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METALLURGY AND CERAMICS
( M-3679, 22nd Ed. )

AEC RESEARCH AND DEVELOPMENT REPORT

# EFFECT OF TRANSFER TIME ON THE RECRYSTALLIZATION BEHAVIOR OF BETA-COOLED URANIUM


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JANUARY 1959

BRASS
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RESEARCH DEPARTMENT
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# EFFECT OF TRANSFER TIME ON THE RECRYSTALLIZATION BEHAVIOR OF BETA-COOLED URANIUM

## SUMMARY

A "critical transfer time" which relates to the time required to immerse samples at a temperature above the beta-alpha transformation in a quench media, directly affects the beta-cooled structure and its ease of recrystallization upon alpha phase annealing.

## OBJECT

The purpose of this investigation is to study the effect of transfer times on the recrystallization behavior of beta-cooled uranium.

## INTRODUCTION

In uranium technology, grain refinement has found wide application in the alteration of cast ingot and fuel element core structures. Techniques for the grain refinement of uranium can be listed in four major categories: 1. hot working in the alpha range, 2. cold working plus alpha phase annealing, 3. multiple beta-quenching, 4. beta-heat treating plus alpha phase annealing.

Refinement of cast structures of ingot and dingot uranium is generally accomplished through the technique of hot forging. The resultant equi-axed structure has been found desirable in the extrusion of billets to solid rod.(1)

In the past, grain refinement of cold and warm drawn rods has been accomplished by alpha phase annealing.(2) Restoration of ductility through grain refinement has been found necessary if additional reductions are anticipated. Further heat treatment, generally of a beta anneal source, is necessary to eliminate or reduce preferred orientation effects.

The importance of multiple beta heat treatments in the refinement of cast structures has been shown by Krashes et al.(3) Possible improvement of beta-quenched structures employing a beta-quench and alpha anneal as detailed by Gardner and Riches is worthy of note.[4] The latter heat-treatment has been reported to eliminate surface roughening of slugs upon irradiation in comparison to the slight roughening encountered among normally beta-heat treated slugs.[5] In addition, the ability of the beta-quench plus alpha anneal to produce greater grain refinement of cast structures than through multiple beta quenching has been indicated by some sites.

The grain refinement of cast structures via beta-quenching and alpha annealing can be likened to the process of recrystallization of cold worked uranium. In the former, internal lattice strains are developed as the result of rapid transformation from the beta to alpha phase. Manisfestations of the strain are found in the heavily twinned and sub-grained structures. Differences in the width of x-ray diffraction lines of beta-quenched and slowly cooled uranium are also indicative of the presence of internal lattice strains. Subsequent alpha phase annealing of the strained, beta-quenched structure results in the recrystallization of the large, irregular alpha grains to a fine grained, equi-axed structure.

The ease of recrystallization of the beta-quenched structure is believed to be dependent on the cooling rate from the beta to alpha phase on quenching. Gardner and Riches studied this effect through use of Jominy end quench specimens and showed that increased cooling rates decrease the activation energy for recrystallization.[6] Unpublished data of a DuPont source has also indirectly corroborated this behavior.[7]

This program was undertaken with the purpose of furthering the contribution to the study of the recrystallization behavior of beta-quenched uranium by incorporating the effects of transfer time from the beta heat-trating bath to the quench media. Because of the apparent versatility and expediency of the quench strain-anneal heat treatment, it is felt that data culled from this program will be of a practical nature and aid in designing of future, large scale billet heat treating programs planned by this site for the improvement of cast structures prior to extrusion.

## PROCEDURE

The variables which this investigation covered resulted in the use of 151 samples of ingot and 151 samples of dingot uranium. These samples were derived from lengths of eleven-inch sections of 3/8-inch alpha extruded rods which were beta-heat treated at 730°C - 1346°F for 15 minutes in a molten lithium carbonate (46 wt. %) - potassium carbonate (54 wt. %) salt bath. The rods were quenched in room temperature water after holding for various delay times in air. The delay or transfer times were 5, 10, 20, 30, 45 and 60 seconds. One-inch of rod was cropped off each end of the section to eliminate any uneven end effects resulting from the quench.

The as-beta treated rods were cut into 3/4"-long samples which were identified according to the respective beta and alpha annealing treatment. Annealing was carried out in a molten Quick Temper #275 Neutral Salt bath at 426°C - 799°F, 450°C - 842°F, and 500°C - 932°F, and a molten Houghton #980 bath at 550°C - 1022°F, 600°C - 1112°F, and 640°C - 1184°F for 15, 30, 60 and 120 minutes.

After heat treatment a transverse face of each sample was rough polished and then macroetched with a 1:1 hydrochloric and 1:1 nitric acid solution and metallographically examined with the aid of a stereomicroscope. After viewing, selected heat-treated series were photographed to depict representative beta-cooled and annealed structures.

Rockwell G hardness readings were taken on the transverse face of all the dingot samples.

A cooling curve slug as shown in Fig. 1 was used to simulate and gain information on the cooling of the 3/8-inch laboratory samples from the beta phase. The slug was heated at 730°C - 1346°F for 15 minutes in a Houghton #980 salt bath and held at prescribed transfer times prior to quenching. The bath temperature was controlled by a Honeywell millivolt recorder. A thermocouple inserted into the milled hole of the slug, and which was then press fitted by packing with uranium chips, was used in conjunction with a direct reading Foxboro potentiometer to follow the cooling of the slug upon transfer and quenching. The laboratory set-up used for this phase of the work as well as the heat treating is shown in Fig. 2.

## RESULTS

Table I provides a comprehensive listing of the heat treating parameters employed in this program and details the effect of transfer times on the recrystallization behavior of beta-cooled dingot and ingot uranium. Miscellaneous information includes Rockwell G hardness and grain size data. In addition to Table I, photomacrographs depicting a cross-section of the structures developed on heat treating are shown in Figs. 4-10. The series of photo-macrographs represent dingot uranium which presented less difficulty during etching. Ingot structures developed under similar heat treating conditions were comparable to dingot, but were more difficult to resolve by etching.

STRUCTURE - Fig. 3 represents a typical macrostructure of alpha extruded dingot uranium stock employed for the heat treating program. The structure is duplex in nature and resembles corresponding ingot uranium stock.

Figs. 4a and b represent typical beta cooled structures developed upon quenching after holding in air for short transfer times (5 and 10 seconds) and longer times (20 to 60 seconds), respectively. Note the columnar, grain structure at the perimeter of the short transfer time sample depicted in Fig. 4a as compared to the more uniform structure of the slow transfer time sample shown in Fig. 4b.

Figs. 5a - d, represent the macrostructure of a typical series of samples quenched after short transfer times (5 and 10 seconds) and then alpha phase annealed in the temperature range 425°C - 500°C (797°F - 932°F) for 15-120 minutes. The beta-cooled structure, typical of samples being quenched after short transfer times, appears unaltered.

Figs. 6a - d, represent the macrostructure of a typical series of samples quenched after short transfer times, but, alpha phase annealed at 600°C - 1112°F for 15-120 minutes. Retention of the beta-cooled structure can be noted in Fig. 6a (600°C - 1112°F - 15 minutes). However, longer annealing times have lead to the development of a fully recrystallized structure as shown by Figs. 6b, c and d.

Figs. 7a - c represent the macrostructure of a series of samples quenched after short transfer times and annealed at the highest temperature employed in this program (640°C - 1184°F). Full recrystallization is apparent.

Figs. 8a - d and 9a - d represent the macrostructure of a series of samples quenched after intermediate transfer times (20 and 30 seconds) and alpha phase annealed at 640°C - 1184°F for 15-120 minutes. The beta-cooled structure is retained up to a 60 minute annealing time. At 120 minutes Fig. 8d, a coarse, recrystallized structure is developed. However, some retention of the beta-cooled structure is evident.

Figs. 10a - d represent the macrostructure of a series of samples quenched after long delay times (45 and 60 seconds) and alpha phase annealed at 640°C - 1184°F for 15-120 minutes. Structurally, this series appears comparable to the previous series, but exhibits a coarser recrystallized structure, Fig. 10d, at the 120 minute anneal. Retention of some beta-cooled structure is also evident.

COOLING RATE - Fig. 11 presents cooling curve data obtained through use of the test slug employed to simulate the cooling of 3/8" samples from the beta to alpha phase. The curves reveal the following information. At transfer times of 5 and 10 seconds, immersion of the slug in the quench media is initiated above the beta-alpha transformation temperature. However, for 20-60 second transfer times, immersion is initiated below the transformation temperature and in the high alpha range.

HARDNESS - Fig. 12 presents the effect of transfer time on the Rockwell G hardness of beta-cooled dingot uranium. The curve reveals a tendency for the hardness to decrease slightly with increased transfer times.

## DISCUSSION OF RESULTS

Analysis of the data presented in Table I and by photomacrographs of representative heat treated series, Figs. 4-10, reveals a number of noteworthy trends. These trends relate to the behavior of uranium upon beta heat treatment and alpha phase annealing.

BETA-COOLED STRUCTURE - At 5 and 10 second transfer times, a columnar structure is prevalent at the perimeter of beta-cooled specimens. The formation of the columnar structure may be attributed to the alignment of newly transformed alpha grains along a steep temperature gradient, that being a radial direction towards the core of the sample. The randomized, coarse grained structure of the core is indicative of less severe cooling across the beta-alpha transformation temperature. At intermediate (20 and 30 seconds) and longer transfer times (45 and 60 seconds) the uniform beta-cooled structure developed is indicative of slow cooling across the beta-alpha transformation temperature. The significance of the columnar structure will be revealed further on in the discussion. It will suffice to say at this point that the presence of a columnar structure is indicative of material that can be readily recrystallized on alpha phase annealing.

ALPHA-ANNEALED STRUCTURE - The recrystallization behavior of beta-cooled uranium as affected by transfer times follows a definite pattern. The trend may be stated as follows: As the transfer time from salt bath to quench media increases, it becomes increasingly difficult to recrystallize beta-quenched uranium. Samples quenched after short transfer times tend to recrystallize at lower annealing temperatures and times than samples quenched after longer transfer times. As the transfer times increase the annealing temperature necessary to produce a recrystallized structure increases. In addition, the grain size of the recrystallized structure produced on annealing long transfer time beta-cooled samples is nearly three times as great as recrystallized structures of short transfer time beta-quenched samples.

The only apparent difference between dingot and ingot behavior is realized for the samples beta-cooled after transfer times of 45 and 60 seconds. Recrystallization is not evident for the ingot samples, alpha phase annealed at 600°C - 1112°F and 640°C - 1184°F.

COOLING RATE STUDY - If the ease or degree of grain refinement can be related to the rate of cooling across the beta-alpha transformation temperature, it appears that a slow cooling rate induced by long transfer times would lead to a less severely strained internal lattice and increase the required activation energy for recrystallization. The corresponding structure would, therefore, be difficult to refine during alpha phase annealing. The cooling rate study phase of the heat treating program was designed to provide practical information pertinent to the above consideration. The information obtained from this aspect of the work as presented in Fig. 9 reveals the following information. Beyond a 15 second transfer time, immersion of the beta-heat treated slugs into the quench media takes place below the beta-alpha transformation temperature of 668°C - 1234°F. Therefore, cooling across the transformation temperature, because it occurs in air, is slow and results in little strain hardening. The difficulty in recrystallizing the structure of samples undergoing similar cooling bears out the above consideration.

If quenching after short transfer times is reviewed from a classical standpoint, a sample could be considered as to pass through three distinct cooling stages. In the first or A stage, a thin vapor film surrounds the specimen and promotes slow cooling. In the second or B stage, the film is broken as evidenced by the formation of bubbles which break away from the surface of the sample. Cooling in this stage is the most severe and occurs through conduction and convection. The third stage appears as the temperature of the specimen drops below the boiling point of the liquid. Transfer of heat is due largely to convection currents within the liquid and is less severe than the B stage. Duwez has shown that by increasing the cooling rate, the transformation temperatures of uranium can be depressed.[8] Therefore, the possibility exists that with increased cooling rates which occur concurrently with short transfer times, the beta-alpha transformation is depressed and occurs in the B stage. Subsequently, an optimum strained lattice is produced. The presence of a columnar structure and its ease of refinement, while not supporting the depression of the transformation temperature or B stage cooling concept, does definitely confirm that fast cooling across the beta-alpha transformation lessens the required activation energy for recrystallization.

If the term "critical transfer time" was applied to beta-cooled uranium, in essence it would refer to the time required to insure immersion of samples at temperatures above the beta-alpha transformation in a quench media. This term can then be related to the ease or degree of recrystallization of beta-cooled uranium.

It must be remembered that this discussion is confined to the characteristic behavior of 3/8-inch samples. Undoubtly, longer transfer times can be tolerated with larger sections, although a limit of cross-sectional penetration exists as to grain refinement.

HARDNESS - With increased transfer times, a decrease in the hardness of beta-cooled structure may be expected. The softening is attributed to the coarsening of the alpha structure due to longer times spent in the high alpha range.

## CONCLUSIONS

A "critical transfer time" which relates to the time required to immerse samples at a temperature above the beta-alpha transformation in a quench media, directly affects the beta-cooled structure and its ease of recrystallization upon alpha phase annealing.

Within the "critical transfer time" the ease of recrystallization and degree of grain refinement of beta-cooled samples as measured by alpha phase annealing temperatures and times are superior to samples quenched beyond the "critical transfer time."

## REFERENCES

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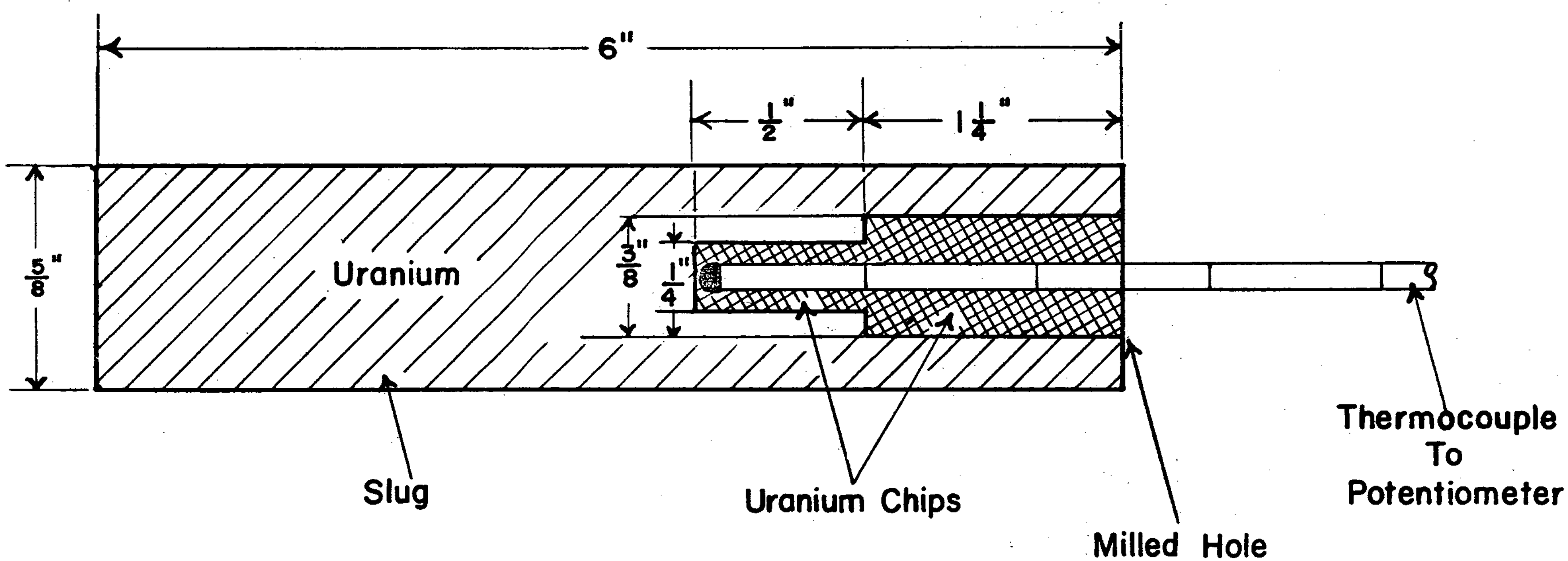
FIGURE — 1
COOLING TEST SLUG

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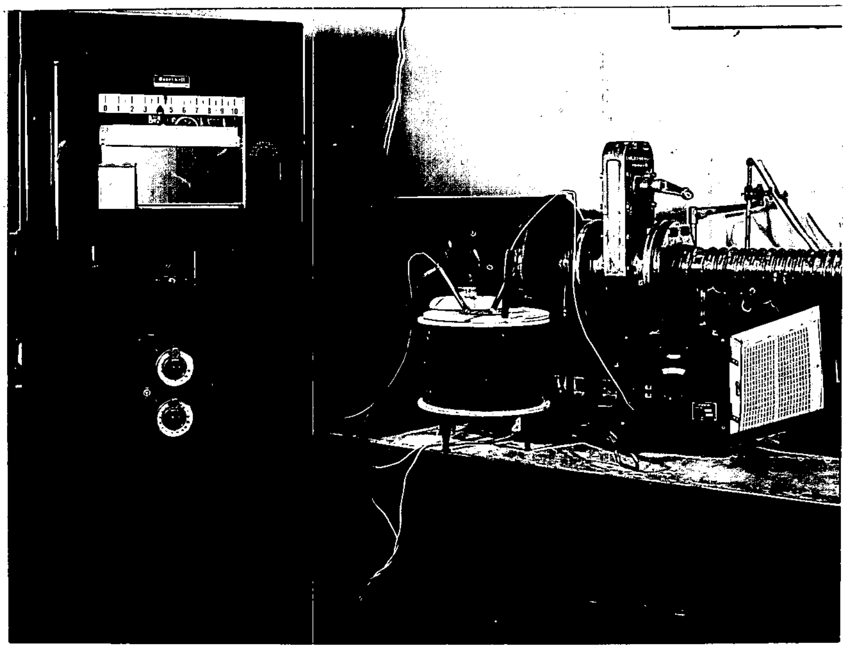

Fig. 2 - Laboratory heat-treating and accessory equipment.

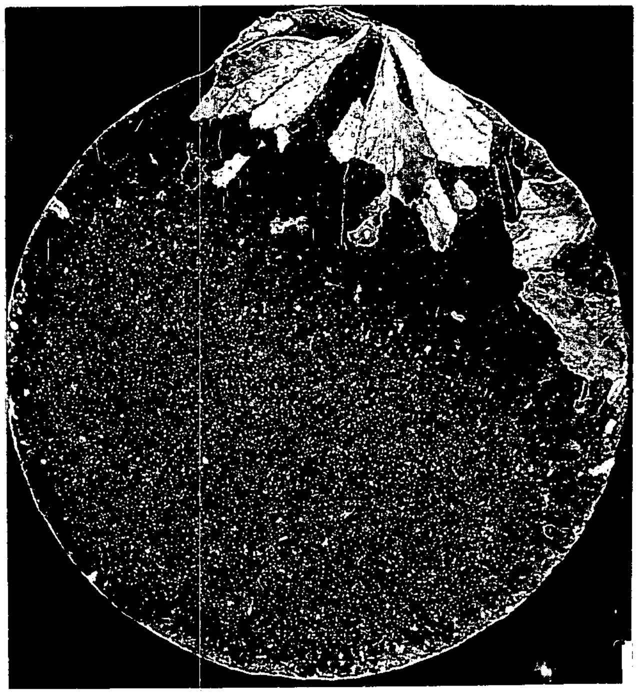

Fig. 3 - Alpha extruded dingot uranium stock.

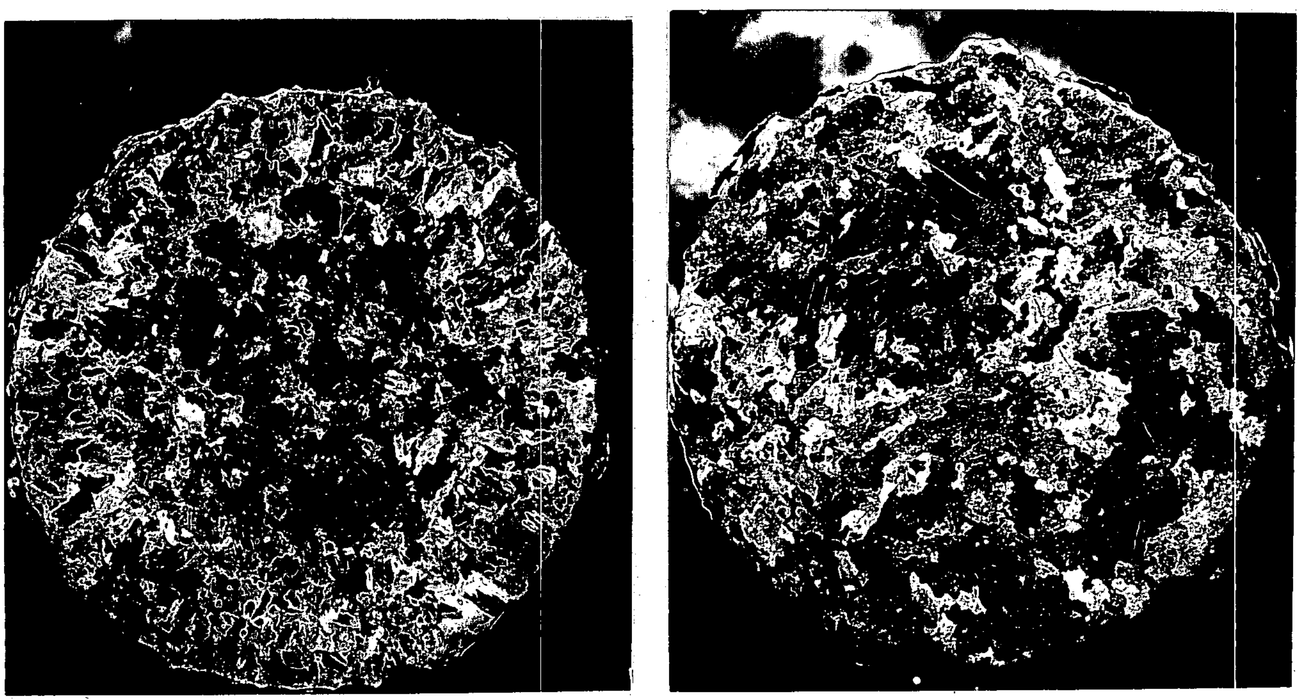

Fig. 4 - As-beta-cooled structure heat treated at 730°C - 15 minutes.

a. Left - Dingot 5 second transfer time.

b. Right - Dingot 30 second transfer time.

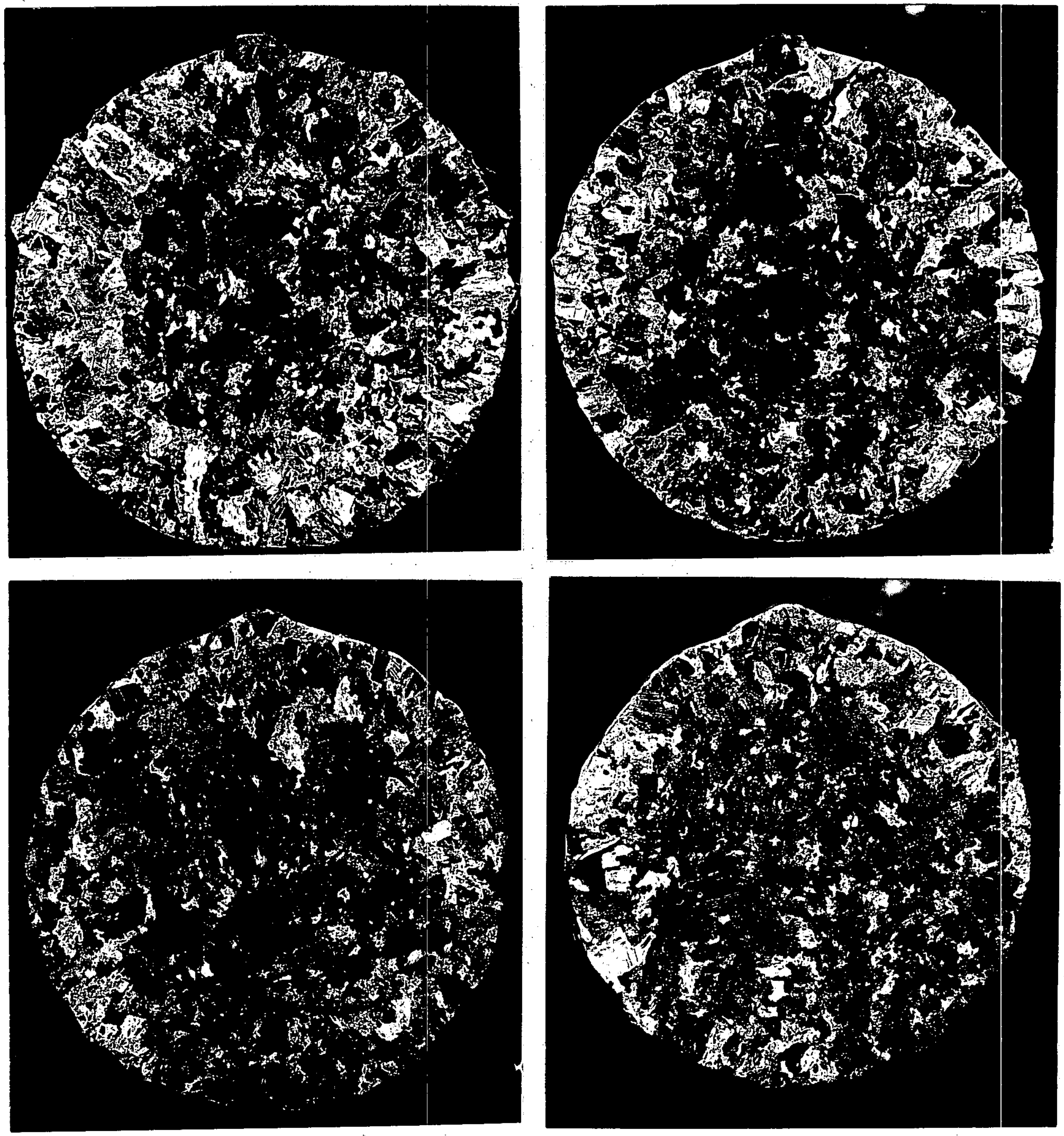

Fig. 5a - d - Effect of a low annealing temperature on the recrystallization behavior of samples quenched after short transfer times. Transfer Time - 5 seconds.

Series 1, 3, 5, and 7 Dingot-Beta-treated 730°C - 15 min.

a. Top Left - Annealed 425°C - 15 min.

b. Top Right - Annealed 425°C - 30 min.

c. Bottom Left - Annealed 425°C - 60 min.

d. Bottom Right - Annealed 425°C - 120 min.

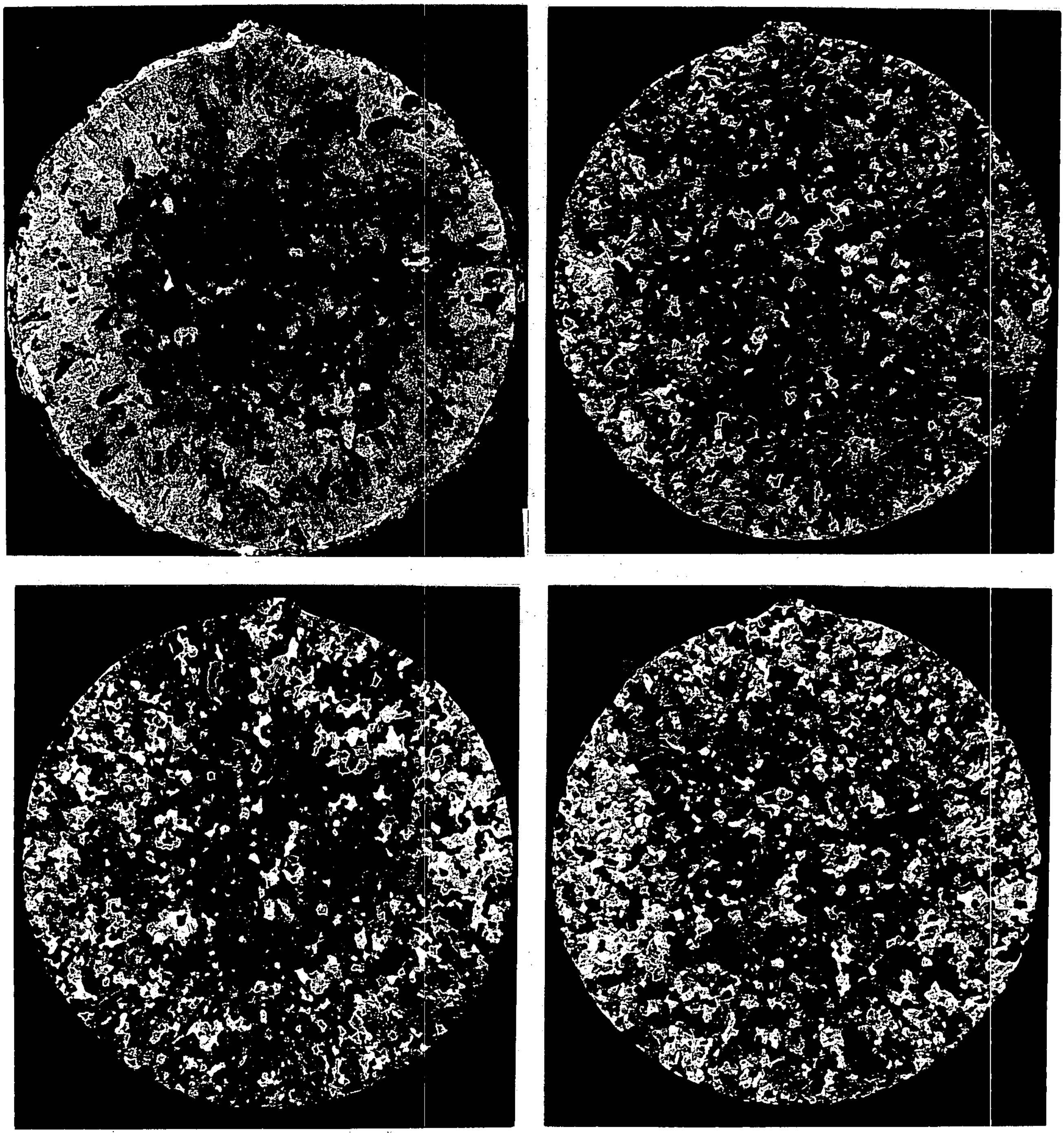

Fig. 6a - d - The effect of high annealing temperature on the recrystallization behavior of samples quenched after short transfer times. Transfer Time - 5 seconds.

Series D Dingot-Beta-treated 730°C - 15 min.

a. Top Left - Annealed 600°C - 15 min.

b. Top Right - Annealed 600°C - 30 min.

c. Bottom Left - Annealed 600°C - 60 min.

d. Bottom Right - Annealed 600°C - 120 min.

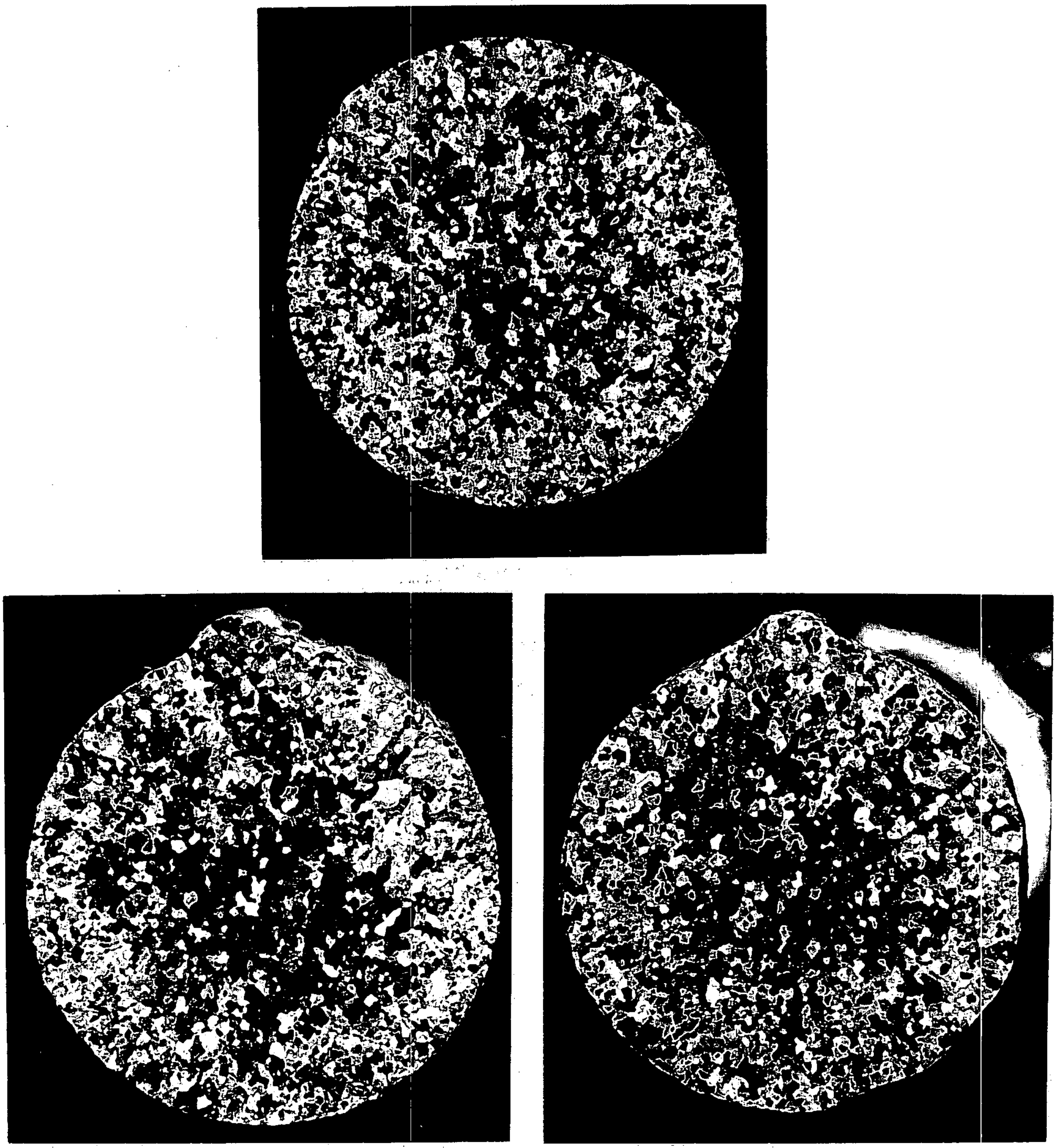

Fig. 7a - c - Effect of a very high annealing temperature on the recrystallization behavior of samples quenched after short transfer times. Transfer Time - 10 seconds.

Series L Dingot-Beta-treated 730°C - 15 min.

a. Top - Annealed 640 C - 15 minutes

b. Bottom Left - Annealed 640°C - 60 min.

c. Bottom Right - Annealed 640°C - 120 min.

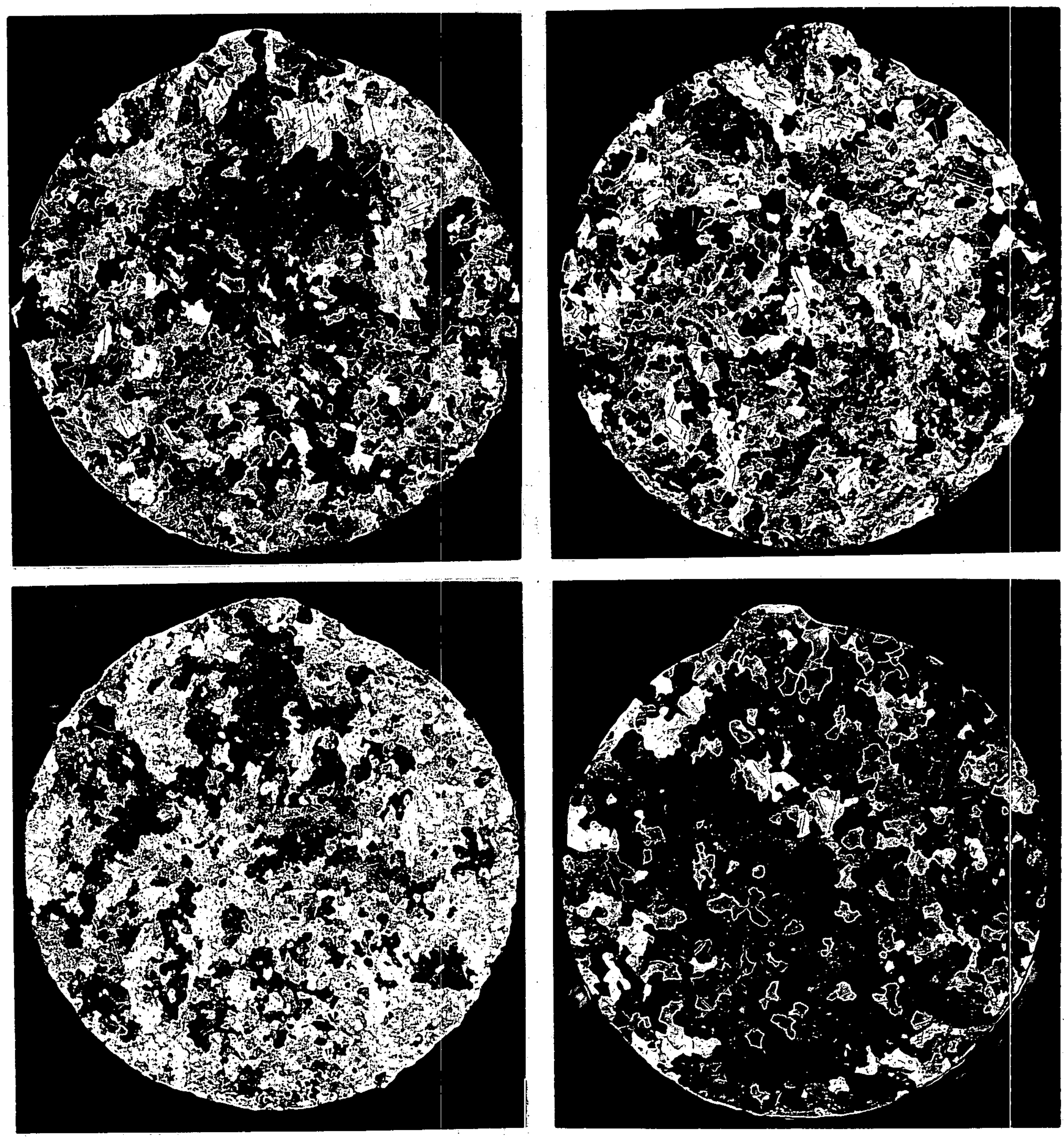

Fig. 8a - d - Effect of annealing temperature on the recrystallization behavior of samples quenched after intermediate transfer times. Transfer Time - 20 seconds.

Series R — Dingot-Beta-treated 730°C - 15 min.

a. Top Left - Annealed 640°C - 15 min.

b. Top Right - Annealed 640°C - 30 min.

c. Bottom Left - Annealed 640°C - 60 min.

d. Bottom Right - Annealed 640°C - 120 min.

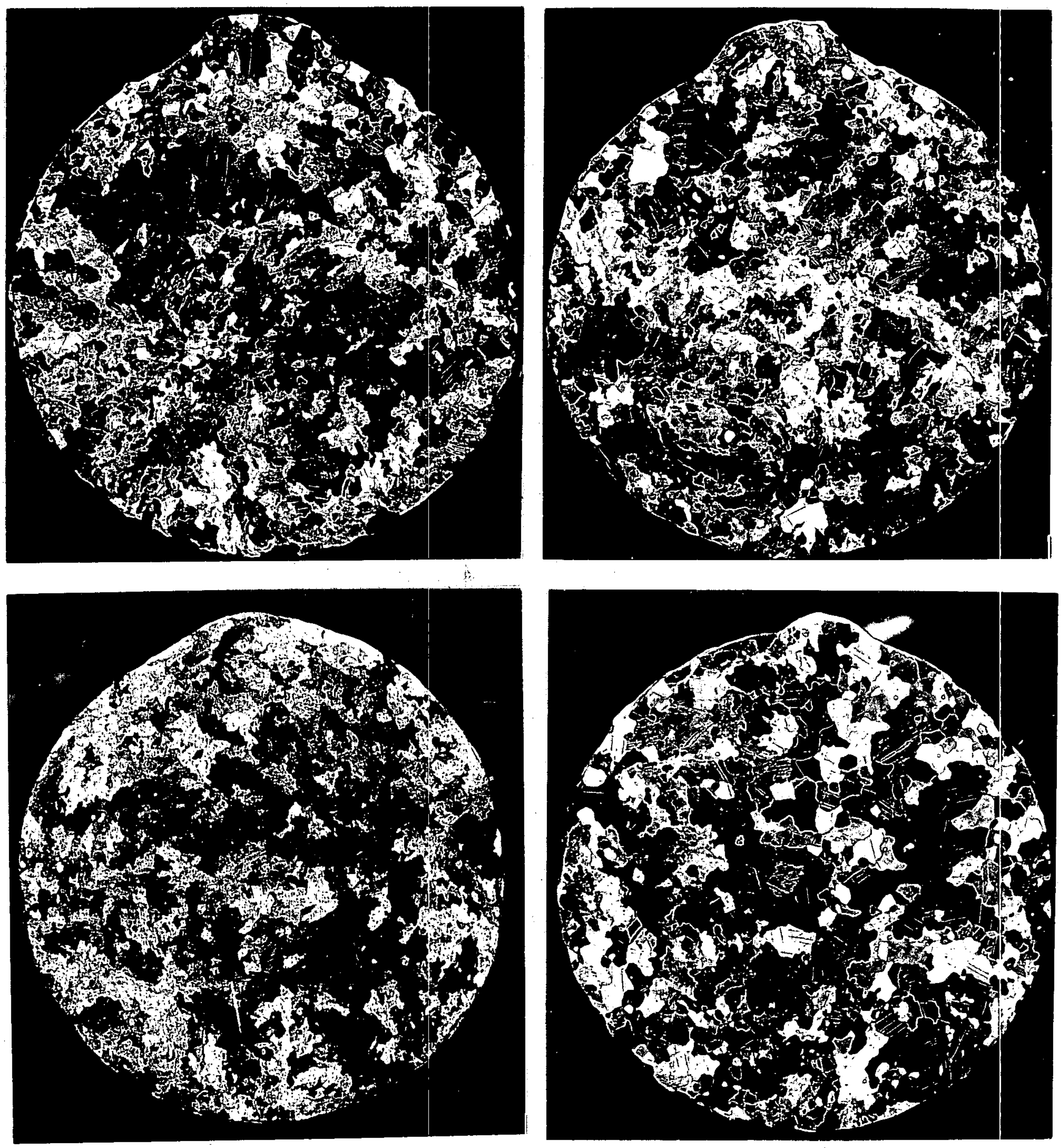

Fig. 9a - d - Effect of annealing temperature on the recrystallization behavior of samples quenched after intermediate transfer times. Transfer Time - 30 seconds.

Series X Dingot-Beta-treated 730°C - 15 min.

a. Top Left - Annealed 640°C - 15 min.

b. Top Right - Annealed 640°C - 30 min.

c. Bottom Left - Annealed 640°C - 60 min.

d. Bottom Right - Annealed 640°C - 120 min.

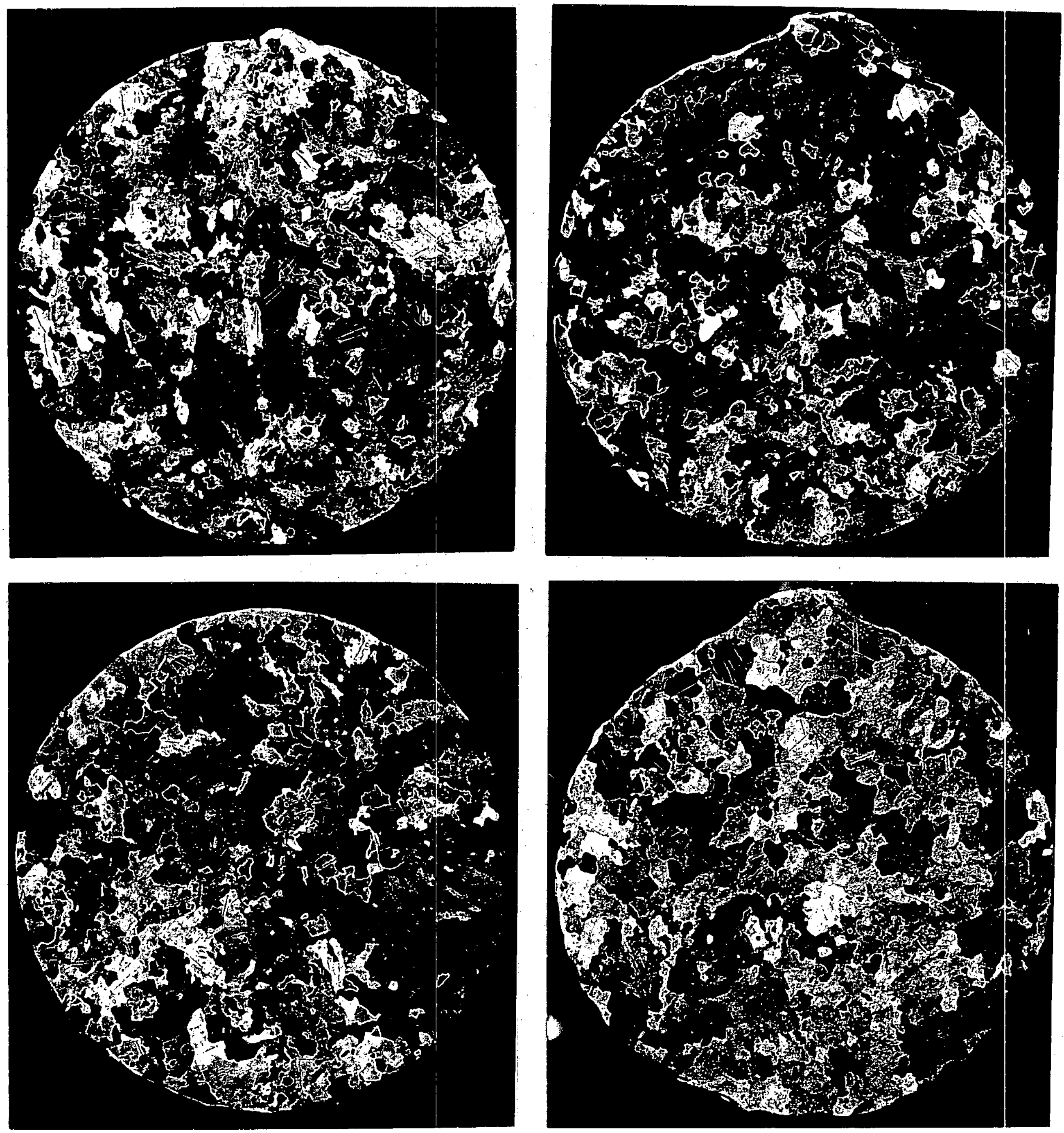

Fig. 10a - d - Effect of annealing temperature on the recrystallization behavior of samples quenched after long transfer times. Transfer Time - 45 seconds.

Series AD Dingot-Beta-treated 730°C - 15 min.

a. Top Left - Annealed 640°C - 15 min.

b. Top Right - Annealed 640°C - 30 min.

c. Bottom Left - Annealed 640°C - 60 min.

d. Bottom Right - Annealed 640°C - 120 min.

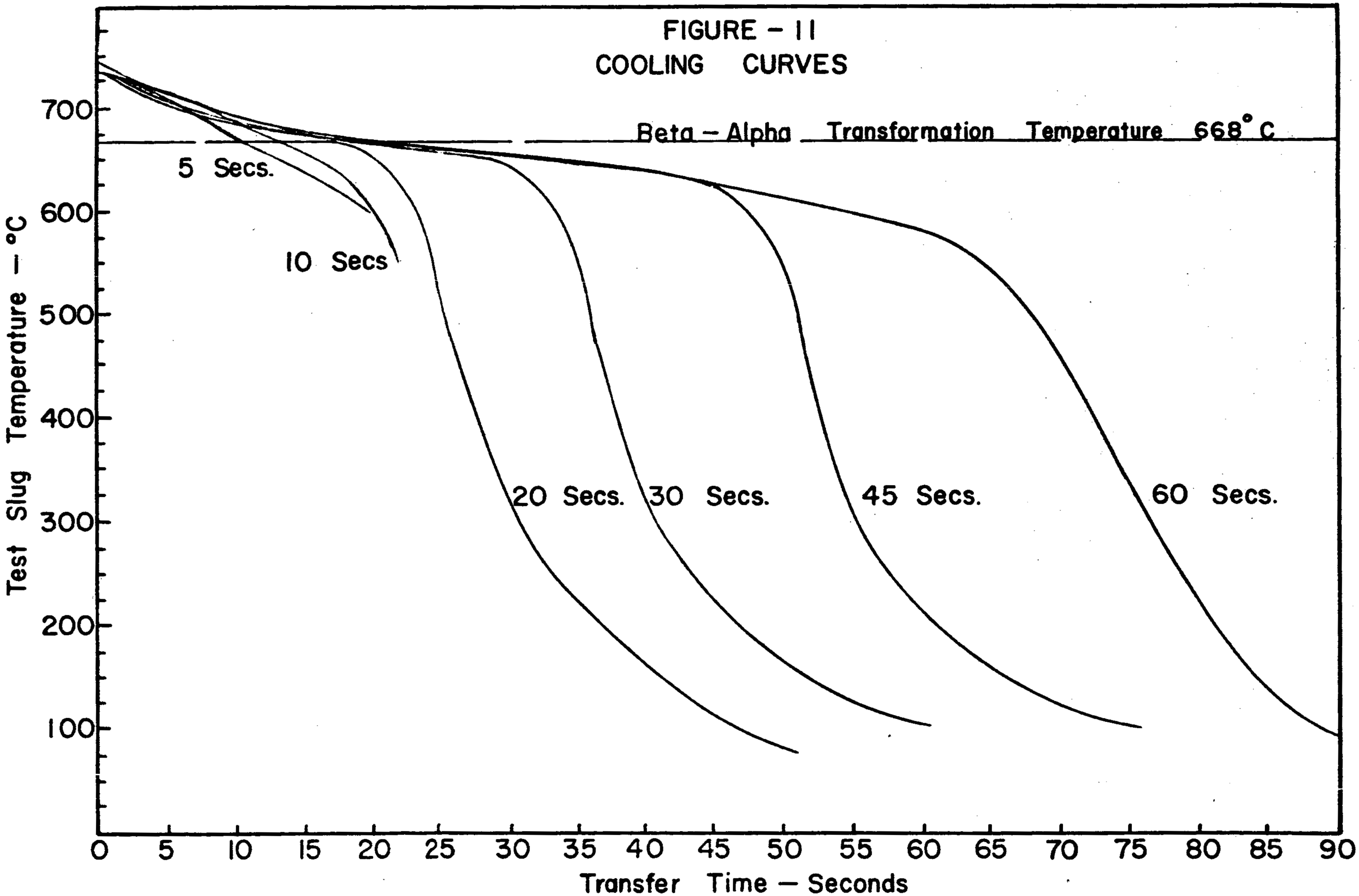


FIGURE – 11
COOLING CURVES

FIGURE - 12
ROCKWELL G HARDNESS VS TRANSFER TIME

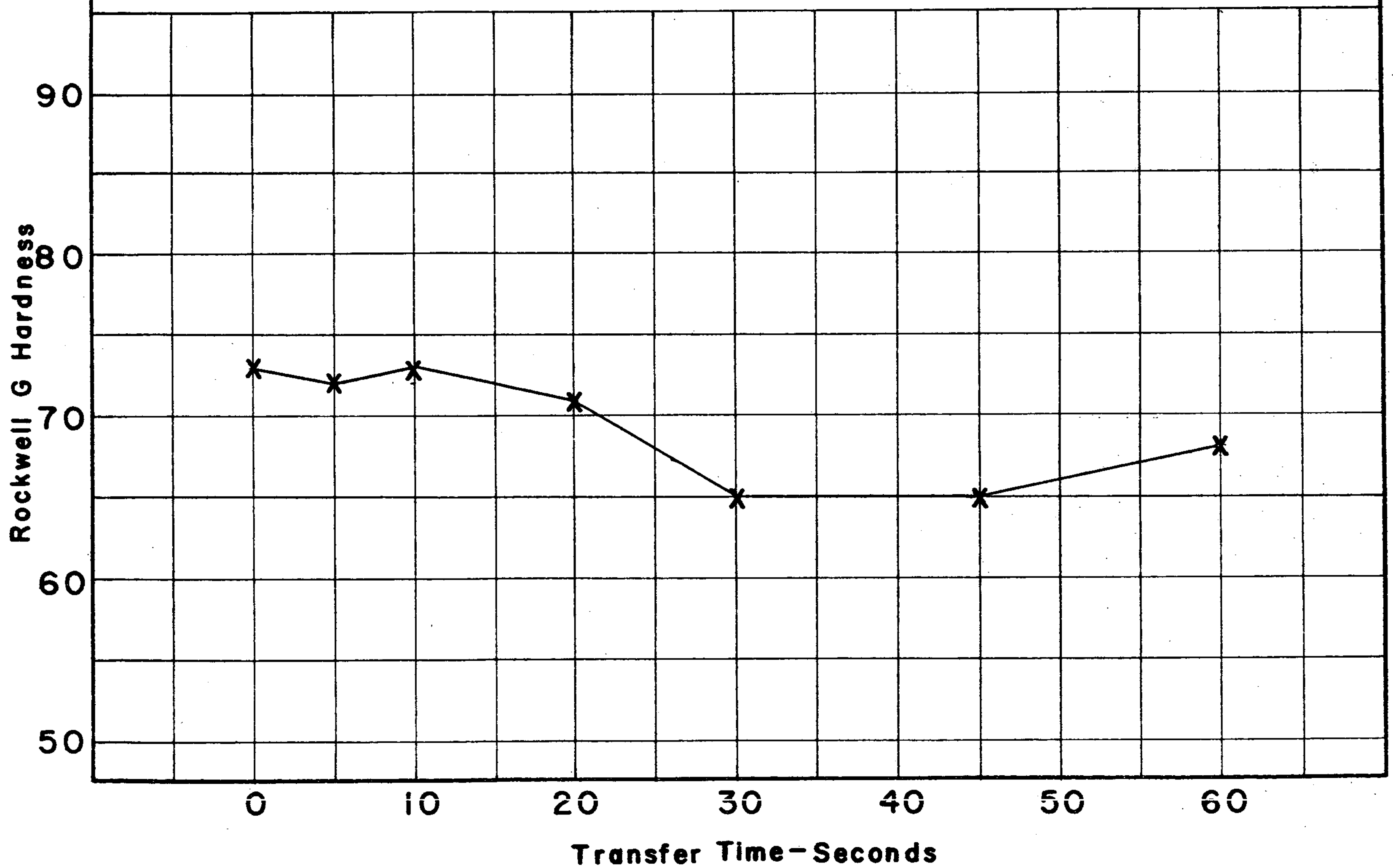


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TABLE I

HEAT TREATING AND METALLOGRAPHIC DATA

| INGOT NO. | HEAT-TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | MACROSTRUCTURAL REMARKS |
|---|---|---|---|
| 2-1 | As-extruded (1175°C) | | One-quarter of sample face exhibits a coarse grained, alpha hot-worked structure, remainder fine grained. |
| | Beta-treated 730°C - 15 min. | | |
| A | | 5 | Typical beta-cooled structure - Coarser at core. |
| 2 | Annealed 425°C for 15 min. | " | Beta-cooled structure, columnar perimeter, coarser at core. |
| 4 | " " 30 min. | " | " " |
| 6 | " " 60 min. | " | " " |
| 8 | " " 120 min. | " | " " |
| 2A | Annealed 450°C for 15 min. | " | Beta-cooled structure, coarser at core. |
| 4A | " " 30 min. | " | |
| 6A | " " 60 min. | " | " " |
| 8A | " " 120 min. | " | " " |
| 2B | Annealed 500°C for 15 min. | " | Beta-cooled structure, coarser at core. |
| 4B | " " 30 min. | " | |
| 6B | " " 60 min. | " | Beta-cooled structure. |
| 8B | " " 120 min. | " | Beta-cooled structure, coarser at core. |
| 2C | Annealed 550°C for 15 min. | " | Beta-cooled structure. |
| 4C | " " 30 min. | " | Uniform beta-cooled structure. |
| 6C | " " 60 min. | " | Beta-cooled structure, columnar perimeter, coarser at core. |
| 8C | " " 120 min. | " | Beta-cooled structure, coarser at core. |



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TABLE I

-Continued-

| INGOT NO. | HEAT-TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | MACROSTRUCTURAL REMARKS |
|---|---|---|---|
| 2D | Annealed 600°C for 15 min. | 5 | Full recrystallization except for partially recrystallized core. |
| 4D | " " 30 min. | " | Full recrystallization, slightly coarser at core. |
| 6D | " " 60 min. | " | " " |
| 8D | " " 120 min. | " | " " |
| 2E | Annealed 640°C for 15 min. | " | Full recrystallization, slightly coarser at core. |
| 4E | " " 30 min. | " | " " |
| 6E | " " 60 min. | " | " " |
| 8E | " " 120 min. | " | " " |
| | Beta-treated 730°C - 15 min. | | |
| C | | 10 | Beta-cooled structure, coarser at core. |
| 2F | Annealed 425°C for 15 min. | " | Beta-cooled structure, columnar perimeter, coarser at core. |
| 4F | " " 30 min. | " | |
| 6F | " " 60 min. | " | Beta-cooled structure. |
| 8F | " " 120 min. | " | Beta-cooled structure, columnar perimeter. |
| 2G | Annealed 450°C for 15 min. | " | Beta-cooled structure, slight columnar perimeter. |
| 4G | " " 30 min. | " | |
| 6G | " " 60 min. | " | " " |
| 8G | " " 120 min. | " | " " |
| 2H | Annealed 500°C for 15 min. | " | Beta-cooled structure, columnar perimeter, coarser at core. |
| 4H | " " 30 min. | " | Beta-cooled structure. |
| 6H | " " 60 min. | " | Beta-cooled structure, columnar perimeter, coarser at core. |
| 8H | " " 120 min. | " | " " |

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TABLE I

-Continued-

| INGOT NO. | HEAT-TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | MACROSTRUCTURAL REMARKS |
|---|---|---|---|
| 2J | Annealed 550°C for 15 min. | 10 | Beta-cooled structure, coarser at center. |
| 4J | " " 30 min. | " | Beta-cooled structure, slightly columnar perimeter. |
| 6J | " " 60 min. | " | |
| 8J | " " 120 min. | " | " " |
| 2K | Annealed 600°C for 15 min. | " | Full recrystallization except for partially recrystallized core. |
| 4K | " " 30 min. | " | |
| 6K | " " 60 min. | " | Recrystallization of core more complete. |
| 8K | " " 120 min. | " | " " |
| 2L | Annealed 640°C for 15 min. | " | Full crystallization. |
| 4L | " " 30 min. | " | " " |
| 6L | " " 60 min. | " | " " |
| 8L | " " 120 min. | " | " " |
| | Beta-treated 730°C - 15 min. | | |
| E | | 20 | Beta-cooled structure, coarser at core. |
| 2M | Annealed 425°C for 15 min. | " | " " |
| 4M | " " 30 min. | " | " " |
| 6M | " " 60 min. | " | Uniform beta-cooled structure. |
| 8M | " " 120 min. | " | " " |
| 2N | Annealed 450°C for 15 min. | " | Uniform beta-cooled structure. |
| 4N | " " 30 min. | " | " " |
| 6N | " " 60 min. | " | " " |
| 8N | " " 120 min. | " | Beta-cooled structure. |

TABLE I

-Continued-

| INGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | MACROSTRUCTURAL REMARKS |
|---|---|---|---|
| 20 | Annealed 500°C for 15 min. | 20 | Uniform beta-cooled structure. |
| 40 | " " 30 min. | " | Beta-cooled structure, radial growth at core. |
| 60 | " " 60 min. | " | " " |
| 80 | " " 120 min. | " | " " |
| 2P | Annealed 550°C for 15 min. | " | Beta-cooled structure. |
| 4P | " " 30 min. | " | " " |
| 6P | " " 60 min. | " | " " |
| 8P | " " 120 min. | " | " " |
| 2Q | Annealed 600°C for 15 min. | " | Beta-cooled structure. |
| 4Q | " " 30 min. | " | " " |
| 6Q | " " 60 min. | " | " " |
| 8Q | " " 120 min. | " | " " |
| 2R | Annealed 640°C for 15 min. | " | Uniform beta-cooled structure. |
| 4R | " " 30 min. | " | Beta-cooled structure, slight evidence of preliminary recrystallization. |
| 6R | " " 60 min. | " | Recrystallization, but coarse in nature. |
| 8R | " " 120 min. | " | Partial recrystallization. |
| | Beta-treated 730°C - 15 min. | | |
| G | | 30 | Beta-cooled structure, coarser at core. |
| 2S | Annealed 425°C for 15 min. | " | Uniform beta-cooled structure. |
| 4S | " " 30 min. | " | Beta-cooled structure, radial growth at core. |
| 6S | " " 60 min. | " | Beta-cooled structure. |
| 8S | " " 120 min. | " | Uniform beta-cooled structure. |

TABLE I

-Continued-

| INGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | MACROSTRUCTURAL REMARKS |
|---|---|---|---|
| 2T | Annealed 450°C for 15 min. | 30 | Uniform beta-cooled structure. |
| 4T | " " 30 min. | " | |
| 6T | " " 60 min. | " | " " |
| 8T | " " 120 min. | " | " " |
| 2U | Annealed 500°C for 15 min. | " | Beta-cooled structure. |
| 4U | " " 30 min. | " | " " |
| 6U | " " 60 min. | " | " " |
| 8U | " " 120 min. | " | " " |
| 2V | Annealed 550°C for 15 min. | " | Beta-cooled structure. |
| 4V | " " 30 min. | " | " " |
| 6V | " " 60 min. | " | " " |
| 8V | " " 120 min. | " | " " |
| 2W | Annealed 600°C for 15 min. | " | Beta-cooled structure, some evidence of recrystallization. |
| 4W | " " 30 min. | " | Beta-cooled structure. |
| 6W | " " 60 min. | " | Beta-cooled structure, some evidence of recrystallization. |
| 8W | " " 120 min. | " | " " |
| 2X | Annealed 640°C for 15 min. | " | Beta-treated structure, some evidence of recrystallization. |
| 4X | " " 30 min. | " | Beta-cooled structure. |
| 6X | " " 60 min. | " | Beta-cooled structure, some evidence of recrystallization. |
| 8X | " " 120 min. | " | " " |
| | Beta-treated 730°C - 15 min. | | |
| J | | 45 | Uniform beta-cooled structure. |



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-Continued-

| INGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | MACROSTRUCTURAL REMARKS |
|---|---|---|---|
| 2Y | Annealed 425°C for 15 min. | 45 | Uniform beta-cooled structure. |
| 4Y | " " 30 min. | " | Beta-cooled structure. |
| 6Y | " " 60 min. | " | " " |
| 8Y | " " 120 min. | " | " " |
| 2Z | Annealed 450°C for 15 min. | " | Beta-cooled structure. |
| 4Z | " " 30 min. | " | " " |
| 6Z | " " 60 min. | " | " " |
| 8Z | " " 120 min. | " | " " |
| 2AA | Annealed 500°C for 15 min. | " | Beta-cooled structure, coarser at core. |
| 4AA | " " 30 min. | " | Uniform beta-cooled structure. |
| 6AA | " " 60 min. | " | Beta-cooled structure, radial growth at core. |
| 8AA | " " 120 min. | " | " " |
| 2AB | Annealed 550°C for 15 min. | " | Beta-cooled structure. |
| 4AB | " " 30 min. | " | " " |
| 6AB | " " 60 min. | " | " " |
| 8AB | " " 120 min. | " | " " |
| 2AC | Annealed 600°C for 15 min. | " | Beta-cooled structure, coarser at core. |
| 4AC | " " 30 min. | " | |
| 6AC | " " 60 min. | " | " " |
| 8AC | " " 120 min. | " | " " |
| 2AD | Annealed 640°C for 15 min. | " | Beta-cooled structure, slight evidence of re-crystallization. |
| 4AD | " " 30 min. | " | Beta-cooled structure. |
| 6AD | " " 60 min. | " | " " |
| 8AD | " " 120 min. | " | " " |



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TABLE I

-Continued-

| INGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | MACROSTRUCTURAL REMARKS |
|---|---|---|---|
| | Beta-treated 730°C - 15 min. | | |
| L | | 60 | Uniform beta-cooled structure. |
| 2AE | Annealed 425°C for 15 min. | " | Beta-cooled structure. |
| 4AE | " " 30 min. | " | " " |
| 6AE | " " 60 min. | " | " " |
| 8AE | " " 120 min. | " | Beta-cooled structure, radial growth at core. |
| 2AF | Annealed 450°C for 15 min. | " | Beta-cooled structure. |
| 4AF | " " 30 min. | " | Beta-cooled structure, radial growth at core. |
| 6AF | " " 60 min. | " | Beta-cooled structure. |
| 8AF | " " 120 min. | " | " " |
| 2AG | Annealed 500°C for 15 min. | " | Beta-cooled structure. |
| 4AG | " " 30 min. | " | " " |
| 6AG | " " 60 min. | " | " " |
| 8AG | " " 120 min. | " | " " |
| 2AH | Annealed 550°C for 15 min. | " | Beta-cooled structure. |
| 4AH | " " 30 min. | " | " " |
| 6AH | " " 60 min. | " | Beta-cooled structure, radial growth at core. |
| 8AH | " " 120 min. | " | Beta-cooled structure. |
| 2AJ | Annealed 600°C for 15 min. | " | Uniform beta-cooled structure, slight evidence of recrystallization. |
| 4AJ | " " 30 min. | " | |
| 6AJ | " " 60 min. | " | |
| 8AJ | " " 120 min. | " | |
| 2AK | Annealed 640°C for 15 min. | " | Uniform beta-cooled structure with more evidence of recrystallization than preceeding series. |
| 4AK | " " 30 min. | " | |
| 6AK | " " 60 min. | " | |
| 8AK | " " 120 min. | " | |

TABLE I

-Continued-

| DINGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1-1 | As-extruded (1175°C) | | 73.0 | One-third of sample face exhibited coarse grained alpha hot-worked structure, remainder fine grained. |
| | Beta-treated 730°C - 15 min. | | | |
| B | | 5 | 72.0 | Uniform beta-cooled structure with columnar grains. |
| 1 | Annealed at 425°C for 15 min. | " | 72.0 | Beta-cooled structure, coarse core, columnar perimeter. |
| 3 | " " 30 min. | " | 72.0 | " " " |
| 5 | " " 60 min. | " | 70.0 | " " " |
| 7 | " " 120 min. | " | 67.0 | " " " |
| 1A | Annealed at 450°C for 15 min. | " | 69.0 | Beta-cooled structure, columnar perimeter. |
| 3A | " " 30 min. | " | 71.0 | " " " |
| 5A | " " 60 min. | " | 67.0 | " " " |
| 7A | " " 120 min. | " | 69.0 | " " " |
| 1B | Annealed at 500°C for 15 min. | " | 71.0 | Beta-cooled structure, columnar perimeter. |
| 3B | " " 30 min. | " | 69.0 | " " " |
| 5B | " " 60 min. | " | 70.0 | " " " |
| 7B | " " 120 min. | " | 68.0 | " " " |
| 1C | Annealed at 550°C for 15 min. | " | 65.0 | Partial recrystallization. |
| 3C | " " 30 min. | " | 68.0 | Beta-cooled structure - No recrystallization. |
| 5C | " " 60 min. | " | 63.0 | Partially recrystallized. |
| 7C | " " 120 min. | " | 67.0 | No recrystallization. |

## TABLE I

-Continued-

| DINGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1D | Annealed 600°C for 15 min. | 5 | 73.0 | Beta-cooled structure, columnar perimeter. |
| 3D | " " 30 min. | " | 67.0 | Fully recrystallized. |
| 5D | " " 60 min. | " | 62.0 | Perimeter fully recrystallized, core partially recrystallized. |
| 7D | " " 120 min. | " | 67.0 | Almost complete recrystallization with a fine grain perimeter. |
| 1E | Annealed 640°C for 15 min. | " | 65.0 | 80% recrystallization, beta-cooled core. |
| 3E | " " 30 min. | " | 63.0 | Complete recrystallization - Grain Size approx. .225 mm. |
| 5E | " " 60 min. | " | 67.0 | Complete recrystallization. |
| 7E | " " 120 min. | " | 66.0 | Complete recrystallization. |
| | Beta-treated 730°C - 15 min. | | | |
| D | | 10 | 73.0 | Beta-cooled structure. |
| 1F | Annealed 425°C for 15 min. | " | 71.0 | Beta-cooled structure, columnar perimeter, coarse core. |
| 3F | " " 30 min. | " | 70.0 | " " " |
| 5F | " " 60 min. | " | 71.0 | " " " |
| 7F | " " 120 min. | " | 68.0 | Uniform beta-cooled structure. |
| 1G | Annealed 450°C for 15 min. | " | 72.0 | Beta-cooled structure, columnar perimeter, coarser core. |
| 3G | " " 30 min. | " | 72.0 | " " " |
| 5G | " " 60 min. | " | 66.0 | " " " |
| 7G | " " 120 min. | " | 68.0 | Uniform beta-cooled structure. |

## TABLE I

-Continued-

| DINGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1H | Annealed 500°C for 15 min. | 10 | 72.0 | Beta-cooled structure, columnar perimeter, coarser core. |
| 3H | " " 30 min. | " | 71.0 | " " " |
| 5H | " " 60 min. | " | 71.0 | " " " |
| 7H | " " 120 min. | " | 73.0 | " " " |
| 1J | Annealed 550°C for 15 min. | " | 67.0 | Beta-cooled structure, columnar perimeter, coarser core. |
| 3J | " " 30 min. | " | 70.0 | " " " |
| 5J | " " 60 min. | " | 67.0 | " " " |
| 7J | " " 120 min. | " | 68.0 | Slight recrystallization along part of perimeter, remainder beta-cooled structure. |
| 1K | Annealed 600°C for 15 min. | " | 68.0 | Almost complete recrystallization, finer perimeter, coarser core. |
| 3K | " " 30 min. | " | 62.0 | " " " |
| 5K | " " 60 min. | " | 68.0 | Recrystallization more complete in core. |
| 7K | " " 120 min. | " | 65.0 | Almost complete recrystallization. |
| 1L | Annealed 640°C for 15 min. | " | 69.0 | Almost complete recrystallization. |
| 3L | " " 30 min. | " | 68.0 | Recrystallized structure appears coarse. |
| 5L | " " 60 min. | " | 71.0 | Complete recrystallization. |
| 7L | " " 120 min. | " | 68.0 | Complete recrystallization. |
| | Beta-treated 730°C for 15 min. | | | |
| F | | 20 | 71.0 | Uniform beta-cooled structure. |

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TABLE I

-Continued-

| DINGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1M | Annealed 425°C for 15 min. | 20 | 65.0 | Uniform beta-cooled structure. |
| 3M | " " 30 min. | " | 71.0 | Beta-cooled structure, coarser core. |
| 5M | " " 60 min. | " | 68.4 | " " " |
| 7M | " " 120 min. | " | 64.0 | Beta-cooled structure. |
| 1N | Annealed 450°C for 15 min. | " | 63.0 | Beta-cooled structure. |
| 3N | " " 30 min. | " | 64.0 | " " |
| 5N | " " 60 min. | " | 63.0 | " " |
| 7N | " " 120 min. | " | 65.0 | " " |
| 1O | Annealed 500°C for 15 min. | " | 66.0 | Beta-cooled structure, radial growth at core. |
| 3O | " " 30 min. | " | 63.0 | Beta-cooled structure. |
| 5O | " " 60 min. | " | 66.0 | Uniform beta-cooled structure. |
| 7O | " " 120 min. | " | 66.0 | Beta-cooled structure, radial growth at core. |
| 1P | Annealed 550°C for 15 min. | " | 65.0 | Beta-cooled structure, slight columnar growth at perimeter. |
| 3P | " " 30 min. | " | 63.0 | " " " |
| 5P | " " 60 min. | " | 67.0 | Uniform beta-cooled structure. |
| 7P | " " 120 min. | " | 66.0 | Uniform beta-cooled structure. |
| 1Q | Annealed 600°C for 15 min. | " | 67.0 | Uniform beta-cooled structure. |
| 3Q | " " 30 min. | " | 68.0 | Beta-cooled structure, finer perimeter, coarser core. |
| 5Q | " " 60 min. | " | 68.0 | Beta-cooled structure. |
| 7Q | " " 120 min. | " | 51.0 | Beta-cooled structure, finer perimeter, coarser core. |

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## TABLE I

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| INGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1R | Annealed 640°C for 15 min. | 20 | 67.0 | Beta-cooled structure. |
| 3R | " " 30 min. | " | 65.0 | Beta-cooled structure with slight evidence of recrystallization. |
| 5R | " " 60 min. | " | 67.0 | Beta-cooled structure with slight evidence of recrystallization. |
| 7R | | " | 62.0 | Recrystallization has taken place, some beta-cooled structure. |
| H | | 30 | 65.0 | Beta-cooled structure with coarse grain offset. |
| 1S | | " | 65.0 | Uniform beta-cooled structure. |
| 3S | | " | 65.0 | " " |
| 5S | | " | 63.0 | " " |
| 7S | | " | 62.0 | " " |
| 1T | Annealed 450°C for 15 min. | " | 67.0 | Uniform beta-cooled structure. |
| 3T | " " 30 min. | " | 69.0 | " " |
| 5T | " " 60 min. | " | 64.0 | " " |
| 7T | " " 120 min. | " | 63.0 | " " |
| 1U | Annealed 500°C for 15 min. | " | 65.0 | Beta-cooled structure. |
| 3U | " " 30 min. | " | 63.0 | " " |
| 5U | " " 60 min. | " | 69.0 | " " |
| 7U | " " 120 min. | " | 57.0 | " " |
| 1V | Annealed 550°C for 15 min. | " | 68.0 | Beta-cooled structure. |
| 3V | " " 30 min. | " | 70.0 | " " |
| 5V | " " 60 min. | " | 66.0 | " " |
| 7V | " " 120 min. | " | 69.0 | " " |

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## TABLE I

-Continued-

| DINGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1W | Annealed 600°C for 15 min. | 30 | 65.0 | Uniform beta-cooled structure. |
| 3W | " " 30 min. | " | 64.0 | Beta-cooled structure with evidence of preliminary recrystallization. |
| 5W | " " 60 min. | " | 70.0 | " " " |
| 7W | " " 120 min. | " | 66.0 | " " " |
| 1X | Annealed 640°C for 15 min. | " | 67.0 | Beta-cooled structure with slight evidence of preliminary recrystallization. |
| 3X | " " 30 min. | " | 65.0 | " " " |
| 5X | " " 60 min. | " | 69.0 | Beta-cooled structure. |
| 7X | " " 120 min. | " | 62.0 | Almost complete recrystallization but coarse in nature. |
| | Beta-treated 730°C - 15 min. | | | |
| K | | 45 | 65.0 | Beta-cooled structure. |
| 1Y | Annealed 425°C for 15 min. | " | 65.0 | " " |
| 3Y | " " 30 min. | " | 69.0 | " " |
| 5Y | " " 60 min. | " | 67.0 | " " |
| 7Y | " " 120 min. | " | 65.0 | " " |
| 1Z | Annealed 450°C for 15 min. | " | 64.0 | Beta-cooled structure. |
| 3Z | " " 30 min. | " | 65.0 | " " |
| 5Z | " " 60 min. | " | 60.0 | " " |
| 7Z | " " 120 min. | " | 65.0 | " " |
| 1AA | Annealed 500°C for 15 min. | " | 63.0 | Beta-cooled structure. |
| 3AA | " " 30 min. | " | 65.0 | " " |
| 5AA | " " 60 min. | " | 62.0 | " " |
| 7AA | " " 120 min. | " | 65.0 | " " |

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TABLE I

-Continued-

| DINGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1AB | Annealed 550°C for 15 min. | 45 | 67.0 | Uniform beta-cooled structure. |
| 3AB | " " 30 min. | " | 67.0 | " " |
| 5AB | " " 60 min. | " | 63.0 | " " |
| 7AB | " " 120 min. | " | 65.0 | " " |
| 1AC | Annealed 600°C for 15 min. | " | 68.0 | Beta-cooled structure, slightly coarser at core. |
| 3AC | " " 30 min. | " | 66.0 | " " " |
| 5AC | " " 60 min. | " | 67.0 | " " " |
| 7AC | " " 120 min. | " | 64.0 | Uniform beta-cooled structure. |
| 1AD | Annealed 640°C for 15 min. | " | 66.0 | Beta-cooled structure. |
| 3AD | " " 30 min. | " | 66.0 | Beta-cooled structure, evidence of preliminary recrystallization. |
| 5AD | " " 60 min. | " | 65.0 | " " " |
| 7AD | " " 120 min. | " | 63.0 | Recrystallization incomplete - Grain size approx. .620 mm. |
| | Beta-treated 730°C - 15 min. | | | |
| M | | 60 | 68.0 | Beta-cooled structure, radial growth at core. |
| 1AE | Annealed 425°C for 15 min. | " | 64.0 | Beta-cooled structure, columnar at core. |
| 3AE | " " 30 min. | " | 62.0 | Beta-cooled structure, slightly coarser at core. |
| 5AE | " " 60 min. | " | 62.0 | " " " |
| 7AE | " " 120 min. | " | 65.0 | Beta-cooled structure, columnar at core. |
| 1AF | Annealed 450°C for 15 min. | " | 63.0 | Uniform beta-cooled structure. |
| 3AF | " " 30 min. | " | 64.0 | Beta-cooled structure, columnar at core. |
| 5AF | " " 60 min. | " | 63.0 | Uniform beta-cooled structure. |
| 7AF | " " 120 min. | " | 60.0 | Uniform beta-cooled structure. |

TABLE I

-Continued-

| DINGOT NO. | HEAT TREATING CONDITIONS | TRANSFER TIME (BETA-HEAT TREATING BATH TO WATER QUENCH) (Seconds) | ROCKWELL G HARDNESS AVERAGE | MACROSTRUCTURAL REMARKS |
|---|---|---|---|---|
| 1AG | Annealed 500°C for 15 min. | 60 | 66.0 | Uniform beta-cooled structure. |
| 3AG | " " 30 min. | " | 62.0 | Beta-cooled structure. |
| 5AG | " " 60 min. | " | 61.0 | Beta-cooled structure, columnar core. |
| 7AG | " " 120 min. | " | 59.0 | Uniform beta-cooled structure. |
| 1AH | Annealed 550°C for 15 min. | " | 62.0 | Beta-cooled structure, coarser at core. |
| 3AH | " " 30 min. | " | 64.0 | Beta-cooled structure. |
| 5AH | " " 60 min. | " | 68.0 | " " |
| 7AH | " " 120 min. | " | 60.0 | " " |
| 1AJ | Annealed 600°C for 15 min. | " | 68.0 | Uniform beta-cooled structure. |
| 3AJ | " " 30 min. | " | 65.0 | Beta-cooled structure, slight evidence of preliminary recrystallization. |
| 5AJ | " " 60 min. | " | 61.0 | " " " |
| 7AJ | " " 120 min. | " | 63.0 | " " " |
| 1AK | Annealed 650°C for 15 min. | " | 66.0 | Beta-cooled structure, columnar core. |
| 3AK | " " 30 min. | " | 66.0 | Beta-cooled structure, slight evidence of preliminary recrystallization. |
| 5AK | " " 60 min. | " | 59.0 | Beta-cooled structure, evidence of preliminary recrystallization. |
| 7AK | " " 120 min. | " | 61.0 | Evidence of coarse recrystallized grains and retained beta-cooled structure. |