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LOS ALAMOS SCIENTIFIC LABORATORY

OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

BOTTOM-POUR RE-USABLE MELTING CRUCIBLES FOR PLUTONIUM CASTING

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LA-2480
METALLURGY AND CERAMICS
TID-4500, 15th ed.

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REPORT WRITTEN: October 1960

REPORT DISTRIBUTED: February 17, 1961

BOTTOM-POUR RE-USABLE MELTING CRUCIBLES FOR PLUTONIUM CASTING

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Contract W-7405-ENG. 36 with the U. S. Atomic Energy Commission

ABSTRACT

Bottom-pour Ta and CaF_2-coated steel melt crucibles for Pu and Pu-rich alloys have been developed and are being used at the Los Alamos Scientific Laboratory. The controlled pour is effected by melting a Pu plug in the bottom spout of the crucible after the desired temperature and vacuum conditions are obtained. This report describes the development of these crucibles which have replaced ceramic crucibles for casting work on the kilogram scale.

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INTRODUCTION

To minimize inclusions in Pu metal, it is desirable to bottom-pour the melt. By doing so, the lighter impurities, which float on top of the melt, will remain behind as part of the skull. Standard Pu casting procedure in the Metal Fabrication Facility(1) of Group CMB-11 at the Los Alamos Scientific Laboratory has been to melt the charge in a ceramic (usually MgO) or CaF_2-coated ceramic crucible in a vacuum furnace using induction heating. When the desired vacuum and temperature are obtained, the melt is gravity-poured by pulling a stopper rod or by melting out a Pu plug in the thick bottom section of the melt crucible. Figures 1 and 2 depict the two casting techniques.

The use of the ceramic melt crucible with the stopper rod pouring technique is objectionable for the following reasons:

1. If the rod does not seat tightly during heating, metal can drip into the mold before the main pour is made.
2. Ceramic material can chip off of the crucible around the stopper rod seat, resulting in inclusions in the casting.
3. Casting feed material must be suitable for loading around a stopper rod. This usually requires the breaking up of feed material or drilling a hole into it for the stopper rod. Further it is time consuming to place the feed material around the stopper rod.
4. Any wedging or binding may cause the ceramic crucible to crack during heating, and initial heating of the ceramic system has to be slow to prevent cracking.

5. After a few runs crucibles usually crack during cooling, resulting in large quantities of ceramic containing recoverable amounts of Pu.

Although the frozen-plug method[2] shown in Fig. 2 does not have the first three disadvantages listed above, the fourth and fifth are still objectionable. All of the above distracting features could be eliminated if a metal melt crucible incorporating the frozen-plug, bottom-pour technique could be developed. This report describes the development work and the resulting metal crucibles which have replaced the ceramics used for the routine pouring of Pu and Pu-rich alloys on the kilogram scale.

DEVELOPMENT

Pours were attempted using Ta crucibles (1.5-in. diameter, 0.030-in. wall, 4-in. long) with a flat bottom to which different diameter spouts had been welded. In all trials the procedure was the same. Pure Pu was placed in the crucible and the furnace was assembled with the bottom of the induction-heating coil level with the top of the spout at the bottom of the crucible. The casting feed was heated to 800°C or higher and held at this temperature for at least 5 min; then the coil was lowered so the bottom of the coil was level with the bottom of the spout. If the Pu plug in the end of the spout did not melt allowing the metal to pour within 5 min after the coil was lowered, the furnace was turned off and the metal allowed to cool. A small section (varying between 0.5 and 1 in.) was then cut from the bottom of the spout and the same melting procedure followed. The spouts were gradually shortened to a length which would permit the metal to pour. Initially a 0.5-in.-long Pu plug was pressed into the end of the spout to form a seal; however, in subsequent runs using the same crucible it was not necessary to re-plug the spout because it never drained completely during a pour.

The following is a list of the size spouts used and the length required to pour the metal within 5 min after lowering the coil using sufficient charge to give a 2-in. head of Pu in the crucible:

1. The 0.190-in. inside diameter, 0.030-in. wall, 4.375-in.-long spout would not pour until it was shortened to 0.5 in.
2. The 0.250-in. inside diameter, 0.030-in. wall, 4.25-in.-long spout would not pour until it was shortened to 2 in.*
3. The 0.375-in. inside diameter, 0.030-in. wall, 6-in.-long spout would not pour until it was shortened to 2 in.*

During this series of melts leaks occurred in the welded zones of the Ta crucibles and two crucibles had to be replaced. Based on the above runs a melt crucible having no welded joints and a 0.25-in. inside diameter, 2.2-in.-long pour spout was designed (Fig. 3). Although a larger diameter pour spout could be used, the 0.25-in. size was selected because it gave a satisfactory pour rate and it was less sensitive to variations in the melt head than a larger opening would be. The spout length was increased slightly (2.2 in. compared to the 2 in. above) to guarantee the plug would hold during the initial heating.

*In some runs the melt was heated to 1000°C and it did not pour until the heater coil was lowered.

Ta MELT CRUCIBLES

The Ta crucibles were fabricated by spinning and had a rough interior surface. This rough surface was ground smooth and the surfaces of the crucibles oxidized by heating in air in a muffle furnace for 2 hr at 500°C. The melt crucibles were then put into service using the heating cycle described for the development work. The initial and subsequent charges poured as predicted when the coil was lowered. Figures 4 and 5 are photographs after the first melt in a Ta crucible. The skull can usually be removed by inverting the crucible and striking it against a flat surface. (Figure 6 is a photograph of the crucible immediately after removing the skull.) The skulls can also be allowed to accumulate for several runs and then burned out by heating on a hot plate. This also re-oxidizes the crucible at the same time. Figure 7 is a photograph of the Ta crucible after 26 runs. This crucible is still in use after 60 runs. The first three Ta crucibles placed into the routine operation have been used to make 9, 60 and 35 castings, respectively, of Pu and Pu-rich alloys.

All three bottom-pour Ta crucibles are working satisfactorily; there is no sign of erosion or wetting with the Pu. The spout length has been shortened to 2 in. They have demonstrated their purpose in that they are used with a controlled pour, the amount of skull is reduced, and MgO residues have been eliminated. Charge load variations from 1 to 6 kg have been successfully poured from the same crucible. It was further found that it was not necessary to initially plug the pour spout. The feed material formed its own plug as the initial charge was heated.

Table 1 lists analytical data of cast feed before melting in the Ta crucible and the as-cast ingot. The charge was heated to 800°C in 15 min, held at 800°C for 10 min, and then poured into a 2-in.-diameter CaF_2-coated graphite rod mold by lowering the induction coil and melting the plug. There is no pick up of Ta or other impurities during the run.

If the cast feed is bulky an extension (Fig. 8, Part 1) can be placed on top of the crucible to facilitate loading. This permits all of the charge to be melted at the same time, thereby eliminating extra melt-downs in order to accommodate the bulky material.

CaF_2-COATED STEEL MELT CRUCIBLES

Recent improvements in the coating of steel molds[3] permitted a re-usable bottom-pour crucible to be designed (Fig. 8) using a low-carbon (AISOC-1019) steel. The spout was split so the inside surfaces could be coated with CaF_2. All surfaces that were to be coated were first plated with 0.002-in.-thick Cr. Figure 9 is a photograph of the assembled crucible after its first casting. The skull in the lower left corner was small and flaky, weighing 15.82 g, and was 1.51% of the total charge. Figure 10 is a photograph of the crucible after disassembly. During use this crucible was heated as high as 900°C. Initially the spout was 3.25-in. long and had two rings to hold it together. After the second casting it was shortened to 2.25 in. and only one ring was used to hold it together; this arrangement worked satisfactorily. A total of 12 melts were made and there was no attack or wetting of steel with the Pu. Although the steel crucible is just as satisfactory as the Ta crucible, it requires disassembly and the coating is repaired between runs. In practice it has been found necessary to re-coat it after every second or third melt.

Table 2 gives analytical data of cast feed before melting in the steel crucible and the cast ingot produced using the same heat cycle and casting crucible as used for the Ta crucibles. It is apparent that there is no pick-up of impurities introduced by the CaF_2 coating or the Cr-plated steel crucible.

Bulky material can be accommodated in this crucible in the same manner as with the Ta crucible.

YIELDS

The skulls remaining in the metal melt crucibles were found to be considerably smaller than those remaining in ceramic crucibles. Although the percent of material remaining in a skull is primarily dependent on the condition of the feed material, skulls from similar feed material average 50% less when the pour is made from metal crucibles rather than ceramic ones.

Table 3 presents data from bottom-pour casting runs made from similar feed using both metal and ceramic melt crucibles. In all runs the melt was held at 800°C for 10 min before pouring by melting a plug in the bottom of the crucible.

SUMMARY AND CONCLUSIONS

The use of the re-usable metal melt crucibles offers the following advantages over the previously used ceramic crucibles:

1. There are fewer residues for recovery in that the skulls contain 50% less Pu, and Pu-contaminated ceramics are eliminated.
2. Sounder castings are produced by the elimination of ceramic particles which frequently chip off of the ceramic melt crucibles and become trapped in the casting.
3. The metal crucibles are easier to use because they are more rugged, a Pu plug is not required for each run, and they may be loaded with almost any shaped feed material.
4. Metal crucibles may be heated faster than ceramic crucibles, and the possibility of cracking during heating due to the expansion of the feed material is eliminated.
5. Leakage of pre-pours does not occur even when the melt is heated as high as 1000°C.

Bottom-pour metal melt crucibles for Pu, using the frozen-plug, induction heating technique, have replaced ceramic melt crucibles for routine Pu casting work on the kilogram scale at the Los Alamos Scientific Laboratory. Both CaF_2-coated steel and Ta crucibles are being used.

The maximum temperature for using this system had been arbitrarily limited to 1000°C. Although the method should be suitable for operating at higher temperatures, the solubility of Ta in Pu and the effectiveness of the CaF_2 coating above 1000°C become problems. These obstacles could be overcome with a suitable high temperature coating material or a more resistant container material than Ta.

REFERENCES

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3. J. W. Anderson and W. C. Pritchard, "The Use of Calcium Fluoride as a Coating for Plutonium Casting Molds," Los Alamos Scientific Laboratory Report LA-2315 (1959) (classified).

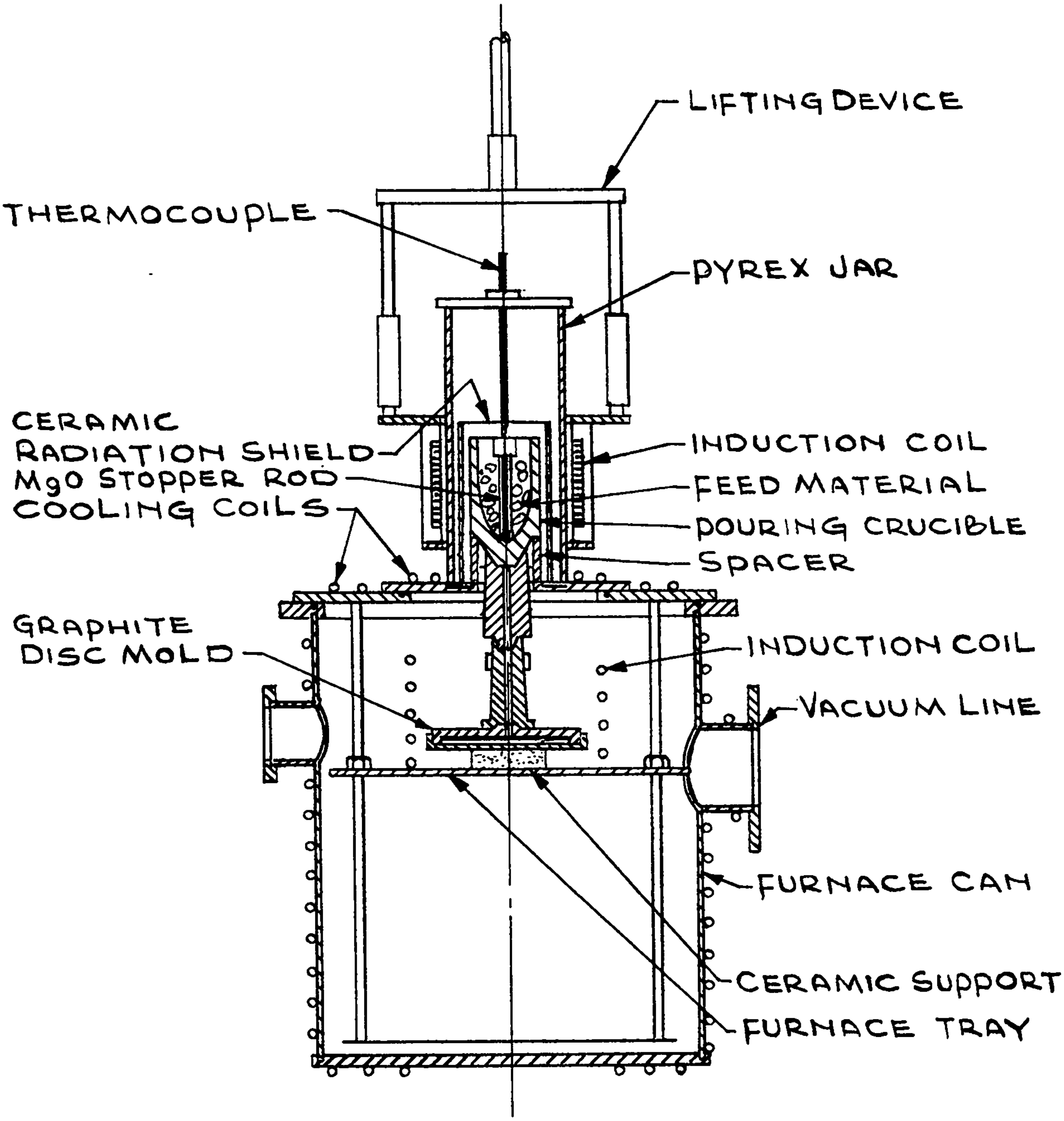

Fig. 1. Pu Vacuum Casting Setup Using Stopper Rod

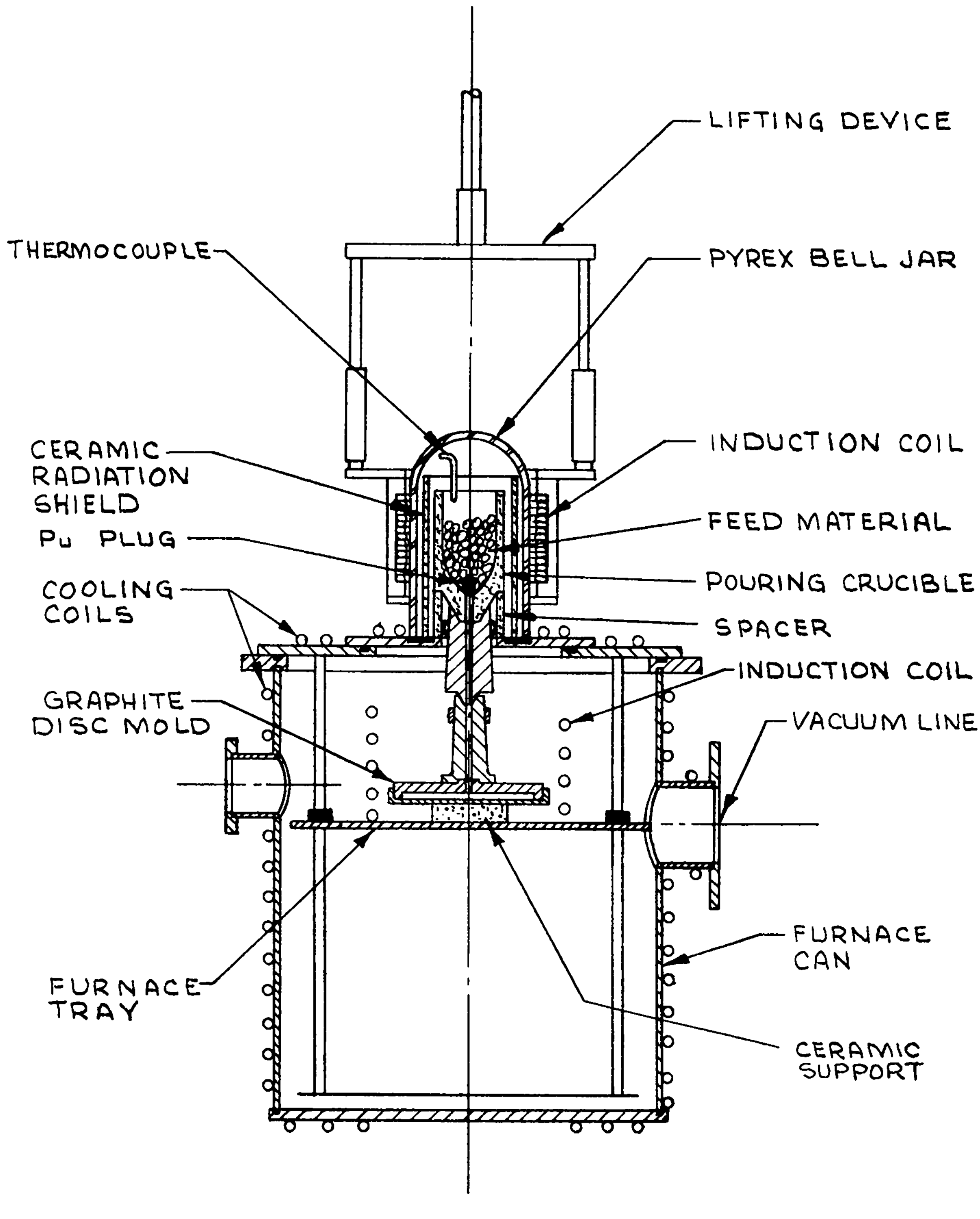

Fig. 2. Pu Vacuum Casting Setup Using Frozen-Plug Technique

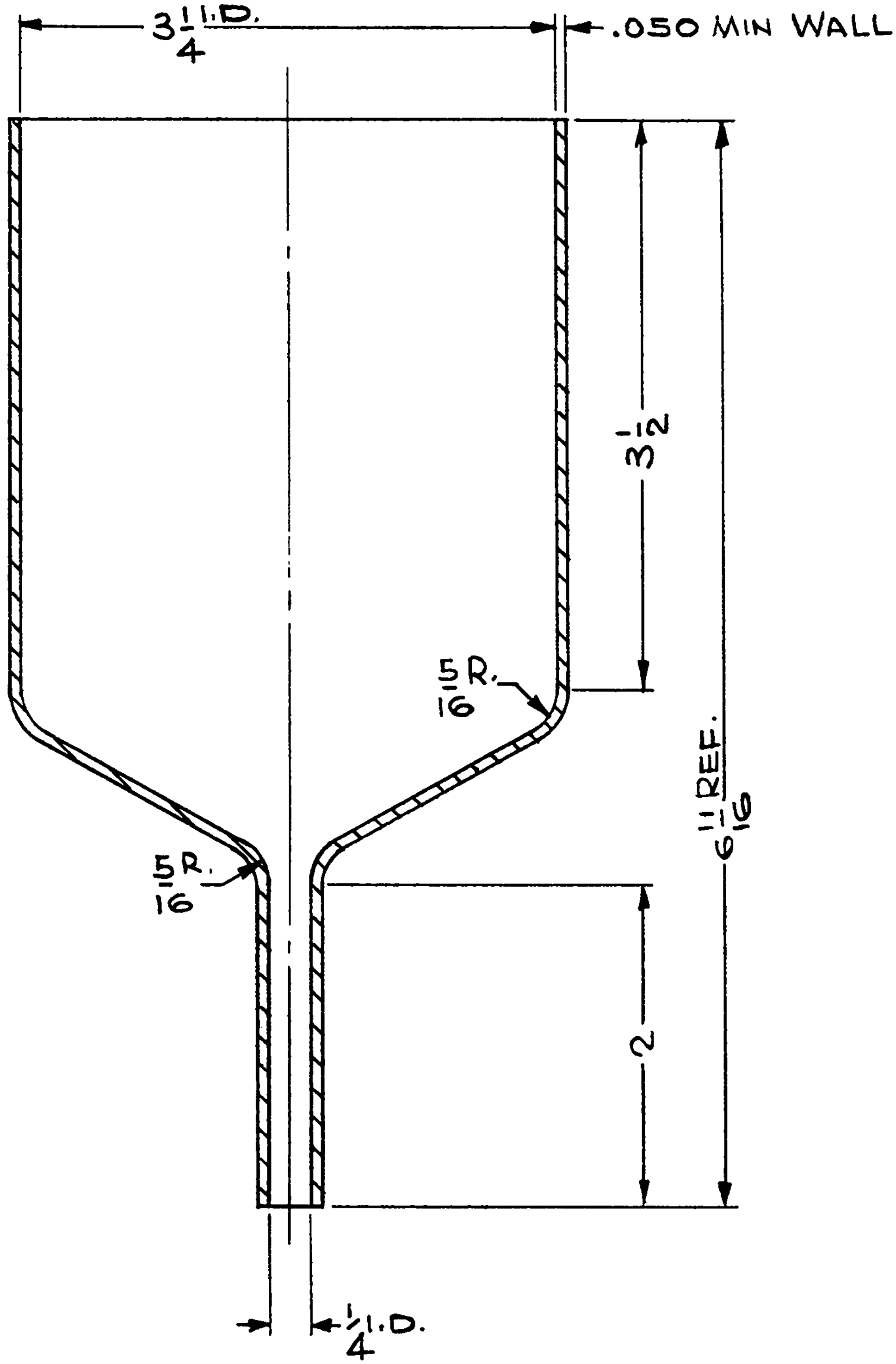

Fig. 3. Bottom-Pour Ta Melt Crucible

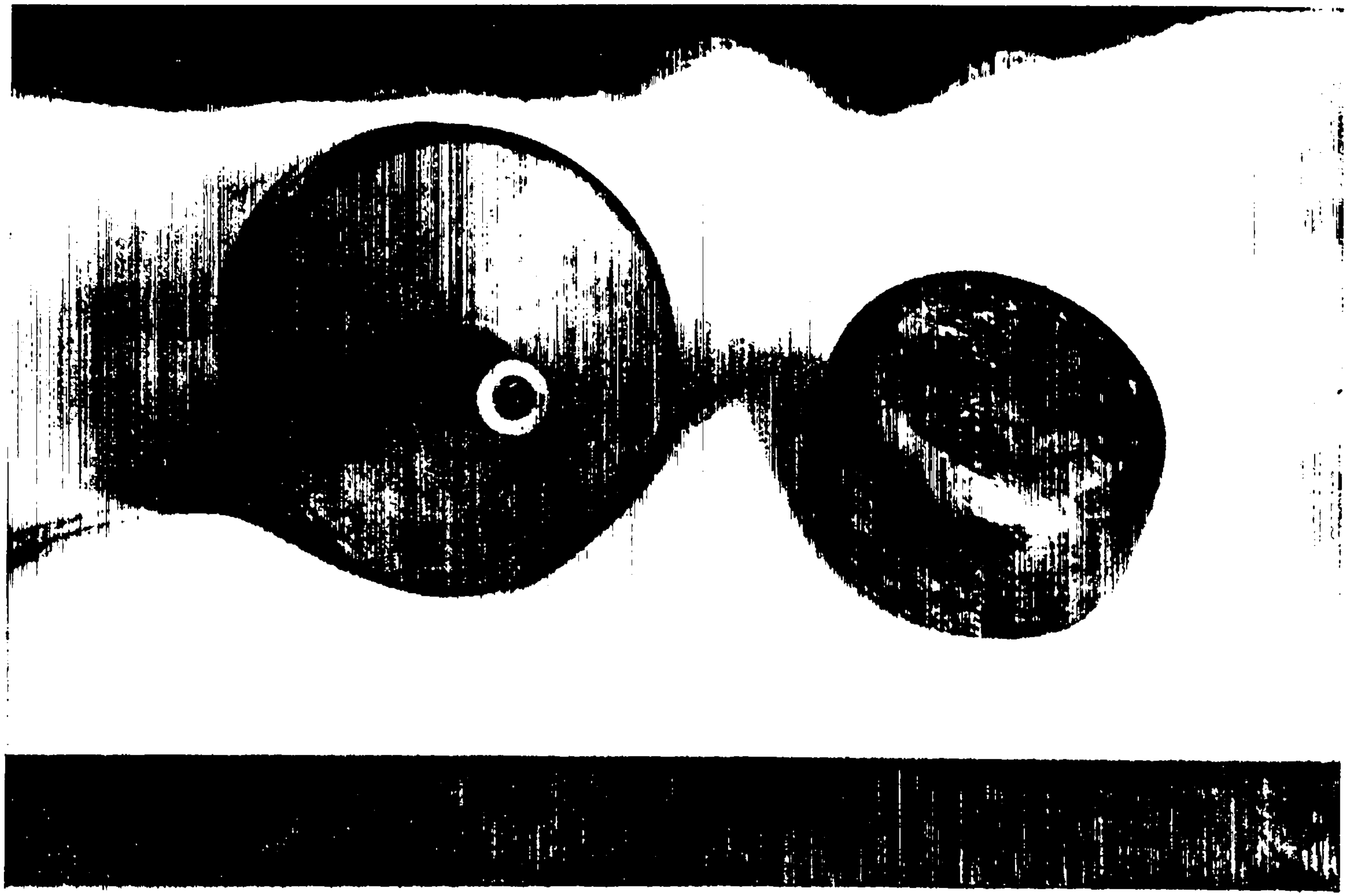

Fig. 4. Ta Melt Crucible (Top View)

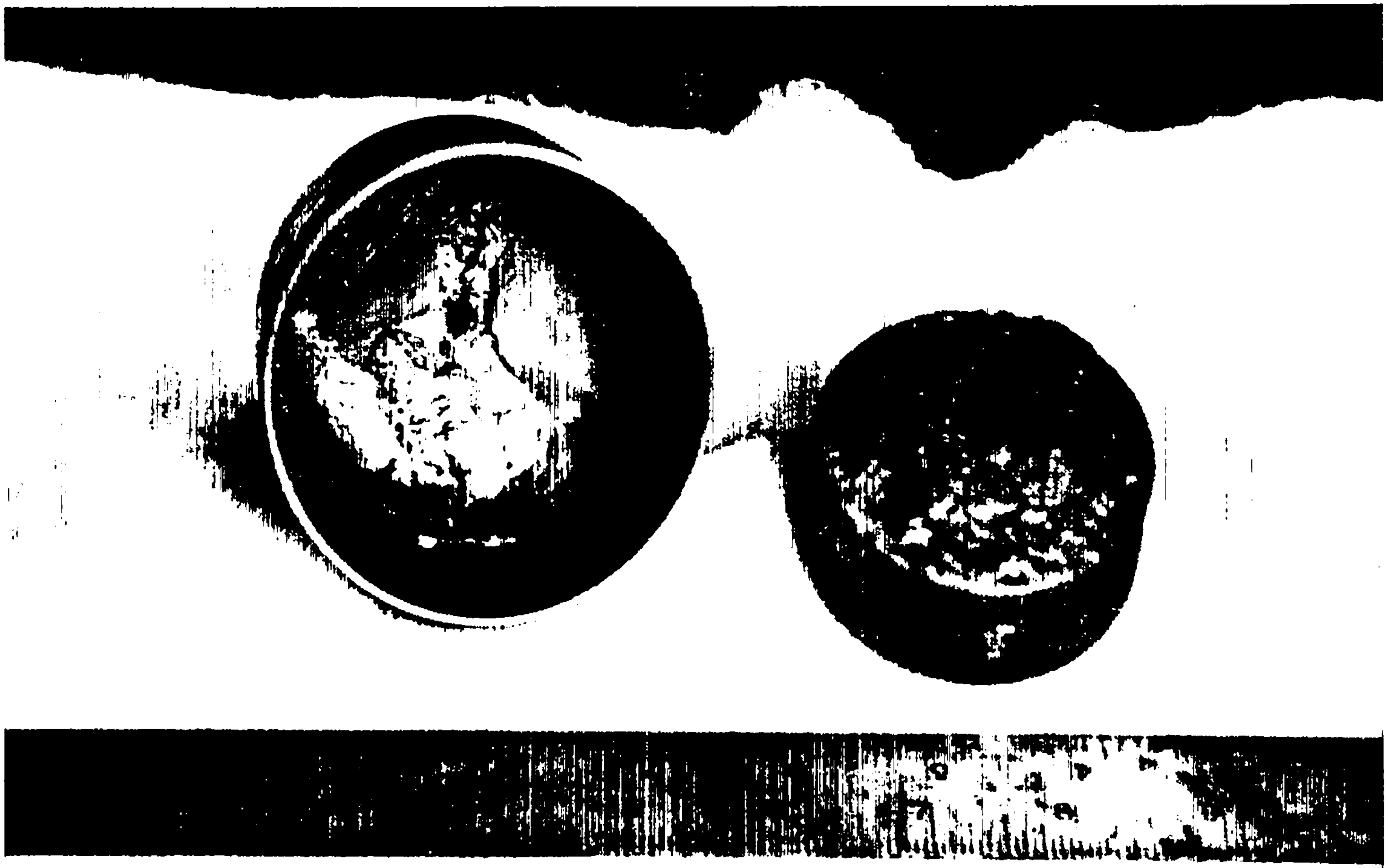

Fig. 5. Ta Melt Crucible (Bottom View)

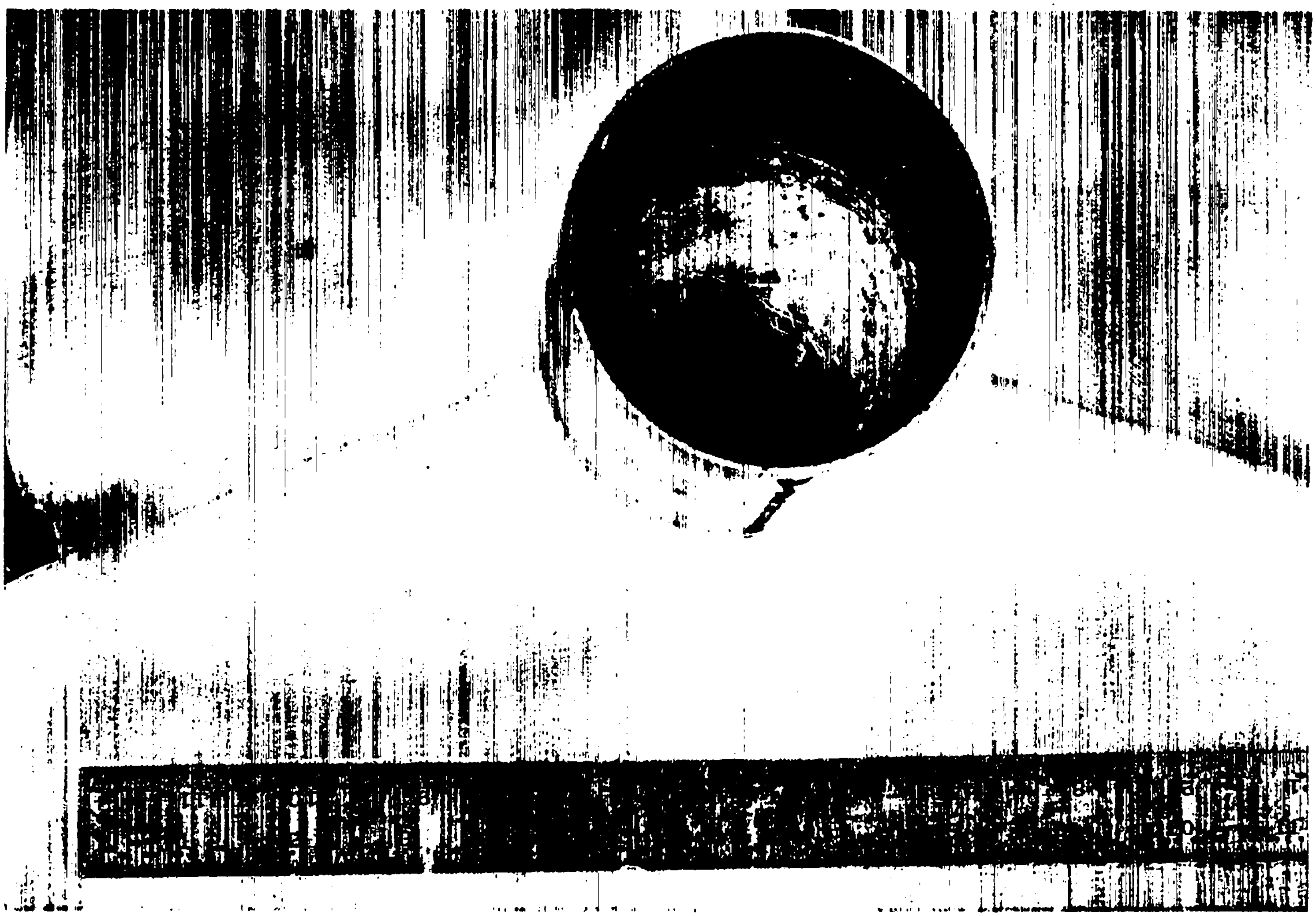

Fig. 6. Ta Melt Crucible - Skull Removed

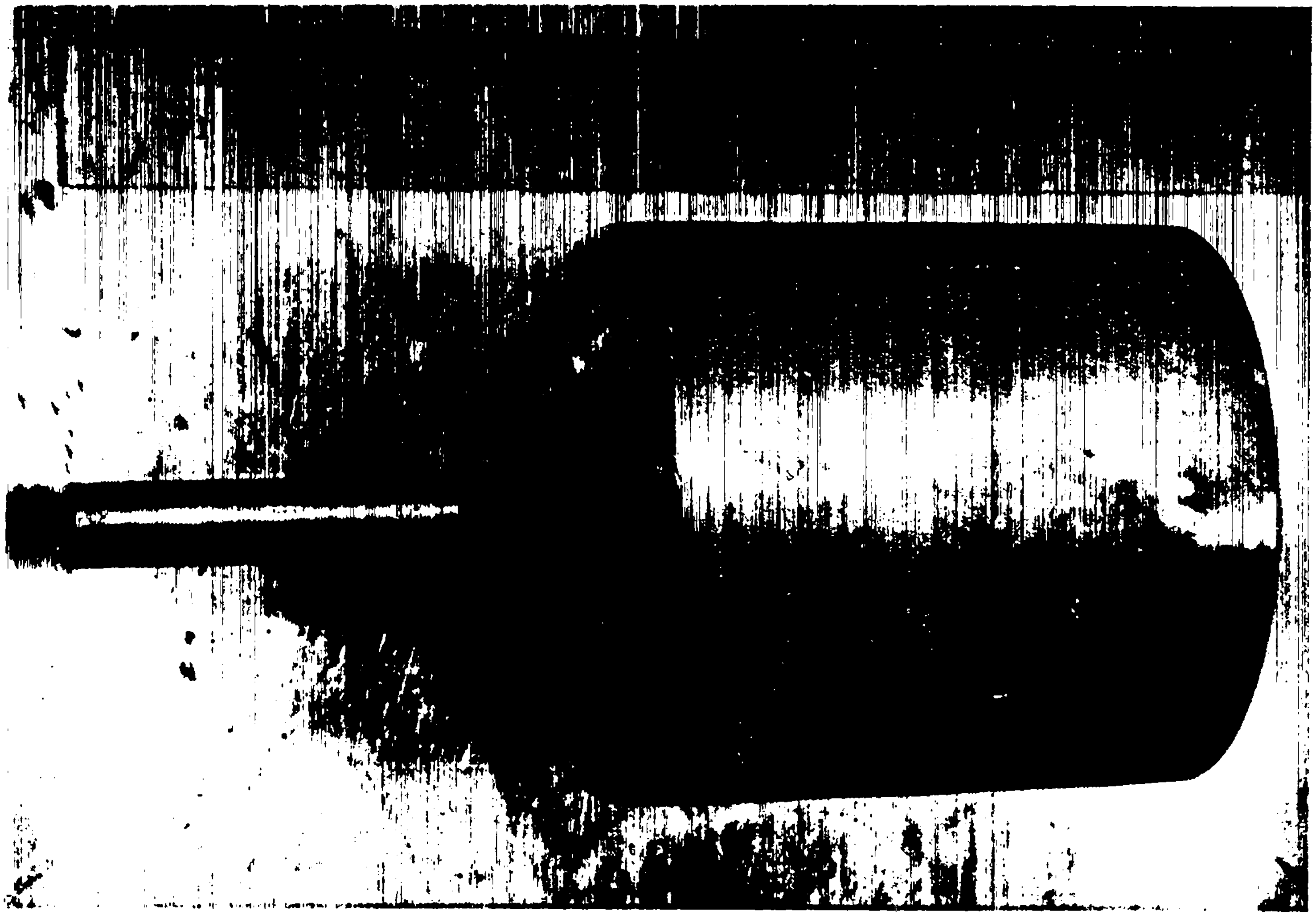

Fig. 7. Ta Melt Crucible After 26 Runs

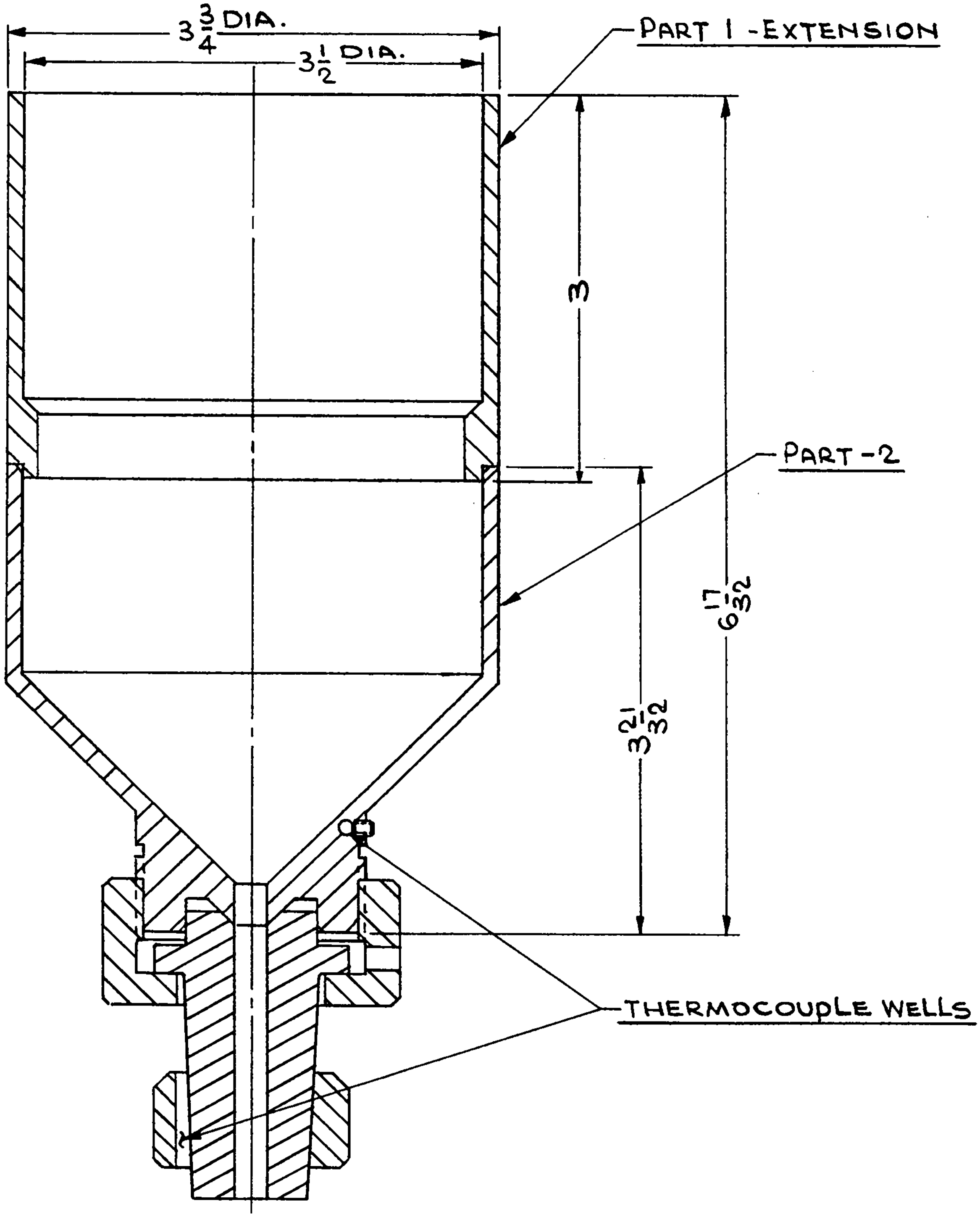

Fig. 8. Melt Crucible Extension Ring on Steel Melt Crucible

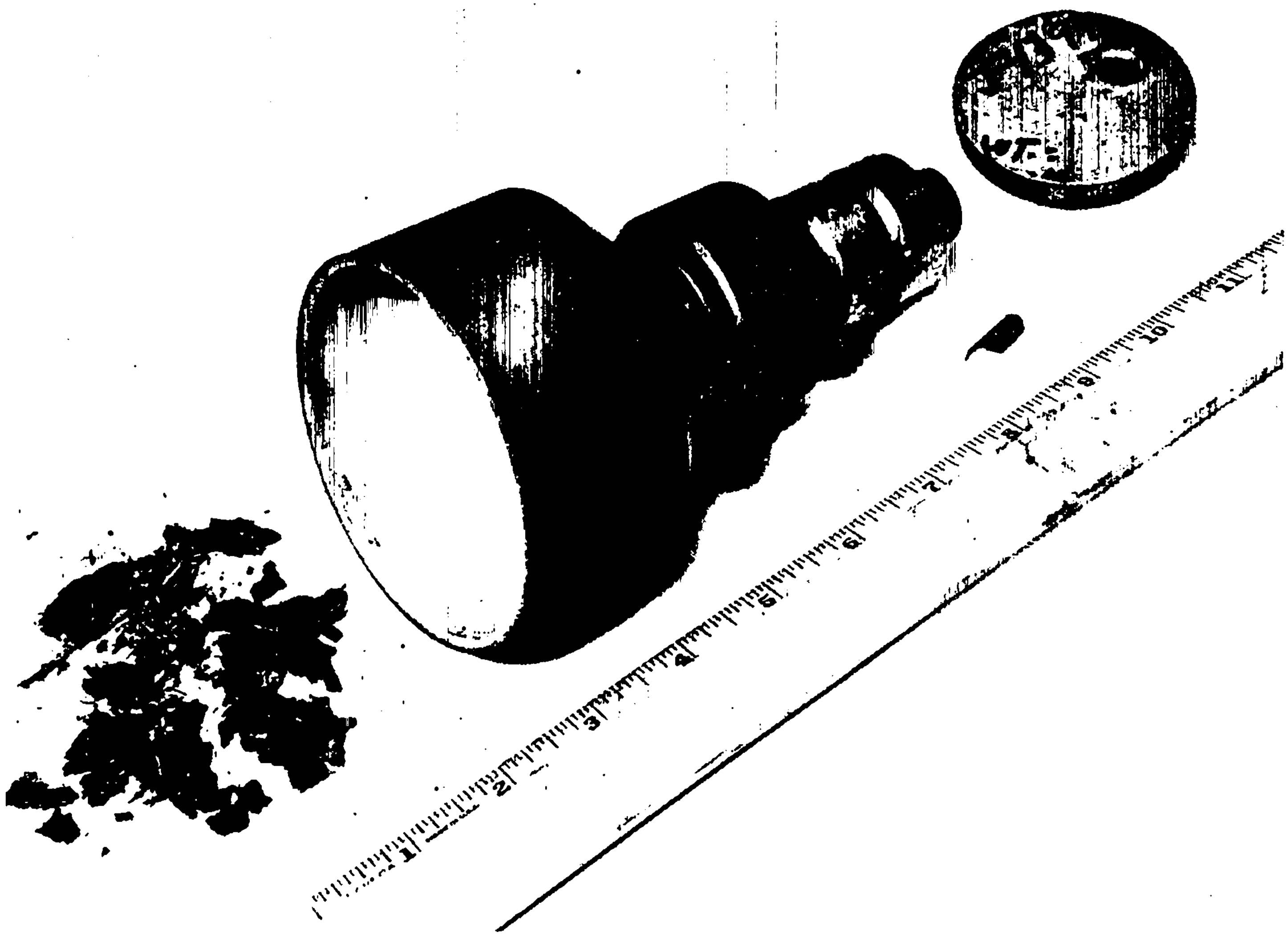

Fig. 9. Steel Melt Crucible

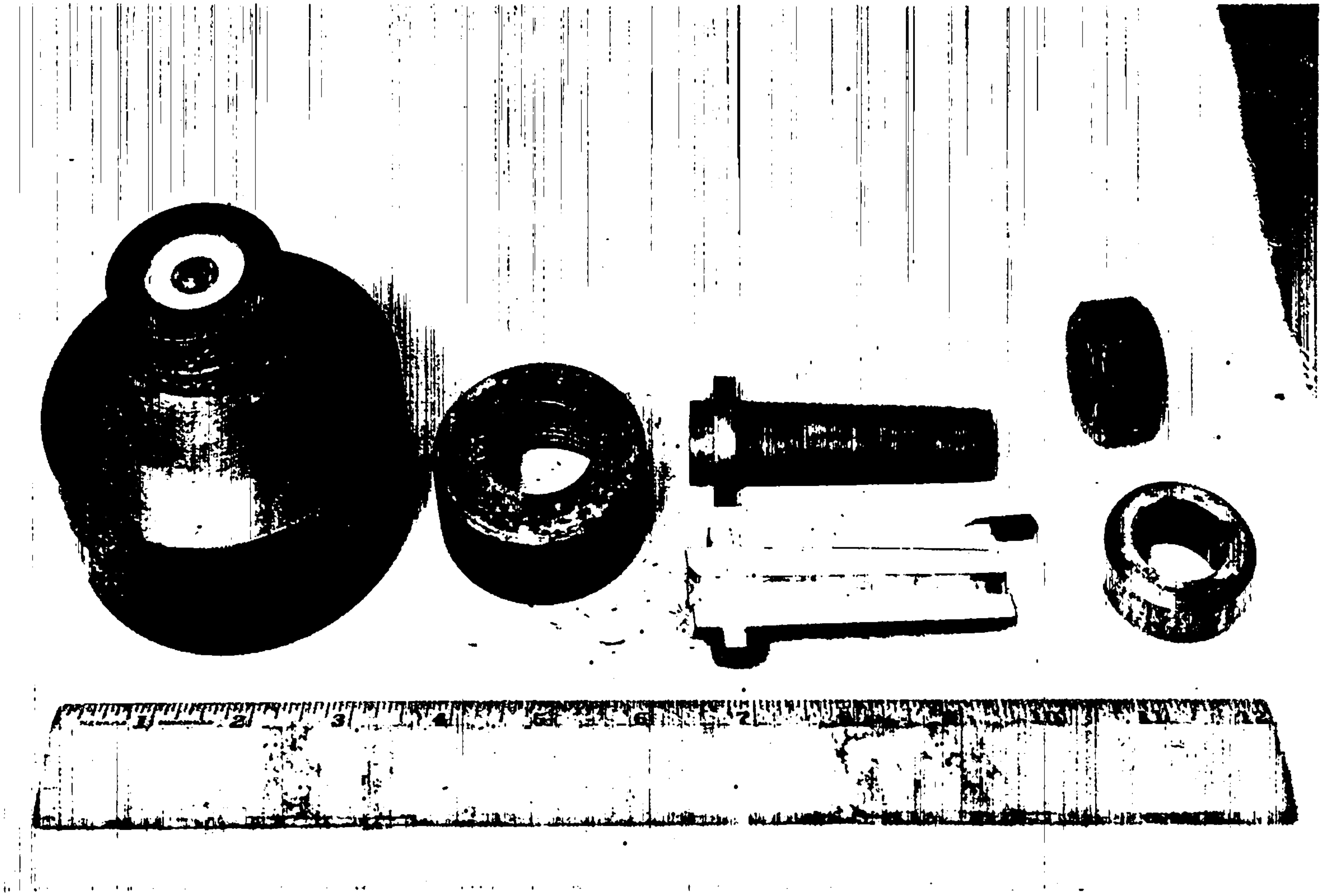

Fig. 10. Steel Melt Crucible (Disassembled)

Table 1

ANALYTICAL DATA FROM Ta MELT CRUCIBLE

Run No. J 4642

Element	Analysis, ppm	
	Feed Material	Cast Ingot
Li	< 0.2	< 0.2
Be	< 0.2	< 0.2
Na	< 10	< 10
Mg	10	< 5
Ca	< 5	< 5
Al	50	35
La	< 10	< 10
Si	110	130
Pb	3	3
Cu	10	20
Cr	55	50
Mn	10	15
Sn	< 1	< 1
Bi	< 1	< 1
Co	< 10	< 10
Zn	10	10
C	155	130
O	85	240
B	0.6	0.6
F	< 2	< 2
Ni	110	110
Fe	350	340
Ta	< 35	< 35

Table 2

ANALYTICAL DATA FROM CaF_2-COATED STEEL MELT CRUCIBLE

Run No. J 4699

Element	Analysis, ppm	
	Feed Material	Cast Ingot
Li	< 0.2	< 0.2
Be	< 0.2	< 0.2
Na	< 10	< 10
Mg	< 5	< 5
Ca	10	< 5
Al	10	25
La	< 10	< 10
Si	25	25
Pb	< 2	< 2
Cu	5	2
Mn	5	5
Sn	< 1	< 1
Bi	< 1	< 1
Co	< 10	< 10
Zn	10	< 10
C	115	125
O	270	75
B	< 0.5	< 0.5
Ni	45	30
Cr	< 20	< 20
F	< 2	< 2
Fe	50	40
Ta	< 35	< 35

Table 3

Pu Skull Data of Bottom-Pour Castings Using Similar Casting Feed in Ceramic and Metal Melt Crucibles

Casting No.	Skull Weight, g	Charge Weight, g		Percent Skull
	Ceramic Melt Crucibles			
J 4055	200	4,590		4.36
J 4060	160	4,615		3.47
J 4316	231	4,270		5.41
J 4326	122	2,591		4.71
J 4350	102	2,074		4.92
J 4363	107	2,117		5.05
J 4376	43	1,267		3.39
J 4419	40	1,610		2.48
J 4430	154	4,563		3.37
J 4463	289	6,034		4.79
Total	1,448	33,731	Average	4.20
	Ta Melt Crucibles			
J 4552	119	5,632		2.11
J 4591	44	2,347		1.87
J 4600	131	5,643		2.32
J 4626	40	2,133		1.88
J 4631	8	1,399		0.57
J 4632	27	1,366		1.98
J 4643	34	1,260		2.70
J 4644	37	1,327		2.79
J 4645	23	1,292		1.78
J 4661	33	1,265		2.61
Total	496	23,664	Average	2.10