SOLIDIFICATION MECHANISM OF URANIUM INGOTS

Topical Report

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ABSTRACT

Solidification of production size uranium ingots (8-inch, 9-inch, and 11-inch diameters) was studied by thermal analyses of experimental data. The temperature distribution in both the ingot and the mold, the progression of the solidification front, solidus isochrones, solidification rates in both the transverse and vertical direction, and volumetric percent of metal solidified with respect to time are shown.

A method of correlating mold wall temperature data to ingot soundness by using a heat transfer mathematical model was developed and is discussed in the report.

1.0 INTRODUCTION

Theoretical¹ and experimental^{2,3,4} work have been reported on the solidification of 7-inch diameter cylindrical uranium ingots cast into cold molds. After the forementioned work was completed, a heated mold technique for 7-inch diameter ingot production was developed and used.^{5,6} At about the same time, techniques were developed for producing the 11-inch diameter cylindrical ingot required for the N-Production Reactor (NPR) at Hanford.⁷ Other fuel core demands required 8- and 9-inch diameter cylindrical castings.

The need for these various ingot sizes made it necessary to achieve a better understanding of their solidification. Thus, a program was initiated to study the solidification of 8-, 9-, and 11-inch diameter cylindrical ingots. Consideration was given to several approaches. The pour-out method and the mathematical analysis method were not considered because they would not give the information desired. Further, the pour-out method was impractical and the mathematical analysis method would have to be based on too many oversimplified assumptions. For example, the mathematical model method used by Fletcher and co-workers¹ had certain limitations in its application which included the adaptability to changes in ingot and mold size and in the effects of using a water-cooled induction coil to heat part of the mold.

The thermal analysis method would provide the specific thermal information desired of both the solidifying metal and mold before and after pour and for this reason it was used in the solidification study. Partial results have been published⁸ and are included in this study.

2.0 SUMMARY OF RESULTS

2.1 Thermal Course in Mold Wall

The study of the thermal course in the mold wall showed that the hotter the pour temperature for a given mold size, the hotter the mold at time of pour. The temperatures within the wall of the molds used with a mold heating coil were uniform. With no thermal barrier between the mold and the water-cooled mold tank, the mold wall had a thermal gradient of up to 45°F. After the mold had reached a maximum temperature after pour, the mold mid-wall and surface temperatures were uniform indicating that the mold heat must be flowing down the mold wall at a greater rate than radiated transversely.

2.2 Thermal Course in Ingot

The study of the thermal course in the ingot showed that the sensible heat of the liquid is lost to the mold in a very short time. After a half-inch thick "skin" of metal had formed, the gap effect and the changing thermal conductivities of both the uranium metal and the graphite mold indicated a complex mode of heat transfer from the liquid. The data implied that a major portion of the heat of fusion must flow down the ingot and out the bottom of the mold or down the mold wall.

2.3 Solidification Front

The progression of the solidification front was studied in depth at about mid-ingot height. The solidification front was very slow to pass from a one-inch thick "skin" of metal to the center-line. The rate of ingot solidification for ingots of a given size is almost equal regardless of the amount of superheat. Superheat merely delays the start of solidification.

Volumetric progression of the solidification front in the ingots decreased as the test ingots solidified. Half of the liquid metal solidified in about 30 percent of the time required for the ingot to completely solidify.

2.4 Solidus Isochrones

The solidus isochrones show an order of magnitude for the relative rates of both the transverse and vertical heat flow from the solidifying front. (Heat flow vectors are normal to the temperature isochrones.)

2.5 Transverse Solidification

The progress of transverse solidification was plotted to ascertain if a parabolic relationship existed. The growth from the mold wall inward to mid-radius was parabolic. At mid-radius, most of the slopes changed. The average of the constant k for the linear portion of the curve was 1.4.

The average transverse solidification rates were calculated and plotted. The effect of the mold coil insulators was apparent. These curves showed various combinations of increasing and decreasing functions of time as solidification proceeded. However, the average rates of solidification for the 11 - inch diameter ingot were increasing functions of time.

2.6 Vertical Solidification

Curves showing the progress of vertical solidification along the ingot centerline were also plotted. There was no parabolic relationship, but the effect of increasing superheat was apparent. As the superheat increased, the curves shifted but retained their relative shape. This again indicated that superheat delays the start of solidification and not the rate of solidification.

The average rates of vertical solidification were calculated and plotted. All the curves plotted were "S-shaped". The middle region of the curve is an increasing function of distance. This is attributed to the fact that in this region the solidification is not only by vertical grain growth but also by the completion of the transverse grain growth.

2.7 Heat Transfer Model

A practical heat transfer model was developed to predict the presence of internal shrinkage voids by using only the mold wall temperatures at various distances from the bottom.

3.0 MATERIALS AND METHODS

3.1 Furnace Components

The Stokes vacuum induction furnace at the National Lead Company of Ohio was the same one used by Foster et al of Battelle Memorial Institute for their large-scale experiments.² A schematic of the mold that Foster used and its position in the furnace with respect to the bottom of the crucible and the water-cooled jacket of the mold tank is shown in Figure 1. Note that, except for the top 11 inches of the mold, there was no thermal radiation barrier between the graphite mold and the water-cooled mold tank walls.

Schematics of the furnacing used in this study are shown in Figures 2, 3, and 4. Standard furnacing was used except for the specially designed thermocouple protection tube assembly and modification of the mold to accommodate these assemblies. The top of the mold — regardless of mold size used — was $\frac{1}{4}$ - to $\frac{1}{2}$ - inch from the bottom of the crucible and all molds were supported by a graphite block in a graphite catch basin. The same mold heating coil was used for both the 8 - inch and the 9 - inch ingot molds. The graphite crucibles used in the test were not coated. The molds were coated with Terrapaint 55M, a zircon mold wash produced by Faseco.

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FIGURE 1 Stokes Vacuum Induction Furnace with 7-Inch Ingot Mold in Position

FIGURE 2 Stokes Vacuum Induction Furnace with 8-Inch Ingot Mold in Position Inside Mold Induction Heating Coil

FIGURE 3 Stokes Vacuum Induction Furnace with 9-Inch Ingot Mold in Position Inside Mold Induction Heating Coil

FIGURE 4 Stokes Vacuum Induction Furnace with 11-Inch Ingot Mold in Position

3.2 Temperature Measurement Techniques

A schematic drawing of the final graphite protection tube is shown in Figure 5. Sealing the hot ends of the graphite thermocouple protection tubes was a problem. At first, a graphite disc $\frac{1}{8}$ -inch thick was cemented to the end of each protection tube with electrode joint graphite cement. When the first ingot was cast, several of the discs came off, and metal leaked into the tubes. Small plugs were then made and cemented into the end of the protection tubes. Very few protection tubes leaked uranium when this technique was used.

FIGURE 5 Schematic Drawing of Protection Tube Assembly

Twenty four protection tubes were mounted through the graphite mold wall. Thermocouples (Pt-6% Rh/Pt-30% Rh) were inserted in each and held in place with threaded graphite follower tubes. The protection tubes were spaced 60° apart throughout the mold length and arranged so that all tubes of a given length were above one another (see Figure 6).

For the 15 thermocouples within the mold wall and one in the stool, $\frac{1}{16}$ -inch holes were drilled to the appropriate depth, capped with $\frac{1}{8}$ -inch thick graphite discs where necessary, and fitted with sheathed chromel-alumel thermocouples. The thermocouples were held in place with electrode joint cement and wire wrapped around the outside of the mold. The completed assembly for a 9-inch ingot is shown in Figures 6 and 7. Schematic views showing the thermocouple location are shown in Figures 8, 9, and 10.

Ingot and mold temperatures were recorded by six multipoint recorders. Two recorders were used for the 16 Cr-Al thermocouples and were of a balance-print type (prints the temperature resulting from a balanced signal regardless of the time) with $\sim \frac{1}{4}$ and $\sim \frac{1}{3}$ minute minimum cycle times respectively to repeat a reading. The other four recorders were identical six-point, time-controlled, print-type recorders (prints on a time cycle regardless of the unbalanced signal) for use with the Pt-Rh thermocouples and required 7.2 seconds to repeat a reading.

FIGURE 6 Top View of Mold Assembly in the Mold Tank

FIGURE 7 Side View of Mold Assembly in the Mold Tank

FIGURE 8 Schematic View Showing
Thermocouple Locations for 8-
Inch Ingot

FIGURE 9 Schematic View Showing
Thermocouple Locations for 9-
Inch Ingot

FIGURE 10 Schematic View Showing Thermocouple Locations for 11-Inch Ingot

4.0 EXPERIMENTAL CASTING PROCEDURE

Eight-, 9-, and 11-inch-diameter cylindrical ingots were produced in this study. Heats of each size were poured at target temperatures of 2500, 2700, and 2900°F. Furnace pressures were maintained at less than 3×10^{-2} torr. The ingots were cast according to standard plant operating procedures except for a few deviations as follows:

- a. The furnace charge was composed entirely of Alloy 501* remelt stock.

* Alloy 501: 100 to 200 ppm Fe and 75 to 140 ppm Si.

- b. Normally the crucible pour hole is in the center of the crucible bottoms. However, to keep the pour stream from impinging on and breaking the internal thermocouple protection tubes, crucibles with off-center pour holes were used.
- c. There were two basic furnace power cycles (Table I) as follows:
- c-1. For the 11 -inch ingot cast from an 1885 lb charge (no external mold heating required):
 170 kw for ~79 minutes or until a liquid pool formed;
 140 kw to casting temperature required;
 Power off for 5 minutes;
 Pour.
- c-2. For the 9 -inch and 8 -inch ingots cast from a 1400 lb charge:
 165 kw to crucible coil and 15 kw to mold coil for 60 minutes;
 Power off mold coil;
 100 kw to crucible coil to casting temperature required;
 Power off for 7 minutes;
 Pour.
- d. All heats monitored with both an immersion thermocouple (5 inches into the melt) and an optical pyrometer. Control of the second part of the power cycle was always based on the immersion thermocouple.
- e. The mold was held under dynamic vacuum for at least 1 hour after pouring before backfilling the furnace. (Normally, the vacuum valves are turned off immediately after casting the ingot, and the furnace is backfilled with nitrogen 5 to 10 minutes later to a furnace pressure of ~100 torr.)

Pertinent data on each of the castings made are listed in Table II.

TABLE I Crucible and Mold Induction Power Cycle Used to Produce the Test Ingots

Ingot Diameter	Target Pour Temp.								
	2500°F			2700°F			2900°F		
	Furnace Power Cycle								
	Crucible Power Cycle kw	Time Power On min	Mold Power Cycle kw	Crucible Power Cycle kw	Time Power On min	Mold Power Cycle kw	Crucible Power Cycle kw	Time Power On min	Mold Power Cycle kw
8 -inch	165	60	15	165	60	15	165	60	15
	103	2	0	103	18	0	103	39	0
	0	7	0	0	7	0	0	7	0
	- Pour -			- Pour -			- Pour -		
9 -inch	145	60	15	145	60	15	145	60	15
	95	20	0	95	48	0	95	64	0
	0	7	0	0	7	0	0	7	0
	- Pour -			- Pour -			- Pour -		
11 -inch	170	78	—	170	78	—	170	74	—
	140	25	—	140	39	—	140	62	—
	0	5	—	0	5	—	0	5	—
	- Pour -			- Pour -			- Pour -		

TABLE II Casting Data

Ingot No.	Ingot Diameter (inches)	Target Control Temperature ($^{\circ}$ F)	Immersion Thermocouple Pour Temperature ($^{\circ}$ F)
20445N	11	2500	—
20444N	11	2700	2680
20446N	11	2900	2885
20454N	8	2500	2440
20455N	8	2700	2630
20456N	8	2900	2810
20460N	9	2500	2415
20461N	9	2700	2610
20463N	9	2900	2790

5.0 EXPERIMENTAL WORK AND RESULTS

The test program was designed so that thermal data could be obtained during the solidification of ingots of three different diameters cast at three different pour temperatures. Some data are missing since the Pt-6% Rh/Pt-30% Rh thermocouple protection tubes failed during the early runs and during later runs a recorder failed a time or two.

Even though the metal used in this study is considered an alloy, it is a dilute alloy with about 1000 ppm total impurities. Typical analyses of the ingots cast for this program were as follows

Carbon	— 500	ppm
Nitrogen	— 25	ppm
Hydrogen	— 10	ppm
Oxygen	— 22	ppm
Aluminum	— 25	ppm
Chromium	— 20	ppm
Copper	— 10	ppm
Iron	— 150	ppm
Nickel	— 40	ppm
Silicon	— 130	ppm

The uranium-carbon phase diagram, according to Blumenthal,⁹ shows that the eutectic is at 2040 $^{\circ}$ F at 500 ppm carbon. Almost all cooling curves obtained in this test showed that the solidus was 2040 $\pm$ 5 $^{\circ}$ F which is similar to the solidus temperatures Foster et al² obtained. Discussion of the experimental work and results follows.

5.1 Temperature Distribution in Ingot and Mold

The thermal gradients in the three ingot sizes cast in this study at three target temperatures are shown in Figures 11 through 23. The figures show the thermal gradients from the ingot centerline

through the ingot mold interface to the mold surface at various times after initiation of pour at various distances from the base of the ingot.

FIGURE 11 Ingot and Mold Thermal Gradients 10 Inches from Base of 8-Inch Ingot Cast at 2500°F

FIGURE 12 Ingot and Mold Thermal Gradients 10 Inches from Base of 8-Inch Ingot Cast at 2700°F

FIGURE 13 Ingot and Mold Thermal Gradients 24 Inches from Base of 8-Inch Ingot Cast at 2700°F

FIGURE 14 Ingot and Mold Thermal Gradients 38 Inches from Base of 8-Inch Ingot Cast at 2700°F

FIGURE 15 Ingot and Mold Thermal Gradients 10 Inches from Base of 8-Inch Ingot Cast at 2900°F

FIGURE 16 Ingot and Mold Thermal Gradients 24 Inches from Base of 8-Inch Ingot Cast at 2900°F

FIGURE 17 Ingot and Mold Thermal Gradients 38 Inches from Base of 8-Inch Ingot Cast at 2900°F

FIGURE 18 Ingot and Mold Thermal Gradients 24 Inches from Base of 9-Inch Ingot Cast at 2500°F

FIGURE 19 Ingot and Mold Thermal Gradients 6 Inches from Base of 9-Inch Ingot Cast at 2700°F

FIGURE 20 Ingot and Mold Thermal Gradients 24 Inches from Base of 9-Inch Ingot Cast at 2700°F

FIGURE 21 Ingot and Mold Thermal Gradients 22 Inches from Base of 11-Inch Ingot Cast at 2700°F

FIGURE 22 Ingot and Mold Thermal Gradients 10 Inches from Base of 11-Inch Ingot Cast at 2900°F

FIGURE 23 Ingot and Mold Thermal Gradients 22 Inches from Base of 11-Inch Ingot Cast at 2900°F

5.1.1 Thermal Course in the Mold Wall — To better understand the heat flow in the mold wall after pour, curves showing the temperature of the mold walls at time of pour (from the stools to the mold tops) were plotted (see Figures 24, 25, and 26).

Figure 24 has an anomaly. Normally, the hotter the pour temperature, the hotter the mold at time of pour. The lower third of the mold for the 8-inch ingot cast at 2500°F was hotter than the molds were when casting at 2700°F or at 2900°F. The power to the crucible induction coil that melted and superheated the melt was too high during the meltdown phase of the cycle. Consequently, the melt was at the 2500°F temperature soon after power cutback (see Table I); there was only a two minute time period at the lower power input during the liquation part of the cycle instead of a minimum 20 minutes that was desired. During this 20 minutes, the lower part of the mold loses heat by radiation and conduction to the mold tank. However, in two minutes only a small amount of heat left the lower part of the mold, resulting in the anomaly.

Except for the anomaly discussed, the hotter the pour temperature for a given mold size, the hotter the mold at time of pour. There are two mold heating sources. One is from the mold induction coil and the other is from the heat that radiates from the bottom of the crucible. Note that the center of the stool of all the test castings was at least 460°F — some were over 700°F.

FIGURE 24 Eight-Inch Ingot Mold Temperature at Start of Pour at 2500°F, 2700°F, and 2900°F for Various Distances from Mold Stool

At time of pour, the temperature within the wall of the 8- and 9-inch ingot molds was nearly uniform from ID to OD. The reason for uniformity was because these molds were inside the mold coil insulator (Figures 2 and 3) which decreased the radial heat loss from the mold. However, a mold coil insulator (Figure 4) was not used when 11-inch ingots were produced; consequently, the heat radiated to the water-cooled mold tank-wall. There was a thermal gradient of up to 45°F across the wall of the 11-inch ingot molds (Figure 23) at time of pour.

FIGURE 25 Nine-Inch Ingot Mold Temperature at Start of Pour at 2500°F, 2700°F, and 2900°F for Various Distances from Mold Stool

After pour, the interior surface of the mold increased in temperature at a faster rate than either the mid-wall or surface thermocouple of the 8- and 9-inch molds. In almost all cases the mid-wall temperature and the mold surface temperature were the same, especially during the period after the mold wall had reached a temperature maximum. Because of the mold coil insulation barrier to radiation heat loss, the mold heat must have flowed down the mold wall at a greater rate than radiated transversely.

A similar increase in the temperature of the inside surface of the 11-inch ingot mold over the temperature increase at mid-wall is shown in Figures 21, 22, and 23. But a thermal gradient existed across the mold wall at all times. This thermal gradient was the result of radial heat loss from the uninsulated mold to the water-cooled mold tank.

Once the molten metal was in the molds, the superheat from the metal appeared to have little or no effect on the manner in which the mold temperature profile progressed except that the higher the superheat, the hotter the mold walls and the slower they cooled. The molds reached a maximum temperature in about 4 minutes after start of pour.

FIGURE 26 Eleven-Inch Ingot Mold Temperature at Start of Pour at 2500°F, 2700°F, and 2900°F for Various Distances from Mold Stool

The experimental results of Foster et al² showed that the gap formed when the solidifying shell was from 0.4- to 0.8-inch thick. The effect of gap formation can be seen in Figures 11 through 23 but the effect was not nearly as severe as has been reported when casting steel into ferrous molds.¹⁰ After the molds reached a maximum temperature, the interior mold walls were about 100°F cooler than the ingot surface near the bottom of the ingot and only about 50°F cooler near the top of the ingot.

5.1.2 Thermal Course in the Metal – The thermal course within the metal depended on the manner in which the heat content of the metal was metered to the mold. The heat can be considered to come from three sources within the solidifying ingot: a) the sensible heat of the liquid metal, b) the heat of fusion, and c) the sensible heat of the solidified metal. The values of specific heat and thermal conductivity for graphite and uranium² are as follows:

<u>Uranium</u>	
a. Specific heat (liquid)	– 0.043 cal/(g)(C)
b. Heat of fusion	– 10.5 cal/g

- c. Specific heat (solid, gamma) — 0.037 cal/(g)(C)
- d. Thermal Conductivity (liquid) — 0.884 cal/(sec)(cm)(C)
- e. Thermal Conductivity (solid, gamma) — $[0.0527 + 5.38 \times 10^{-5} T]$ cal/(sec)(cm)(C)

Graphite

- a. Specific Heat — 0.40 cal/(g)(C)
- b. Thermal Conductivity — $[0.259 - 1.429 \times 10^{-4} T]$ cal/(sec)(cm)(C)

As the metal poured from the crucible into the mold, the sensible heat of the liquid was rapidly lost because the thermal conductivity of liquid uranium is almost 8 times greater than for uranium right after freezing. The maximum temperature recorded (shown in Figure 22) of metal in the mold was 2460°F when poured out of the crucible at 2900°F into an 11 - inch ingot mold.

The liquid metal lost heat rapidly to the graphite mold, the rate being dependent on the mold temperature. Too, there was a rapid accumulation of heat in the mold near the interface that appeared as a sharp temperature rise because the thermal conductivity of the mold is 7 to 8 times smaller than for liquid uranium. The top section of the mold inside the crucible support refractory was usually at a temperature of 1800 to 2000°F, depending on the power cycle and pour temperature. However, the lower half of all the molds used in this study was below a temperature of 1000°F.

After the molten uranium froze to a half-inch depth,² a gap formed and the mode of heat transfer of the solidifying metal inside the solidified shell changed and was even more complex. According to Figure 27, solid uranium and graphite have the same thermal conductivity at 1920°F.

FIGURE 27 Thermal Conductivity of Graphite and Uranium

As the temperature decreases, the thermal conductivity of uranium decreases and that of graphite increases. The flatness of the thermal gradients in the mold wall in Figures 11 through 23 indicates that a major portion of the heat of fusion of the solidifying uranium must flow down the ingot and out the bottom stool or down the mold wall.

5.2 Progression of Solidification Front

Cooling curves of the uranium at about mid-ingot height of each ingot size at the various pour temperatures were plotted (Figures 28, 29, and 30). The cooling curves show that the metal cooled quickly to the freezing point. At about 1-inch from the metal-mold interface the heat of fusion was sufficient for a slight arrest, indicating that the skin formed fairly rapidly. The temperature of the thermocouples further in the interior of the casting remained much longer at the freezing point. The passage of the solidification front followed as each thermocouple in turn dropped below 2040°F.

The start and end freeze curves for the same castings at the same ingot levels according to pour temperature are shown in Figures 31, 32, and 33. The solidification front was very slow to pass

FIGURE 28 Cooling Curves 24 Inches from Base of 8-Inch Ingots Cast at 2700°F and 2900°F

FIGURE 29 Cooling Curves 18 Inches from Base of 9-Inch Ingots Cast at 2500°F and 2700°F

FIGURE 30 Cooling Curves 16 Inches from Base of 11-Inch Ingots Cast at 2700°F and 2900°F

FIGURE 31 Effect of Superheat on Transverse Solidification - Start and End of Freeze 24 Inches from Base of 8-Inch Ingot Cast at 2700°F and 2900°F

FIGURE 32 Effect of Superheat on Transverse Solidification - Start and End of Freeze 18 Inches from Base of 9-Inch Ingot Cast at 2500°F and 2700°F

FIGURE 33 Effect of Superheat on Transverse Solidification - Start and End of Freeze 16 Inches from Base of 11-Inch Ingots Cast at 2700°F and 2900°F

from the one-inch thick skin to the centerline. The reason is complex but, oversimplified, it is a combination of effects due to (1) gap formation, (2) thermal conductivity of uranium decreasing as the temperature decreases, and (3) thermal conductivity of graphite being lower than uranium above 1920°F (Figure 27).

The effects of superheat on the progression of the solidification front can be seen in the cooling curves and start-and-end freeze curves. The superheat was lost rapidly during the initial for-

mation of the skin. Since the mold had a limited capacity for accepting heat, the greater the superheat, the hotter the graphite mold, and the lower the thermal conductivity of the mold, resulting in a decrease in the rate of flow of the heat of fusion through the solidified metal. This heat flow reduction rate can be seen in Figures 31, 32, and 33 as a delay in the start of solidification as the superheat increased. As soon as the superheat was lost, solidification began but the rate of solidification for ingots of a given size became almost equal regardless of the pouring temperature.

Although variations in mold thickness were not studied in this test, the effect of decreasing the mold wall thickness, thereby decreasing the mold ratio, would be expected to increase the time necessary for complete solidification to occur. Decreasing the mold ratio or increasing the superheat should have practically the same effect on the time for solidification to occur; however, the mechanism by which each effect solidification is much different. The solidification retarding effect of decreasing the mold ratio would be attributed to the lower heat capacity of the graphite molds and the inability of the molds to conduct heat away rapidly. But increasing the superheat would only retard the solidification process during the initial stages of solidification.

5.3 Isochrones

To show the progression of the solidifying front in each ingot produced in the test, solidus isochrones were plotted as shown in Figures 34, 35, and 36. As the superheat increased for a con-

FIGURE 34 Temperature Profiles of 8-Inch Ingots - Solidus Isochrones at Time after Pour

FIGURE 35 Temperature Profiles of 9-Inch Ingots – Solidus Isochrones at Time after Pour

FIGURE 36 Temperature Profiles of 11-Inch Ingots – Solidus Isochrones at Time after Pour

stant ingot size, or as the mold ratio decreased in conjunction with a decrease in the height to width ratio, the slope of the isochrones (above the bottom end effect) decreased. Because the heat flow vectors are normal to the temperature isochrones,¹¹ an order of magnitude of the relative rates of both the transverse heat flow and vertical heat flow from the solidifying front can be seen. This does not necessarily mean that the heat lost in the transverse direction from the solidifying front was equal to the heat radiating from the mold surface transversely.

However, when the 11-inch ingots were cast, there were no radiation barriers and heat was lost transversely. The solidus isochrones (Figure 36) of the 11-inch ingots show that molten metal was able to feed the solidifying front because of the low mold ratio of 5:1 and height-to-width ratio of 3:1.

The solidus isochrones for all the 8- and 9-inch ingots also indicate that molten metal was able to feed the solidifying fronts in those ingots. As previously discussed, the transverse radiation barrier of the mold coil insulator probably decreased the heat lost transversely just enough so that even with relatively poor height-to-width ratios, the transverse solidification rate was favorable for the production of ingots without internal voids. (One internal void was observed in an 8-inch ingot cross-section.⁸)

5.4 Uranium Solidification Rates

Chipman and Fondersmith¹² showed that the progress of the solidification front in a steel ingot followed a parabola of a general form

$$D = k\sqrt{t} - C \quad (1)$$

where

D = depth of solidified metal

t = time

k = constant that varies with the material solidifying and the type and size of mold

C = time delay constant directly related to the amount of superheat

The progress of transverse solidification of the ingots studied was plotted in Figures 37 through 42 to show the extent to which the parabolic relationship is valid. The growth from the mold wall inward to mid-radius was plotted as being parabolic (although only two data points were available). In most cases the slope of the line changed at the mid-radius. The constant k for the linear portion of the curves is given. The k values ranged from 0.87 to 2.6 with an average of 1.4. The superheat time delay value, C, can also be obtained from the curves. In all cases the higher the superheat, the greater the X intercept value.

Curves showing the progress of vertical solidification along the centerline of all ingots cast (providing data were available) were plotted to ascertain if a parabolic relationship was valid for the solidification of the ingots in a vertical direction. The curves in Figures 43, 44, and 45 show that there was no parabolic relationship.

FIGURE 37 Progress of Transverse Solidification for 8-Inch Ingots Cast at 2700°F

FIGURE 38 Progress of Transverse Solidification for 8-Inch Ingots Cast at 2900°F

FIGURE 39 Progress of Transverse Solidification for 9-Inch Ingots Cast at 2500°F

FIGURE 40 Progress of Transverse Solidification for 9-Inch Ingots Cast at 2700°F

Any time a portion of such curves have an infinite or negative slope, voids are probably present because the ingot solidified to the center or "bridged over" at a higher level. Bridging occurred because the transverse solidification growth from the mold wall — especially in ingots with an unfavorable height-to-width ratio — was parallel to the mold wall. Consequently, no tapered feed channel existed to feed the solidifying center portion of the ingot.

The effect of increasing superheat was apparent. As the superheat increased, the curves were shifted to the right (Figures 43 and 44). However, the curves retained their relative shape. Again, they show that superheat increased the time for the ingot to solidify by delaying the start of freezing and not by decreasing the rate of freezing.

FIGURE 41 Progress of Transverse Solidification for 11-Inch Ingots Cast at 2700 °F

FIGURE 42 Progress of Transverse Solidification for 11-Inch Ingots Cast at 2900 °F

FIGURE 43 Progress of Vertical Solidification for 8-Inch Ingots Cast at, A-2500 °F; B-2700 °F; and C-2900 °F

FIGURE 44 Progress of Vertical Solidification for 9-Inch Ingots Cast at, A-2500 °F; and B-2700 °F

FIGURE 45 Progress of Vertical Solidification for 11-Inch Ingots Cast at 2900°F

5.4.1 Average Transverse Solidification Rates – The parabolic relationship implies that the rate of transverse solidification decreases as time increases. Differentiation of equation 1 gives

$$\frac{dD}{dt} = \frac{k}{2\sqrt{t}} \quad (2)$$

Equation 2 gives the rate of solidification at a given point any time after solidification begins and shows that the rate is a decreasing function of time. When the transverse solidification front is parallel to the mold wall, the average rate-of-solidification curves should show a decreasing function of time. Conversely, as the solidification front changes from being parallel to the mold wall as the heat flow direction changes downward, the average rate-of-solidification curves should show an increasing function of time.

The average transverse solidification rates of the 8-, 9-, and 11-inch ingots at various levels were calculated and plotted in Figures 46 through 52. The effect of the mold coil insulators on the rate of solidification is apparent. The curves of the average solidification rates at various levels for both the 8- and 9-inch ingots show much variability as solidification proceeded. Figures 51 and 52 show that the average rates of transverse solidification for the 11-inch ingots were increasing functions of time for all points.

The average rates of transverse solidifications are quite erratic for 8-inch ingots cast at 2500°F and 2700°F and for the 9-inch ingot cast at 2700°F as shown in Figures 46, 47, and 50. However, in Figure 48 for the 8-inch ingot cast at 2900°F, almost all the curves show an increasing

FIGURE 46 Average Rate of Transverse Solidification at Various Levels from Base of 8-Inch Ingot Cast at 2500°F

FIGURE 47 Average Rate of Transverse Solidification at Various Levels from Base of 8-Inch Ingot Cast at 2700°F

FIGURE 48 Average Rate of Transverse Solidification at Various Levels from Base of 8-Inch Ingot Cast at 2900°F

FIGURE 49 Average Rate of Transverse Solidification at Various Levels from Base of 9-Inch Ingot Cast at 2500°F

FIGURE 50 Average Rate of Transverse Solidification at Various Levels from Base of 9-Inch Ingot Cast at 2700°F

FIGURE 51 Average Rate of Transverse Solidification at Various Levels from Base of 11-Inch Ingot Cast at 2700°F

FIGURE 52 Average Rate of Transverse Solidification at Various Levels from Base of 11-Inch Ingot Cast at 2900°F

function with time. In Figure 49, for the 9-inch ingot cast at 2500°F, all the curves but one show a decreasing function for half the radius, then an increasing function to the center of the ingot. In both Figures 51 and 52 for the 11-inch ingots cast at 2700°F and 2900°F, all curves show an increasing function with time.

Increasing function with time was not predicted by the parabolic law. Equation 2 is for growth normal to the mold wall and does not account for the effect of heat flow downward, which actually occurred in the solidifying ingots. Therefore, a deviation from the expected decreasing function of time occurred when the rate of heat flow downward was considerable.

The degree to which the deviation from a decreasing function of time occurred is apparent in Figure 52. The solidification front tends to move in a more transverse manner as the front progresses up the ingot because the slope of the curve decreases as the ingot level increases. The rate of transverse solidification tends to approach the decreasing function of time characteristic.

Figures 46 through 52 also reveal the effect of superheat on the rate of transverse solidification. As the superheat was increased, the average rate-of-solidification curves shifted downward.

However, this was expected because increasing superheat causes a delay in the start of solidification as discussed in Section 5.2.

Since the ingots solidify from the bottom to the top, the rate of transverse solidification should be greater at the bottom levels within the ingot than the top levels if a void-free casting is to be produced. The path of the rate of transverse solidification from the mold wall to the ingot centerline is relatively unimportant compared with the rate at the end of freezing along the ingot centerline. A gradation of final freezing rates should exist along the ingot centerline with the rates at the bottom being greater than those at the top.

This gradation existed as seen in Figures 46 through 52 except in Figures 47 and 49. In Figure 47, the 17-inch and 24-inch level completed solidification at the same time. A void was observed 17 inches from the bottom in this ingot. In Figure 49, the curve for the 12-inch level indicated that the center was solid before the center at the 9-inch level. No voids were visible in the ingot at this level. However, small voids certainly could have been present but not exposed during the sawing.

5.4.2 Average Vertical Solidification Rates — The average rates of vertical solidification were calculated and plotted for the test ingots (Figures 53, 54, and 55). These average rates were computed in the same manner as the average rates for transverse solidification as described in Section 5.4.1.

The effect of increasing the superheat resulted in a decrease in the average rates of vertical solidification (Figures 53 and 54). The slopes of the curves in a given figure are similar. The

FIGURE 53 Average Rate of Vertical Solidification Along Centerline of 8-Inch Ingots Cast at 2500°F, 2700°F, and 2900°F

FIGURE 54 Average Rate of Vertical Solidification Along Centerline of 9-Inch Ingots Cast at 2500°F and 2700°F

FIGURE 55 Average Rate of Vertical Solidification Along Centerline of 11-Inch Ingot Cast at 2900°F

shifting of curves again indicates that superheat delays the start of solidification with a decrease in solidification rates. The fact that the rates also decrease is attributed to the effect that the solidified metal has on the calculations of these rates. The average rates are calculated from the time the pour started and are not instantaneous rates. A downward shift in the relative position of the curves would be expected.

In general, all the curves are "S-shaped" with relative maximum and minimum points. The middle region of the curves is an increasing function of distance (time). This function is attributed to the fact that the solidification in this region was not by grain growth in the vertical direction but was by the completion of transverse grain growth. The rate of vertical solidification was, therefore, dependent upon the rate of transverse solidification. Inspection of the isochrones (Figures 34, 35, and 36) support this in that the solidification front in the central portion of the ingot length was near a vertical line moving in a transverse direction toward the ingot centerline. This is very evident in the case of the 8- and 9-inch diameter ingots (Figures 34 and 53, Fig-

ures 35 and 54) because of the unfavorable height-to-width ratios of 5.5 to 1 and 3.8 to 1, respectively.

The region of decreasing rates near the bottom of the 8- and 9-inch ingots is attributed to grain growth from the ingot bottom. This region of decreasing slope reaches a relative minimum at the point where vertical grain growth was restrained by transverse grain growth. Apparently this did not occur in the 11-inch ingot cast at 2900°F. The isochrones in Figure 36 indicate that the solidifying front in the center portion of the 11-inch ingot was much more tapered than the solidifying fronts that moved to the center in the 8- and 9-inch ingots.

The decreasing slope region near the top of the ingot is attributed to the grains beginning to grow in a more vertical direction. The movement of the solidification front as shown in the isochrones (Figures 34, 35, and 36) near the completion of freezing is almost horizontal and progresses vertically.

5.5 Volumetric Per Cent of Metal Solidified

In order to clarify a possible misconception of solidification when linear rates only (Figures 53, 54, and 55) are considered, the volumetric progression of the solidification of the ingots was calculated and plotted (Figures 56, 57, and 58). The volumetric solidification rates decreased during the time the metal froze; however, linear solidification rates became rapid as the freezing cycle progressed (Figures 53, 54, and 55). The increase in the average rates of vertical solidification resulted from heat losses to the mold wall manifested as a decreasing perimeter of the solidifying front which decreased to a point when the center was reached. Half the ingot had solidified in approximately 30% of the time necessary for complete solidification to occur. Thus, as the perimeters decrease, the volume likewise decreases.

FIGURE 56 Effect of Superheat on the Volumetric Progression of Solidification for 8-Inch Ingots

FIGURE 57 Effect of Superheat on the Volumetric Progression of Solidification for 9-Inch Ingots

FIGURE 58 Effect of Superheat on the Volumetric Progression of Solidification for 11-Inch Ingots

5.6 Correlation of Mold Wall Temperature Data to Ingot Soundness

To obtain a desired solidification pattern, the rate at which the metal loses its heat to the mold must be greater at the bottom of the ingot than at the top. The degree to which this desired heat transfer pattern occurs depends largely on the vertical temperature gradient along the length of the mold before pour. A mold which is hot at the top and cold at the bottom would facilitate rapid freezing at the bottom since the transverse temperature gradient is larger at the bottom than at the top.

The desired manner in which the metal should lose its heat to the mold is shown in Figure 59. If the ingot is to exhibit directional solidification, the solidification front must move upward and toward the centerline of the ingot as shown. The mold will absorb the heat from the molten metal first by conduction and then by radiation because of the air-gap. Heat will also flow through the solidified metal to the mold bottom by conduction. However, this amount decreases as the solidification front moves farther away from the mold bottom because the thermal conductivity of solid uranium decreases as the temperature decreases.

The mold can dissipate heat in one of three ways: 1) by radiation to the furnace wall, 2) by conduction up the mold wall, and 3) by conduction down the mold wall to the mold bottom. The latter mechanism is preferred because radiation to the furnace wall could produce transverse solidification which would cause the metal to bridge-over and create shrinkage voids. Conduction of heat up the mold wall could occur if the crucible bottom cooled faster than the top of the mold and would also result in bridge-over and shrinkage voids.

FIGURE 59 Features of Transverse and Vertical Solidification Required to Achieve "Directional" Solidification

The above discussion leads to the development of a practical heat transfer model by which mold wall temperature data can be correlated to ingot soundness. Assuming that there is no radiant nor conductive heat losses from the mold once the metal has been poured, Fourier's Law of heat transfer by conduction

$$Q = kA \frac{dT}{dx} \quad (3)$$

where

$$\begin{aligned} Q &= \text{rate of heat transfer} \\ k &= \text{thermal conductivity} \\ A &= \text{area perpendicular to heat flow} \\ \frac{dT}{dx} &= \text{temperature gradient} \end{aligned}$$

can be applied to the mold in Figure 60*. The metal at the bottom of the mold freezes first, and the solidification gradient $\left(\frac{\Delta T}{\Delta x}\right)$ is the driving force for heat transfer, it is not a complete measure of the amount of heat transferred in this case since the thermal conductivity (k) of graphite is a function of the actual temperature. Therefore, it was necessary to calculate the heat transferred by conduction between points T_1 and T_2 , T_2 and T_3 , and T_3 and T_4 .

Accordingly, the rate of heat transfer Q_{2-1} should be greater than Q_{3-2} , which in turn should be greater than Q_{4-3} . It follows that all values of the rate of heat transfer should be positive.

FIGURE 60 Schematic Diagram Showing Heat Lost from Metal to Mold and Showing Heat Dissipated Down Mold Wall

* Where temperature $T_1 < T_2 < T_3 < T_4$.

Furthermore, the slope of the curve for the rate of vertical heat transfer in the mold wall versus distance from the top of the mold will be positive at all points on the curve (Figure 61). The equation used to calculate the rate of heat transfer is

$$Q_{t-b} = \frac{(k_g)(A)(\Delta T)}{X}, \text{ BTU/sec} \quad (4)$$

where

- Q_{t-b} = rate of heat transfer (top to bottom)
- k_g = thermal conductivity of graphite
- A = cross-sectional area of mold
- ΔT = temperature difference between the two mold levels in consideration
- X = distance between the mold levels being considered.

Q was calculated at the time when the peak temperature after pour occurred at the topmost thermocouple location because at this time the combined effects of air-gap formation, lost superheat and mold chilling are maximized.

The thermal conductivity of graphite is given by an equation which for the convenience of the units was converted to

$$k_g = 0.00146 - 4.445 \times 10^{-7} T \text{ (BTU/(sec)(in)(°F))} \quad (5)$$

FIGURE 61 Relationship of Heat Transfer Rate to Distance Along Mold Wall if Conformity to Heat Transfer Model is Achieved

where k_g = thermal conductivity of graphite at temperature, T . The temperature (T) was taken as the average temperature between the two mold levels of interest. Substituting equation 5 into equation 4 yields

$$Q = \frac{\left[0.00146 - 4.445 \times 10^{-7} \left(\frac{T_2 + T_1}{2} \right) \right] (A) (T_2 - T_1)}{X} \text{ BTU/sec} \quad (6)$$

As a measure of heat flow down the mold wall, the expression for Q was divided by X to obtain the intensity of vertical heat transfer per unit length, or

$$\frac{Q_{t-b}}{X} = \frac{\left[0.00146 - 2.222 \times 10^{-7} (T_1 + T_2) \right] (A) (T_2 - T_1)}{X^2} \quad (7)$$

FIGURE 62 Relationship of Heat Transfer Intensities to Furnace Components for 8-Inch Ingots Cast at 2500°F, 2700°F, and 2900°F

where T_2 and T_1 are the average temperatures of the ID and OD thermocouples at the upper and lower levels being considered. No plots of the type described in equation 7 are shown in Figures 62, 63, and 64 for the three ingot diameter studied at various pour temperatures.

The amount of superheat did not affect the shape of the curves for the 8- and 9-inch diameter ingots (Figures 62 and 63). In each case, the proportional relationships expected did not appear. In fact, negative values were found in both the 8- and 9-inch diameter castings. These values signified that the transfer of heat was up the mold wall instead of down, as was also expected. These negative values are attributed to the fact that heat is lost into the space between the furnace support table and the top of the mold heating coil. This heat loss causes a depression in the curves and prevents conformity to a directly proportional relationship as expected.

The shape of the curves in Figure 64 (11-inch ingot) vary as the superheat increases. However, the general shape of the curves is the same as those for the 8- and 9-inch diameter ingots (Figures 62 and 63). The decreasing portions of these curves are attributed to the large radiant heat

FIGURE 63 Relationship of Heat Transfer Intensities to Furnace Components for 9-Inch Ingots Cast at 2500°F, 2700°F, and 2900°F

FIGURE 64 Relationship of Heat Transfer Intensities to Furnace Components for 11-Inch Ingots Cast at 2500°F, 2700°F, and 2900°F

FIGURE 65 Conformity of Insulated and Noninsulated Molds Based on Proposed Heat Transfer Model

losses since there is no mold heating coil to retain the heat. Negative values were shown for the amount of heat transferred when pouring at 2500°F, again signifying heat transfer up the mold wall. Apparently, there were excessive amounts of heat lost upward by conduction and, thus, the negative values.

Since radiant heat losses appear to have the greatest detrimental effect on the model proposed, prevention of this type of heat loss should make the plots conform to give an increasing slope and the relationship desired. Data on 11-inch ingots cast for another program were analyzed for their conformity or nonconformity to the proposed model. The results are shown in Figure 65.

Ingots in this test were cast using insulated and uninsulated molds. The curves for the uninsulated molds show a decreasing slope while the insulated molds show the desired increasing slope. The effect of the insulation is attributed to keeping the radiant heat losses low and forcing the heat to be transferred downward. Two different crucible heating cycles were used and both appear not to disturb the conformity or nonconformity to the heat transfer model.

6.0 CONCLUSIONS

1. The developed heat transfer model can be used to predict the presence of internal shrinkage voids using only a series of mold wall thermocouples.
2. The temperature varies only slightly, if at all, across a graphite mold wall 1- to 1½-inch thick, up to time of pour if the mold walls are insulated. The transverse mold wall temperature gradient may increase up to 50°F when casting into 11-inch ingot molds that are not insulated.
3. The procedure of increasing superheat, with all other conditions being equal, would increase the liquation time in the mold and delay the start of freezing. After the initial stages, solidification proceeds at almost a constant rate regardless of the superheat.
4. The solidification process can be delayed by increasing superheat, increasing mold temperature at pour, or decreasing heat loss transversely from the mold wall.
5. The cooling curves of interior mold surfaces can be used to predict the cooling of ingot surfaces to within 50 to 100°F.
6. The molds with a larger mold-wall ratio act as more of a chill than the molds with a smaller mold-wall ratio.
7. The thermal course within the mold wall is affected by the increased amount of heat which is radiated from the crucible bottom when the pour temperature (superheat) is increased.
8. The parabolic relationship appears to be valid until the solidification front has progressed about one-half the distance to the ingot centerline.

9. The rate of transverse solidification at the ingot centerline can be correlated to the occurrence of shrinkage voids. Excessive radiant heat losses from the mold wall are the suspected cause of promoting the occurrence of voids in ingots with large height-to-width ratios.

10. The uranium ingots cool slowly because of the specific values of the heat capacities and thermal conductivities of graphite and uranium.

11. The average rates of vertical solidification along the 8- and 9-inch ingot centerlines show that solidification in the central portion of these ingots occurs by the completion of transverse solidification rather than grain growth from the ingot bottom. The volumetric percent of metal solidified shows that the metal solidifies at a decreasing rate, a fact which is not apparent from the average rates of vertical solidification curves.

7.0 RECOMMENDATIONS

1. Using the rate of superheat transfer as found by the equation

$$Q_s = \frac{(W)(C_p)(T_p - T_{mp})}{\text{Time}} \text{ BTU/sec} \quad (8)$$

where

Q_s = superheat transfer rate
 C_p = heat capacity of liquid metal
 T_p = temperature at pour
 T_{mp} = temperature at melting point
 Time = time of pour
 W = charge weight

and the ratio of the rate of vertical heat transfer near the bottom of the mold to that at the top, construct a diagram similar to the one that Patenaude developed for beryllium ingots¹³ as shown below:

On the diagram, locate the points of monitored ingots and knowing whether or not these ingots are sound, determine the threshold values (dotted lines) of Q_b/Q_t and Q_s necessary to obtain a void free casting. Therefore, by varying the time of pour, degree of superheat, and mold temperature distribution, the critical values for obtaining a void-free ingot can be determined.

2. Investigate the effects of a bottom chill to promote directional solidification and eliminate voids in ingots.

3. Insulate molds on the OD along their length in an effort to promote directional solidification by promoting heat transfer down the mold wall to the mold bottom.

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