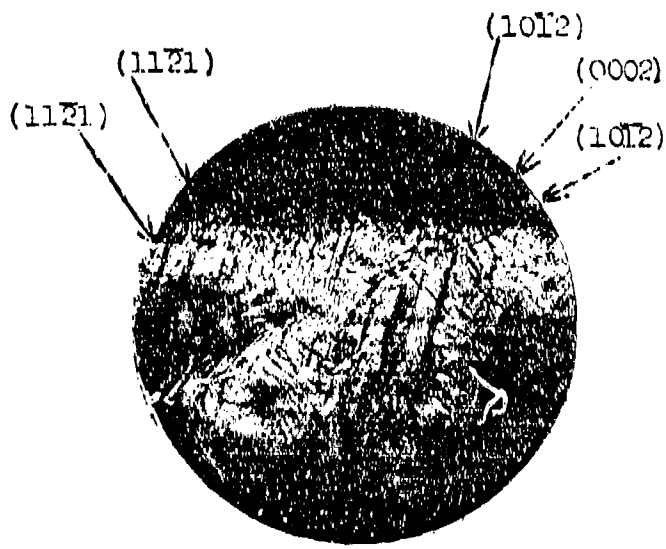


Figure 28 - TiX1-2 - Showing (0002) Slip and (10$\bar{1}$2) and (11$\bar{2}$1) Twinning.
M35690 #2 - 75X

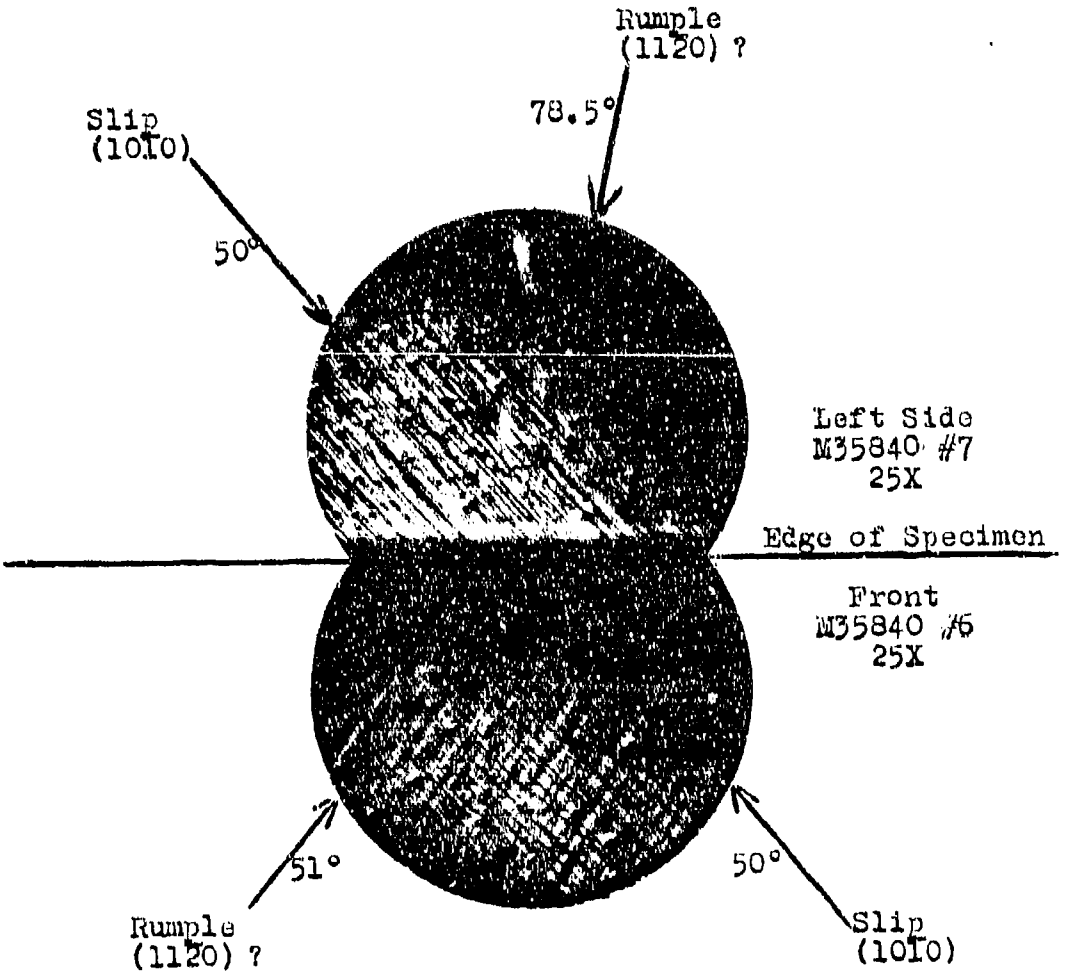


Figure 29 - Micrographs (25X) of Surfaces of Ti-Al-10A Pulled to 12.5% Elongation Showing Deformation Markings

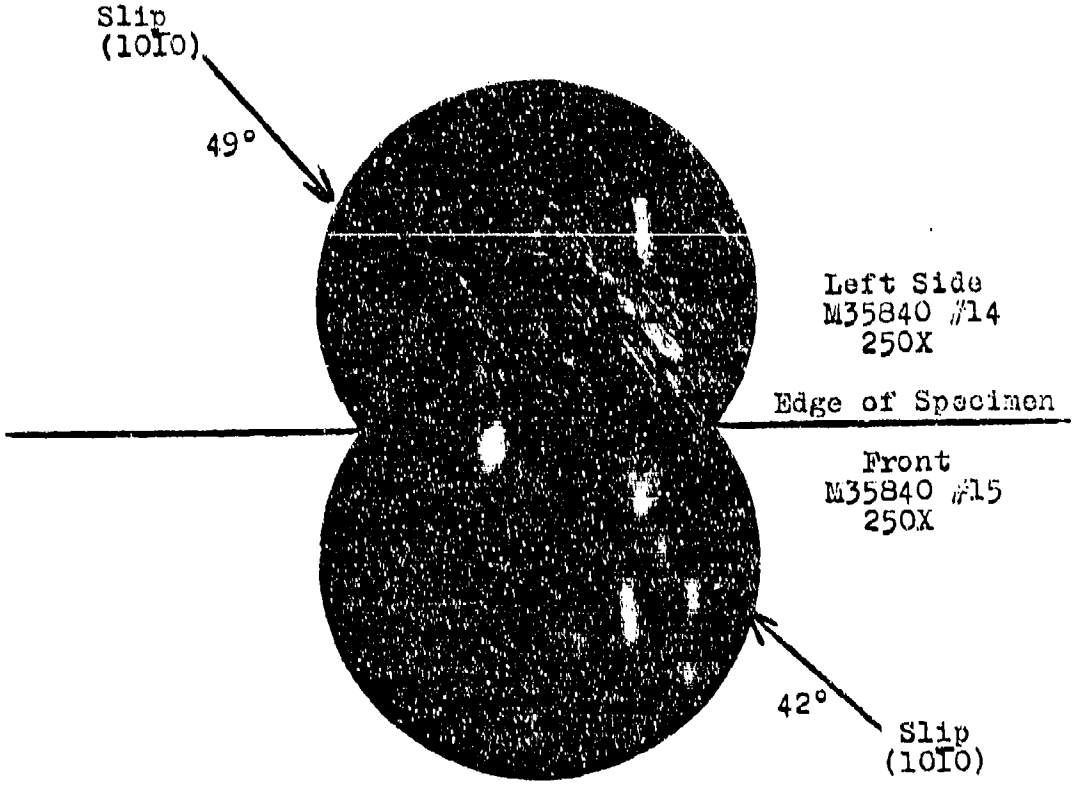
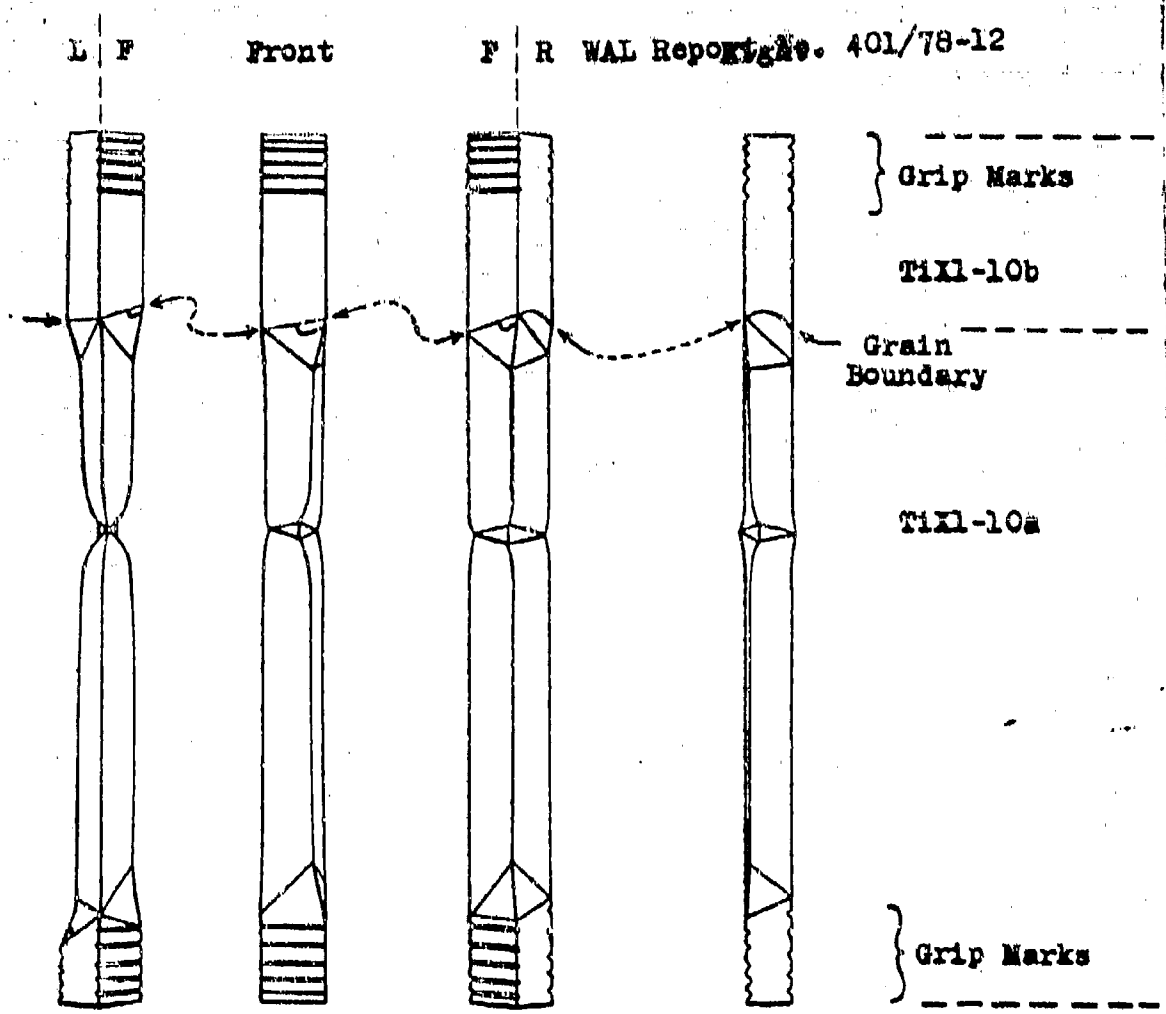


Figure 30 - Micrographs (250X) of Surfaces of TiX1-10A
Pulled to 49.3% Elongation
Note Irregularities in Surface



2X Actual Size

Figure 31 - Appearance of Ti-10 After Final Extension

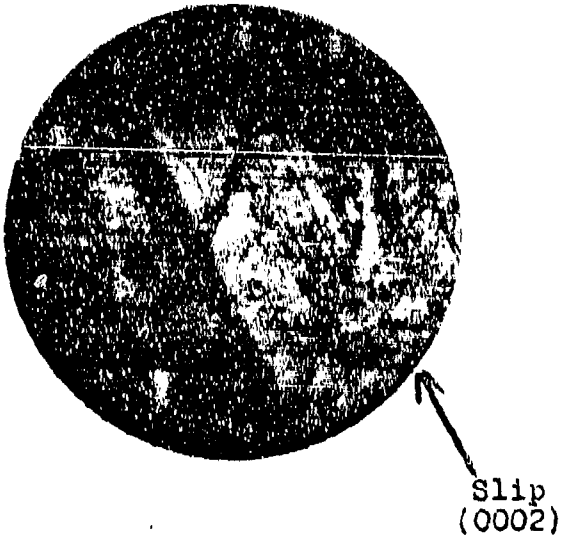


Figure 32 - Back Surface of TiX1-17 - Compressed 1.65% (36,400 p.s.i. Stress) - Showing (1122) Twinning and (0002) Slip
M35903 #1 75X

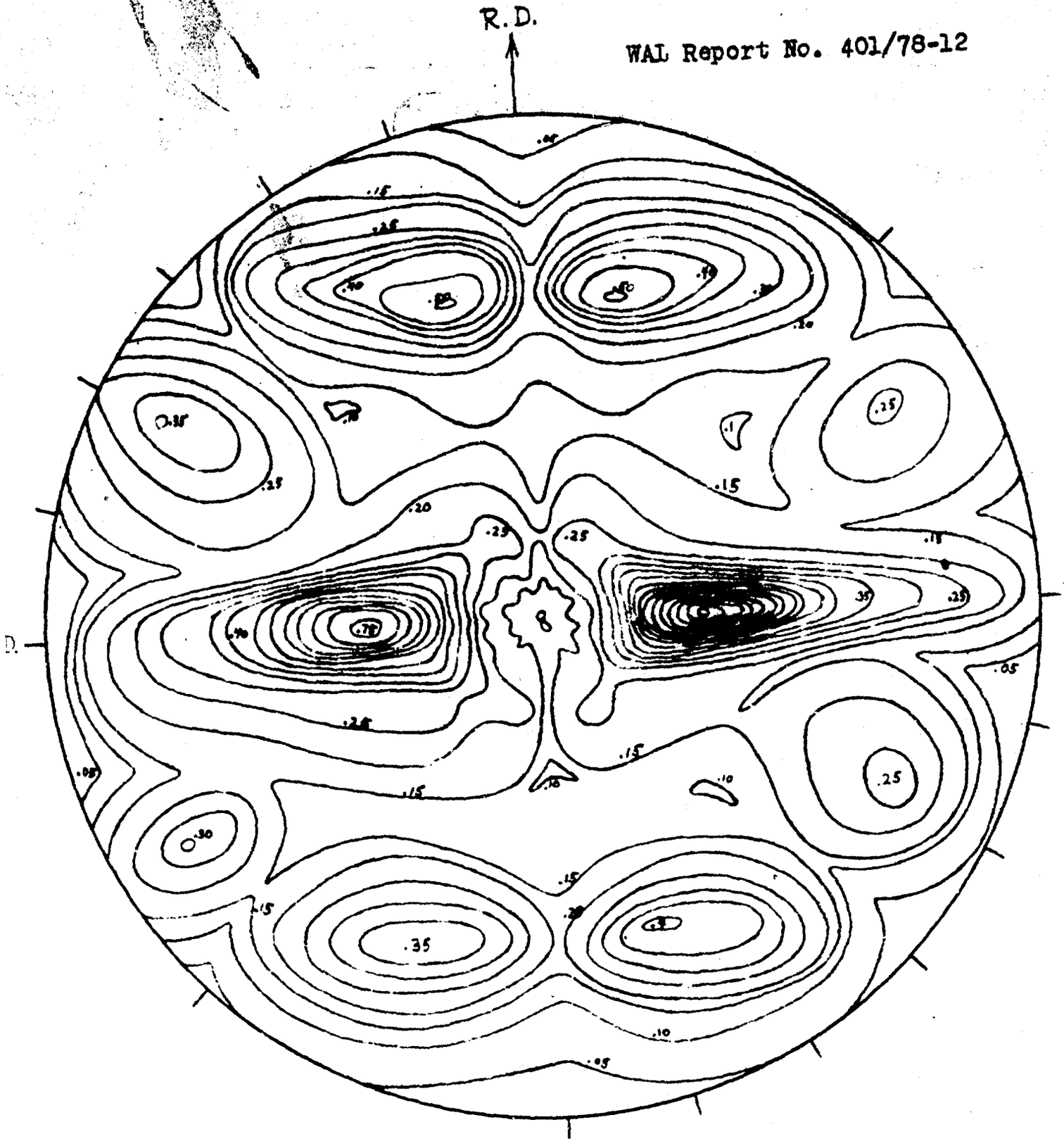


Figure 33 - (0002) Pole Figure for Iodide Titanium Arc Melted, Forged, and Colled Rolled 96.5% from 1.1 Inch to 0.038 Inch

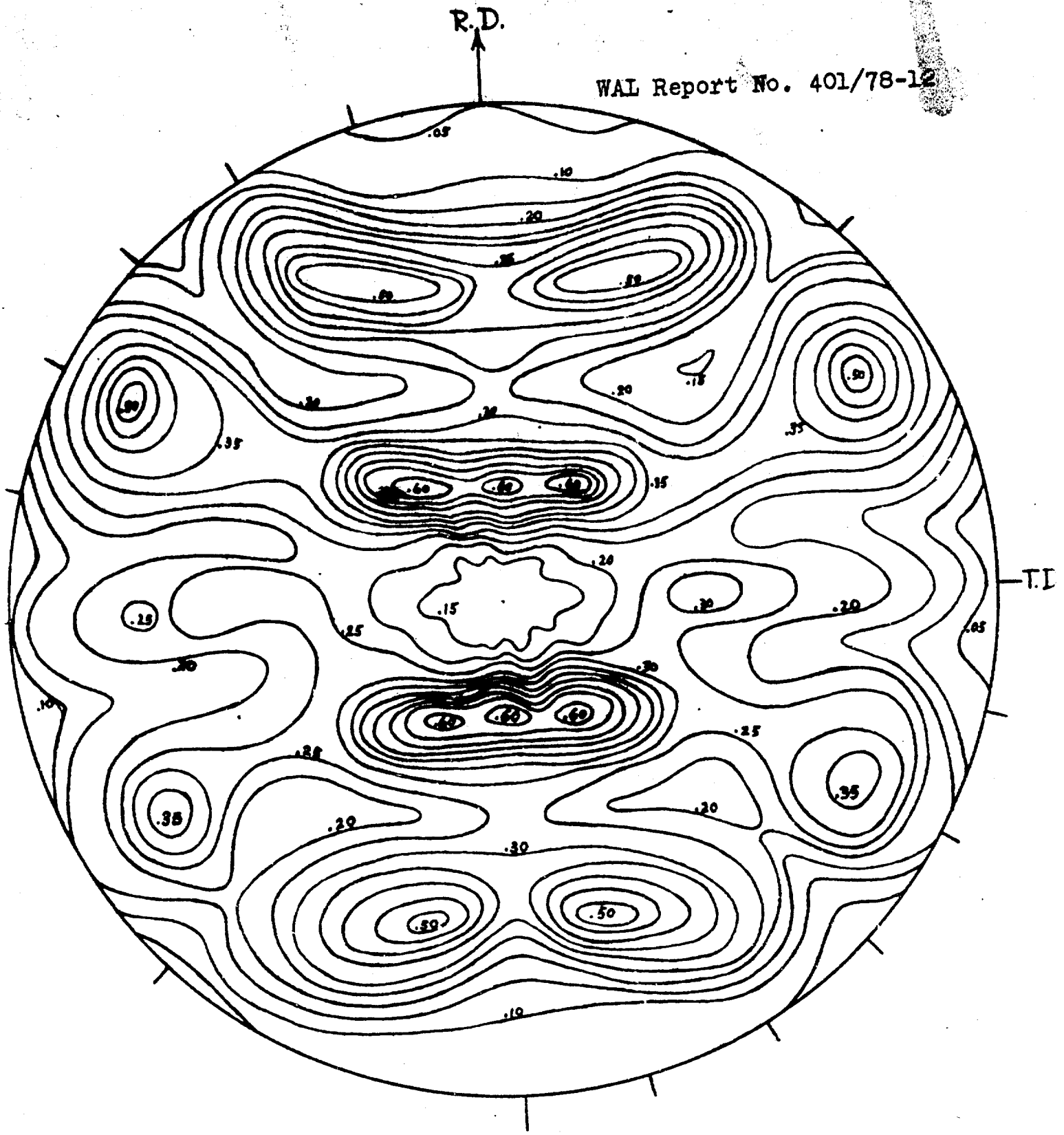


Figure 34 - (10$\bar{1}$1) Pole Figure for Iodide Titanium
Arc Melted, Forged, and Cold Rolled 96.5%
from 1.1 Inch to 0.038 Inch

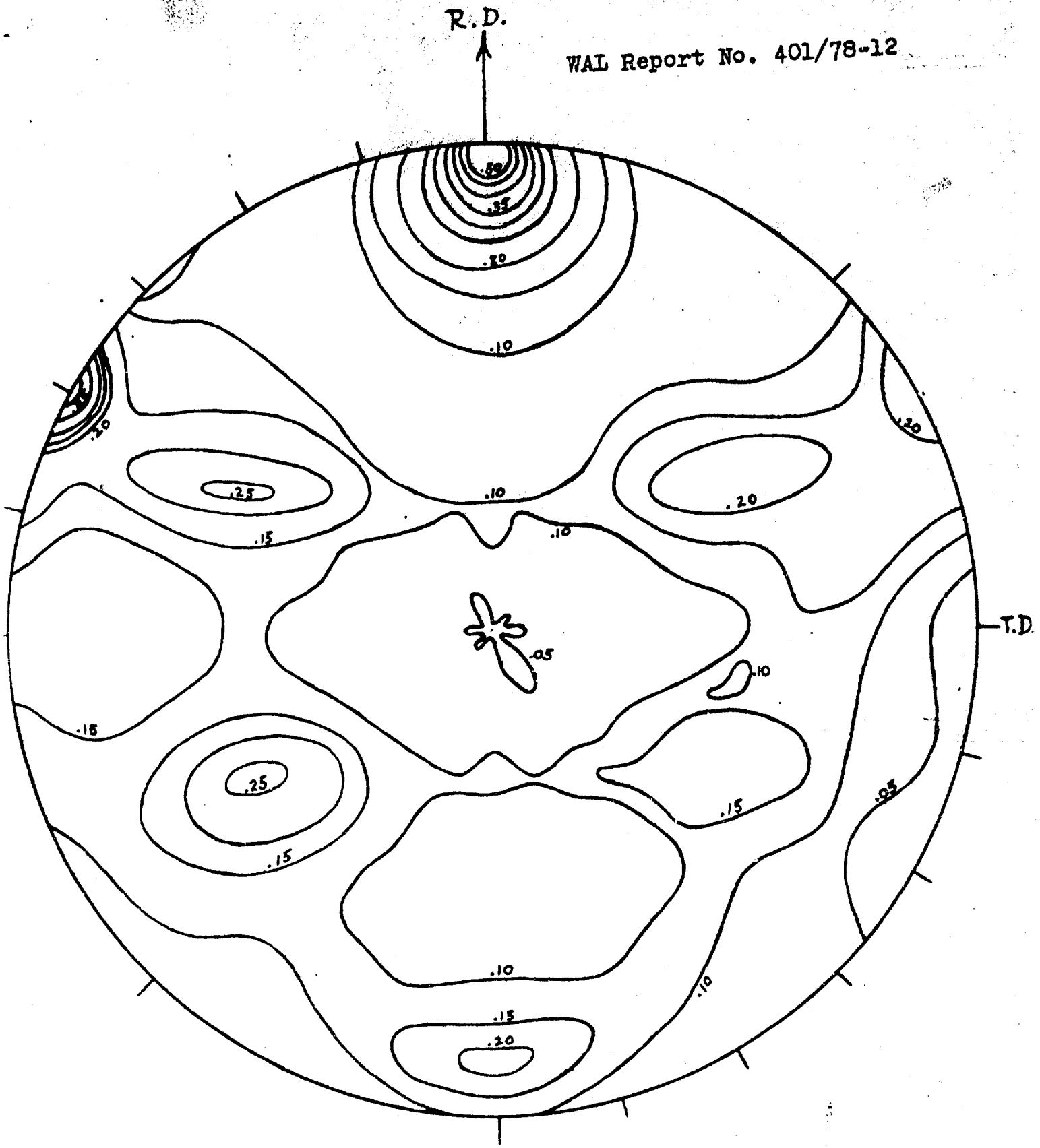


Figure 35 (10$\bar{1}$0) Pole Figure for Iodide Titanium Arc Melted, Forged, and Cold Rolled 96.5% from 1.1 Inch to 0.038 Inch

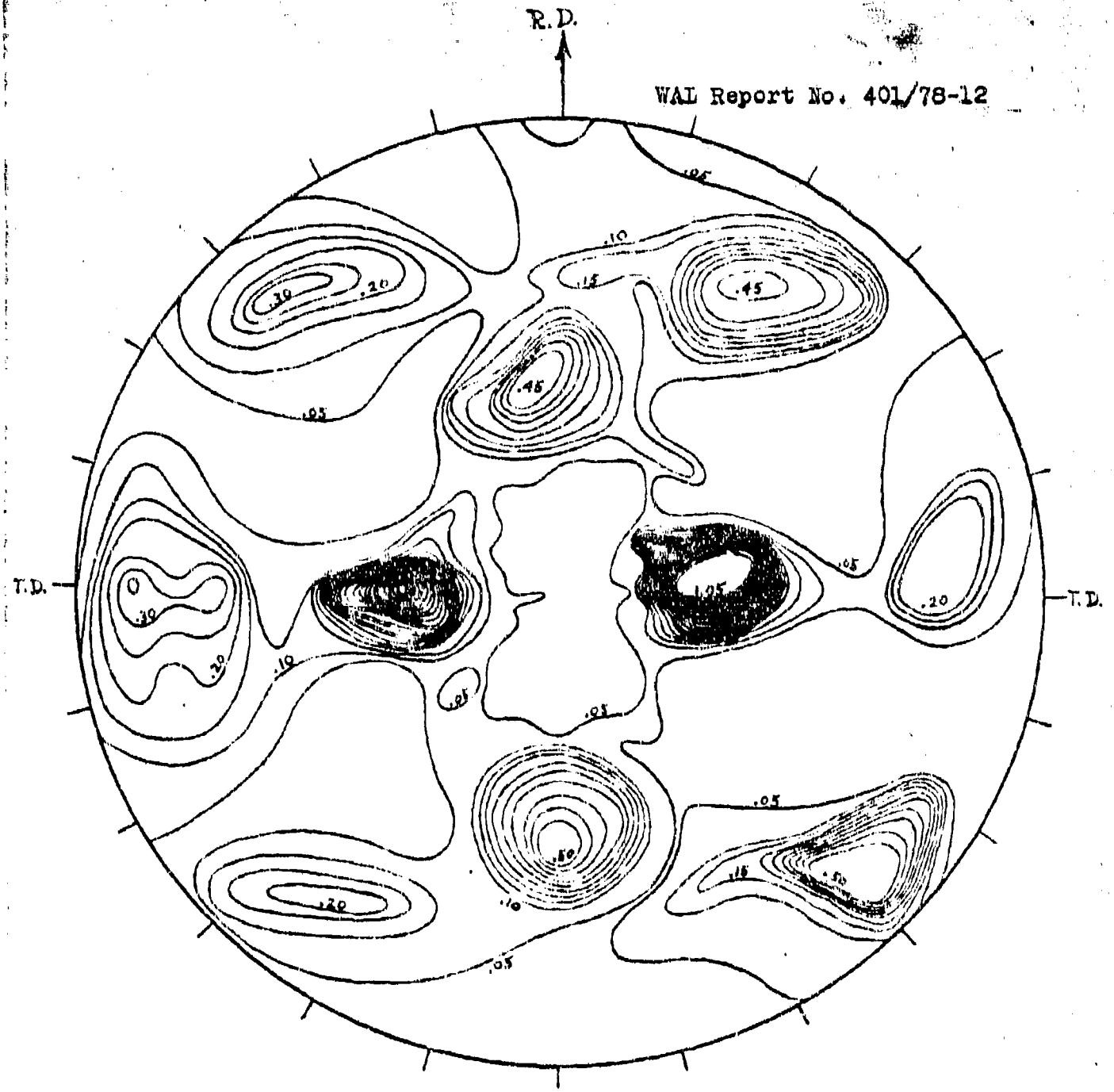


Figure 36 - (0002) Pole Figure for Iodide Titanium Arc Melted, Forged, Cold Rolled 96.5% from 1.1 Inch to 0.038 Inch and Annealed in Argon for 1 Hour at 825°C.

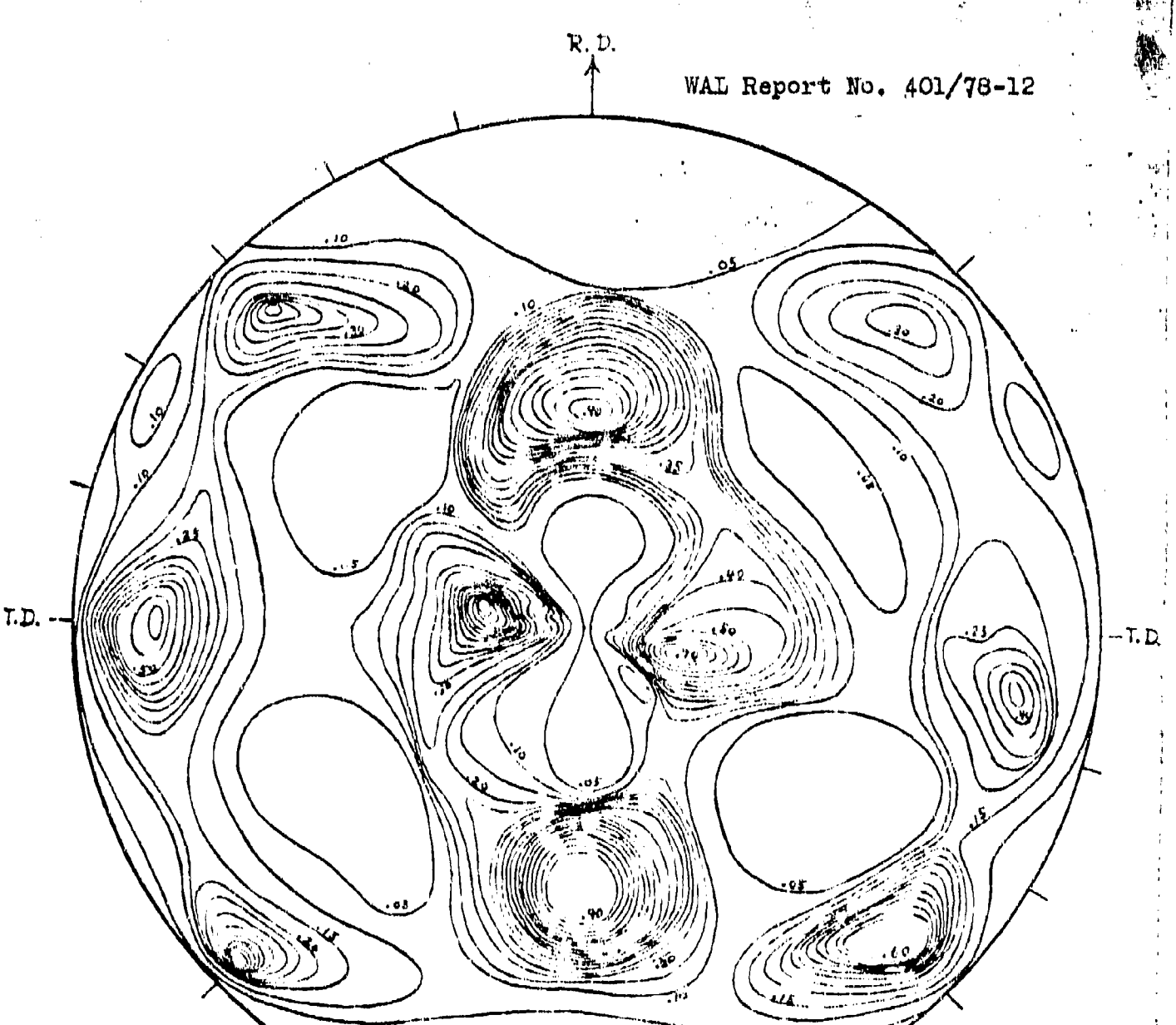


Figure 37 (10$\bar{1}$1) Pole Figure for Iodide Titanium Arc Melted, Forged, Cold Rolled 96.5% from 1.1 Inch to 0.038 Inch and Annealed in Argon for 1 Hour at 825°C.

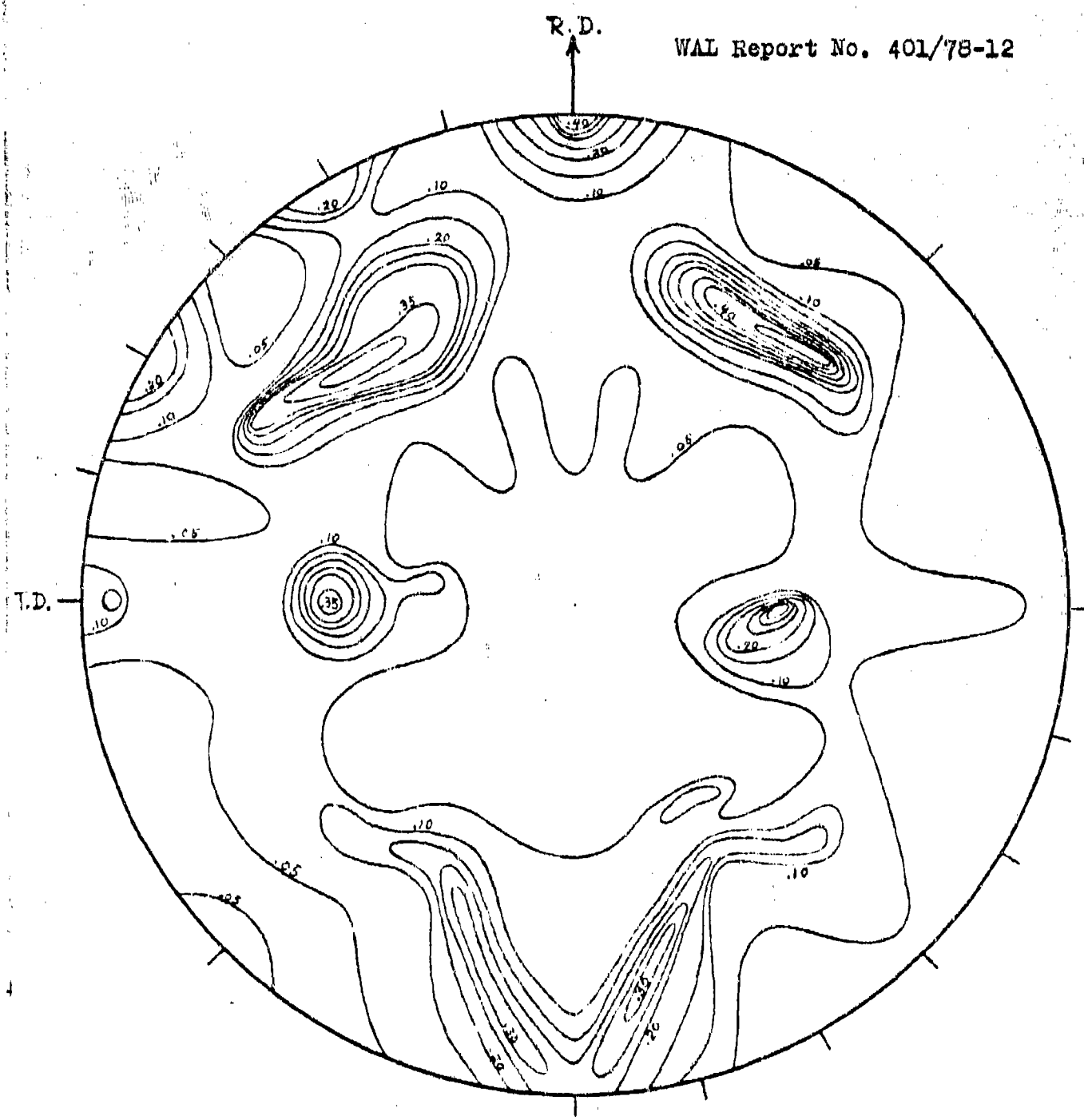


Figure 38 (10$\bar{1}$0) Pole Figure for Iodide Titanium Arc Melted, Forged, Cold Rolled 96.5% from 1.1 Inch to 0.038 Inch and Annealed in Argon for 1 Hour at 825°C.

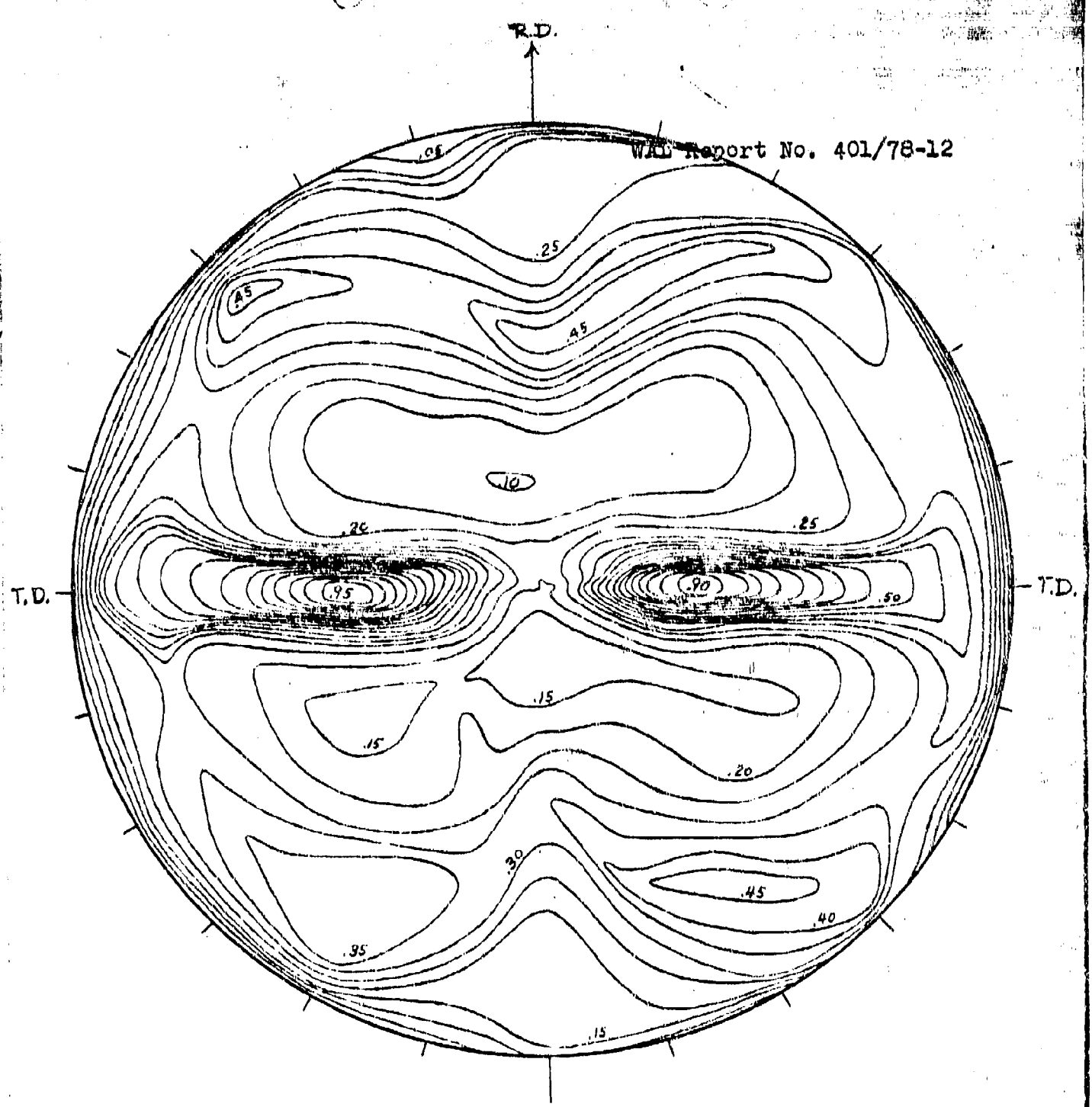


Figure 39 - (0002) Pole Figure for Surface of Iodide Titanium Arc Melted, Forged, and Cold Rolled 96.5% from 1.1 Inch to 0.038 Inch

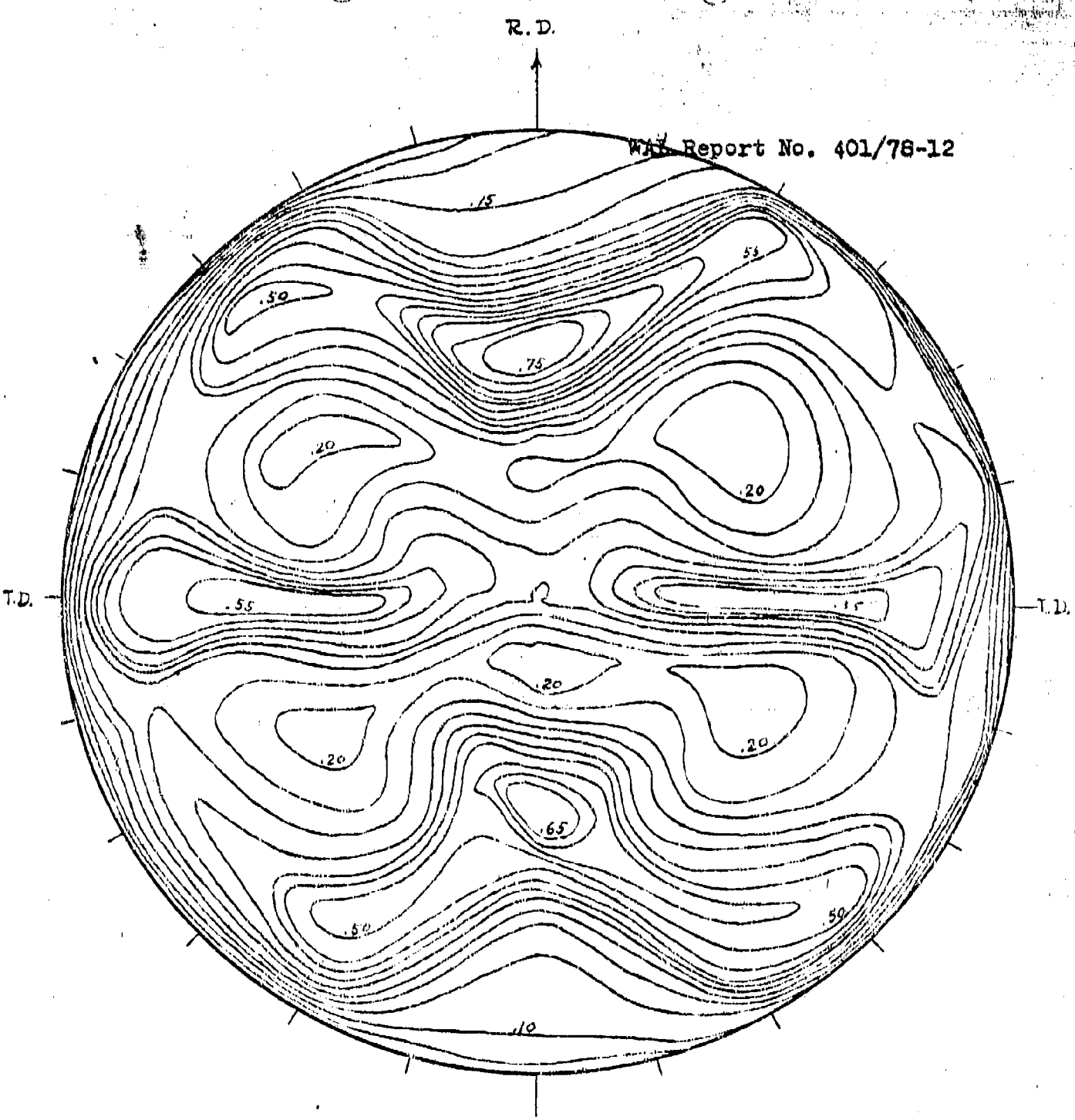


Figure 40 - (10$\bar{1}$1) Pole Figure for Surface of Iodide Titanium Arc Melted, Forged, and Cold Rolled 96.5% from 1.1 Inch to 0.038 Inch

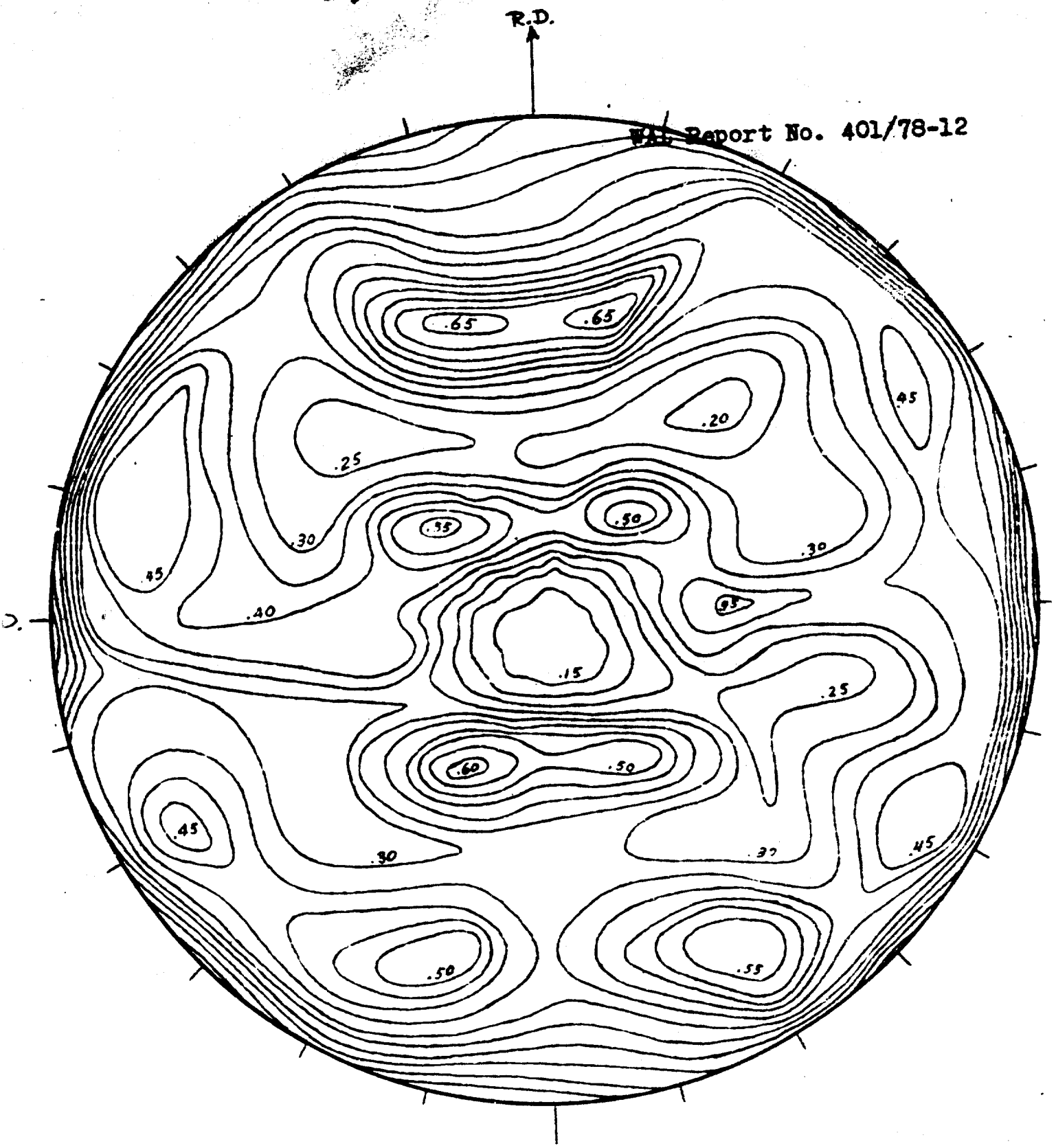


Figure 41 - (10$\bar{1}$1) Pole Figure for Material 0.006" Below Surface of Iodide Titanium Arc Melted, Forged, and Cold Rolled 96.5% from 1.1 Inch to 0.038 Inch

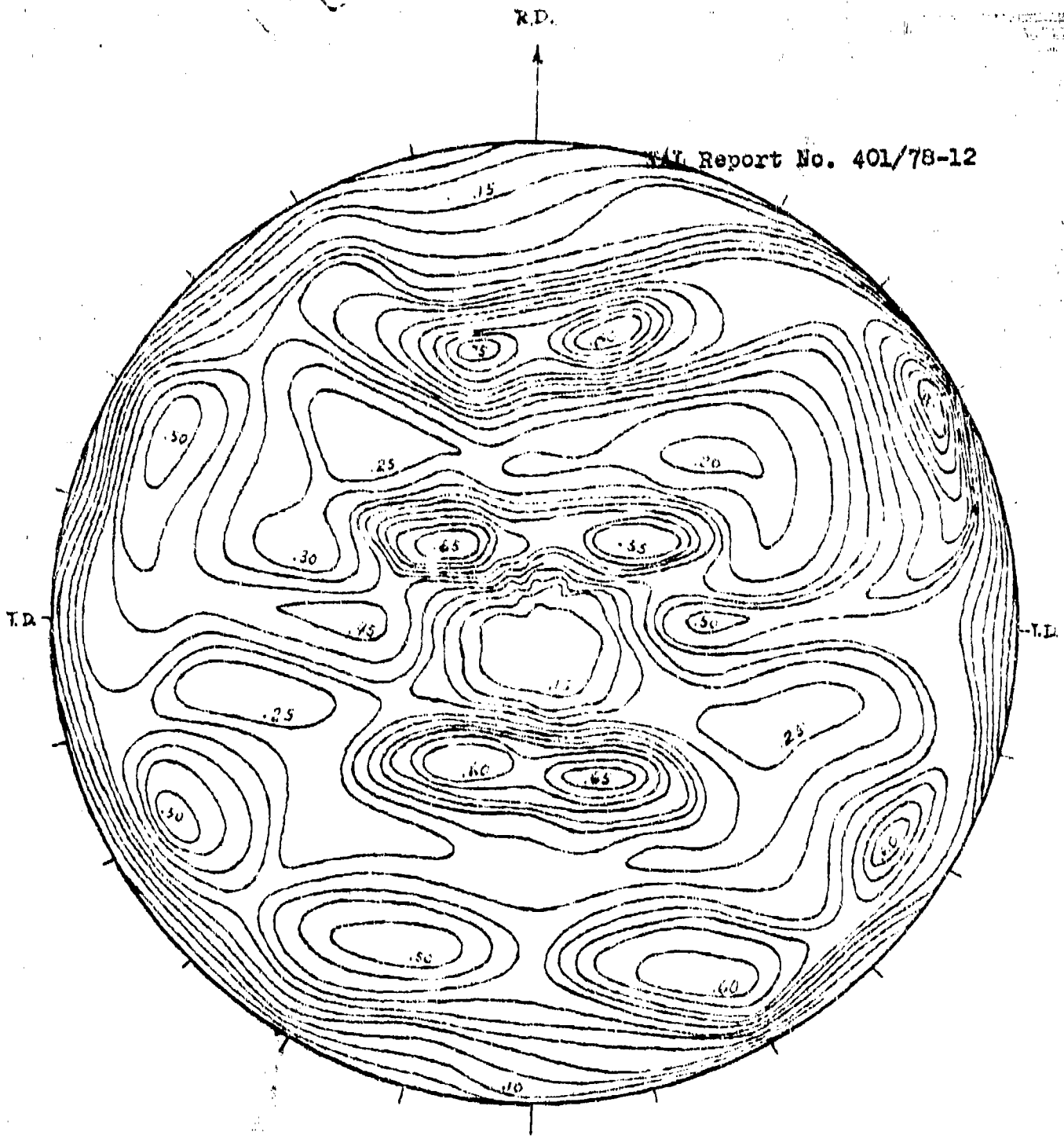


Figure 42 - (10$\bar{1}$1) Pole Figure for Material of Figure 41 Annealed in Argon for 1 Hour at 400°C.

Figure 43 - (10$\bar{1}$1) Pole Figure for Material of Figure 42 Annealed in Argon for 1 Hour at 450°C.

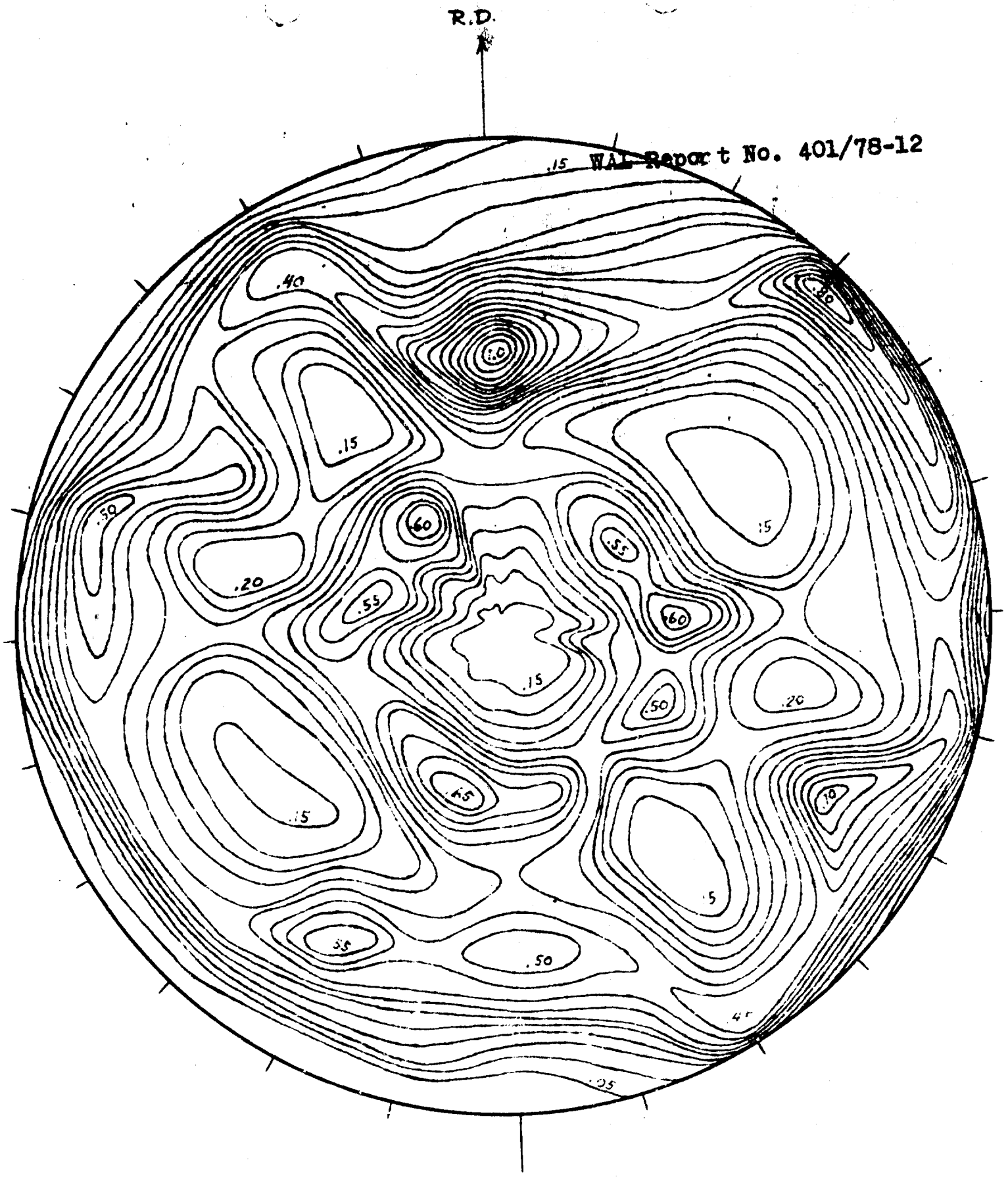


Figure 44 - (10$\bar{1}$1) Pole Figure for Material of Figure 43 Annealed in Argon for 1 Hour at 500°C.

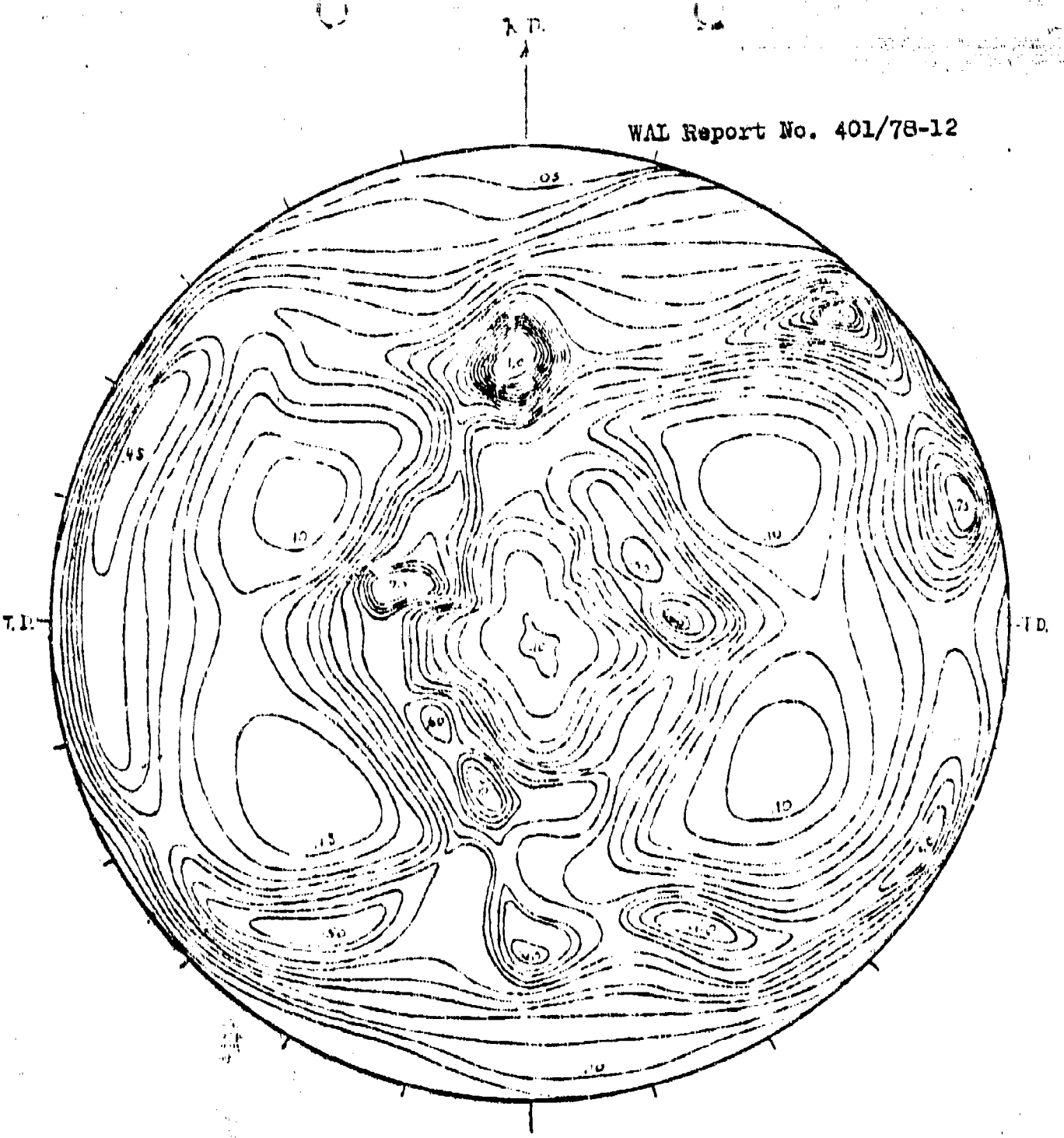


Figure 45 - (10$\bar{1}$1) Pole Figure for Material of Figure 44 Annealed in Argon for 1 Hour at 550°C.

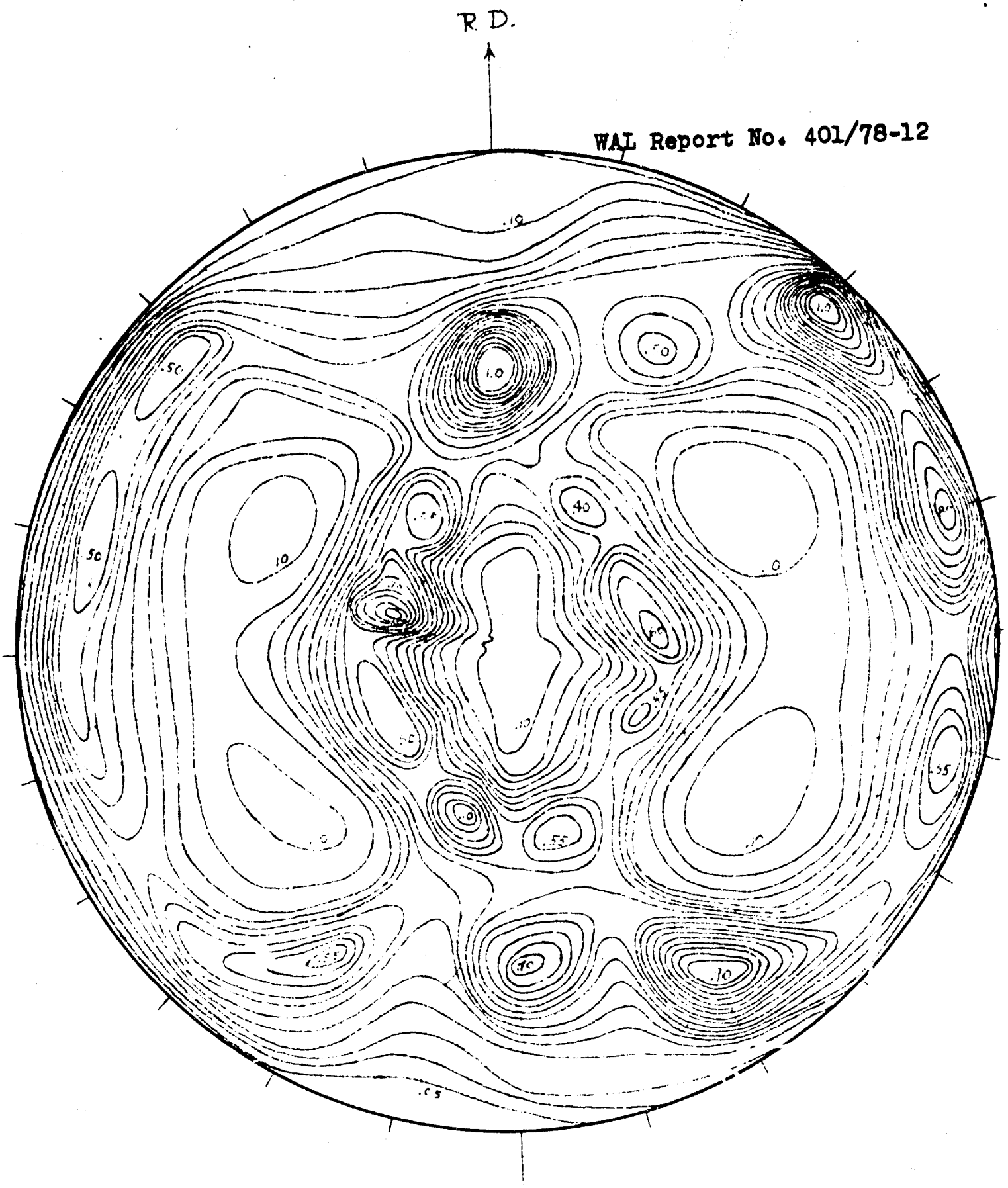


Figure 46 - (10$\bar{1}$1) Pole Figure for Material of Figure 45 Annealed in Argon for 1 Hour at 600°C.

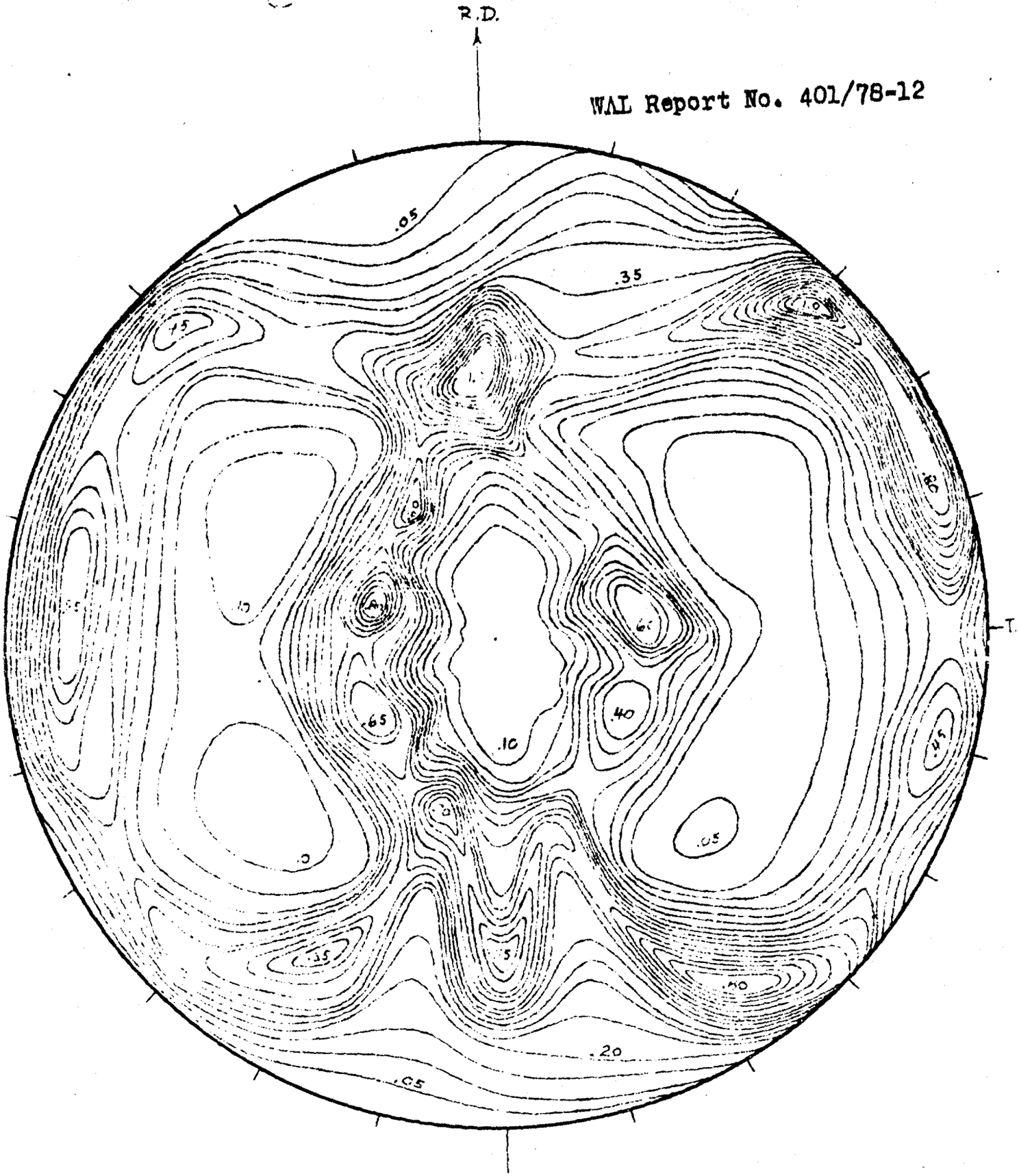


Figure 47 - (10$\bar{1}$1) Pole Figure for Material of Figure 46 Annealed in Argon for 1 Hour at 650°C.

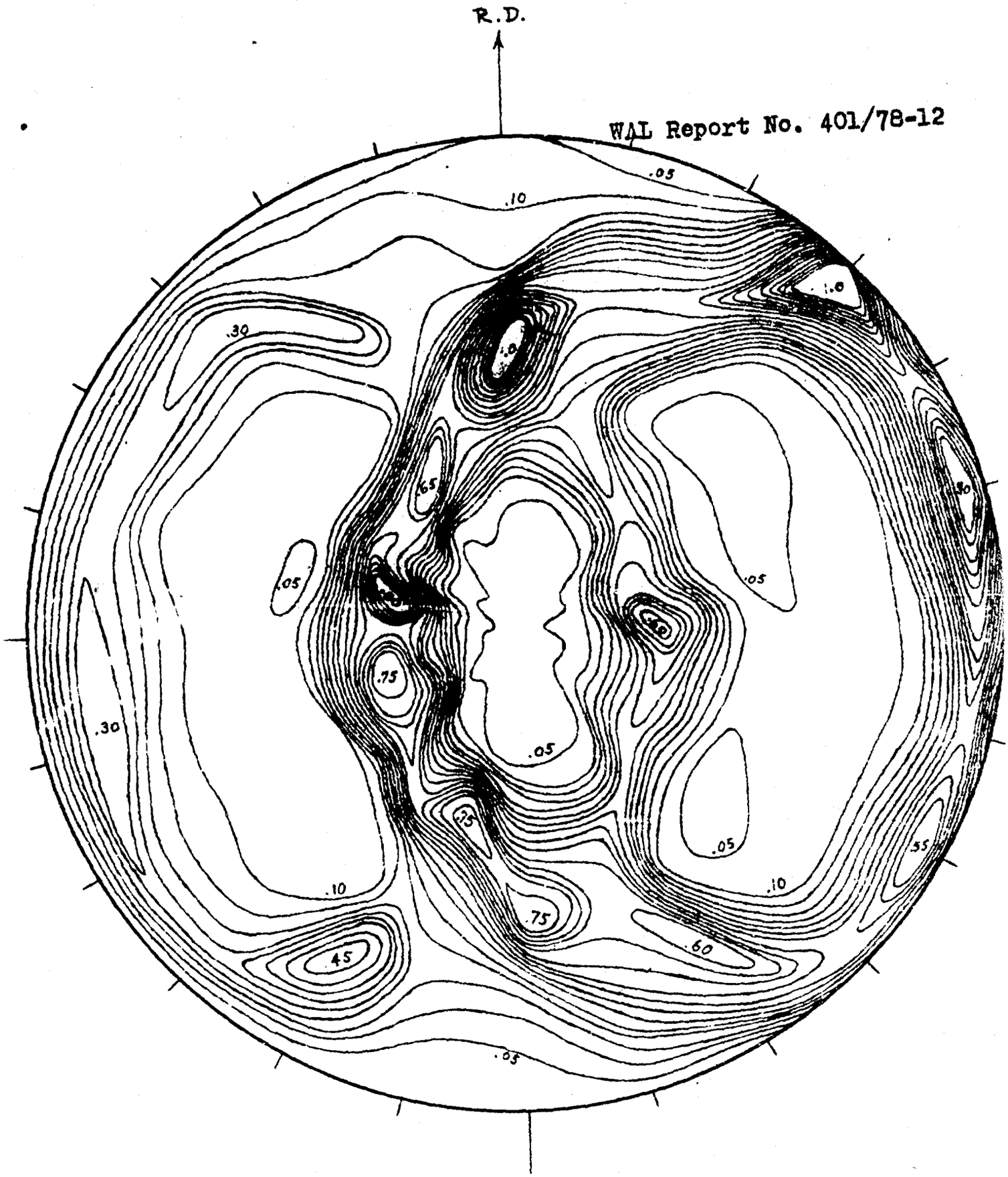


Figure 48 - (10$\bar{1}$1) Pole Figure for Material of Figure 47 Annealed in Argon for 1 Hour at 800°C.

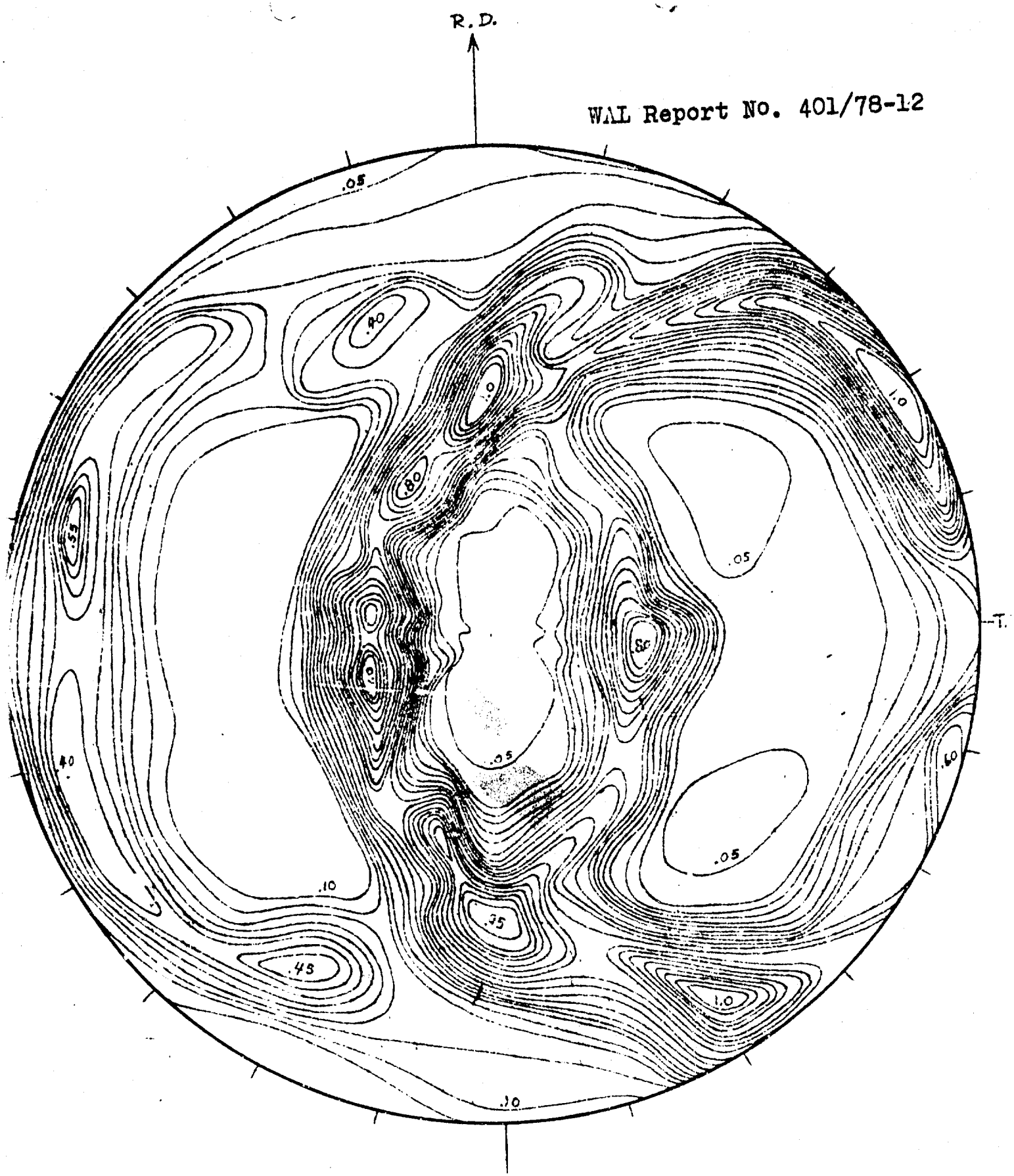


Figure 49 - (10$\bar{1}$1) Pole Figure for Material of Figure 48 Annealed in Argon for 1 Hour at 900°C.

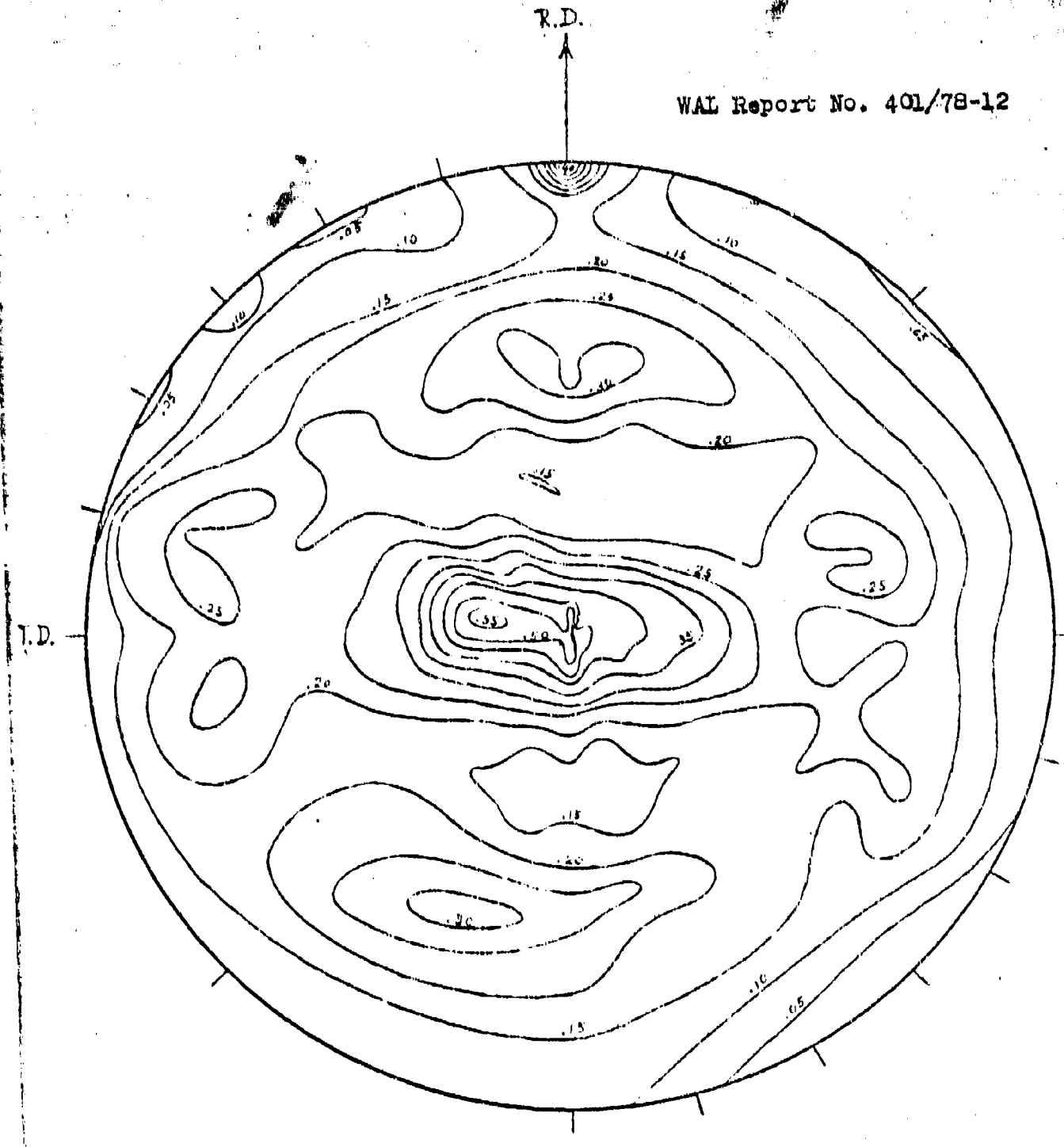


Figure 50 - (0002) Pole Figure for Some Commercial Titanium Sheet

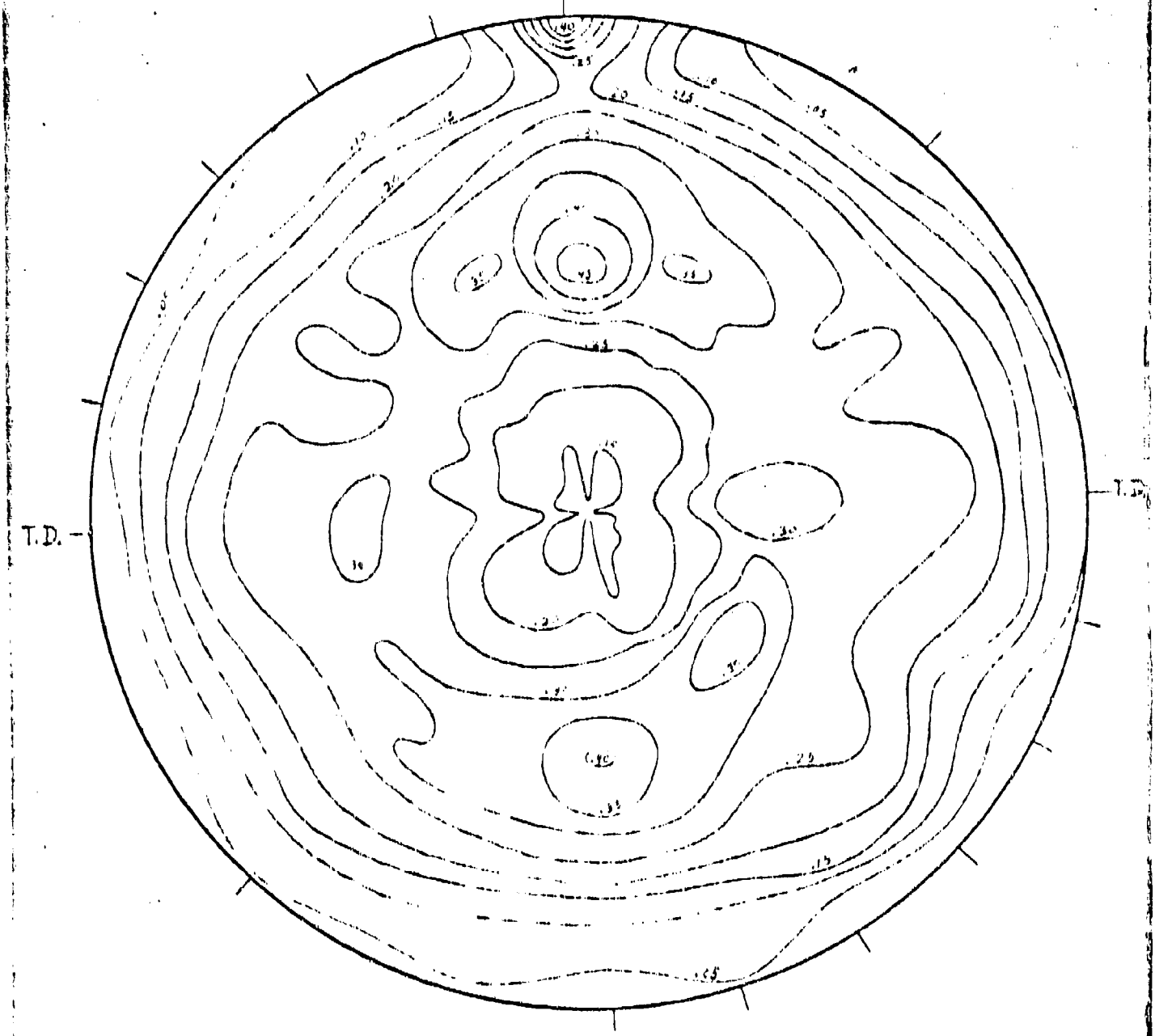


Figure 51 - $(10\bar{1}1)$ Pole Figure for Some Commercial Titanium Sheet