CONFIDENTIAL

UNCLASSIFIED

C -C.

AEC RESEARCH AND DEVELOPMENT REPORT

CLASSIFICATION CANCELLED

DATE 1-29-60

For The Atomic Energy Commission

.74. R. Cansel

Chief, Declassification Branch fl

Photostat Charge \$ 4.59 for Access Permittees

Available from
Technical Information Service
P. O. Box 1001, Oak Ridge, Tennessee

Contract No. W-7401-eng-37

Section To IV

VACUUM CASTING OF ALUMINUM-SILICON COATING ON TUBALLOY

(Final Report on a part of P.A. #390-ML-54-S, F.S. 17)

D. L. Schwartz and L. Kurland

This report was prepared as a scientific account of observations are the comment-sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights. The Commission assumes no liability with respect to the use of, or from damages resulting from the use of, any information, apparatus, method, or process disclosed in this report.

RESTRICTED DATA

This document contains restricted data as defined in the Atomic Energy Act of 1954.

Its transmittat or the disclosure of its contents in any manner to an unauthorized person is prohibited.

This document contains Confidential-Restricted Data relating to civilian applications of atomic energy.

Report received: March 1, 1945; Figures received: March 12, 1945

Issued: MAR 23 1945

CONFIDENTIAL

UNCLASSIFIED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

CONFIDENTIAL

Table of Contents

α ·																	Ž	Page
Summa	y .	c	•	•	•	•	•	•	•	٥	•	•	•	•	•	٠	9	3
I.	Intro	duct	ion	.	٠	٥.	6	•	•	•	•	•	•		Ð	•	•	4
· II.	Inves	tiga	tio	ns	•	•	•		•	5	•	٠	•		•	•	•	<u> </u>
	A.	Gla								ts	•	. 🌣		٠	•	•	a	5
	B.	Ste	el :	Mol	d :	ලිද	pari	men	ts	•	•	•	٠	G	۰	•	9	6
		1.	• •	Aut Mol	Q=(ter			odu	cti	OM	Ces	tin	Œ			
		_	•				•			•	•	.•	• ,	•	٥	•	•	8
							Cen		_			۰	•	0	0	•	•	9
		, 3,					Pro					•	٠	•	٠	•	•	9
		4.	, !	l'op	Co	ni	cal	Ce	nte	rin	g	•	•	•	•	•	o	10
III.	Summar	y of	R	esu.	lte	3	0	•	•	•	•	•	0	•	•	•	9	10
IV.	Recomm	enda	tic	ons.	•	•	٥	۰	•	9	٠	•	۰		•			11
٧.	Refere	nces	,	• .	9	•	•	0	•	•	•	•	0	ย	۰	•	•	11
	Append	ix:																
	Tabl Figu	.es I res					• late	Il		•	•	•	.•	٠	٠	•	12	∞ 1 5

The successful coating of tuballoy is a problem that has been approached in many different ways. Basically, the problem resolves itself into a wetability study. How best to prepare a clean and active tuballoy surface such that wetting, that is, metallurgical bonding, shall always occur between the coating material and the tuballoy, has been the underlying motive for this research. Of equal importance has been the adaptation of the techniques of vacuum casting to successful coating of W slugs on a production basis.

The approach to this investigation has been guided by these objectives:

- 1. Preparation of a clean tuballoy surface.
- 2. Use of a vacuum to retain the previously cleaned tuballoy surface.
- 3. Make the vacuum serve the additional function of providing the means for bringing molten metal around a slug that is centered in a suitable mold.

The initial experiments were carried out in evacuated glass capsules and made use of one-half lengths of X slugs. These investigations served to indicate how best to prepare the surface of the tuballoy for obtaining metallurgical bonding between it and the coating material, such as Al/Si, and the nature of the difficulties to be encountered with coating by vacuum methods.

As a result of these findings and because of the added impetus at that time to find suitable means for coating W slugs, several steel molds were designed. Tests were made and subsequent changes in design afforded solution to most of the mechanical problems. The end product of these changes is the production vacuum casting mold.

The final and complete solution of all the difficulties has not yet been realized and because the problem of wetting is not an important one any longer, probably the vacuum coating of W slugs with Al/Si will never achieve production status. Nevere theless, the fundamentals of the process have been shown to be sound and the utility of the general techniques for straight casting is recognized.

Apart from the direct coating of tuballoy slugs with metals of moderate melting points, use of the techniques here developed is foreseen in casting high melting metal billets and composite billets of tuballoy and other metals in which the project might become interested.

Prepared by Leonard Kulaust
Submitted by Albuniant.
Group Leader

Approved by:

Section Chief

702

Associate Division Director

division Director

I. Introduction

Coating and bonding W slugs in a single operation by the techniques of vacuum casting was undertaken as a possible process for use in place of the multioperation aluminum silicon canning process. Several difficulties inherent to the
latter process were seen to be overcome by the former.

- 1. Internally heated corrosion tests had uncovered the serious difficulty of swelling of the Al jackets as corrosion proceeded underneath the jacket. Since Al/Si is considerably less ductile than 2S Al, an Al/Si coat, when subjected to the pressures developed by corrosion product formation, would probably crack and flake off rather than swell. This would prevent jamming of the water annulus in the pile.
- 2. The slug is never in contact with the bath so the bath composition remains constant.
- 3. Since wetting occurs almost instantaneously, uniformity of bonding is more consistently obtained over the slug surfaces.
- 4. Some flexibility is allowed in that vacuum insulator or conductor ends can be applied with about equal ease.

The early investigations showed that the responsibility for failure to obtain consistently uniform wetting was due to mechanical difficulties such as off centering in the mold, occluded gas, defective slugs etc. The experiments that are discussed have been simed at evolving a mechanically perfect mold assembly that is a practical tool. Into its design has been taken cognizance of the limitations to which an oxide free tuballoy surface can be prepared end maintained. This mold has been designated as the auto-centering production casting mold.

While a complete article has not yet been turned out, valid explanations of defects can be made; and it is interesting to note that they are dependent on mechanical difficulties and not chemical or metallurgical problems.

II. Investigations

The experimental work may be conveniently divided into two stages. The glass capsule experiments during which the methods of surface preparation were correlated to the ease and extent of wetting of the tuballoy; and second, the steel mold experiments, where the mechanical problems were treated and out of which were developed some significant features pertinent to mechanically fluent vacuum casting.

A. Glass Capsule Experiments

There are two standard procedures for getting a visually clean tuballoy surface:

- 1. Cleaning with warm (70°C) HNO3.
 - 2. Shot blasting with 100 mesh grit.

Both methods of cleaning were investigated. A factor for important consideration in conjunction with this problem was that the cleaned slug had to remain clean, that is, as free as possible from any influence that would prevent wetting between the tuballoy and coating material while undergoing processing before actual casting. Unless otherwise stated, Al/Si eutectic was in all instances the coating material to which references is made.

The circumstances under which it was found that a shot blast slug offered the best means for obtaining a good surface for Al/Si casting can be illustrated by several glass capsule experiments.

Acid cleaning of slugs was done in place. The tuballoy was sealed in a 35 mm. glass tube with a ground glass joint on one end to connect to the vacuum line and with a straight open outlet tube on the other end. The open end was clamped off and warm 50:50 HNO3 was brought in contact with the slug until the surfaces were silvery. Then the acid was discharged and the slug was given, in the order named, a cold water rinse, an acetone wash, and a blast of dry air (compressed air passed through a bed of activated alumina held in a Lectro-dryer). As soon thereafter as possible, a thin bulb was blown on the open end inlet tube and the capsule inserted into the vacuum line. Plicene cement was used to seal the ground glass joint.

After a short period of evacuation to about 5 microns, cutgassing of the assembly was attempted by heating under kinetic vacuum with periodic flushes of purified argon. Purification consisted of passage through hot copper turnings, drierite, ascarite, P2O5, and in some instance, Ca ships and Tu turnings. Degassing was done at 350-400°C under a vacuum of about 4 microns. The slug surfaces always became colored; the change was from silver to teal blue to canary yellow. These films appeared to be stable and underwent no change on standing in air.

In the other instance of cleaning by shot blasting with 100 mesh shot, mechanical abrasion is responsible for disengaging the oxide on the tuballoy surface and so obviates any subsequent oxide formation as a consequence of the particular cleaning operations. Development of a technique for sealing off a shot blast slug and attaching it to the vacuum line with a minimum of oxidation was accomplished by sealing off under a flow of argon. Using this method, slugs were outgassed up to 450°C without visible change in the brightness of the surface and were subsequently vacuum cast in Al/Si. On breaking the inlet bulb beneath the metal bath surface, the capsule is filled with metal in a fraction of a second. In all cases at least partial wetting of the tuballoy was obtained. The extent to which the total surface was bonded was dependent on mechanical features such as how well the clug was centered in the glass tube and on whether or not the glass cracked before motal flowed up through the capsule.

The glass ampule studies, which are partially tabulated in the Appendix, Table I, allowed the following general observations:

- 1. A tuballoy slug surface that is properly shot blasted provides a satisfactory surface for securing very rapid wetting between it and Al/Si by vacuum coating methods.
- 2. Degassing of the tuballoy and its container before casting is essential to sound casting and good wetting.
- 3. The technique of vacuum coating, wherein the vacuum serves the triple purpose of maintaining a clean surface, preventing inclusion of dross and scum in the cast material, and serving as the mechanism for causing metal to encase a slug, is workable.
- 4. The preparation of a uniformly clean tuballoy surface by hydride formation and subsequent decomposition is apparently not amenable to large scale procedures for coating slugs. More development work is necessary for a valid appraisal.

- 5. Preheating the slug is not a necessary prerequisite for securing metallurgical bonding if sufficient reservoir space is allowed to ensure enough washing action by the rising molten metal to break through any film that might have been retained on the slug surface.
- 6. The uniformity of wetting is a function of the uniformity of the annulus between the slug and the mold walls. This is further clarified by restatement in the following way. The most serious difficulty is the problem of centering and maintaining the centering of the slug throughout the casting operation.

These observations served as a guide for the design of steel molds for coating W slugs.

B. Steel Mold Experiments

Coating of full size W slugs was first attempted using a quartz tube as a mold. The bore allowed an average annulus of 140 mils. Several experiments were run with this set-up but each time cracks developed along the upper portions of the mold at the instant of dipping and vacuum was lost. Quartz molds were abandoned in favor of cold rolled steel molds.

Casting with the mold designated as E.C.M. and shown in Figure 1 gave proof that metal could be brought up through a 140 mil annulus and thereby encase a W slug. The initial trials did not provide any reservoir atop the mold and when encased slugs were chiseled, wetting was not found. Al/Si bath temperatures ranging from 600-800°C failed to alter the previously reported results and it became clear that the practical difficulties of maintaining an oxide free surface would necessitate a washing action across the tuballoy surface. Therefore a reservoir was bolted to the mold that allowed for approximately 10 times the volume of metal in the 140 mil annulus.

The assembly of the slug in the mold is illustrated in Figures 1 and 5. The slug was held in place by aluminum plugs that were fitted into holes drilled in the ends of the slug. The bottom plug fitted into a recess in the bottom plate and the top plug was fitted to a two-spoke wheel that lay in a recess in the mold wall. Vacuum was secured between the component parts by using annealed 25 Al gaskets. A bearing surface on one of the two mating pieces bit into the gasket and made a vacuum tight seal. Fusible aluminum plugs sealed the inlet and melted on dipping below the surface of the bath. This allowed the molten metal to rise in the mold and reservoir, and since the metal was drawn from below the surface, none of the dross entered the mold.

Some of the stages in the development of centering devices are shown photographically in Figures 11 and 12. Freedom of metal flow was aimed at by the changes in centering devices. Vacuum insulator type end plugs have been designed and could be used as well as the conductor type ends that have been used during the course of this investigation. Examples of this type of centering end plug is shown in Figures 11 and 12.

Two very serious difficulties remained with this mold.

1. The inability to obtain consistent true alignment of the bottom plate recess with the rest of the mold assembly and melting out of the bottom pin did not make for symmetrical coatings.

2. The imbedding of two spiders in the casting did not make this method of centering a practical production method.

Several other developments at this time were the use of 3/4" inlet tube with rolled fusible Al-clad diaphrams, 15-25 mils thick, in place of the 1/4" inlet with pressure fitted Al plugs, and the use of a modified Al/Si bath containing 0.1% Be. Bath temperatures of 800-815°C were used and melting of the diaphram occurred in 2-3 seconds as compared to the 20-25 seconds required for the Al plugs. Obviously, the time to melt through the diaphram is dependent on the melting point of the material, the diaphram thickness, and the bath temperature. Filling of the mold and reservoir occurred almost instantaneously after melting of the diaphram.

Bonding was accomplished on the initial trial of the mold-reservoir.

Figures 2, 3, 4 and 5 show the slug and part of the cast structure of the first experiment with the reservoir. An interesting and significant feature was here observed. The few unwetted portions of the slug surface were directly adjacent to large cracks in the tuballoy surface, and parallel to strings of gas pockets in the coat. This was the first positive indication of the source of the previously noted blow-holes in the cast material. On the basis of these observations, slugs were subjected to high temperature outgassing under kinetic vacuum before subsequent surface treatment and assembly in the mold. Sample outgassing data and warp measurements are shown in Table II.

Experimental results verified the aforementioned observations and future castings with previously outgassed slugs had good sound coats, free of blow-holes. Refer to Figures 2 and 6 for comparison between coatings on untreated and treated slugs.

It was next attempted to cast directly 40 mil coats on W slugs using a small annulus steel mold. Because of the rapid conduction of heat through the mold walls, the metal froze part way up the mold and it was necessary to use higher bath temperatures. Offcentering further complicated the problem for where the annulus was much smaller than the 40 mils, the metal would freeze and in some instances would not even begin to flow in these constricted regions. The use of the 140 mil annulus mold with suitable low thermal conductivity sleeve inserts was forseen as a plausible solution to this problem.

Using the standard leading and cleaning procedures and inserting a glass sleeve between the mold wall and the slug to allow a 40 mil annulus, castings were made and good bending on parts of the slug was observed. The glass, by nature of its low thermal conductivity, allowed the molten metal to retain more of its heat while flowing past the slug, thereby not freezing prematurely. The glass usually shattered on cooling and in most instances adhered to the Al/Si. Off-centering was emmipresent and completely coated slugs were not prepared. The off-centering was of the magnitude to allow a coat of 25 mils on one side and 60 mils on the opposite side; and in some instances was so thin on one side that no metal flowed over part of the slug. This same type of experiment was repeated in an effort to secure a completely coated article, but off-centering prevented attainment of this objective. A photograph of a slug coated in a glass lined mold is shown in Figure 8.

The difficulty of satisfactorily removing the glass from the coating gave rise to tests of other materials of low thermal conductivity as sleeves. Stainless steel (18-3) sleeves with about 1/3 the thermal conductivity of cold rolled steel and annuli of 40 and 60 mils were used. Film oxidation of the steel sleeves was done in an effort to prevent metallurgical bonding with the Al/Si. Nevertheless, the Al/Si wetted the stainless-steel sleeves and the sleeves were removed only by the use of the compression testor and the application of 30,000 lbs. to the sleeve. In so

doing, the outside surfaces of the coat were scarred and the sleeves ruined. The remaining coat was chiseled and the bond was found to be continuous over most of the slug, and the cast structure sound. A few discontinuities in the bond were adjacent to small holes in the coat. Figures 6 and 7 show the slug and coating material.

Several photographs of coated slugs and mold reservoir assembly are included in appendix to this report.

While demonstrating more conclusively then ever before the feasibility of vacuum coating of W slugs, these first steel molds also showed the practical mechanical problems to be overcome in designing a production mold.

1. Auto-Centering Production Casting Mold

The P.C.M. was designed to be a production tool. Its design eliminated most of the mechanical difficulties of the original steel mold.

- 1. There were no steel spiders to get embedded in the casting.
- 2. The inexact alignment of the bottom plate with the mold was obviated by making the mold and inlet a single permanent unit.
- 3. Clumsy mating of parts by bolting against Al gaskets was replaced by the more efficient clamping against a neoprene gasket.
- 4. The greater utilization of the mold capacity was arranged by having replaceable inlet tips (see Figures 13 and 16).
- 5. The rapid assembly and disassembly of the mold was achieved.
- 6. A split centering ring was used to hold the top tapered key and was readily removed from the casting (see Figure 15).
- 7. Slight tapers of the reservoir and inlet surfaces facilitated the rapid removal of the casting.
- 8. Cowee type seal was utilized for maintaining a vacuum seal end yet allow transverse motion of the metal-step shaft (Figures 13 and 18).
- 9. Split or continuous sleeves that have a lower thormal conductivity than cold rolled steel can be effectively used to control annulus dimensions and promote better wetting. These include glass, stainless steel, and nitralloy.
- 10. The technique for rolling fusible seals was reduced to a standard routine practice.
- 11. Centering by conical surfaced keys provided a means for selfalignment.

2. Conical Centering

The method of centering the slug in the mold derives its name from the fact that conical surfaces are formed on aluminum keys and fit into like tapered holes in the bottom of the mold and in the split locating ring at the top, illustration of which is shown in Figures 11, 12, 17 and 18. The slots in the slug are at right angle to one another so that the assembly assumes the most stable arrangement possible.

The keys are originally blanked out with pyrimidal sides. The taper of the sides is 150, the same as the taper of the conical hole. The forming die shown in Figure 15, squeezed the excess metal on the blanked key, upsetting and forming at once. This operation was carried out by using the compression testor and applying about 3000 pounds. After these operations the keys are held fast in place such that severe jarring and dropping do not dislodge them*. The slug guide sleeve shown in Figure 15 is about 10 mils larger on a diameter than the slug, so that alignment of the key in the slug slot is not yet absolutely concentric with the slug circumference. This could easily be done by clamping the bottom of the slug in a fixed position relative to the die head each time and thereby form the key on center.

3. Coating Procedure

The procedure for assembly and loading for a typical run was as follows. A replaceable inlet tip was screwed onto the bottom of the inlet section of the mold and vacuum was held by seating against an annealed Al gasket. The mold was then set in place on the vacuum bench and the shot blast slug, previously fitted with die-formed, conical surfaced, Al centering keys, was lowered against a neoprene gasket and tightened in place by three C clamps. The vacuum inlet was opened to the line and the metal stop valve was adjusted through the Cowee seal to permit rapid evacuation of the mold. Vacuum was read on a Pirani gage. The assembly is shown in Figure 13.

The time and extent of evacuation was dependent on the effectiveness of the Cowee seal. Normally, evacuation to 20-50 microns was made in several minutes. For best results with Cowee seals it was found that the thickness of the rubber gasket should be approximately equal to \$\frac{1}{2}\$ the diameter of the gasket minus the shaft hole.

Initial tests were made without using any sleeves so that an annulus of about 125 mils was obtained. With bath temperatures about 810°C, the mold and reservoir always filled to yield a casting similar to that shown in Figure 15. Chiseling of the coats showed the slug to be off-centered in the casting, and the responsibility for this occurrence was definitely placed on melting out of the bottom key thereby allowing the slug to drop out of the locating ring and orient itself in random fashion. To overcome this difficulty, a steel tapered key was used to center the slug at the bottom, and under these new conditions, the slug was centered within the limits of the original warp of the slug before being coated. A split nitralloy "G" sleeve was interposed between the mold and the slug allowing a split nitralloy "G" sleeve was interposed between the mold and the slug allowing a split nitralloy. The slugs used in these experiments had less than 10 mils warp and typical measurements of the coat were: top, 58-59; middle, 56-61; bottom, 56-62.

*In one instance a keyed slug was dropped on a concrete floor and landed on the corner edge of key without moving or displacing the key.

The nitralloy sleeves were exidized with dichromate-sulfuric acid solution and as such did not wet and were disengaged from the coated slug easily. More complete wetting was obtained with the narrower annuli. Whether that is due to the fact that more metal flows nearer the slug in an annulus that is narrow, or whether better centering is responsible for the more complete wetting has not been absolutely determined. The several examples of bonded slugs shown in the appended section appear under the microscope to have continuous bonding. The outward coat is seen visually to have a very bright spangled appearance.

The most recent experiments with casting done at 86800 have given rise to the unexplained coating on the bottom third of the slug surface. Illustration is seen in Figure 10. X-ray analysis has shown this material, which is coarse and granular appearing, to be TuAl3. As yet no explanation for this phenomenon is available.

4. Top Conical Centering

Several schemes for top centering only, thereby obviating offcentering due to melting out of the bottom key, have been tried and show development possibilities. Figure 19 shows the most recent development along this line. A 4-way key with a taper that is inverted with respect to the original tapered keys, is formed in place by a suitable split die and assembled in similarly inverted-tapered, split centering ring.

This assembly provides a stable arrangement because the 4 making surfaces do not allow any sidewise slip and the pressure of the top of locating ring against the key prevents it from being jarred out of position when impact is made against the slug by the rising metal.

With such an arrangement, composite billet casting of high melting metals and alloys should be more readily accomplished.

III. Summary of Results

The practicability of a process for coating W slugs with Al/Si by vacuum casting has been demonstrated. This method of coating slugs offers greater flexibility than other processes because:

- 1. Thickness of coat is easily varied and controlled by the convenient use of removable insert sleeves.
- 2. A single action is required to produce a coated slug (the qualification necessary here is that complete vetting of the top end of the slug may necessitate a closure action of some sort).
- 3. Centering plugs can be either of the conductor or vacuum insulator type.
- 4. Condition of the bath surface is unimportant since metal is drawn from beneath the bath surface.

The following observations were made with regard to the conditions affecting wettability of Tu by Al/Si with vacuum coating techniques:

- l. Shot blasting of tuballoy provides a satisfactory surface for securing very rapid reaction between the tuballoy and Al/Si.
- 2. Outgassing of the tuballoy and its container before casting is essential to sound casting and good wetting.
- 3. Preheating the slug is not a necessary prerequisite for securing metallurgical bond if sufficient reservoir space is allowed to insure enough washing action by the rising metal to break through any film that might have been retained on the slug surface.
- 4. The uniformity of wetting is a function of the uniformity and size of the annulus between the slug and the mold wall.

IV. Recommendations

Coating W-slugs by the methods and devices described can be accomplished on a production basis. Use of the technique is foreseen in straight vacuum casting of billets. The method should be readily applied to casting billets and composite billets of other metals and alloys with which the project is interested for future fabrication purposes.

V. References

A. Literature

MUC-FF-204

B. Secret Notebooks

D. L. Schwartz - #251-B, pp. 17, 42-44, 47, 51, 57, 58, 59, 60, 63, 64, 65-66, 67, 73, 75, 77 and 82.

- B. Sawyer #311-B, p. 8-12.
- L. Kurland #421-B, p. 15-95.

Table I - Glass Capsule Experiments

Experimental

1. Surface Preparation: 50:50 HN03

Heated to 220°C and purged with

purified argon (tank argon —> H2SO4

bubbler —> Cu turnings furnace

—> drierite, ascerite, P2O5).

Vacuum: 4 M*
Outgassing Temperature: 220°C approx.
Preheat Temperature: Not known.

- 2. Surface Preparation: 50:50 HN03
 Outgassing Temperature: 220°C.
 Vacuum: 4 M
 Purified argon was flushed through
 the capsule (tank argon —> H2SO4
 bubbler drierite, ascarite,
 F2O5—> Ca chip furnace—> tuballoy turnings furnace).
 Preheat Temperature: 575°C.
- 3. Surface Preparation: 50:50 HN03
 Slug was allowed to oxidize and
 form the multicolored surface
 layers.

Hydride Formation: A closed end Hg manometer was attached to the capsule to indicate hydrogen consumption and subsequent release on hydride decomposition. Initial hydrogen consumption began at 254°C. The temperature was increased to 274°C and maintained there until signs of reaction disappeared.

Decomposition: Decomposition was made at 460°C.

Vacuum: 5 M
Preheat Temperature: < 460°C.

Results

- A. General Observations:
 Slug turned teal blue when argon was admitted into capsule. On continued heating, the end of the slug nearest the vacuum system became canary yellow. After cooling in Vacuo, the slug was allowed to set in air and no color change occurred on standing.
- A. General Observations:
 The surface was still bright just prior to the argon admission.
 After several argon flushes and evacuations, one end of the slug had become light brown.
- B. Metallographic #666 Vacuum cast in Al/Si at 650°C. Bond of Al/Si to slug was poor. Some compound was formed at the bottom of the slug but none on the upper 2/3 of the 4" slug.
- A. General Observations:

 As hydrogen was consumed the colors disappeared and in their stead a dark brownish black powder dotted the surface. On decomposition surface became somewhat lighter.
- B. Metallographic #675
 Vacuum cast in Al/Si at 650°C. The Al/Si coat was easily peeled off of the slug leaving a dark surface with a few bright spots through which the compound layer had cracked. On some spots, Al/Si was retained and showed a spheroidized structure. The familiar Al/Si compound layers were found.

* Microns

(continued on following page)

Experimental.

4. Surface Preparation: Shot Blast
Hydride Formation: Purified H2
(H2SO, bubbler -> Pt-Asbestos
furnace -> drierite, ascarite,
P2O5--> Ca chip furnace --> tuballoy turnings furnace) was admitted when capsule was at 189°C.
Temperature was increased to and
maintained at 260°C.

Hydride Decomposition: The temperature was increased to 390 when the first signs of decomposition were noted. Final decomposition was made at 450°C.

Outgassing Temperature: 438°C. Vacuum: 5 M

 Surface Preparation: Sand Blast Outgassing Temperature: 450°C.
 Vacuum: 4 M
 Preheat Temperature: <450°C.

- 6. Surface Preparation: Same as #5
 Outgassing Temperature: 475°C.
 Vacuum: 5 M
 Preheat Temperature: 4475°C.
- 7. Surface Preparation: Shot blast and Ca metal was used as a getter to remove any retained gas and oxide left in the capsule.



Evacuation was made to 4 M and the capsule was sealed off. The Ca was vaporized at 554°C. After a while the Ca was condensed away from the slug.

Preheat Temporature: <554°C

(continued on following

Results

- A. General Observations:
 As hydrogen was consumed, a greyishblack powdery material formed on the
 previously shiny, smooth tuballoy.
 On decomposition of the hydride,
 the surface color became lighter,
 but did not become either as smooth
 or bright as originally.
- B. Metallographic #674
 On vacuum casting in Al/Si at 650°C, the lower section of the bulb broke under the surface of the molten Al/Si and the slug fell to the bottom of the bath. When removed from the bath, sections of the Al/Si peeled off (some actually popped off) leaving bare spots that became a dull red color.

 A single compound was formed and this layer was 1/32" thick.
- A. General Observations:
 Surface remained visibly unchanged.
- B. Metallographic #677

 Vacuum cast in Al/Si at 650°C. The coating was peeled off and the slug surfaces were bright. Compound formation was found all over the slug with slight discontinuities at the top.
- A. General Observations: Same as #5
- B. Metallographic #680

 Bonding was bad only on bottom half of slug. Two compound layers formed.
- A. General Observations:
 After contact with the Ca vapor, the surfaces still appeared uniformly bright with a very faint blue tinge (not to be confused with the teal blue previously mentioned in #1 and #2).
- B. Metallographic #685
 Vacuum cast in Al/Si at 650°C. A
 new sort of compound at the Tu-Al/Si
 interface was seen. In other places
 the two familiar Al/Si compounds
 were recognized and near the top of
 ... the flug were unbonded areas.

702-013

Experimental

8. Surface Preparation: Shot blast and inserted onto vacuum line and sealed off under a flow of dry argon.

Outgassing Temperature: Luminous gas flame.

Vacuum: 5 M Preheat Temperature: 386°C.

Results

- A. General Observations:
 The slug surfaces remained undiminished in brightness throughout the operations. The technique used here appeared to be the most sound approach yet taken to the problem of maintaining a clean surface.
- B. Metallographic #694
 Vacuum cast in Al/Si at 70000. The
 metal filled the capsule and remained
 molten even at the top of the ampule
 for several seconds.
 A continuous bond was seen over all
 the slug surfaces. The usual compound layers were found to be somewhat thicker, being at the thinnest,
 2,7 mil.

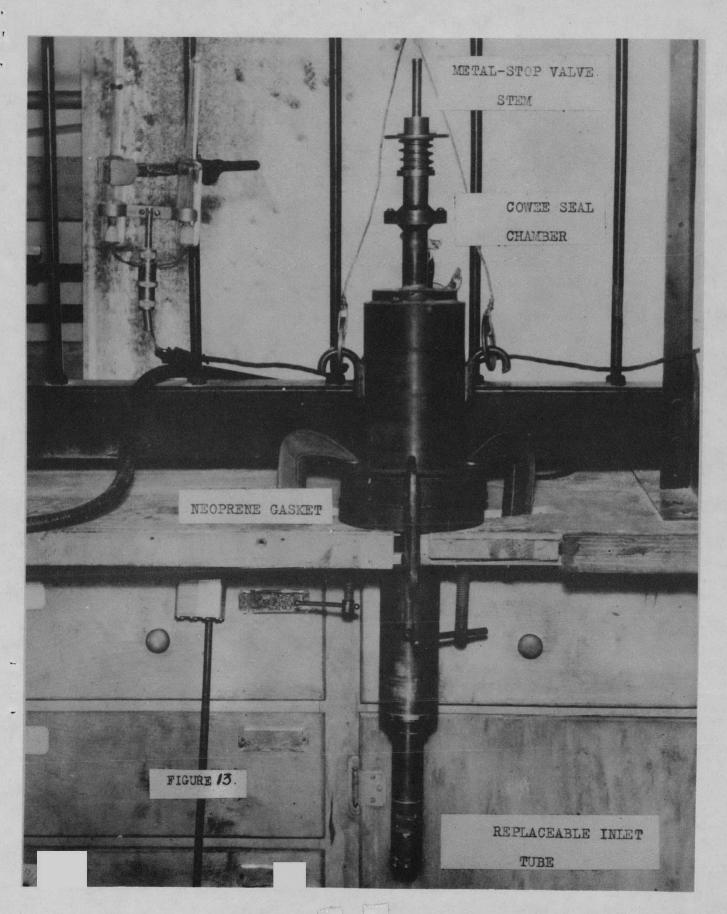
<u>Table II</u>
Warpage of Tuballoy Slugs, Annealed in Kinetic Vacuum (MUC-FF-204)

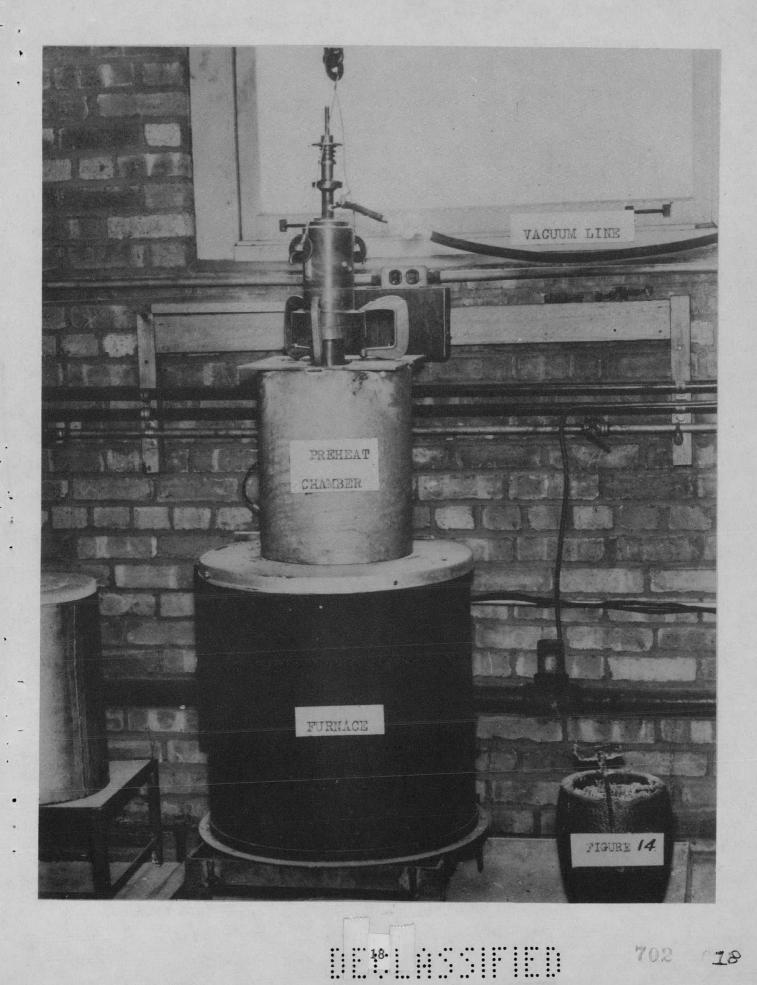
Slug #	Temp. oc	Right End	Center	Left End	•
1	602	。0000	0000	.0000	Baforo
		。 0010	.0070	0020	After
2	·æ	.0000	.0000	.0000	Before
	63	.0000	.0030	.0015	After
3	842	.0000	.0000	。0000	Before
,	<u>.</u>	.0040	。0060	.0040	Alter
4	co	.0000	。0005	.0000	Before
•	, e	.0020	.0090	.0050	After
9	950	.0000	.0000	.0000	Before
	œ	.0100	。0120	,0 06 0	After
10	ess	.0000	。0000	。0000	Before
	cs	.0050	.0090	.0040	After
11	700	.0000	.0000	.0000	Before
	.	。0010	.0025	.0010	After
12	, 623	。0000	.0000	.0000	Befor
	ga	0010	.0025	.0010	After

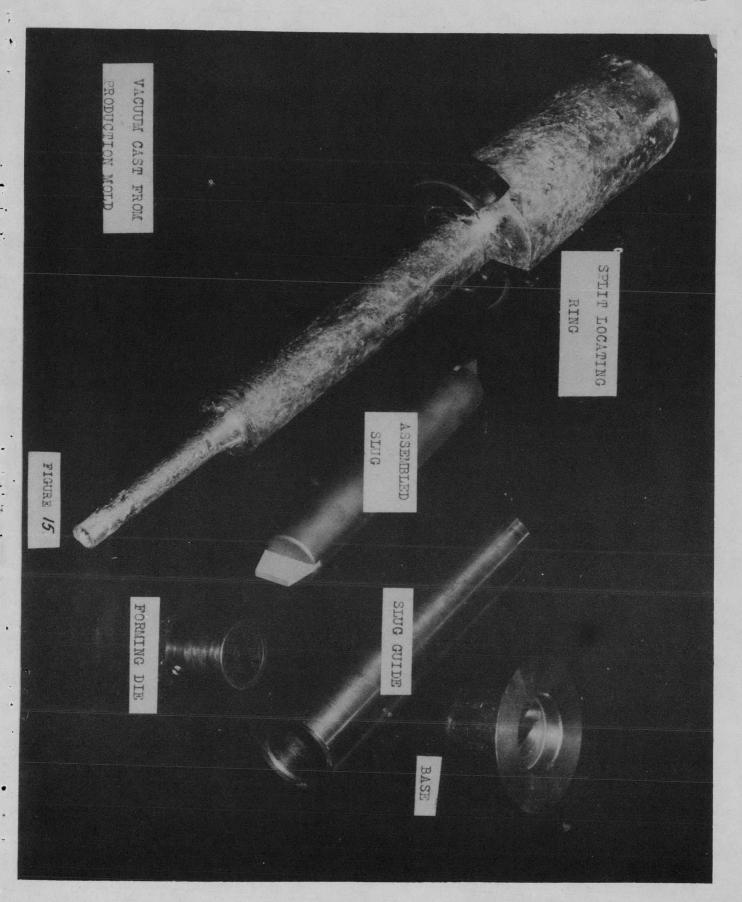
FIG. I EXPERIMENTAL VACUUM CASTING MOLD

60

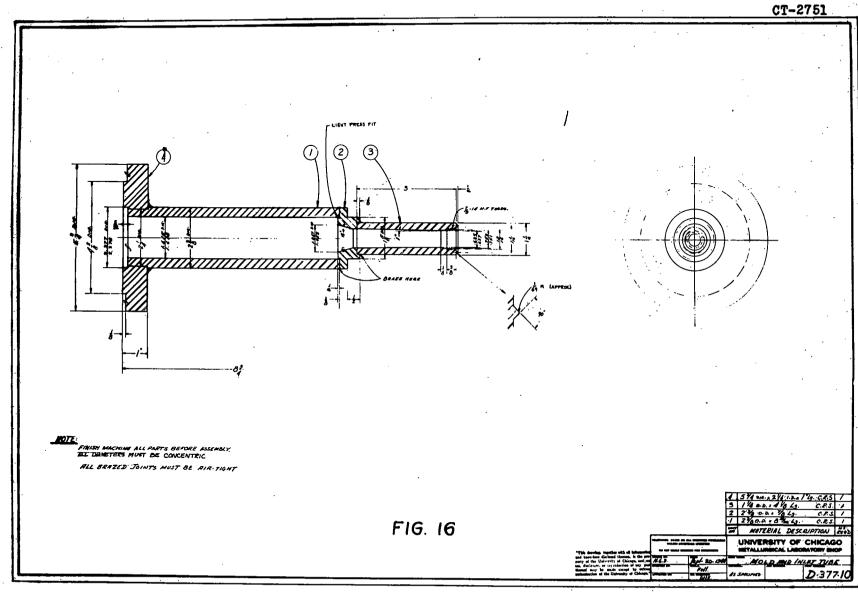
USIBLE SEAL







702-019

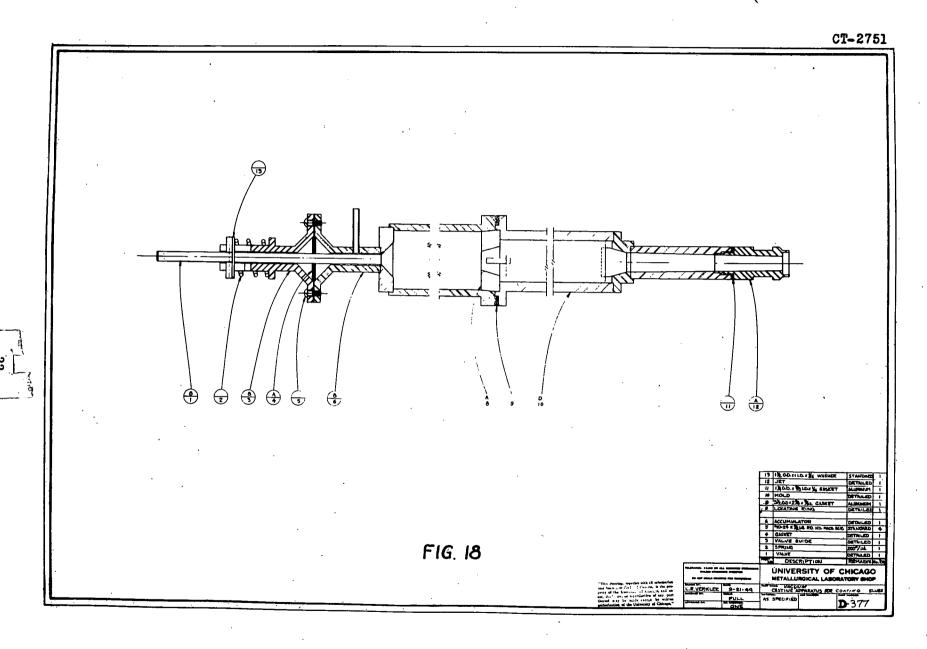


.L 4 40 "

2 C)

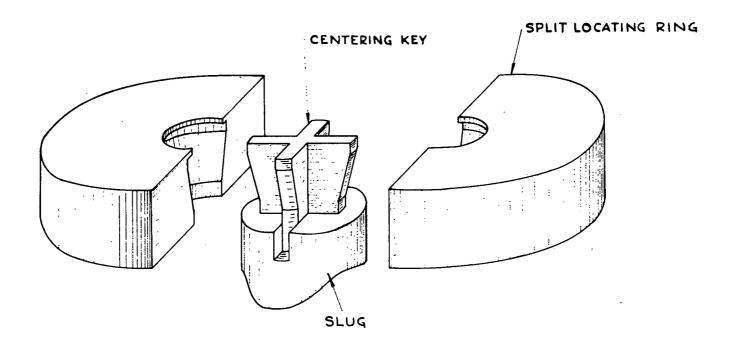
:::::

•;



こい

FIG. 19



TOP SINGLE KEY CENTERING

300

りとび

PLATE NO I

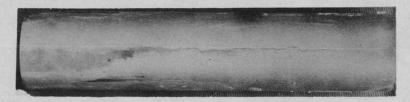


FIGURE 2. FIRST W SLUG COATED USING E.C.M. MOLD-RESERVOIR.
COAT HAS BEEN CHISELED OFF EXPOSING COLFOUND
FORLATION AND SHOWING CRACKS IN SLUG AND AREAS
WHERE WETTING DID NOT TAKE PLACE.

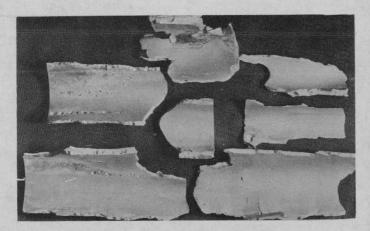


FIGURE 3. COAT OF SLUG SHOWN ABOVE. BLOW HOLES ARE SEEN TO BE ADJACENT TO CRACKS IN SLUG.

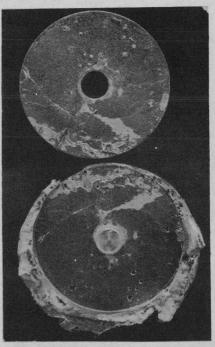


FIGURE 4 TOP PICTURE SHOWS DEPTH OF CRACK
ON BOTTOM END OF SLUG. BOTTOM
PICTURE SHOWS CONDITION OF CAST
MATERIAL ON THE BOTTOM SLUG END.

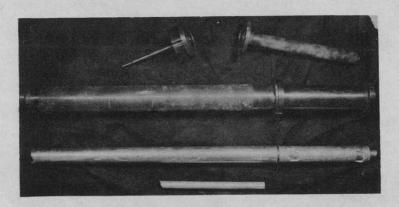


FIGURE 5. MOLD-RESERVOIR PARTS AND FIRST CASTING. EMBEDDED TOP SHIDER IS SEEN IN CASTING.

PLATE NO II

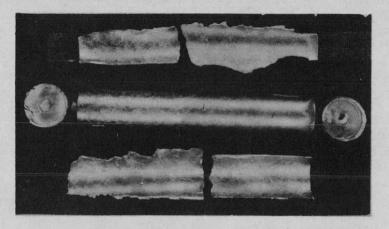


FIGURE 6. W SLUG COATED IN E.C.M. AND USING A STAINLESS STEEL SLEEVE INSERT. BOTTOM OF SLUG IS LEFT SIDE. NOTE SPANGLED APPEARANCE OF COMPOUND LAYER.

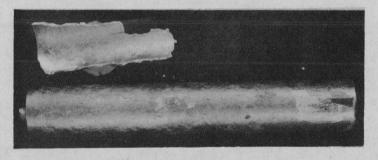


FIGURE 7 SAME SLUG AS SHOWN AROVE, BUT ROTATED 180°. SLIGHT DISCONTIN-UITIES FOLLOW HOLES IN CAST STRUCTURE. ABRASION AT TOP OCCURRED WHILE DISENGAGING THE SLEEVE INSERT.



FIGURE $\mathcal B$. TYPICAL COATED SLUG FROM A GLASS LINED MOLD, ILLUSTRATING GLASS ADHERENCE TO THE A1/Si.

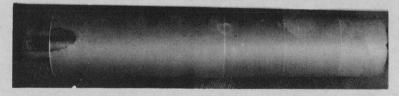


FIGURE 9. COAT HAS BEEN MACHINED DOWN IN STEPS. COMPOUND IS EXPOSED ON RIGHT. WARP OF THE SLUG AS A RESULT OF OUTGASSING AND OFF-CENTERING IN THE MOLD CAUSED THE TO BE EXPOSED ON THE LEFT. A FEW SMALL HOLES ARE SEEN ON THE CAST MATERIAL. SUBSEQUENT REMOVAL OF THE COAT SHOWED THE SLUG TO BE COMPLETELY WETTED EXCEPT ON THE TOP END FACE.

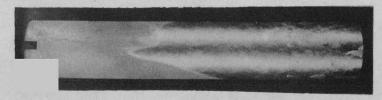


FIGURE AZ SLUG COATED IN P.C.M. WITH A SPLIT HITRALLOY "G" SLEEVE.
BOTTOM IS LEFT SIDE. COARSE LOOKING MATERIAL ON BOTTOM THIRD
HAS BEEN AMALYZED BY K-RAY AND IS UNDICATED TO BE TWATY.

702-025

PLATE NO III STRUCTURE COMPARISON AI/SI COATS



A. VACUUM CAST

BOTTOM SHOWS PART OF COMPOUND LAYER THAT REMAINED WITH THE CAST MATERIAL ON BEING REMOVED FROM THE SLUG. THE STRUCTURE IS SOUND AND HAS LITTLE PORCSITY.



B. PRESSURE DIE CAST---BONDED

RATHER EXTENSIVE POROSITY IN THE Al/Si IS SEEN.



C. PRESSURE DIE CAST -- UNBONDED

PRONOUNCED POROSITY IN THE CAST MATERIAL CLOSEST TO SLUG SURFACE.

702-026

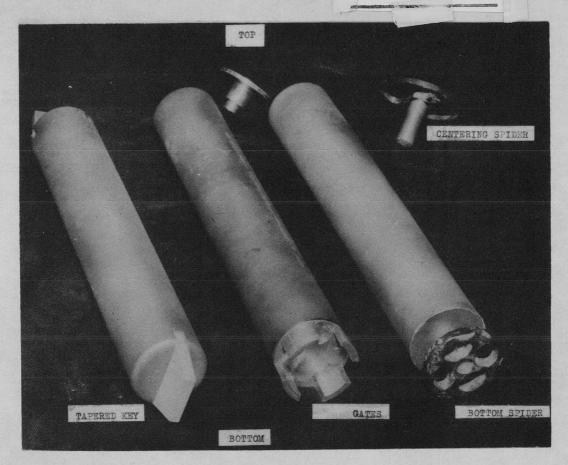


FIGURE //. PARTIAL SEQUENCE OF CENTERING DEVICE DEVELOPMENT. LATEST DEVELOPMENT IS SHOWN AT LEFT.

