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ANL-5673

Metallurgy and Ceramics

M-3679 (20th Ed., Rev. 1)

This document consists of 37 pages.

No. 177 of 216 copies. Series A.

ARGONNE NATIONAL LABORATORY

P. O. Box 299

Lemont, Illinois

A COATED CAST IRON CRUCIBLE FOR USE WITH EUTECTIC
Al-Si ALLOY IN THE TEMPERATURE RANGE 595° - 650° C

by

F. L. Yaggee

Metallurgy Division

Program 7.7.4

Portions of the material in this report have appeared
in the following Metallurgy Division Quarterly Reports:

ANL-4576	(126)	December 1950
ANL-4618	(103-105)	March 1951
ANL-4664	(112-113)	June 1951
ANL-4705	(113)	September 1951
ANL-4755	(116-117)	December 1951
ANL-4804	(114-116)	March 1952
ANL-4860	(11)	June 1952

November 1957

Operated by The University of Chicago
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Contract W-31-109-eng-38

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JTB, date 3-12-60

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A COATED CAST IRON CRUCIBLE FOR USE WITH EUTECTIC Al-Si ALLOY IN THE TEMPERATURE RANGE 595° - 650°C

by

F. L. Yaggee

ABSTRACT

The feasibility of the coated metal crucible as a container for eutectic Al-Si alloy has been proven by test. Small, enamel-coated cast iron pots have successfully withstood the chemically aggressive Al-Si alloy and the adverse influence of an oxidizing atmosphere for a period of 3 months at 725°C. A similarly coated cast iron crucible containing 450 pounds of eutectic Al-Si alloy was successfully tested for 144 days in a jacketing operation conducted at 595°-650°C. Under the same conditions, the normal service life of clay-bonded graphite and silicon carbide crucibles rarely exceeds 45 days.

The coating material is a commercially available enamel capable of withstanding temperatures up to 790°C (1450°F). It is readily applied to the surface of a variety of ferrous metals and alloys; however, best results are obtained with alloys low in chromium and nickel which also have a low thermal expansion coefficient.

INTRODUCTION

The investigation into the feasibility of the coated metal crucible, described herein, was conducted during the period November 1951-July 1952. Its undertaking was a natural adjunct to an existing program of development relating to a jacketing process for the production of metallurgically bonded reactor fuel slugs. The motivating forces were the restricting effect of a short ceramic crucible life on the unit output of the jacketing process and the adverse effect of a constantly decreasing crucible conductivity on process efficiency and product quality.

The popularity of ceramic crucibles in molten metal applications is based primarily upon a single characteristic of this class of materials, namely, an inherent ability to resist chemical attack by a variety of molten metals and alloys. The mediocre thermal conductivity is usually compensated by an increased thermal input and preserved by operation within the reducing atmosphere of a gas-fired furnace. Rapid deterioration in an

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oxidizing environment is a natural consequence for these materials. Their characteristically low initial conductivity is further jeopardized by the depletion of their carbon content, occurring either in the combined form or as free graphite.

This type of crucible deterioration was observed in clay-bonded graphite and silicon carbide crucibles at temperatures in excess of 600°C. At the time, the clay-bonded graphite crucible was being used to contain molten lead and molten eutectic Al-Si alloy, the former under a covering layer of Al-Si alloy to minimize oxidation of the lead surface. Use of the silicon carbide crucible, on the other hand, was restricted to eutectic Al-Si alloy, because of the incompatibility of this material with molten lead as exemplified in Figure 1. The entire jacketing operation was conducted within the temperature range 595°-650°C in the course of which both crucibles were exposed to the oxidative character of the natural atmosphere.

Under normal operating conditions, using an industrial-type resistance-wound furnace (Hevi-Duty type HD-2430), the clay-bonded graphite crucible was found to have a useful service life ranging between 25 and 30 days. Furthermore, the observed longevity was found to be completely independent of the molten metal being contained and solely a function of the operating temperature: more precisely, the temperature in the immediate vicinity of the furnace windings. The first perceptible decrease in thermal conductivity occurred, with regularity, after three or four days of continuous operation. Thereafter, the conductivity continued to drop at an estimated uniform rate necessitating periodic advances in the controller setting to maintain the desired bath temperature. After three weeks of continuous operation, the degree of carbon depletion was such as to make the already difficult task of temperature control a virtual impossibility. The hairline surface cracks which appeared during the fourth week of service proved to be fairly accurate signs of impending failure. In the few instances where the warning was ignored, failure occurred within a period of 5 to 7 days after the first cracks were observed. The consequence of such failure was the complete penetration of the crucible wall by the molten metal bath.

The thermal conductivity of the silicon carbide crucible was less drastically affected by the oxidizing environment, accounting for the somewhat longer service life of 35 to 45 days. In spite of this fact, the terminal point in the service life of this crucible was similarly preceded by the appearance of surface cracks.

An example of the surface cracking which has been seen in both types of ceramic crucibles as a result of extended exposure to an oxidizing environment is illustrated in Figure 2.

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The nature and completeness of the carbon depletion suffered by the clay-bonded graphite crucible is shown in Figure 3. This crucible lost 48 pounds during four weeks of service, representing 40% of its original weight. Within the same period, the crucible conductivity is believed to have been decreased by an estimated 86%: from an estimated initial conductivity of $0.019 \text{ cal-cm/cm}^2\text{-sec-}^\circ\text{C}$ ($56 \text{ BTU-in/ft}^2\text{-hr-}^\circ\text{F}$) to a final conductivity of $0.003 \text{ cal-cm/cm}^2\text{-sec-}^\circ\text{C}$ ($8.1 \text{ BTU-in/ft}^2\text{-hr-}^\circ\text{F}$) when the graphite has been burned out.⁽¹⁾

The initial conductivity of the silicon carbide crucible is reported to be in the vicinity of $0.034 \text{ cal-cm/cm}^2\text{-sec-}^\circ\text{C}$ ($100 \text{ BTU-in/ft}^2\text{-hr-}^\circ\text{F}$)⁽¹⁾ or approximately twice that of the clay-bonded graphite crucible. Information regarding the decrease in conductivity of silicon carbide upon prolonged exposure to an oxidizing environment has not been volunteered by the manufacturer, and no attempt was made to obtain such information in the course of this investigation. Since a less drastic decrease in conductivity was actually experienced with silicon carbide, and since the conductivity and weight appear to be intimately interrelated properties, it can reasonably be assumed that the weight loss suffered by the silicon carbide crucible was not nearly so great as that experienced with clay-bonded graphite.

PRELIMINARY TESTS

Testing Set-Up

The relative merits of the coated metal crucible were first checked in an engineering test set-up such as that shown in Figure 4. It was the intention that this preliminary test set-up should serve as a scaled-down facsimile of the full size equipment, duplicating, as nearly as possible, the conditions which exist in the jacketing process itself. Toward this end, an ordinary resistance-wound furnace was used as the source of the heat applied to the coated metal pot and its contents. Power to the furnace was controlled by an "on-off" type controlling instrument, as demanded by the controlling thermocouple located near the bottom of the coated metal pot and external to it. The testing temperature was continuously recorded with a second thermocouple which was immersed in the molten bath, within a similarly coated metal tube, the latter extending within one-half inch of the bottom of the coated metal pot.

Crucible Materials Tested

Cast metal containers (pots) of two alloy types were selected for the initial tests. The two alloy compositions were: (1) a 1% chromium cast iron and (2) a 28-32% chromium stainless steel falling within the ferritic stainless steel composition range. This choice of alloys was not a totally arbitrary one, but one based upon information regarding the

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commercial usage of these materials in molten metal and molten salt applications. Cast iron, for example, is the most resistant of the common structural metals and alloys to attack by molten aluminum and its alloys.⁽²⁾ In addition, it possesses a relatively low coefficient of thermal expansion. On the other hand, high chromium stainless steels enjoy wide usage in the containment of molten lead and the exceedingly corrosive mixed chloride salts at temperatures up to 850°C.

The physical dimensions and chemical compositions of the small alloy pots are given in Table I, as are the names of the vendor supplying each. The coated metal pots and the coated metal thermocouple tubes are shown in Figures 5 and 7 as they appear prior to testing. All items were commercially enamel coated by the Charles H. Milles Foundry Company of Chicago, Illinois.

The enamel-coated cast iron pots were tested as possible containers for three different molten metal baths, namely: (1) eutectic Al-Si alloy, (2) molten lead under eutectic Al-Si, and (3) molten bronze (composition 49-45 w/o Cu - 51-55 w/o Sn) under a protective layer of a mixed chloride salt. Two duplicate tests were conducted in enamel-coated high chromium alloy pots containing the above-mentioned eutectic Al-Si alloy and the molten bronze, respectively. The bronze bath was included in these tests because of its interest in an alternate jacketing process.

The container-thermocouple tube combinations tested are listed below.

	Molten Bath		
	Eutectic Al-Si	Lead under Eutectic Al-Si	Bronze
T.C. Tube	Cast Iron	446 S.S.	Cast Iron
Container	Cast Iron	Cast Iron	Cast Iron
T.C. Tube	Cast Iron	No Test	None
Container	Michiana Alloy 55		Michiana Alloy 55

Test Conditions

The test conditions used in these small-scale preliminary tests were purposely made more severe than those likely to be encountered in the jacketing process itself. This course was made necessary by the

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relatively high boron content in the enamel coating material, a reported 0.5 w/o. The presence of this amount of boron in the coating material was viewed with some concern because of the possibility of boron contamination in the jacketed fuel slug assemblies.

The testing temperature was arbitrarily fixed at 700°-725°C. This represents an estimated 120°C increase over the normal operating temperature of the jacketing process, and is 75°C in excess of the maximum idling temperature for the eutectic Al-Si bath when not in use. At the higher temperature, any latent tendency toward boron removal from the coating material would be magnified for easy detection by the increased aggressive character and small volume of the molten metal bath. The ratio $\left(\frac{\text{enamel contact area}}{\text{molten metal volume}} \right)$, which is essentially an expression of the probability of boron contamination, was increased ten-fold over the value expected in the full size crucible: 2.85:1 as compared to 0.25:1.

The eutectic Al-Si bath was sampled at regular intervals and spectrochemically analyzed for boron. Virgin Al-Si alloy was used as a standard for comparison.

The testing variables are summarized in Table II. Spectrochemical results for boron are given in Table III. These results were obtained from three Al-Si bath samples taken after 20, 40 and 54 days of testing.

RESULTS OF PRELIMINARY TESTS AT 700°-725°C

A. Coated Cast Iron with Eutectic Al-Si

The condition of an enamel-coated cast iron pot upon exposure to eutectic Al-Si at 700°-725°C for 54 days is shown in Figure 6. Section A is taken at the Al-Si liquid level where chemical attack would be expected to be most severe. Section B is taken at a point 1" beneath the Al-Si liquid level. Neither section shows any evidence of serious chemical attack upon the underlying cast iron.

The coated metal thermocouple tube used in this test is shown in Figure 7 as it appeared before and after testing. Transverse sections of this tube taken at and above the Al-Si liquid level are shown in Figure 8. There is some evidence of incipient attack occurring at the Al-Si liquid level, but it is not serious enough to have warranted the replacement of this thermocouple tube. That attack which is in evidence (Figure 8, Section A) is localized, suggesting that the protective coating may have been damaged, through physical abuse, prior to the test.

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B. Coated Cast Iron and Type 446 Stainless Steel with Lead Under Eutectic Al-Si

Two transverse sections of an enamel-coated cast iron pot containing lead under a 1" thick layer of eutectic Al-Si alloy at 700°-725°C are shown in Figure 9. The duration of this test was 60 days. Section A, taken at the lead/Al-Si interface, shows some evidence of attack, whereas section B, taken at a point beneath the lead level, is completely devoid of similar evidence. The extent of the localized attack, which is in evidence in Figure 10, is limited to a portion of the container above the lead/Al-Si interface.

There is reason to believe that the observed attack is the ultimate result of an initial interaction, at the lead/Al-Si interface, between the coating material and lead oxide, resulting in localized exposure of the underlying metal to the aggressive action of the Al-Si covering layer. Subsequent attack begins at the lead/Al-Si interface and continues upward into the Al-Si layer. The proposed interaction between the coating material and lead oxide was first observed when such a pot was put to use as a heat-treating bath containing unprotected lead at 600°C. After a few days the lead surface was covered with a soft, "taffy-like" substance which, when water quenched, took on a dark green color and the brittle characteristics of glass. An X-ray diffraction pattern obtained from this material consisted of extremely diffuse lines, reminiscent of a super-cooled liquid (glassy substance). This pattern was not otherwise capable of strict interpretation.

It is possible that the proximity of the lead/Al-Si interface to the exposed Al-Si surface was primarily responsible for the attack that occurred, and that a thicker Al-Si covering layer (much thicker than 1 inch) may have prevented such attack by more effective exclusion of atmospheric oxygen from this interface. This possibility is supported by the observed fact that a similarly coated cast iron thermocouple tube survived a three-month exposure to such a bath at 595°-650°C. At the conclusion of this period, the thermocouple tube was without evidence of attack at the lead/Al-Si interface and at the Al-Si surface. This thermocouple tube is shown in Figures 19 and 20. The Al-Si covering layer was approximately 10" deep, and the lower end of the thermocouple tube extended approximately 2" below the lead/Al-Si interface.

The coated 446 stainless steel thermocouple tube used in this test was completely penetrated by the eutectic Al-Si alloy in less than four weeks. The damaged tube is shown in Figure 11. It is particularly significant that the attack occurred at the Al-Si surface and that the coating appears to have melted in spots above the Al-Si level. The greenish-yellow deposit found in each area of melting was identified, by X-ray diffraction, as a sodium chromate-sodium chromate tetrahydrate mixture ($\text{Na}_2\text{CrO}_4\text{-Na}_2\text{CrO}_4\cdot 4\text{H}_2\text{O}$).

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The same phenomenon was not observed with the coated cast iron pot, suggesting that this interaction with the coating material is peculiar to the high chromium alloys (chromium contents of 23-32%), and occurs in the temperature range 700°-725°C. Specifically, the reaction is one occurring between the sodium in the coating material and the chromium in the alloy container, resulting in a lowering of the melting point of the enamel coating and subsequent exposure of the underlying metal. Severe intergranular attack results when exposed materials, especially Type 304 and Type 446 stainless steel, are contacted by molten Al-Si alloy. The nature and severity of the chemical attack suffered by unprotected stainless steels upon exposure to Al-Si alloy is clearly seen in Figures 12 and 13.

C. Coated Cast Iron with Tin Bronze

Neither the enamel coating nor the cast iron was able to withstand the corrosive nature of the mixed chloride flux that was used as a protecting layer over the surface of the molten bronze. This test was concluded in 11 days, with the results shown in Figure 14. The same test, when checked after 9 days at temperature and at the conclusion of the working week, showed no signs of impending failure other than the deterioration of the enamel coating on both the container and the thermocouple tube, above the flux level. The condition shown in Figure 14 was observed on the morning of the 12th day of the test: the bronze bath had completely drained from the container.

Although this test has demonstrated the incompatibility of the enamel coating material and casting material and cast iron with the mixed chloride salts, it has yielded no definite information regarding the feasibility of containing tin bronze in such a coated metal container in the absence of the corrosive salts. It is quite possible that the coated cast iron could effectively contain the tin bronze (composition 49-45 w/o Cu-51-55 w/o Sn) at these temperatures if the integrity of the coating is not adversely affected. This point of view is, in some measure, supported by Figure 16, which shows the enamel coating to be intact. On the other hand, it is less likely that the same bronze bath could be successfully contained in unprotected cast iron.

D. Coated Michiana Alloy 55 (28-32 w/o Cr) with Eutectic Al-Si

This test was terminated after 12 days upon evidence of localized attack being initiated at the liquid Al-Si surface. The enamel coating appeared to have melted in localized areas, exposing the underlying metal in much the same manner as shown in Figure 11. These areas of enamel melting occurred at and above the liquid Al-Si level, as well as over the entire outside surface of the coated container. The high chromium content is considered detrimental to the integrity of the enamel coating.

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Unprotected, Michiana Alloy 55 can be expected to exhibit the same susceptibility to intergranular attack by eutectic Al-Si as that shown by the 446 and 304 stainless steels in Figures 11, 12 and 13, respectively.

E. Coated Michiana Alloy 55 (28-32 w/o Cr) with Tin Bronze

The adverse effect of a chloride environment upon the protective enamel coating is again illustrated in Figure 15. This test was concluded after 9 days when the furnace windings failed as the result of corrosive attack by the chloride vapors; the 1" thick chloride flux layer used to prevent excessive surface oxidation of the bronze bath is gone.

Transverse sections of this alloy container taken at the bronze/flux interface and well below this interface are shown in Figure 16. The enamel coating is intact in both sections, and there is no evidence of attack of the underlying alloy by the bronze bath.

FULL-SCALE TEST

Testing Set-Up

The decision to invest in and test a full-size coated metal crucible was based upon the favorable results obtained in the preliminary tests. The actual testing of such a crucible was conducted under the normal operating conditions of the fuel jacketing process. Except for a difference in the physical size of the crucible and furnace, the full-scale set-up is accurately represented in Figure 4.

The enamel-coated crucible and its contents were heated in a 50-kw Hevi-Duty electric furnace (Hevi-Duty type HD-2430). Electric power to the furnace was again controlled with an "on-off" type of controller-recorder. The controlling thermocouple was immersed in the molten bath to a depth of 12" and protected within a coated metal tube. Bath temperature readings were taken at frequent intervals with a second thermocouple and portable potentiometer. A thin-wall, Type 304 stainless steel tube was used to protect the second thermocouple during the brief periods of immersion.

Crucible Description

The large enamel-coated cast iron crucible was cast and coated especially for ANL by the Charles H. Milles Foundry Company of Chicago, Illinois. It was 20 $\frac{1}{4}$ " in diameter (O.D.) and 24" deep: the wall thickness was 1 $\frac{1}{4}$ " at the top and increased to approximately 2" at the base, where the sides tapered inward. The empty crucible weighed 505 pounds, approximately four times the weight of a corresponding ceramic crucible

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(clay-bonded graphite); the rated capacity was equivalent to 450 pounds of molten aluminum. Because of the combined weight of the crucible and its contents, the usual ceramic base block was replaced by one of cast iron.

Test Conditions

The test conditions are summarized in Table II. As mentioned earlier, the testing temperature of 595°-650°C is 75°C below the testing temperature used in the preliminary tests. Likewise, the calculated value for the ratio $\left(\frac{\text{enamel contact area}}{\text{molten metal volume}}\right)$ is approximately one-tenth as large.

The Al-Si alloy bath was sampled at three-day intervals and spectrochemically analyzed for boron and iron. Each bath sample analysis was accompanied by a simultaneous analysis of virgin Al-Si as a standard for comparison. The results of these analyses are given in Table III for regular intervals of approximately 10 days each.

RESULTS OF FULL-SCALE TEST AT 605°-650°C

Coated Cast Iron with Eutectic Al-Si

The physical appearance of the full-size enamel-coated cast iron crucible after 144 days of continuous service can be seen in Figure 17 and 18. This test was terminated involuntarily when trouble developed in a Micromax controller, allowing the Al-Si bath temperature to reach 1120°C during an otherwise normal week-end idling period at 650°C. The consequences of this overheating are clearly seen in the above-mentioned photographs, namely, collapse of the cast iron base block (cast iron m.p. 1260°C), and total melting of the protective enamel coating. The maximum temperature attained by the molten bath was approximately 310°C above the recommended operating temperature for the enamel coating used.

An inspection of the inside of this crucible after the overheating incident revealed that approximately 2/3 of the area in contact with the molten Al-Si was chemically attacked. Penetration was mostly limited to a depth of less than 1/16". The most severe penetration occurred in two localized areas at the Al-Si liquid level and was no more than 1/8" deep. Almost all of the observed chemical attack is believed to have occurred during the 28 hours that the underlying cast iron lay exposed to the chemically aggressive Al-Si at 1120°C.

BATH CONTAMINATION

The analytical results obtained from the Al-Si bath samples taken during the course of the preliminary and large scale testing are tabulated in Table III.

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The reported values for boron (in parts per million) in the small-scale test show essentially no boron buildup in the bath after 54 days at temperature. A similar condition was found to prevail in the large scale test. The constancy of the difference in the reported values for the successive samples and standards in both tests is considered sufficient proof that eutectic Al-Si does not remove boron from the enamel coating at temperatures up to 725°C.

Table III also shows there was some buildup in the iron content of the Al-Si bath in the large scale test, reaching a reported 1.0 w/o after 51 days of continuous bath operation. The iron buildup stops at this point and shows essentially no increase for the last 92 days (3 months) of the test. This iron contamination is considered to be, in part, the result of chemical attack upon certain unprotected steel fixtures used in the jacketing operation and, in part, of the attack suffered by a coated thermocouple tube and a coated carbon steel scraping fixture, the latter being used in a completely submerged position. Both items displayed localized attack at spots where it appeared as though the enamel coating had been damaged by physical abuse. The replacement of these items with new fixtures of like kind coincided with the observed "leveling-off" of the reported iron contamination.

Although the Al-Si bath was not sampled after the overheating incident, the contribution to the contamination by the resulting attack upon the crucible can be estimated by calculation.

Using the following data

Area of Observed Attack:	$1175 \times \frac{2}{3} = 784$ sq. in.
Average Penetration:	0.0625 in.
Cast Iron Density (ρ):	0.258 lb per cu. in.
Wt. of Al-Si in bath:	450 lb

The weight of cast iron dissolved by the Al-Si bath as a result of the chemical attack observed at the conclusion of the test is

$$784 \times 0.0625 \times 0.258 = 12.54 \text{ lb}$$

If it is assumed that this amount of iron is homogeneously distributed throughout the Al-Si bath, the resulting iron contamination, in per cent, will be

$$\frac{12.54}{450} \times 100 = 2.79 \text{ w/o}$$

The maximum iron contamination reported after 144 days was only 1.10 w/o, or less than half of the calculated 2.79 w/o. Therefore, it is reasonable to assume that the chemical attack observed upon termination of the test

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occurred during the estimated 28 hours after the overheating incident and not during the 144 days prior to it. The probable final iron content of this Al-Si bath can be estimated by adding the calculated value of contamination due to the overheating incident to the analytical results reported just prior to the termination of the test, namely:

$$1.10 + 2.79 = 3.89 \text{ w/o Fe.}$$

Since the Al-Si bath was not sampled for analysis immediately after the overheating incident, the above estimate of the probable final iron content was not verified.

BATH TEMPERATURE GRADIENTS

In the course of the large-scale test of the coated cast iron crucible, a comparison was also made of the temperature gradients occurring in the Al-Si bath in this crucible and in a silicon carbide crucible of identical size, shape and capacity. The two crucibles were put into service on the same day: each was loaded with approximately 450 pounds of eutectic Al-Si alloy and allowed $1\frac{1}{2}$ days to reach thermal equilibrium. Controller settings of 630°C and 660°C were used, the higher setting representing an attempt to compensate for the lower thermal conductivity of silicon carbide.

The temperature-measuring apparatus consisted of an arrangement of five, pre-calibrated thermocouples - each in its own thin-wall stainless steel tube - spaced at equal intervals across the diameter of each molten Al-Si bath. The individual thermocouples from each bath were connected to a portable potentiometer through a multi-contact switch. Temperature measurements were made at depths of 1, 5, 9, 13 and 17 inches below the Al-Si surface. Each arrangement of five thermocouples was allowed 10 minutes for achieving thermal equilibrium at the successive levels prior to the act of temperature recording.

The temperature gradients found to exist in an Al-Si bath contained in the two types of crucibles are presented in tabular form in Table IV and in graphical form in Figure 21.

Temperature gradients in the region 5 to 13" below the Al-Si surface are of particular importance to the fuel-jacketing process in question. It is evident from Figure 21 that the temperature gradients, within this region, in the Al-Si bath contained in the coated metal crucible are smaller, as represented by the flatter curves. Of equal importance is the fact that the temperature gradients within the Al-Si bath contained in the cast iron crucible remain substantially unchanged with time throughout the service life of the metal crucible. In the case of the silicon carbide crucible, these gradients will increase appreciably as the thermal conductivity of the ceramic decreases with time.

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ENAMEL COATING MATERIAL

The enamel coating material is a commercially available product, secured for the present tests from the Charles H. Milles Foundry of Chicago, Illinois. An analysis of this material, showing its major constituents, is given in Table V. The recommended maximum working temperature is 790°C (1450°F).

Ferrous metals and alloys such as cast iron, carbon steel, Armco iron and low alloy steels are best suited for coating. Austenitic stainless steels (18-8 type) and high chromium steels (28-32 w/o Cr) can also be coated, but the integrity of the coating is not as reliable. In general, metals and alloys having a low chromium and nickel content and a low expansion coefficient are most successfully coated.

REMARKS

The information obtained from the tests described herein and the agreement between the large and small-scale tests are considered sufficient to demonstrate the feasibility and practicality of the enamel coated cast iron crucible as a container material for eutectic Al-Si alloy in the temperature range 595°-650°C.

Some of the more obvious advantages to be derived from the coated metal crucible are:

- 1) a longer crucible service life as demonstrated by actual test;
- 2) better bath temperature control due to the inherently higher thermal conductivity of the metal crucible and its capacity for storing heat (greater mass);
- 3) lower power cost made possible by a thermal conductivity which is essentially unchanged by time;
- 4) excellent thermal shock properties.

Although the large coated metal crucible used as one of the subjects of the present tests was made to order for ANL, its cost compared very favorably with the cost of identical ceramic crucibles of silicon carbide or clay-bonded graphite. Furthermore, these ceramic crucibles have approximately one-fourth the service life under the stated conditions of temperature and atmosphere environment.

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ACKNOWLEDGEMENT

The technical assistance of Earl W. Hoyt and Howard V. Rhude in performing the work on which this report is based is gratefully acknowledged. Credit is also due J. Goleb of the Laboratory Chemistry Division for the analytical data.

REFERENCES

- (1) Private Communication: Letter to F. L. Yaggee from the Joseph Dixon Crucible Company, August 20, 1952.
- (2) Kelman, L. R., Wilkinson, W. D., and Yaggee, F. L., "Resistance of Materials to Attack by Liquid Metals," ANL-4417 (July 1950).

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TABLE I.

Enamel-Coated Metal Containers Used in the Preliminary Tests

Vendor	Dimensions (in.)			Alloy	Cr	Alloy Composition (w/o)						
	I.D.	Depth	Wall Thick.			Mo	Mn	C	Si	Fe	P	S
Charles H. Milles Foundry Chicago, Ill.	2½	4½	3/8	Cast Iron	1.00	0.50	0.72	3.00 3.10	2.06 2.10	Bal	0.074	0.04
Michiana Products Corp.	2½	4½	1/4	Alloy 55*	28.00 32.00	-	1.50	0.50 Max.	-	Bal		

*Composition of Michiana Alloy No. 55 is within the ferritic stainless steel range.

TABLE II.

A Summary of Conditions Used in Testing the Feasibility of Enamel-Coated Metal Crucibles

Testing Variables	Enamel-Coated Small Metal Pots (2½" I.D. x 3/8" wall x 4½" deep)	Full-Size Enamel-Coated Cast Iron Crucible (18½" I.D. x 1½" wall x 24" deep)
Molten Metal Volume (cu. in.)	16.2*	4680*
Enamel Area in Contact with Molten Metal (sq. in.)	46.2*	1175*
Ratio $\frac{\text{Enamel Area}}{\text{Metal Volume}}$	$\frac{2.85}{1}$	$\frac{0.25}{1}$
Testing Temp. (°C) (°F)	700-725 1292-1337	595-650 1103-1202
Duration of Test (days)	51-60	144

*Calculated values based on the dimensions of the coated metal container and the thermocouple tube. Enamel surface assumed to be smooth.

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TABLE III.

Results of Spectrochemical Analyses of Al-Si Contained in an Enamel-Coated Metal Pot and Crucible

Small Enamel-Coated Cast Iron Pot (2½" I.D. x 4½" Deep)						Full-Size Enamel-Coated Cast Iron Crucible (450-pound Capacity)					
Al-Si Sample No.	Test Time (Days)	B (ppm)		Fe (Percent)		Al-Si Sample No.	Test Time (Days)	B (ppm)		Fe (Percent)	
		Al-Si Sample	Virgin Al-Si	Al-Si Sample	Virgin Al-Si			Bath Sample	Virgin Al-Si	Bath Sample	Virgin Al-Si
-	-	-	-	-	-	1	1	1	2	0.40	0.20
-	-	-	-	-	-	5	10	1	2	0.60	0.20
1	20	4	2	-	-	10	20	1	2	0.60	0.30
-	-	-	-	-	-	15	31	1	2	0.70	0.30
2	40	3	3	-	-	19	41	1	2	0.80	0.30
3	54	4	4	-	-	24	51	1	2	1.00	0.30
NOTE: Each boron and iron determination followed by a similar determination on virgin Al-Si as a standard for comparison.						28	62	1	2	1.00	0.30
						31	71	1	2	1.00	0.30
						35	80	1	2	1.10	0.30
						38	90	1	2	1.10	0.30
						42	100	1	2	0.90	0.30
						46	111	1	2	1.20	0.30
						50	120	1	2	1.10	0.30
						54	129	1	2	1.00	0.30
						57	144	1	2	1.00	0.30

TABLE IV

A Comparison of Temperature Gradients Occurring in Molten Al-Si Contained in a Cast Iron Crucible and a Silicon Carbide Crucible.

Depth Below Al-Si Surface (inches)	AL-SI BATH TEMPERATURE IN °C									
	(Readings at points along crucible diameter measured from center)									
	CAST IRON CRUCIBLE					SILICON CARBIDE CRUCIBLE				
	7"	3-1/2"	At Center	3-1/2"	7"	7"	3-1/2"	At Center	3-1/2"	7"
1	605	605	603	607	605	612	603	606	611	606
5	607	609	609	609	607	615	616	612	617	614
9	607	609	607	610	607	614	616	612	619	614
13	607	609	609	610	608	614	616	616	619	615
17	607	609	609	610	608	617	618	616	619	614

Each crucible contained approximately 450 lb of eutectic Al-Si alloy.

Cast iron crucible - controller setting 630°C

Silicon carbide crucible - controller setting 660°C

TABLE V

Spectrochemical Analysis Results
Listing the Major Constituents of
the Enamel Coating Material

Element	w/o
Boron	0.50
Calcium	G 1.00
Sodium	G 1.00
Lead	G 1.00
Silicon	GG 1.0

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FIGURE 1. View of the Inside of a Silicon Carbide Crucible Showing a Fissure Caused by Reaction with Molten Lead. This crucible failed without warning after 17 days at 605°C.

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FIGURE 2. Hairline Cracks Occurring on the Surface of a Clay-Bonded Graphite Crucible after Four Weeks of Service in an Oxidizing Atmosphere.

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FIGURE 3. Section of a Clay-Bonded Graphite Crucible Showing the Degraphitization Occurring at 595°-650°C. This crucible lost 48 pounds in four weeks of operation in an oxidizing atmosphere.

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FIGURE 4. Equipment Setup Used for the Small Scale Preliminary Tests.

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FIGURE 5. Coated Metal Containers Used in the Small Scale Preliminary Tests. Testing temperature range 700°-725°C.

11773

Approx. 1/3X

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FIGURE 6. Sections of an Enamel Coated Cast Iron Pot after 54 days of Testing at 700°C-725°C.

SECTION A

Taken at the Al-Si
Liquid Level

11879

Approx. 1X

SECTION B

Taken 1" below the
Al-Si Liquid Level

11878

Approx. 1X

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FIGURE 7. An Enamel-Coated Cast Iron Thermocouple Tube.
Top: Before exposure to Al-Si.
Bottom: After 54 days exposure to Al-Si
at 700^o-725^oC.

11777

Approx. 1/3X

FIGURE 8. Transverse Sections of the Thermocouple Tube Shown in Figure 7.
Section A: Taken at the Al-Si Liquid Level.
Section B: Taken above the Al-Si Liquid Level.

A.

B.

11847

Approx. 2X

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FIGURE 9. Sections of an Enamel-Coated Cast Iron Pot Tested for Two Months at 700° - 725°C as a Container for Lead under Al-Si.

SECTION A

Taken at the Lead/Al-Si interface.

11884

Approx. 1X

SECTION B

Taken below the Lead/
Al-Si interface.

11883

Approx. 1X

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FIGURE 10. Longitudinal Section of the Coated Cast Iron Pot Shown in Figure 9. The observed localized attack begins at the Lead/Al-Si interface and extends upward into the Al-Si covering layer.

11964

Approx. 1X

FIGURE 11. Condition of an Enamel-Coated Ferritic Stainless Steel (Type 446) Thermocouple Tube after Exposure to Lead under Al-Si at 700°-725°C.

11880

Approx. 1X

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FIGURE 12. Type 304 Stainless Steel Thermocouple Tube.
 Top: Before exposure to Al-Si.
 Bottom: After less than 24 hours exposure
 to Al-Si at 595°-650°C.

11775

Approx. 1/3X

FIGURE 13. Metallographic Sections of a Type 304 Stainless Steel Thermocouple Tube.

11732

100X

SECTION A: Before exposure to Al-Si.

11731

100X

SECTION B: After less than 24 hours exposure to Al-Si at 595°-650°C.
 Note intergranular type attack.

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FIGURE 14. Condition of an Enamel-Coated Cast Iron Pot Containing Bronze (49-45% Cu--51-55% Sn) after 11 days at 700-725°C. The bronze surface was covered with a 1" thick layer of a mixed chloride flux.

11772

Approx. 1/3X

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FIGURE 15. An Enamel-Coated Alloy Steel Pot Used to Contain Bronze (49-45% Cu--51-55% Sn) for 9 days at 700°-725°C. The bronze surface was covered with a 1" thick layer of a mixed chloride flux.

11771

Approx. 1/3X

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FIGURE 16. Transverse Sections of the Coated Alloy Steel Pot Shown in Figure 15.

SECTION A

Taken at the bronze/
flux interface.

11886

Approx. 1X

SECTION B

Taken below the
bronze/flux
interface.

11882

Approx. 1X

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FIGURE 17. Full-Size Enamel-Coated Cast Iron Crucible Used to Contain Al-Si at 595-650°C. This test was terminated after 144 days when the furnace overheated.

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FIGURE 18. View of the Inside of the Coated Cast Iron Crucible
Shown in Figure 17.

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FIGURE 19. An Enamel-Coated Cast Iron Thermocouple Tube Exposed to Lead under Al-Si at 595°-650°C.

Top: Before exposure.

Bottom: After 3-month exposure.

11746

Approx. 1/3X

FIGURE 20. Transverse Section of Thermocouple Tube Shown in Figure 19.

11848

Approx. 2X

SECTION A: Taken at the Pb/Al-Si interface. SECTION B: Taken at the Al-Si liquid level.

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FIGURE 21. A Graphical Representation of Temperature Gradients Occurring in Molten Al-Si Contained in a Cast Iron and Silicon Carbide Crucible.

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