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R. G. Nelson
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Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
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SUMMARY

This report describes several encapsulation casting processes that were developed or used at the Pacific Northwest Laboratory to embed simulated high-level wastes of two different forms (glass marbles and ceramic pellets) in metal matrices. Preliminary evaluations of these casting processes and the products are presented.

Demonstrations have shown that 5- to 10-mm-dia glass marbles can be encapsulated on an engineering scale with lead or lead alloys by gravity or vacuum processes. Marbles ∿12 mm in dia were successfully encapsulated in a lead alloy on a production scale. Also, 4- to 9-mm-dia ceramic pellets in containers of various sizes were completely penetrated and the individual pellets encased with aluminum-12 wt% silicon alloy by vacuum processes. Indications are that of the casting processes tested, aluminum 12 wt% silicon alloy vacuum-cast around ceramic pellets had the highest degree of infiltration or coverage of pellet surfaces.

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INTRODUCTION

The safe disposal of nuclear waste is essential for the continued use of nuclear energy in the United States and abroad. Nuclear high-level liquid waste (HLLW) is produced when spent-fuel from nuclear reactors is reprocessed. In this process, the spent fuel assemblies are disassembled, the fuel rods opened, and the fuel dissolved in an acid solution. Various elements are then extracted from the solution, leaving behind a liquid rich in nuclear-fission products.

The solidification of this HLLW and its immobilization are the key elements in most proposed, radioactive-waste management systems. The primary purpose of solidification is to create an inert waste form that minimizes the migration of radionuclides from the waste to the biosphere. The immobilization or encapsulation of the HLLW is designed to assure that the final solid-waste form is compatible with all phases of waste management, including interim storage, transportation and emplacement of the waste form in a repository.

During the past 20 yr, numerous waste forms and processes have been proposed for solidifying and/or encapsulating high-level wastes (HLW). Recently, processes applicable to defense wastes were identified and evaluated by Stone, Goforth and Smith (1979), and commercial-waste processes were identified and evaluated by Treat et al. (1980) and E. R. Johnson Associates, Inc. (1980). A number of the waste forms and processes currently under consideration utilize the concept of embedding solid high-level waste forms in a metal matrix that provides multiple layers of isolation.

The specific objective of the metal-matrix casting effort in the Pacific Northwest Laboratory's (PNL) Alternative Waste Forms (AWF) Program is to develop acceptable casting procedures on an engineering scale that can be applied full scale in remotely operated and maintained production processes and facilities. These casting methods are to be compatible with specific waste forms and their integrated processes.

This report describes the scale-up and development of different casting procedures to demonstrate the feasibility of selected processes for encapsulating simulated HLW glass marbles with lead and lead alloys. Also, procedures to encapsulate simulated wastes in the glass-marble or ceramic-pellet form with an aluminum-12 wt% silicon alloy are discussed. Demonstrations on laboratory, engineering and full scale were conducted with glass marbles and ceramic pellets, and a full-scale integrated process with marbles was demonstrated. Discussions of these demonstrations, their results and the conclusions based on these tests are presented.

CONCLUSIONS

Two basic casting procedures (gravity and vacuum) are feasible for encapsulating and embedding waste forms in metal matrices. Results showed that compared to the gravity process, the vacuum process offered greater penetration of the matrix metal into the interstices of the marble waste-form stack. Of both processes tested, the vacuum-casting of aluminum-12 wt% silicon alloy around ceramic pellets had the highest degree of infiltration or coverage of the pellet surfaces. All of the processes developed or used are adaptable to remote operation and are capable of being scaled up to full production requirements.

Results of laboratory-, engineering-, and full-scale demonstration tests using these processes with glass marbles and ceramic pellets are listed below:

Glass Marbles

- Glass marbles 5 to 10 mm in dia were encased and encapsulated in 20-cm-dia x 80-cm-long containers with lead or lead alloy. These engineering-scale tests used gravity and vacuum processes.
- Simulated waste-glass marbles ∿12 mm in dia were successfully encapsulated on a production scale with a lead alloy in a 40-cm-dia x 213-cm-long canister.
- Simulated waste-glass marbles 5 to 10 mm in dia were infiltrated or encapsulated in a 10-cm-dia x 15-cm-long container with aluminum-12 wt% silicon. This laboratory-scale test used capsule vacuum-immersion techniques that did not melt the marbles. The resulting composite was free of voids.

Ceramic Pellets

- Ceramic pellets 4 to 9 mm and 2 to 4 mm in dia were completely infiltrated or encapsulated with aluminum-12 wt% silicon alloy on an engineering scale using a vacuum-casting process.
- Ceramic pellets 2 to 4 mm in dia were completely infiltrated with aluminum-12 wt% silicon alloy on a laboratory scale using vacuum capsule immersion techniques.

- Ceramic pellets 4 to 5 mm in dia could not be gravity-cast with aluminum-12 wt% silicon alloy using laboratory-scale capsule immersion techniques.

BACKGROUND OF METAL-MATRIX CASTING CONCEPT

One of the primary goals of the AWF Program at PNL is the development of waste forms that will provide multiple layers of isolation or protection. Such waste forms would be compatible with the multibarrier concept illustrated in Figure 1. The inner core of the waste form would consist of an inert solid, such as a "supercalcine" pellet or a glass marble (Rusin 1978). This inner core could be coated with additional leach- and oxidation-resistant materials, cast in a metal matrix, and then placed in a metal canister. The resulting composite waste form would exhibit improved thermal stability and mechanical strength, and the added layers would greatly improve leach resistance.

High-level liquid waste can be converted to solids by calcination at temperatures ranging from 500 to 600°C. The resulting product, or calcine, is thermally stable but is a dispersible powder in the form of particles that range in size from 2 to 500 µm. Also, the powder has a relatively high solubility in water (Jardine and Steindler 1978). Because of these characteristics--in addition to its poor conductivity and the presence of some residual nitrates--additional processing or treatment of the calcine is required to make a waste form whose properties are suitable for safe handling, transportation and long-term storage.

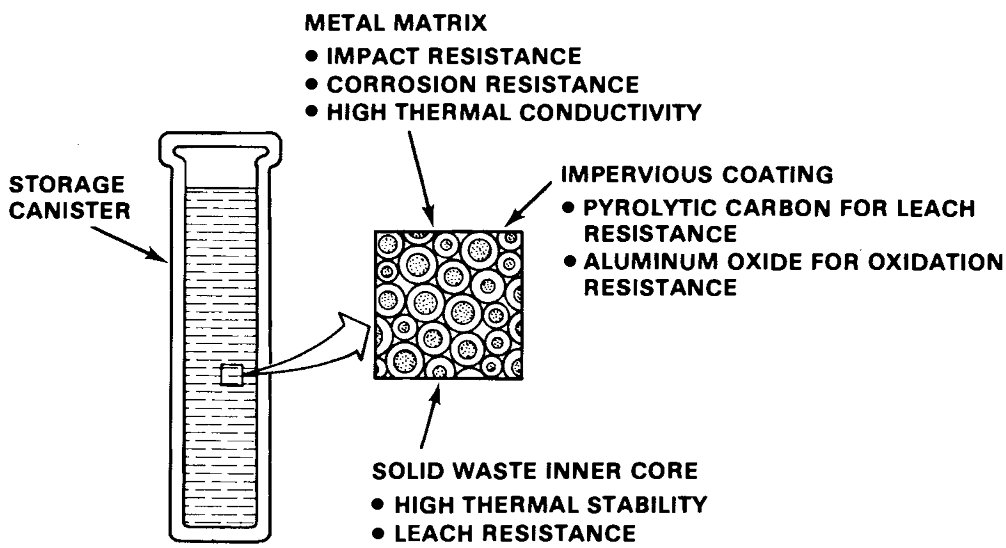


FIGURE 1. Multibarrier Concept for Isolating High-Level Waste

In addition to the problems above, difficulties were experienced in the past (Rhodes 1969) with the incomplete infiltration or encasement of the calcine with a metal matrix because of the small size of the calcine particles. Also, other investigators found that infiltration by casting was incomplete when calcine fines were present in concentrations greater than $\sim$10 wt% (Bell, Cole and Samsel 1976; Berreth, Cole, Samsel and Lewis 1976; VanGeel, Eschrich and Detilleus 1975). However, when larger particles were used to simulate processed calcine waste, successful infiltration was achieved (Sump 1976a and b; Sump and Begej 1976).

At PNL in 1976, laboratory-scale castings (1.3 to 3.2 cm in dia by 1.3 to 3.2 cm long) of matrix materials (lead, lead-tin, aluminum-silicon, and stainless steel or copper) containing either glass marbles or ceramic pellets were made and evaluated (Rusin et al. 1979). The 5- to 12-mm-dia borosilicate glass marbles are made from calcine and suitable glass frits (Treat et al. 1980). The 4- to 9-mm-dia ceramic pellets are made by adding soluble compounds of aluminum, calcium, silicon and strontium to the HLLW before calcination. After calcination, the calcine is pelletized and sintered to form hard, dense, spherical ceramic pellets. This final material is referred to as "supercalcine" (McCarthy 1977; Treat et al. 1980). Researchers at PNL also made and evaluated 1-L-volume, laboratory-scale, multibarrier waste forms of simulated waste-glass marbles and coated and uncoated ceramic pellets (Rusin et al. 1978).

The purpose of the demonstration discussed here is to identify the feasibility of encapsulating HLW borosilicate glass marbles and "supercalcine" ceramic pellets cast in a metal matrix on a production basis.

METAL-MATRIX ENCAPSULATION DEMONSTRATIONS

Many demonstrations have been conducted on metal-matrix casting techniques at PNL in the last 2 yr. These have used simulated HLW glass marbles and ceramic pellets and two basic alloys as matrix materials--lead and aluminum-12 wt% silicon. A range of waste-form sizes has been used in laboratory-, engineering- and full-scale tests.

LEAD ALLOY--GLASS MARBLES

A series of tests used commercial and/or simulated waste-glass marbles and lead alloys. These alloys were used because the softening temperature of borosilicate glass ranges between 500 and 550^{o}C and almost all other candidate alloys melt above this temperature. The first tests were on an engineering scale and included molten-metal casting and lead-shot casting. Subsequently, a full-scale test was performed using lead-shot casting. These tests and their results are described below.

Engineering-Scale Tests--Molten Metal

Figure 2 is a concept of a metal-matrix casting station. The canister has an inner container that retains the waste particles. The container is positioned to allow a gap between the walls and the bottom of the canister, and has a screened bottom and a covered screened vent on the top. Matrix metal is poured into the top of the canister and is deflected to the annulus between the container and the canister by the deflector plate. The metal flows down the annulus, across the bottom of the canister, and up through the bottom of the container into the waste-form particles, forcing air out the upper screened vent. This concept also provides for an optional vacuum-assisted vent system.

The molten-metal-matrix casting demonstration equipment is shown in Figure 3. This equipment consists of a furnace to melt the metal, a canister assembly, and a water-cooled canister stool. The casting furnace, schematically shown in Figure 4, is a 30-kW, resistance-wound, bottom-pour furnace. The furnace has a batch capacity of 45 kg of aluminum or 180 kg of lead.

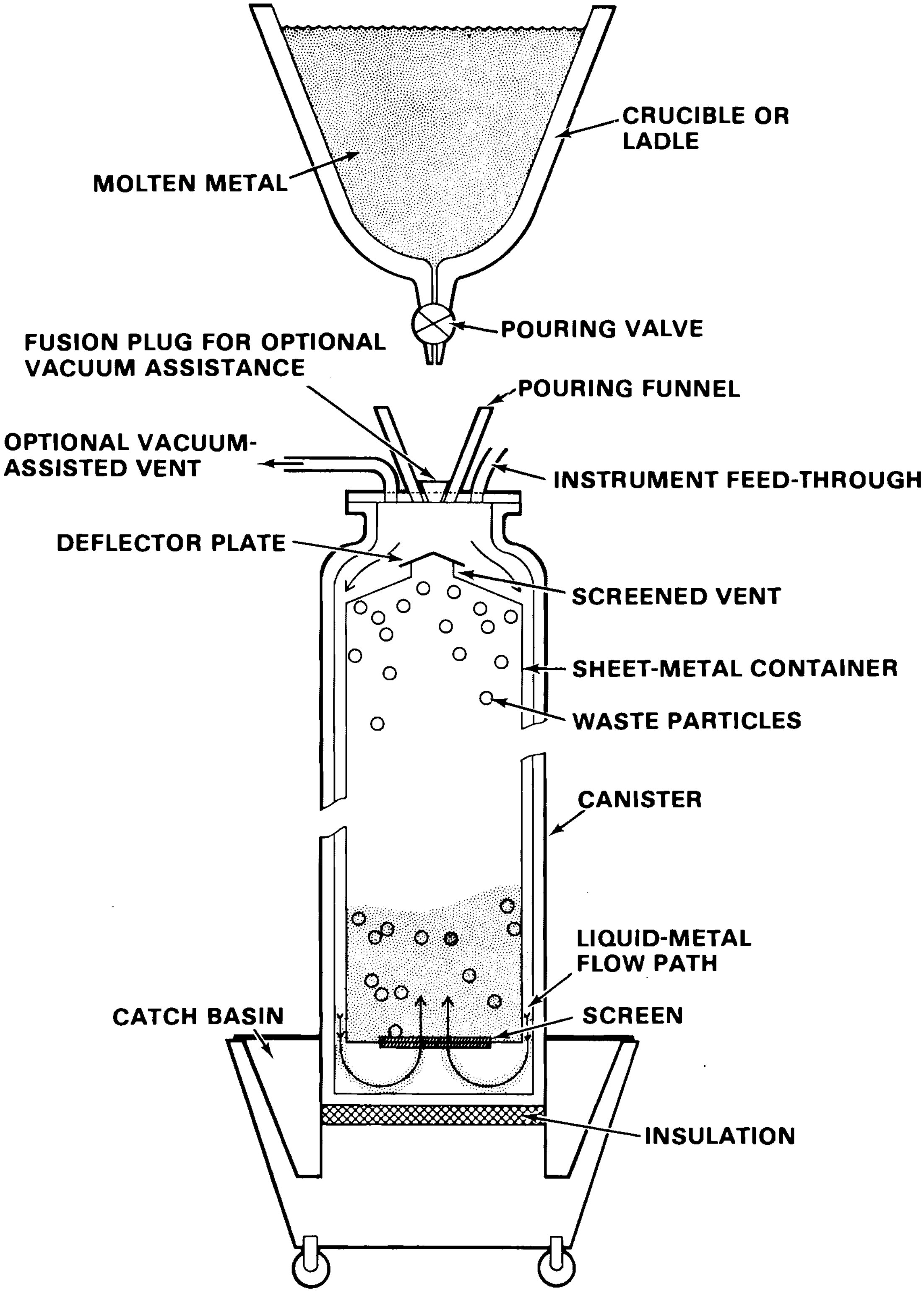


FIGURE 2. Metal-Matrix Casting Station

FIGURE 3. Engineering-Scale Metal-Matrix Casting Equipment

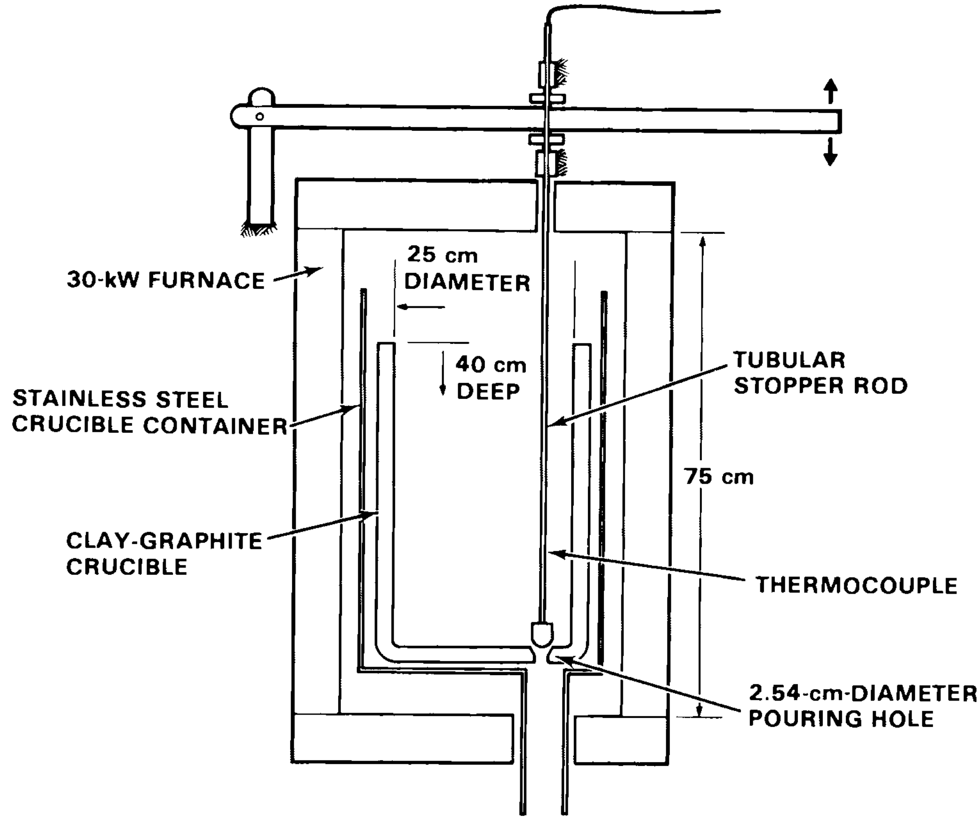


FIGURE 4. Metal-Melt Furnace

The canister assembly and water-cooling stool are shown in Figure 5. The canister is a Schedule 10 stainless steel pipe with an annulus between the inner waste container and the canister wall. To provide effective seals in a test setup that could be assembled and disassembled efficiently, flanged assemblies with soft copper gaskets were used. The water-cooling stool supports the canister assembly and contains nozzles that spray water directly onto the lower end of the canister. The vacuum-pouring funnel (Figure 5) is equipped with a fusion disk that allows the canister to be evacuated prior to metal-matrix pouring. Pouring of the heated matrix-metal melts the fusion disk, and by keeping the funnel full during pouring, a full dynamic vacuum can be maintained throughout the pour. The vacuum for this test setup was provided from a nearby vacuum-induction melting system having a volume of about 6 m^3.

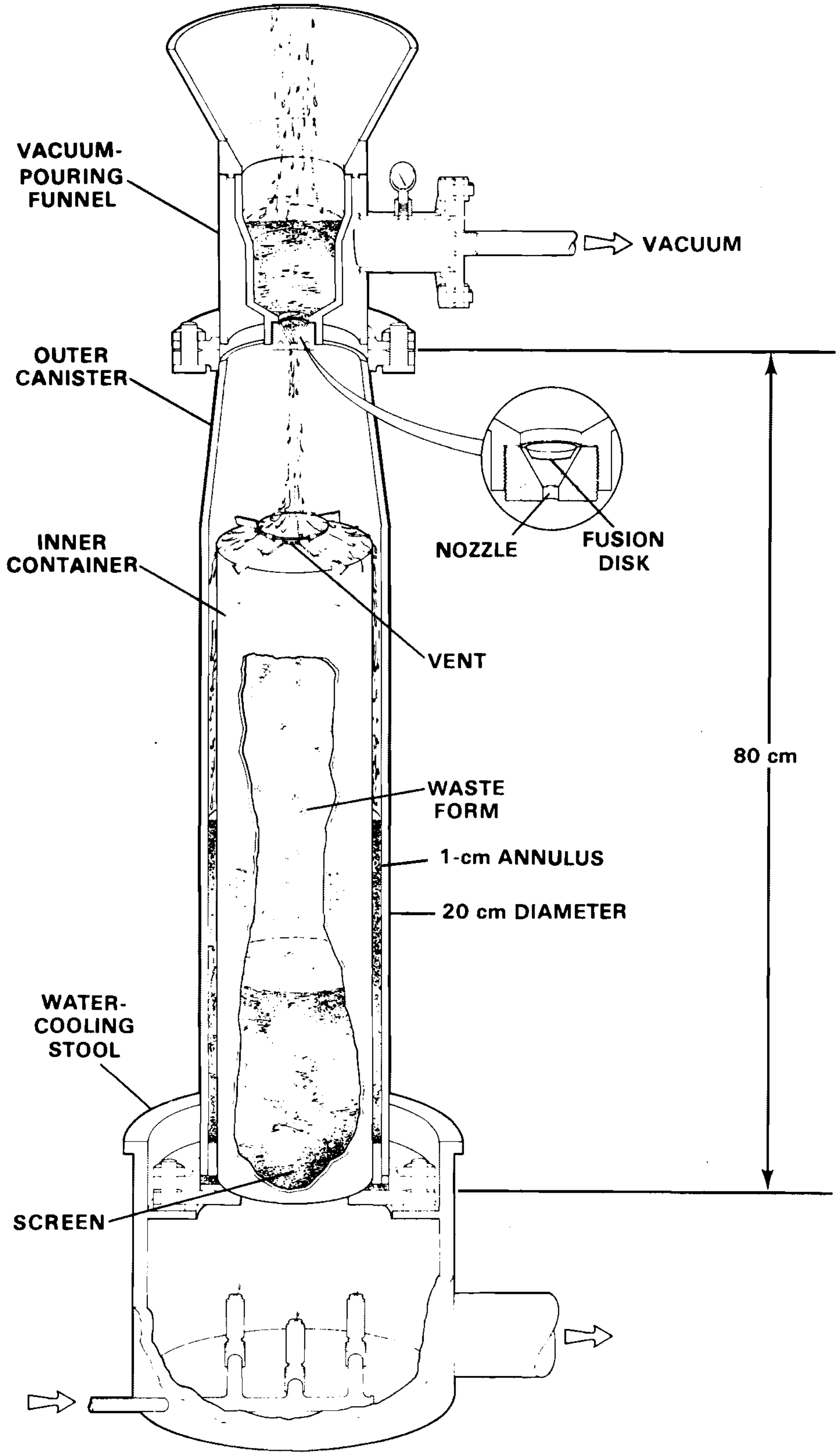


FIGURE 5. Waste Encapsulation by Molten Metal

Test runs for canister heatup times showed that 10 to 12 h were required to bring a canister loaded with simulated waste-glass marbles to the 400°C temperature felt to be required for adequate infiltration of the lead or lead alloy. A summary of these tests is given in Table 1.

The first successful test was Number 4. It consisted of a gravity infiltration of 35 kg of glass marbles with 182 kg of lead. (The composition of the glass marbles is noted in the Appendix.) Canister preheat and metal-pouring temperatures were 400°C.

Heat Number 5 was a test run to check the operation of the vacuum-pouring funnel. The funnel was placed on an empty canister with a special container inside to catch the molten metal and thus permit future use of the canister. The nozzle configuration for lead-alloy pouring shown in Figure 6A was used on this lead-10 wt% tin, 66-kg heat. A full vacuum was maintained throughout the pour that took only 27 s.

Heat Numbers 6 (vacuum) (Figure 7) and 7 (gravity) were the demonstration pours for the waste-glass marbles and Pb-10 wt% Sn matrix-alloy combinations. These heats were performed using the procedures developed in Heat Numbers 5 (vacuum) and 4 (gravity) and listed below.

Engineering-Scale Casting Procedure

1. Load inner container with waste-form particles.
2. Crimp deflector plate in place.
3. Assemble outer canister over container and attach lower flange.
4. Insert into preheat furnace.
5. Melt matrix alloy and bring to pouring temperature.
6. Remove canister assembly from preheat furnace.
7. Attach pouring funnel with nozzle and fusion disk in place.[a]
8. Place canister on cooling stool.
9. Position canister and cooling stool under pouring hole of casting furnace.
10. Attach vacuum line.[a]
11. Open vacuum valve.[a]

(a) For vacuum pouring only.

TABLE 1. Summary of Metal-Matrix Casting Demonstration Tests

Test or Heat Number	Matrix Alloy	Waste Form	Pour Temp., °C	Preheat Temp., °C	Type of Pour	Remarks
1	Pb, 46 kg	None	400	None	Gravity	Furnace checkout made and air sampling conducted for lead fumes.
2	Pb, 155 kg	Mixture of waste-glass marbles and commercial lime glass, 34 kg	416	407	Gravity	Flotation hold-down clips gave way. Inner can floated up out of open canister, causing a molten-metal spill.
3	Pb	Waste-glass marbles, 35 kg				Due to leaky pouring valve, test was scrubbed. Loaded canister reusable.
4	Pb, 182 kg	Waste-glass marbles, 35 kg	400	400	Gravity	Excellent heat. Very little splashing during pour.
5	Pb 10 wt% Sn, 66 kg	None	400	None	Vacuum	Test pour for vacuum funnel. Sixty lead-40 tin fusion disk 7.1 mm (0.28 in.) thick; 9.5-mm (0.375-in.)-dia nozzle; 27-s pour. Full vacuum and valve wide open all the way.
6	Pb 10 wt% Sn, 178 kg	Waste-glass marbles, 35 kg	400	400	Vacuum	Fusion disk and nozzle as above. Fifty-five-second pour; full vacuum all the way; used insulating blanket on upper 15 cm of canister.
7	Pb-10 wt% Sn, 186 kg	Waste-glass marbles, 34 kg	400	400	Gravity	Fifty-eight-second pour. Used insulating blanket on upper portion of canister.
8	Al-12 wt% Si, 27 kg	Ceramic pellets, 4 kg; waste-glass marbles, 1.5 kg	700	700 for ceramic pellets, 400 for glass marbles	Immersed capsules. Filled by gravity and vacuum	Gravity-filled one capsule containing 4- to 5-mm-dia ceramic pellets with an unidentified foreign coating. Vacuum-filled one capsule containing 2- to 4-mm-dia ceramic pellets. Vacuum-filled one capsule containing 5- to 10-mm-dia glass marbles.
9	Al-12 wt% Si, 42 kg	Ceramic pellets, 2 to 5 mm dia, 22 kg	700	700	Vacuum	Pour failed to penetrate 3-mm (0.125-in.)-thick 6061 aluminum fusion disk because vacuum pouring funnel was not preheated.
10	Al-12 wt% Si, 42 kg	Canister loaded same as that in Heat No. 9	700	700	Vacuum	Preheated canister with pouring funnel attached. No fusion disk used. Heat failed because vacuum sucked metal into upper part of canister and the vacuum line. Very little matrix metal infiltration of the pellets.
11	Al-12 wt% Si, 9 kg	None	700	500	Vacuum	Preheated canister with pouring funnel attached. Small 15-cm (6-in.)-dia test. Canister with 3-mm (0.125-in.)-thick Al-12 wt% Si alloy fusion disk and 11-mm (0.438-in.)-dia nozzle. Test was scrubbed because aluminum leaked out of furnace pouring hole.
12	Al-12 wt% Si, 8 kg	None	700	500	Vacuum	Repeat of Heat Number 11. Good results. Matrix metal poured through Al-12 wt% Si fusion disk. Eleven millimeter (0.438-in.)-dia nozzle was too large. Periodically lost vacuum during pour.

TABLE 1. (contd)

Test or Heat Number	Matrix Alloy	Waste Form	Pour Temp., ^{o}C	Preheat Temp., ^{o}C	Type of Pour	Remarks
13	Al-12 wt% Si, 11 kg	Ceramic pellets, 2 to 5 mm dia, 2.2 kg	730	500	Vacuum	Small test canister with Al-12 wt% Si fusion disk and 9.5-mm (0.375-in.)-dia nozzle. Nozzle diameter too large since some vacuum breaks occurred during the pour. Excellent results. Full infiltration of the waste particles.
14	Al-12 wt% Si, 45 kg	Ceramic pellets, 4 to 9 mm dia, 34 kg	730	500	Vacuum	Full-size canister with Al-12 wt% Si, fusion disk and 8.7-mm (0.344-in.)-dia nozzle. Thirty-seven-second pour. Vacuum all the way with no vacuum breaks. Excellent results with full infiltration.
15	Pb-3 wt% Sb, 8 mm dia, 175 kg	Simulated waste-glass marbles, 35 kg	400	400	Gravity	Inner container hold-down broke and container floated up in canister. Average shot introduction rate 75 kg/h.
16	Pb-3 wt% Sb, 179 kg	Waste-glass marble, 35 kg	450	450	Gravity	Successful demonstration. Average shot introduction rate 94 kg/h.
17	Pb-2 wt% Sb, 3.3 mm dia, 1429 kg	SRL TDS calcine and 211 frit, 310 kg	400-450	450	---	Full-production-scale demonstration. Average shot introduction rate 455 kg/h.

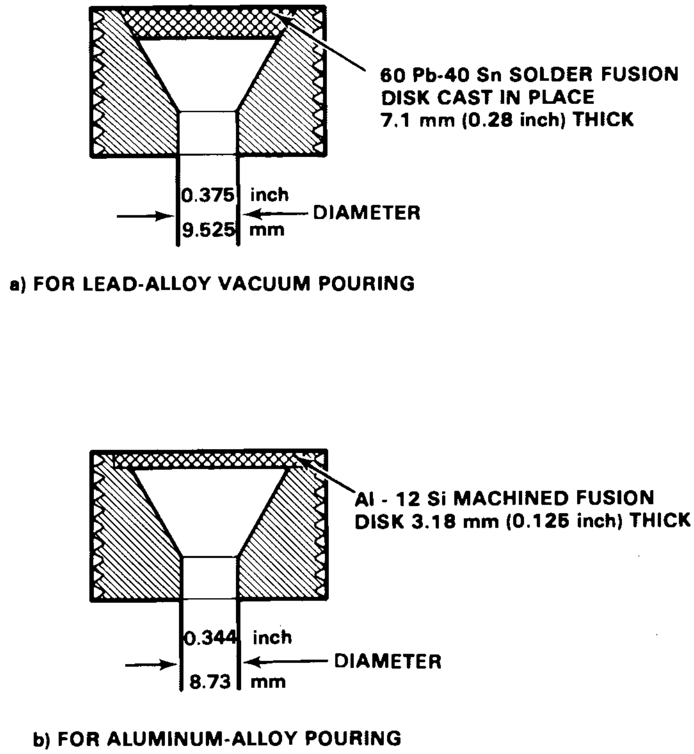


FIGURE 6. Nozzle Configurations for Vacuum Pouring

12. Attach water supply and drain lines.
13. Pour metal.
14. Open water-cooling valve.
15. Add insulating blanket to upper portion of canister.

(a) For vacuum pouring only.

Engineering-Scale Tests--Lead Shot

Tests were performed to demonstrate the feasibility of melting the metal matrix in the HLW-process primary storage canister (i.e., the technique of In-Can Encapsulation or ICE). ICE encapsulation would eliminate the handling of molten metal in a remote-process cell. In the ICE technique, the metal matrix is slowly added to the canister in the form of particles (shot) while the canister and its inner container, loaded with a solid waste form, are in a preheat furnace. Such a test setup is depicted in Figure 8.

Waste Form: 35 kg Simulated Waste-Glass Marbles
Matrix: 178 kg Lead-10 wt%-Tin Alloy
Heat Number: 6

FIGURE 7. Vacuum-Cast Lead-Tin Matrix

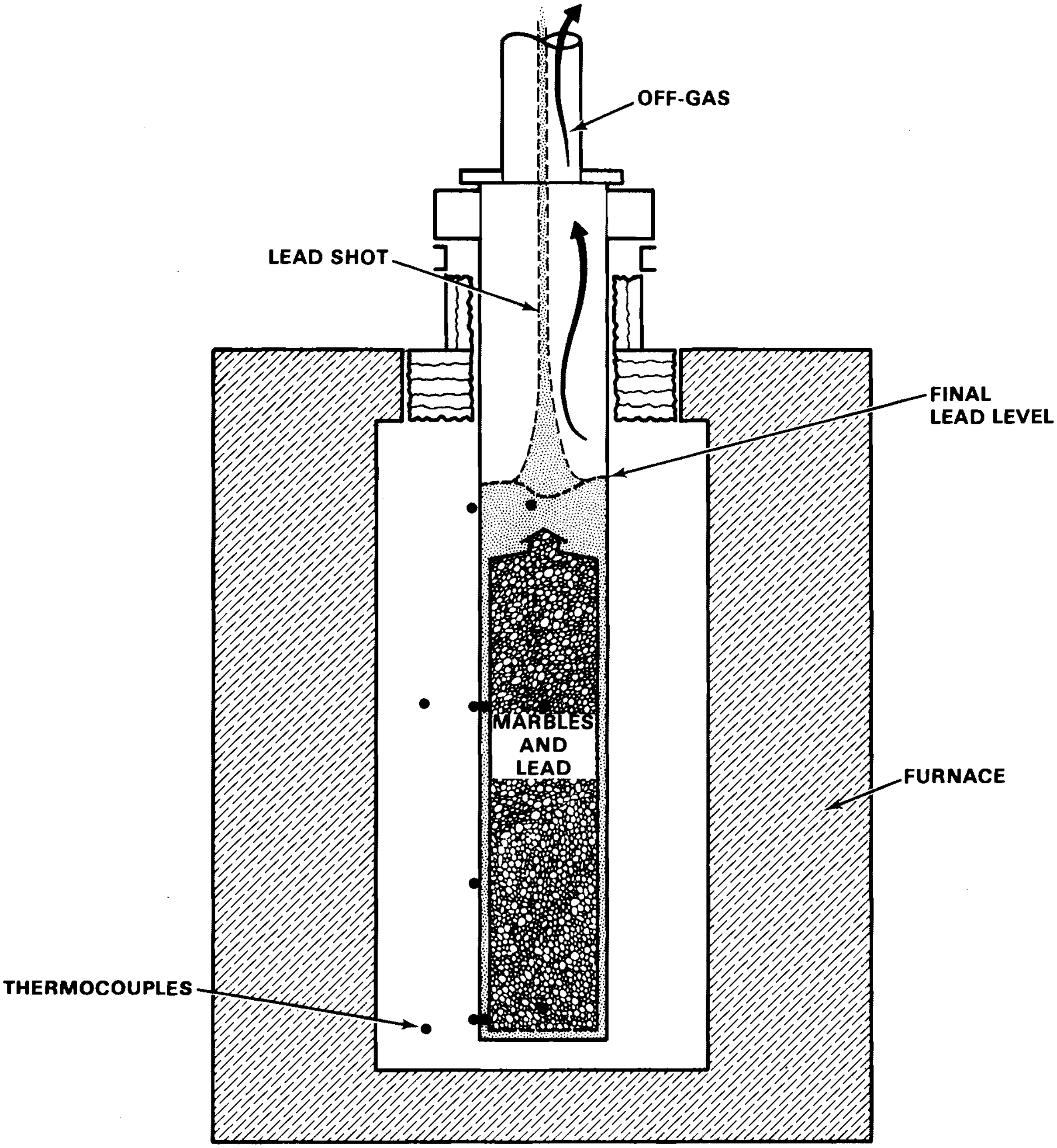


FIGURE 8. Engineering-Scale In-Can Encapsulation of Glass Marbles

Inner containers identical to those used for the previous engineering-scale tests were used. These containers were filled with waste-glass marbles ~12 mm in dia. The filled container was inserted in an open-top 20-cm canister having a length compatible with the existing pilot-scale In-Can Melter

(ICM). The first ICE test is summarized as Heat Number 15 in Table 1. The test assembly was instrumented as shown in Figure 8.

While a slight vacuum was held on the system (∿2.5 cm H_2O), the test setup was preheated to bring the temperature of the marbles to 400°C. Initially, 34 kg (75 lb) of 8-mm-dia lead-antimony shot were poured in. Then, after a 15-min hold to assure that all internal temperatures were above 400°C, 11.34 kg of lead shot were introduced about every 10 min. During the pouring of Bag Number 7 of shot (68 to 79 kg), one of the lower thermocouples oscillated ∿10°C and then dropped ∿20°C. Later, it was found that the inner container had broken loose from its hold-downs and had floated up about 20 cm. After 15 bags (170 kg) of lead shot had been poured in, the test setup was held at temperature for 30 min and then disconnected. The canister was removed from the furnace and its upper portion insulated to simulate zone cooling. Then the bottom 10 cm. of the canister was placed in flowing water for a period of ∿3 h.

Because the inner container in this first test had broken loose from its original position, a second test was performed. This test, identified as Heat Number 16 in Table 1, followed the same procedure, except the assembly was preheated to 450°C, which took ∿11 h. Two-millimeter-diameter lead-antimony shot was used, and the marble container was welded to the canister to assure that it would stay in place. After 34 kg of lead shot had been poured in, and after a 15-min hold to assure temperature, an additional 136 kg of lead shot was inserted in 11.34 kg batches in the next 90 min. A total of 179 kg of shot was used. As indicated in Figure 9, the metal matrix fully infiltrated the stack of marbles.

<u>Full-Scale Demonstration Test--Lead Shot</u>

The purpose of this test setup and demonstration was to show the feasibility of in-can encapsulation of marbles in a lead matrix on a scale that would be required in a production process.

In the ICE technique, the glass for the marbles is melted in the Advanced Glass Forms Melter (AGFM) (Dierks 1980), and the molten glass is poured continuously into a marble-making machine (Platt and Powell 1980). Acceptable

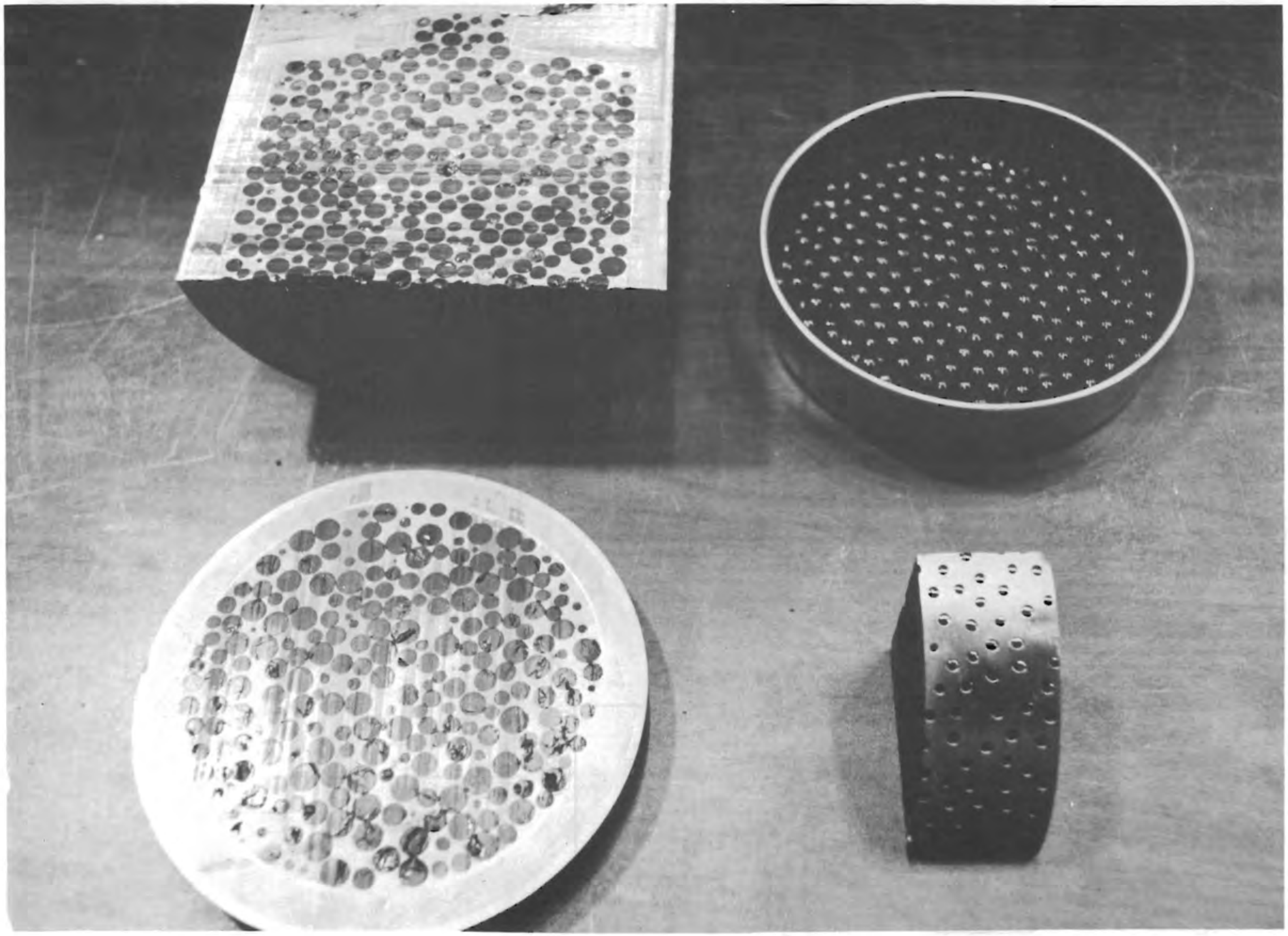


FIGURE 9. Marbles Gravity-Cast With Lead-Shot Feed

marbles are fed to a basket or container inside a canister. A schematic of this process is shown in Figure 10.

In the full-scale ICE demonstration test, the marble-filled canister was placed in a furnace which heated the marbles to ~450°C. (Heating of the

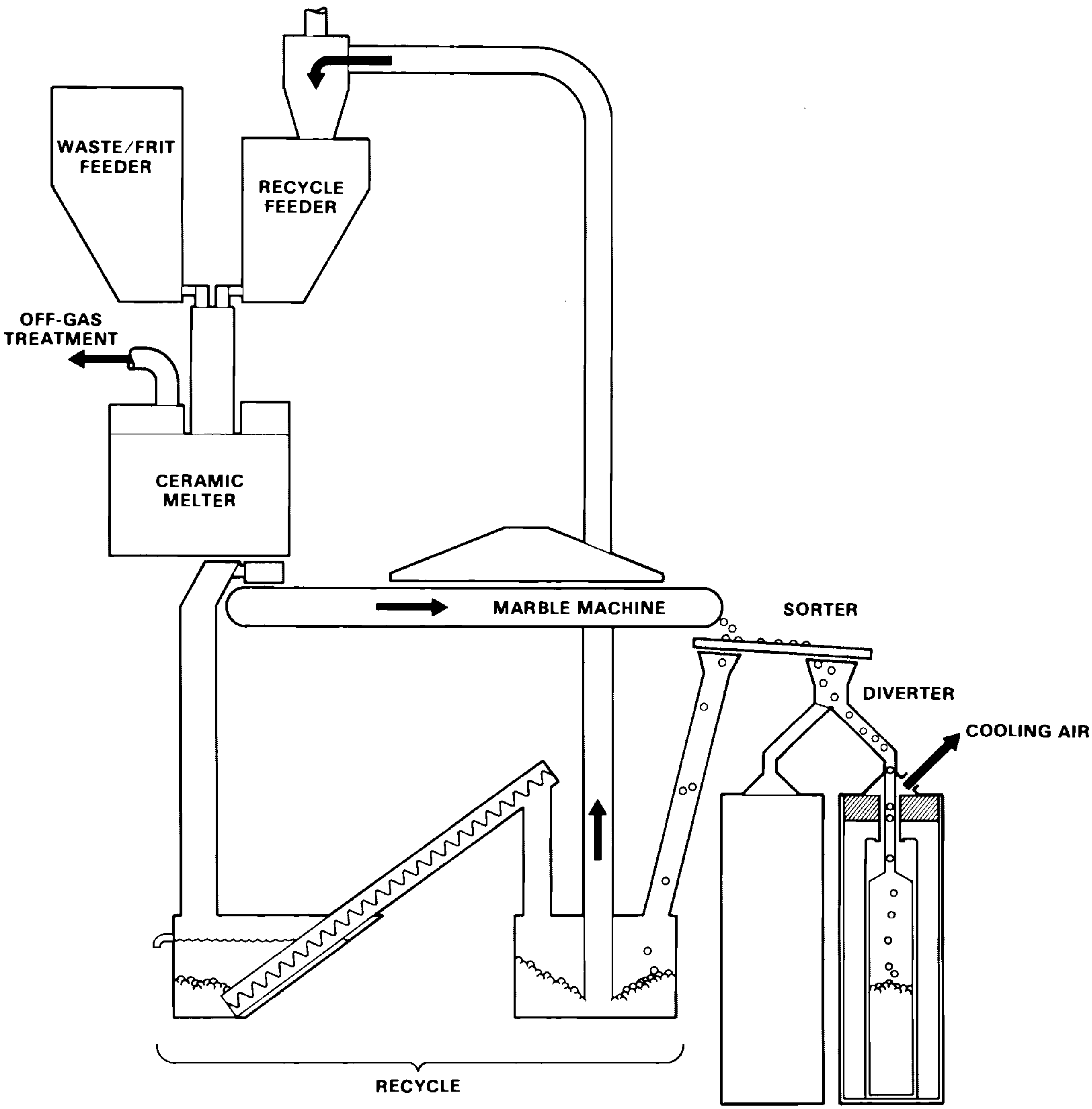


FIGURE 10. Glass Marble Production Schematic

marbles is enhanced by blowing hot air through the marble mass.) When the mass of marbles reached the desired temperature range, lead shot was added to the canister. The test is summarized as Heat Number 17 in Table 1.

The product of this demonstration was a 40-cm (16-in.)-dia by 213-cm (7-ft)-long stainless steel canister containing ∿310 kg of glass marbles encapsulated in ∿1429 kg of lead. Figure 11 is a photograph of the filled canister. The waste-glass marbles, ∿12 mm in dia, were made from simulated Savannah River TDS calcine and 211 frit in the composition noted in the Appendix. Number 4 lead shot (3.3 mm dia) of the composition noted in the Appendix was used.

This full-scale canister of lead-encapsulated marbles was produced in an existing ICM furnace (Figure 12) that contains six separate heating zones. The test assembly was instrumented as shown in Figure 13. The temperature of the test assembly was raised by powering all of the heating zones in the ICM and by heating air that flowed into the assembly up to $450^{\circ}C$. The temperature in the marbles was brought up to $450^{\circ}C$ in ∿7 h and held overnight.

A vacuum of ∿2.5 cm H_2O was established in the test assembly and lead shot was gravity-fed through an airlock in 57-kilogram batches. As the lead falls in the annulus between the inner container and the canister, it melts. As Figure 14 shows, a total of 1383 kg (3050 lb) of lead shot was added in 2 h and 50 min for an overall rate of 488 kg/h. No long-term temperature changes were noted, except that the thermocouple located just above the inner container never registered above $300^{\circ}C$ while the shot was being added.

Next, the power was turned off in the two lowest heating zones, and forced-air cooling was introduced in the lowest zone. Target temperatures for each of the six zones were monitored, and zone heating and cooling were controlled for a 9-h period. This permitted molten lead to fill voids that are created when the lead densifies as it passes from liquid to solid state. During this cooldown step, an additional 46 kg of lead shot were added to maintain the desired level of lead in the canister.

FIGURE 11. Full-Scale In-Can Encapsulation of Marbles in Lead

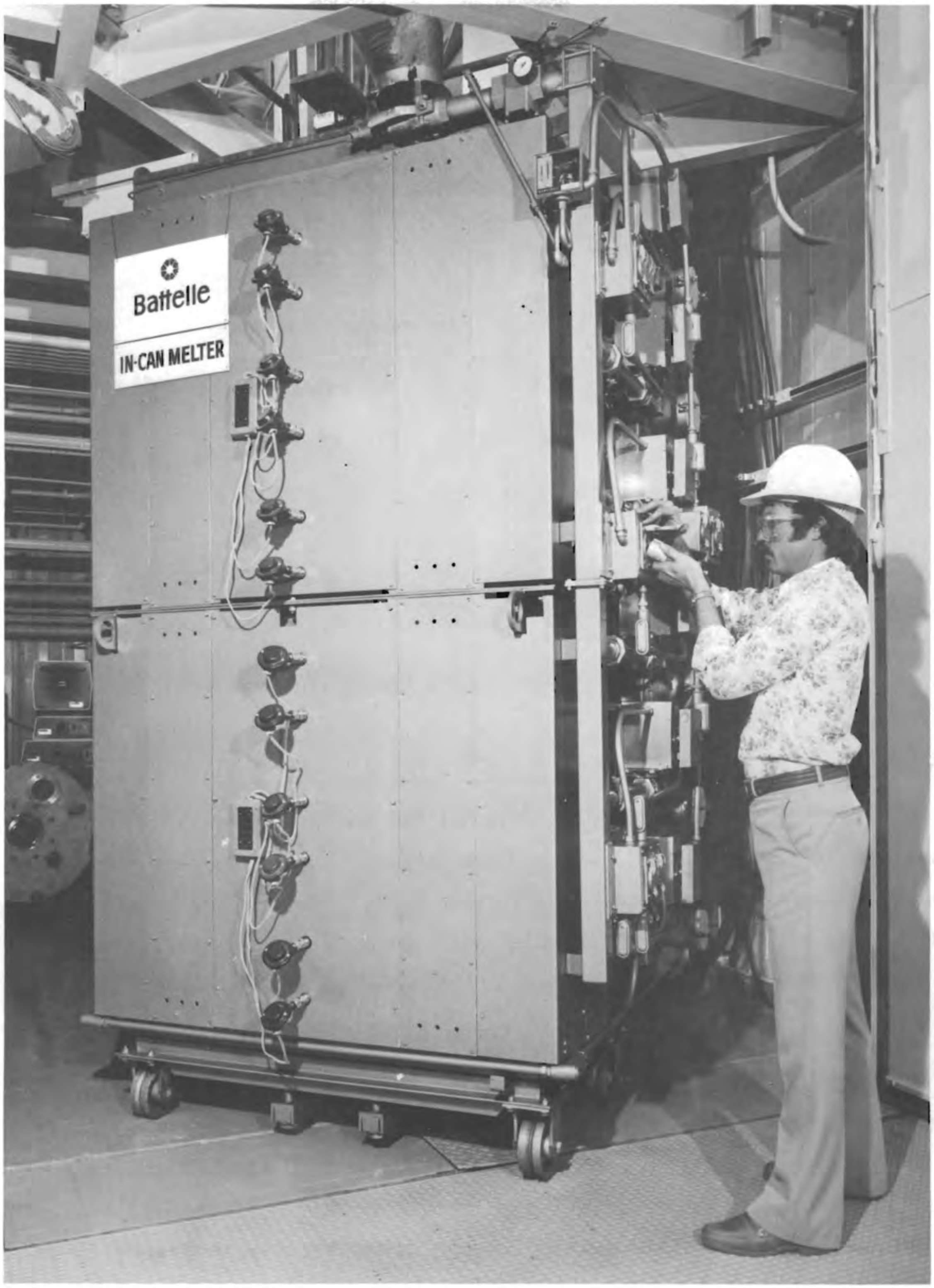


FIGURE 12. Full-Scale In-Can Melter

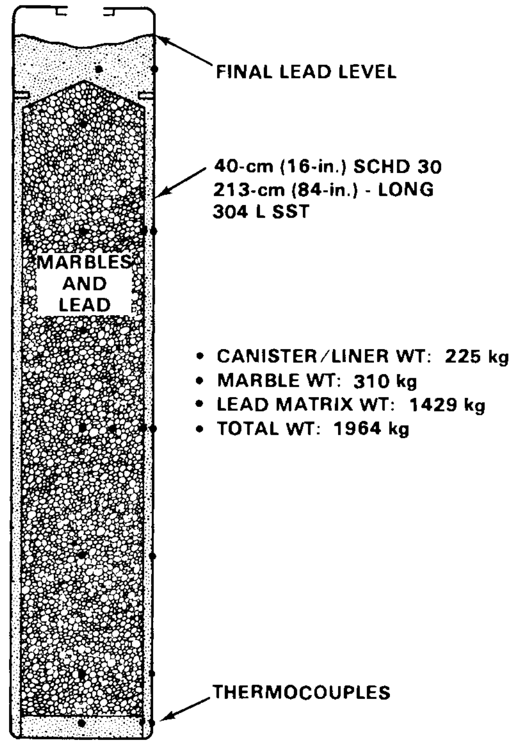


FIGURE 13. Full-Scale Marble-In-Lead Canister

ALUMINUM-12 WT% SILICON ALLOY--CERAMIC PELLETS AND GLASS MARBLES

This series of tests demonstrated the encapsulation of simulated waste pellets in an aluminum-12 wt% silicon alloy. Both laboratory-scale and engineering-scale tests were performed using gravity- and vacuum-casting of molten metal. Also, glass marbles were encapsulated in the aluminum alloy on a laboratory-scale.

Laboratory-Scale Aluminum Alloy--Ceramic Pellets and Glass Marbles

In the laboratory-scale tests, capsules 10 cm (4 in.) in dia by 15 cm (6 in.) long containing ceramic pellets and glass marbles were infiltrated with metal matrix by immersion in a furnace. A summary of the capsule tests is shown as Heat Number 8 in Table 1. The method used to fill these capsules with Al-12 wt% Si matrix metal is shown schematically in Figure 15 and is described below.

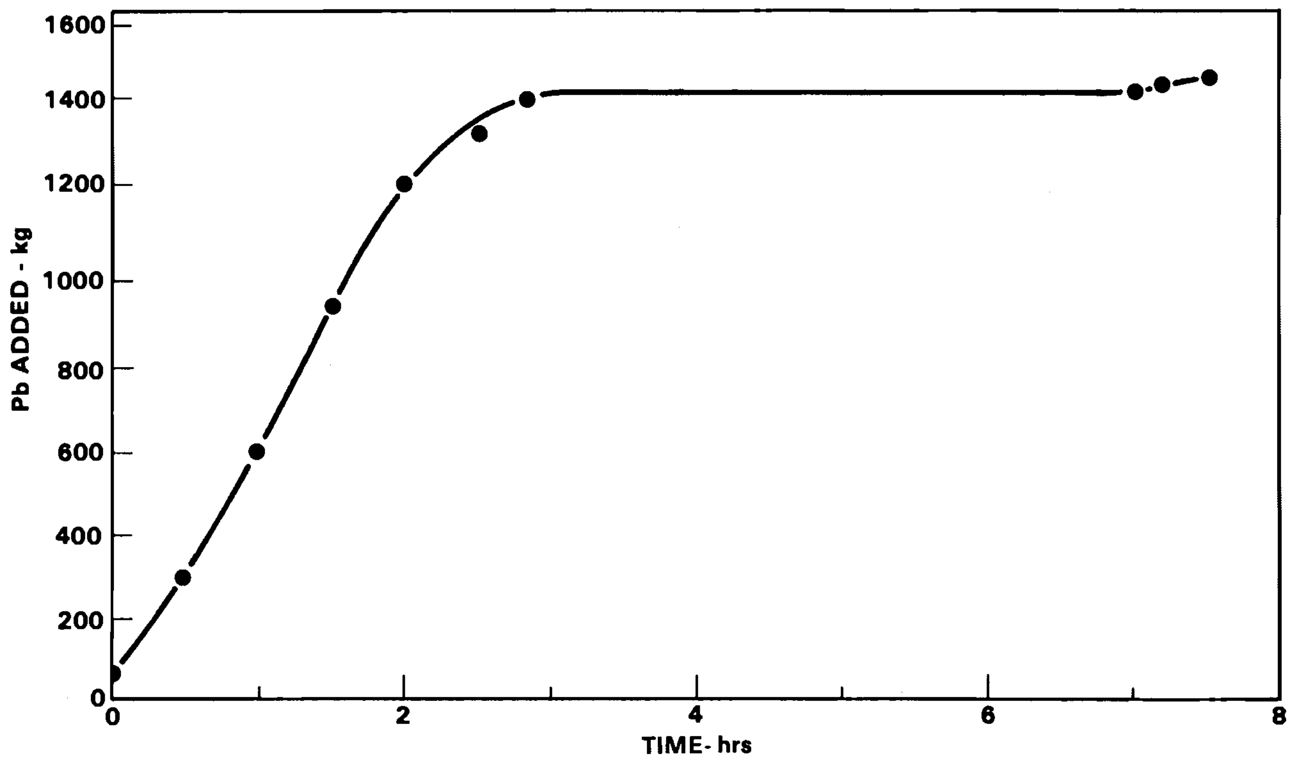


FIGURE 14. Full-Scale Lead-Filling Rate

Capsule-Filling Procedure

1. Preheat Capsule: 700°C for ceramic pellets
   400°C for waste-glass marbles
2. Add Al-12 wt% Si alloy fusion plug[a]
3. Evacuate capsule[a]
4. Crimp vacuum line partially closed[a]
5. Immerse capsule in Al-12 wt% Si 700°C matrix metal
6. Remove and water-quench bottom surface.

(a) For vacuum-filling only

The first two capsule experiments showed that:

- ceramic pellets 2 to 4 mm in dia can be successfully vacuum-infiltrated with Al-12 wt% Si matrix alloy (see Figure 16).

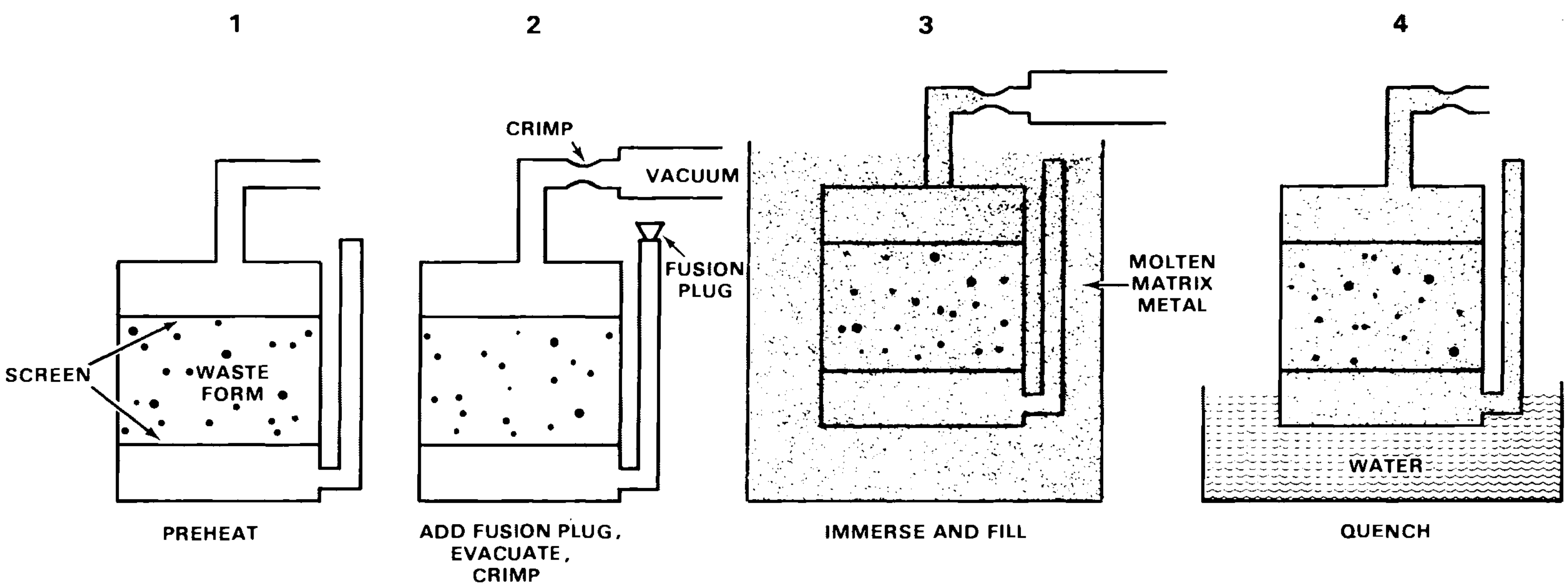
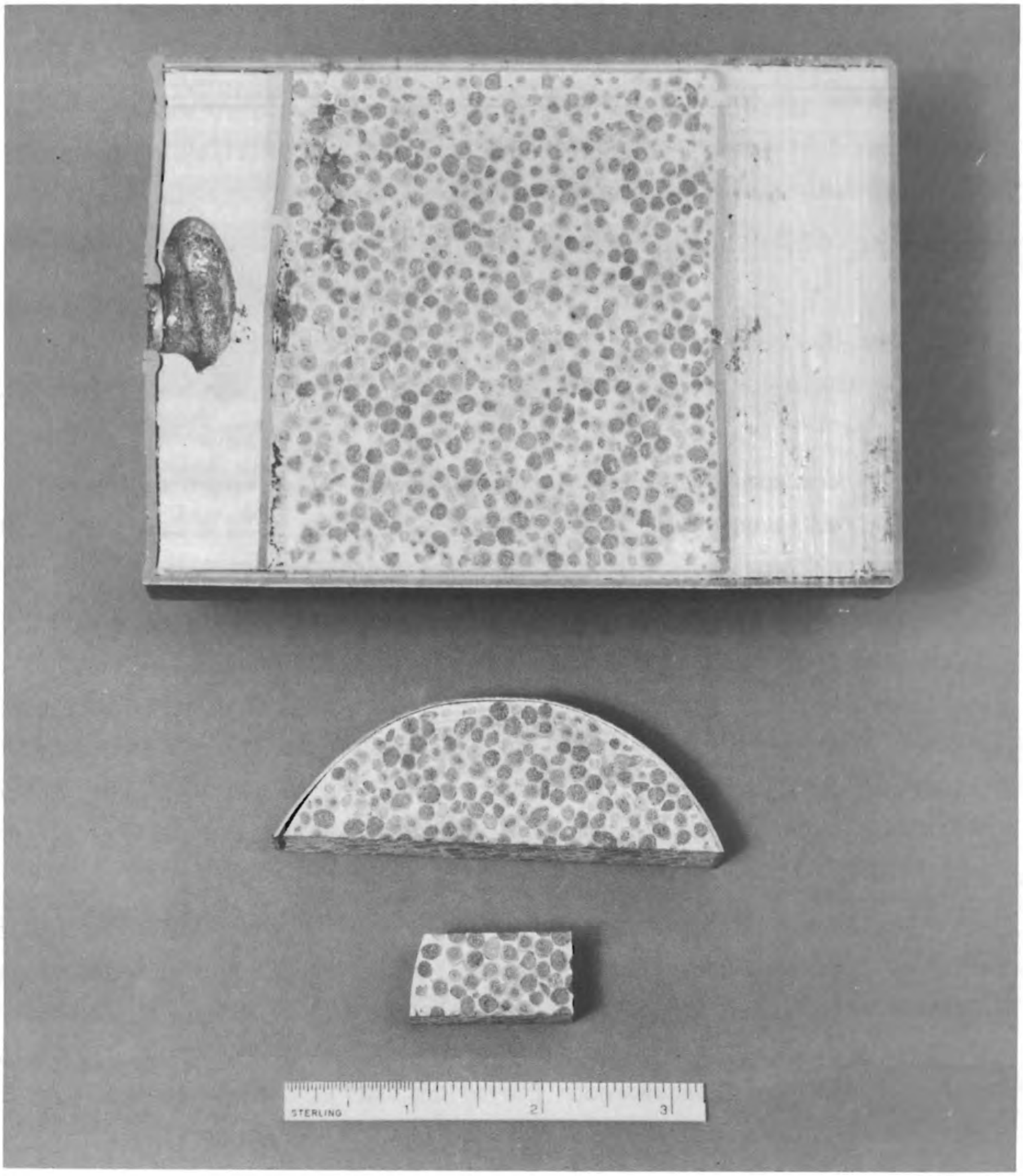


FIGURE 15. Schematic of Capsule-Filling Technique



Waste Form: Ceramic Pellets 2 to 4 mm in Diameter
Matrix: Aluminum 12 wt% Silicon Alloy
Heat Number: 8

FIGURE 16. Vacuum-Immersion Infiltrated Capsule--Pellets

- ceramic pellets 4 to 5 mm in dia could not be gravity-infiltrated with Al-12 wt% Si matrix alloy.

A third capsule containing 5- to 10-mm-dia simulated waste-glass marbles was successfully vacuum-infiltrated with Al-12 wt% Si alloy at a capsule preheat temperature of 400^{o}C (Figure 17).

Engineering-Scale Aluminum Alloy--Ceramic Pellets

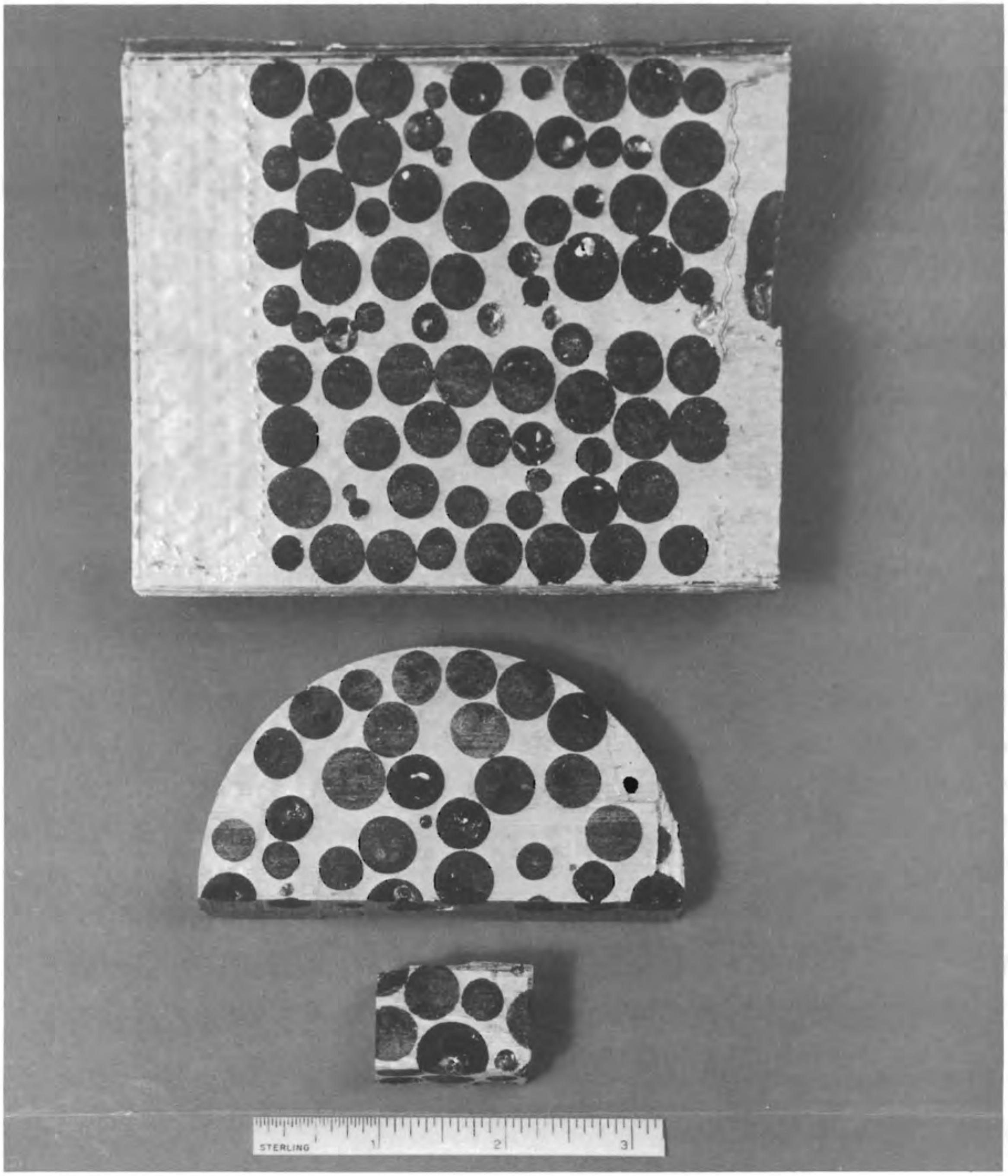
Because of the successful vacuum-infiltration results obtained with the 2- to 4-mm-dia ceramic pellets, development efforts were directed toward vacuum infiltration in larger canisters--20 cm (8 in.) dia. The test approach and the equipment were the same as those used for lead alloy-glass marbles.

Test runs conducted on canister heatup times indicated that 9 to 11 h were required to bring the mass of ceramic pellets up to a temperature of 700^{o}C.

Heat Number 9 (see Table 1) was the first attempt to vacuum-infiltrate a 20-cm (8-in.)-dia canister containing ceramic pellets with Al-12 wt% Si alloy. The loaded canister was preheated to 700^{o}C. The vacuum funnel had a 6061 aluminum fusion disk and a 9.5-mm (0.375-in.)-dia nozzle. The unheated funnel was attached to the preheated canister just prior to pouring. When the 700^{o}C metal matrix was poured, it froze in the funnel and, consequently, did not penetrate the fusion disk.

Heat Number 10 was an attempt to eliminate the fusion disk because of the extremely rapid pumpdown time (<5 s) of the canister. For this procedure, the canister with funnel attached was preheated to 700^{o}C. Then, the canister vacuum system was opened immediately after attaching the vacuum line, and the metal was poured. This approach failed because as soon as the metal reached the canister it was carried upwards and into the vacuum port.

Because of the limited supply of ceramic pellets and the need for additional testing, a small 15-cm (6-in.)-dia by 38-cm (15-in.)-deep test canister was fabricated. A 3-mm (0.125-in.)-thick Al-12 wt% Si fusion disk with a melting point of 577^{o}C was used with an 11-mm (0.438-in.)-dia nozzle. The canister-vacuum funnel assembly was preheated to 500^{o}C before pouring. Heat



Waste Form: Glass Marbles 5 to 10 mm in Diameter
Matrix: Aluminum 12 wt% Silicon Alloy
Heat Number: 8

FIGURE 17. Vacuum-Immersion Infiltrated Capsule--Marbles

Number 12 was a test of this pouring configuration and preheat temperatures. The 700°C matrix metal poured through the Al-12 wt% Si fusion disk without any problems. However, the nozzle was apparently too large since the sound of periodic vacuum breaks were evident during the pour.

Heat Number 13 was similar to Heat Number 12. A 13.4-cm (5.3-in.)-dia by 13.4-cm (5.3-in.)-long inner container loaded with 2- to 5-mm-dia ceramic pellets was inserted in a 15-cm (6-in.)-dia test canister. The nozzle diameter was reduced to 9.5 mm (0.375 in.). The loaded canister was preheated at 500°C overnight and the vacuum-funnel assembly preheated at 500°C just before pouring. The matrix metal was poured at 730°C. There were some vacuum breaks during the pour, which indicated that the nozzle diameter was still too large. However, excellent results were achieved with full encapsulation of the waste particles, as shown in Figure 18.

Ceramic pellets 4 to 9 mm in dia were used for Heat Number 14. A 20-cm (8-in.)-dia canister was loaded with 34 kg of pellets. Forty-five kilograms of Al-12 wt% Si alloy at 730°C were vacuum-poured into the canister in 37 s. A nozzle with a diameter of 8.7 mm (0.344 in.) (Figure 6B) was used, and no vacuum breaks were detected during the pour. Excellent results were achieved with full metal-matrix penetration of the pellet stack, as shown in Figure 19. The casting procedure used for lead alloy-glass marbles (see page 12) was used for vacuum-infiltrating these ceramic pellets during Heat Numbers 9 through 14. However, an additional step, 4a, was included in the procedures as listed:

4a. Two to four hours prior to pouring, remove canister from preheat furnace and attach funnel assembly with fusion disk in place. Return assembly to preheat furnace.[a]

(a) It is not known at this time what the effect of the prolonged (9 to 11 h) preheat time required to bring the ceramic pellets up to temperature will have on the integrity of the 3-mm (0.125-in.)-thick Al-12 wt% Si fusion disk. Therefore, preheat time for the funnel assembly is kept to a minimum.

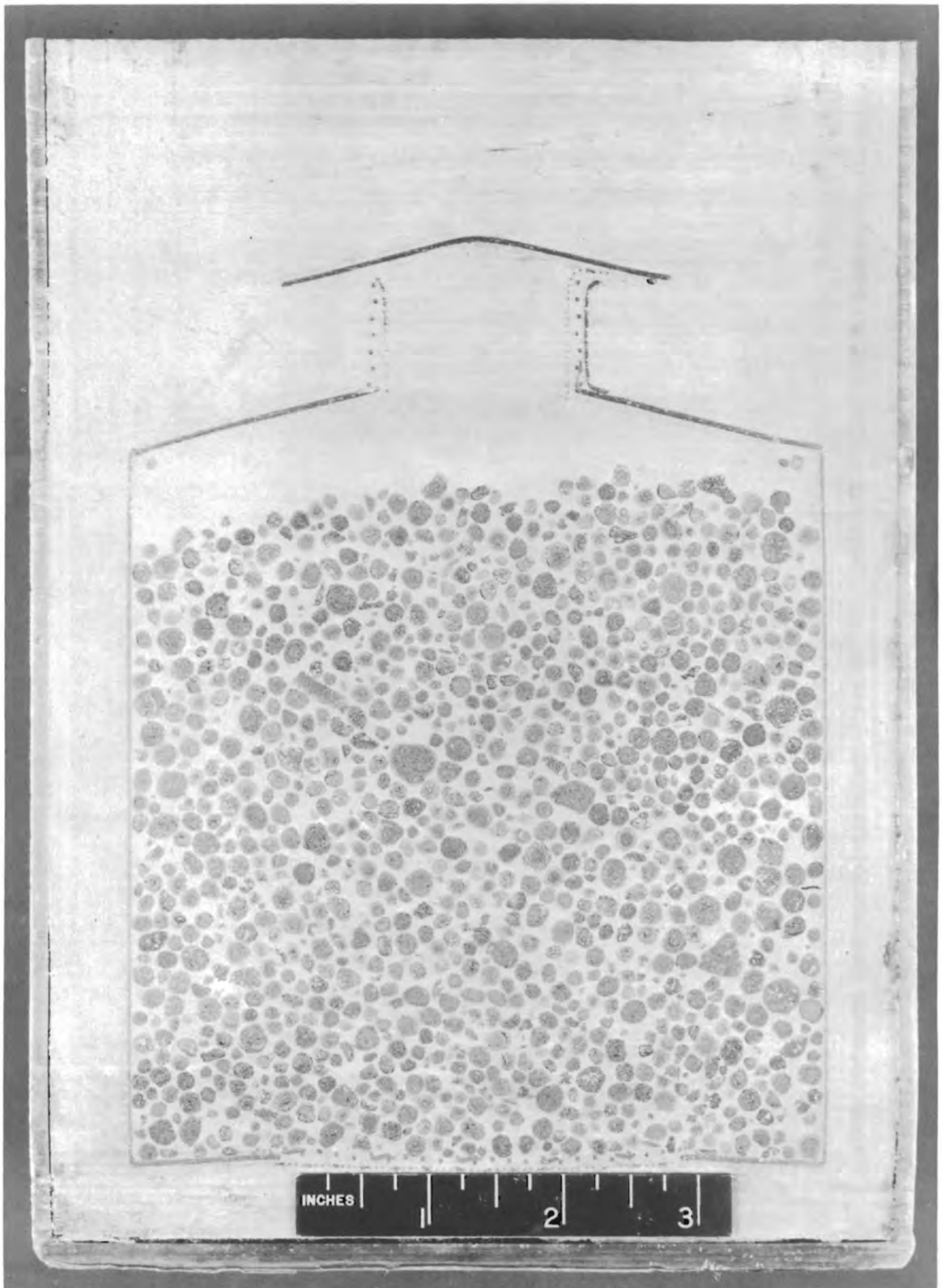
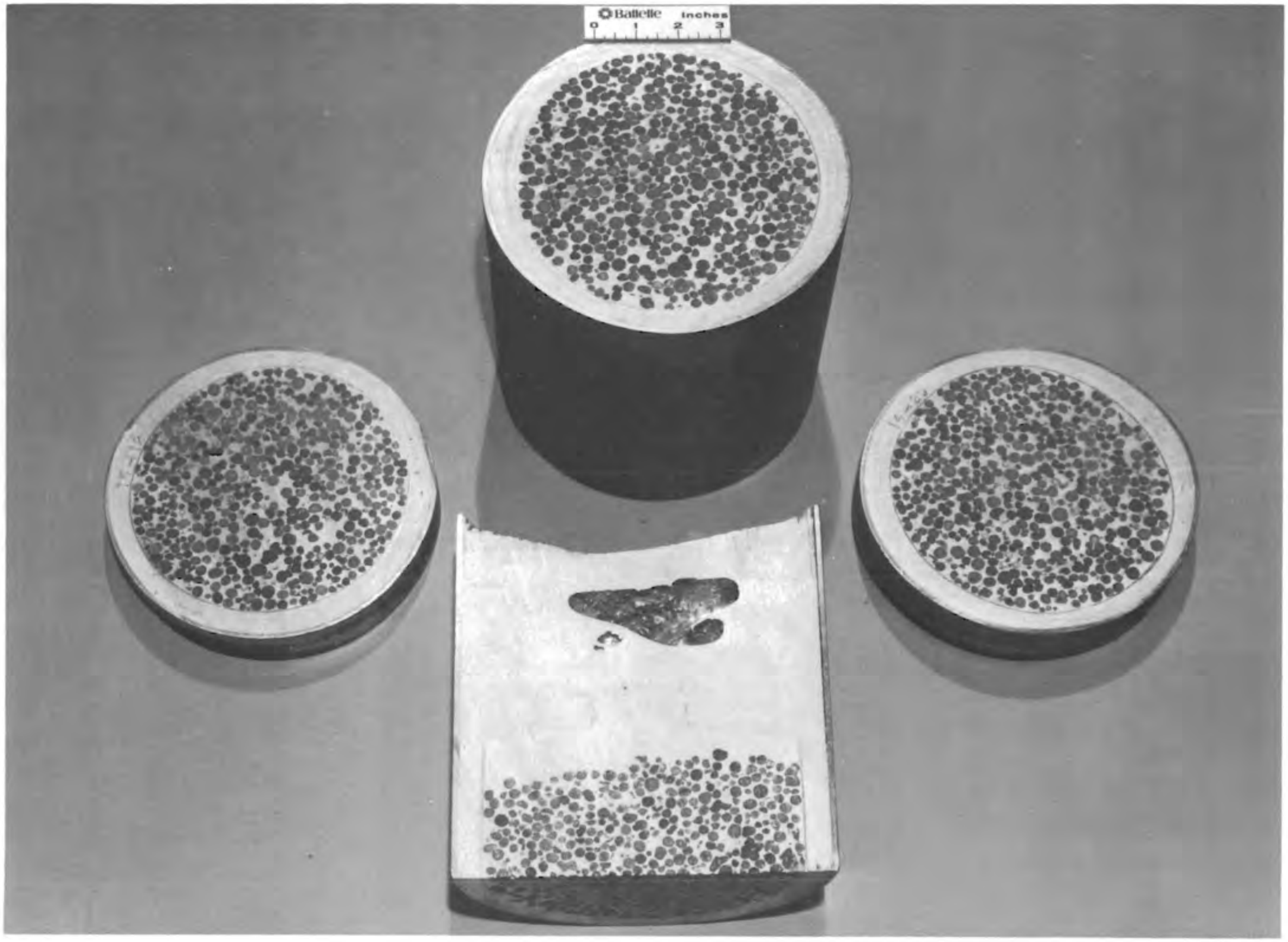


FIGURE 18. Vacuum Infiltration of Al-12 Wt% Si Ceramic Pellets



Waste Form: 34 kg Ceramic Pellets 4 to 9 mm in Diameter
Matrix: 45 kg Aluminum 12 wt% Silicon Alloy
Heat Number: 14

FIGURE 19. Vacuum-Cast Aluminum-Silicon Alloy Matrix--Pellets

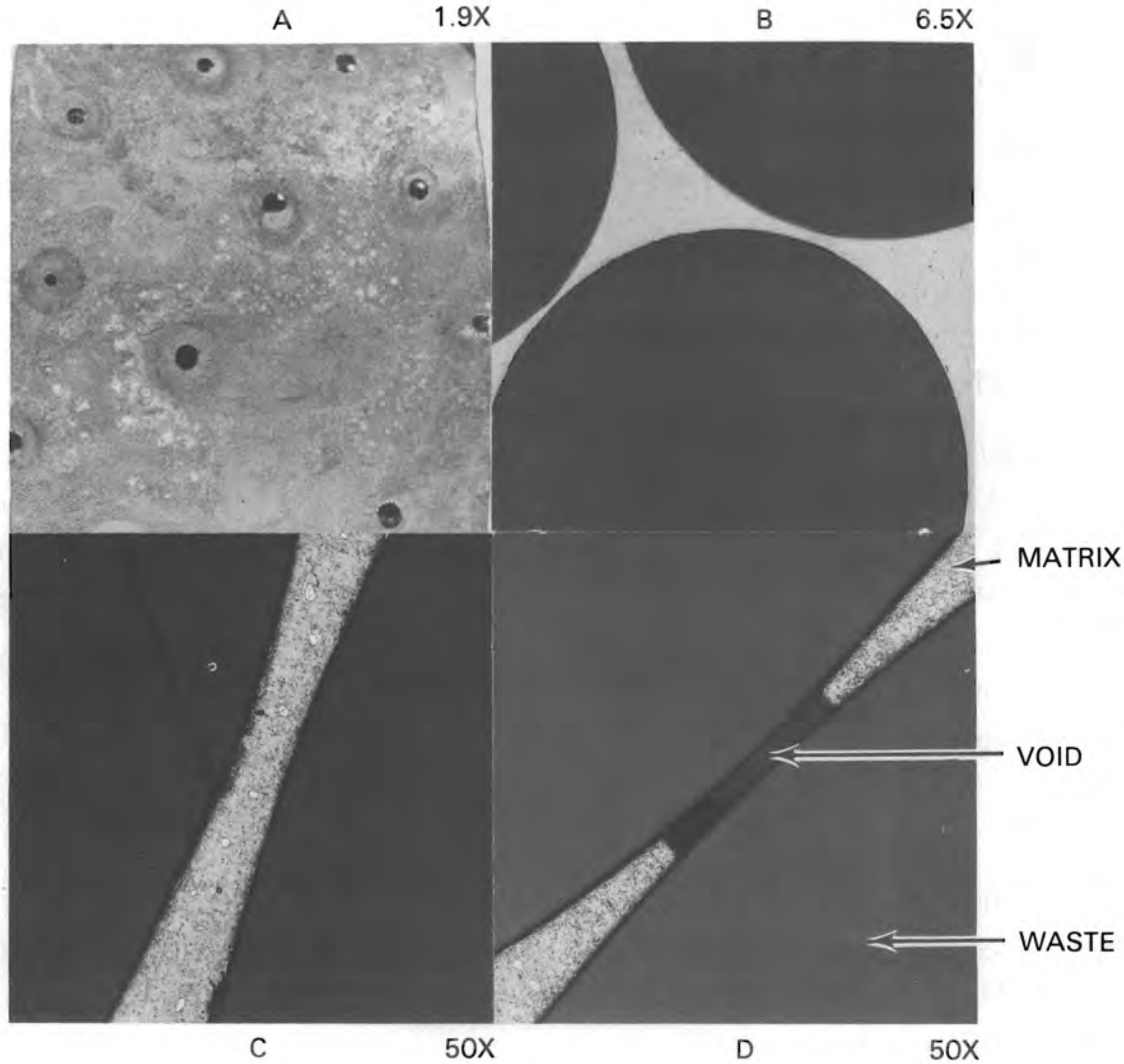
PRODUCT EVALUATION

Several cross sections, both vertical and horizontal, were cut in the various test containers and canisters so that the results of the various casting procedures could be evaluated. Typical sections are shown in Figures 7, 9, 16, 17, 18 and 19.

The alloys used in this investigation have excellent casting characteristics. As shown in the Appendix, commercial casting grades were used. The cast matrix-metal quality showed no evidence of gross impurities or porosity.

Figures 7 and 20 show sections of the engineering-scale vacuum casting performed on simulated waste-glass marbles with lead-tin alloy (Heat Number 6). Figure 21 indicates the configuration of the marbles in gravity-cast lead-tin alloy (Heat Number 7). Figures 9 and 22 show some of the sections and micrographs made after the enginering-scale tests were performed with waste-glass marbles in lead shot (Heat Numbers 15 and 16). These figures show that the lead encapsulated the marbles very well. No voids or inclusions are in the lead, and only occasional small gaps are evident at some of the marble contact points.

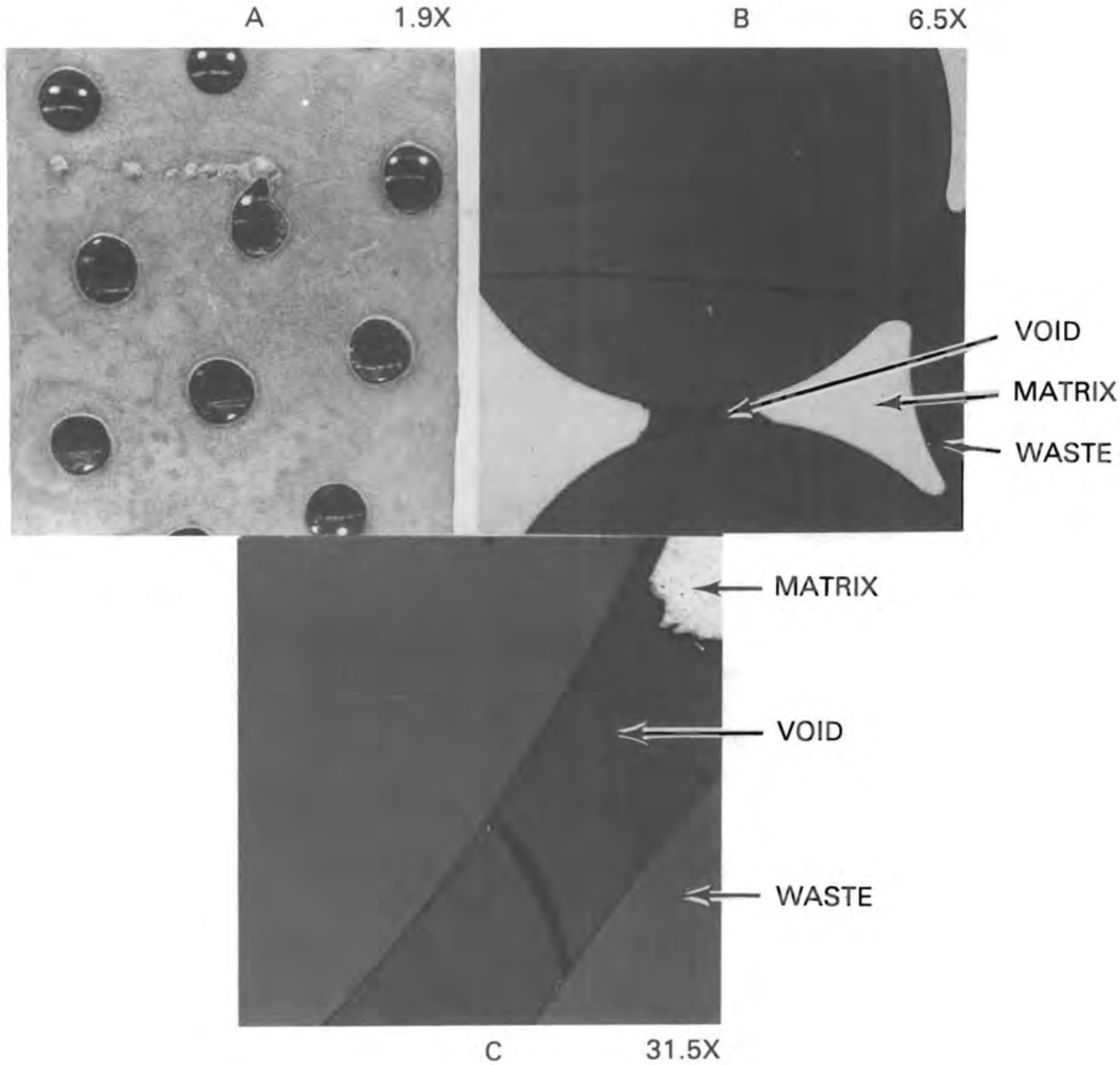
Figures 19 and 23 depict the configuration of ceramic pellets vacuum cast in an Al-Si alloy (Heat Number 14). These photographs show that the simulated waste pellets are entirely encapsulated by the Al-Si matrix and that there are no detrimental voids or inclusions in the metal matrix. Figures 20A, 21A, 22A and 23A are photographs of the contact surface between the waste-matrix mixture and the inner container wall. The diameters of the exposed waste particles are an indication of the degree of infiltration. Figures 20 and 23 are of vacuum-cast, metal matrix, whereas Figures 21 and 22 are of gravity-cast metal matrix. Bonding of the matrix metals or the waste forms to the container did not occur. All container components were fabricated from 304L stainless steel. Figure 24 is a section of Figure 17 showing the infiltration by the capsule-immersion technique of waste-glass marbles with 12 wt% Si alloy. By limiting the preheat temperature of the glass marbles to 400^{o}C and instantaneously



WASTE FORM: SIMULATED WASTE-GLASS MARBLES
MATRIX: LEAD-10 WT% TIN ALLOY
HEAT NUMBER: 6

FIGURE 20. Vacuum-Cast Lead-Tin Matrix

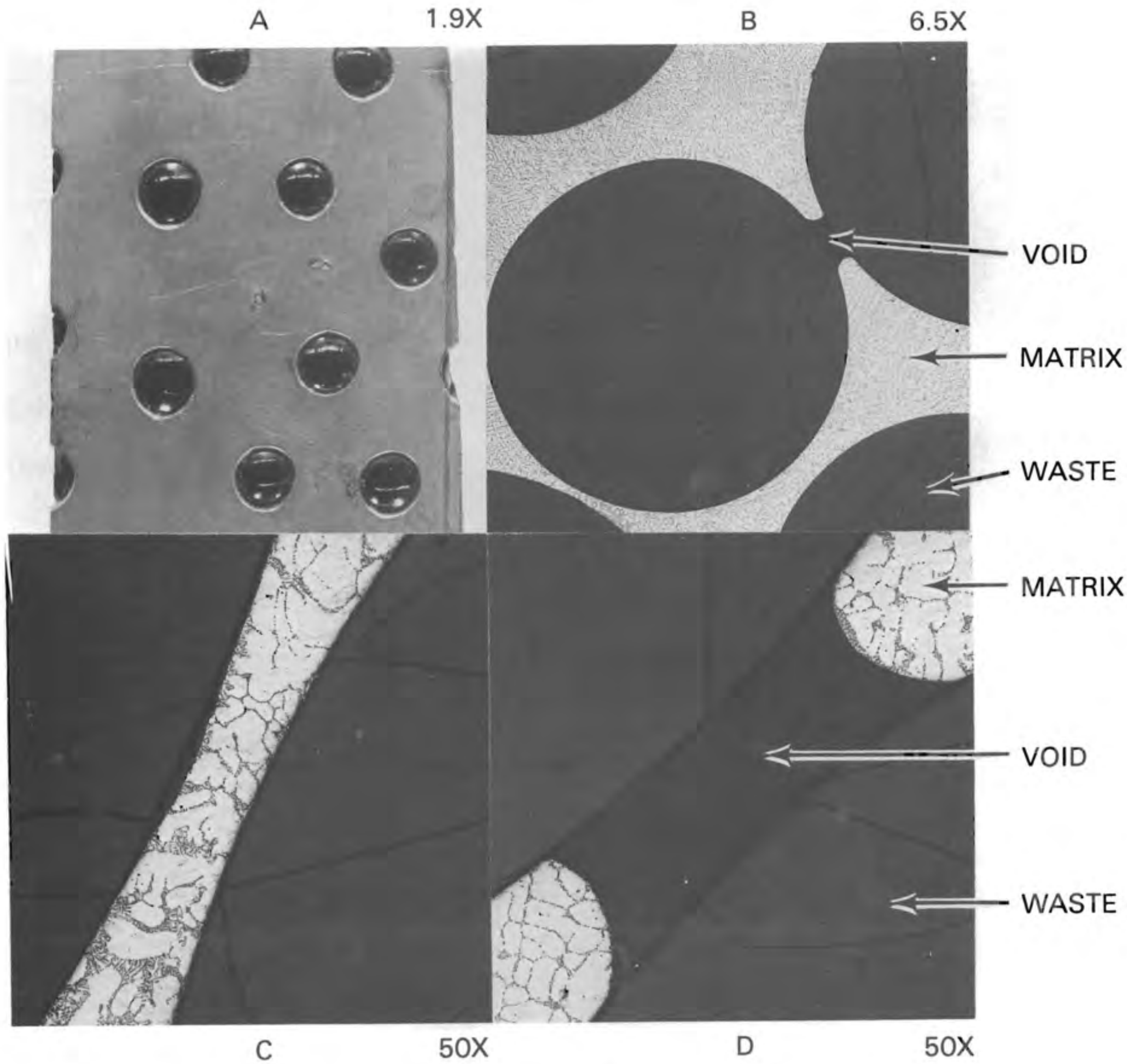
infiltrating the marble stack with 700^{0}C matrix metal, the distortion of the low softening point (∿500 to 550^{0}C) of the glass marbles is minimized. Occasional welding of the glass marbles to each other at their contact points is noted in Figure 17.



WASTE FORM: SIMULATED WASTE-GLASS MARBLES
MATRIX: LEAD-10 WT% TIN ALLOY
HEAT NUMBER: 7

FIGURE 21. Gravity-Cast Lead-Tin Matrix

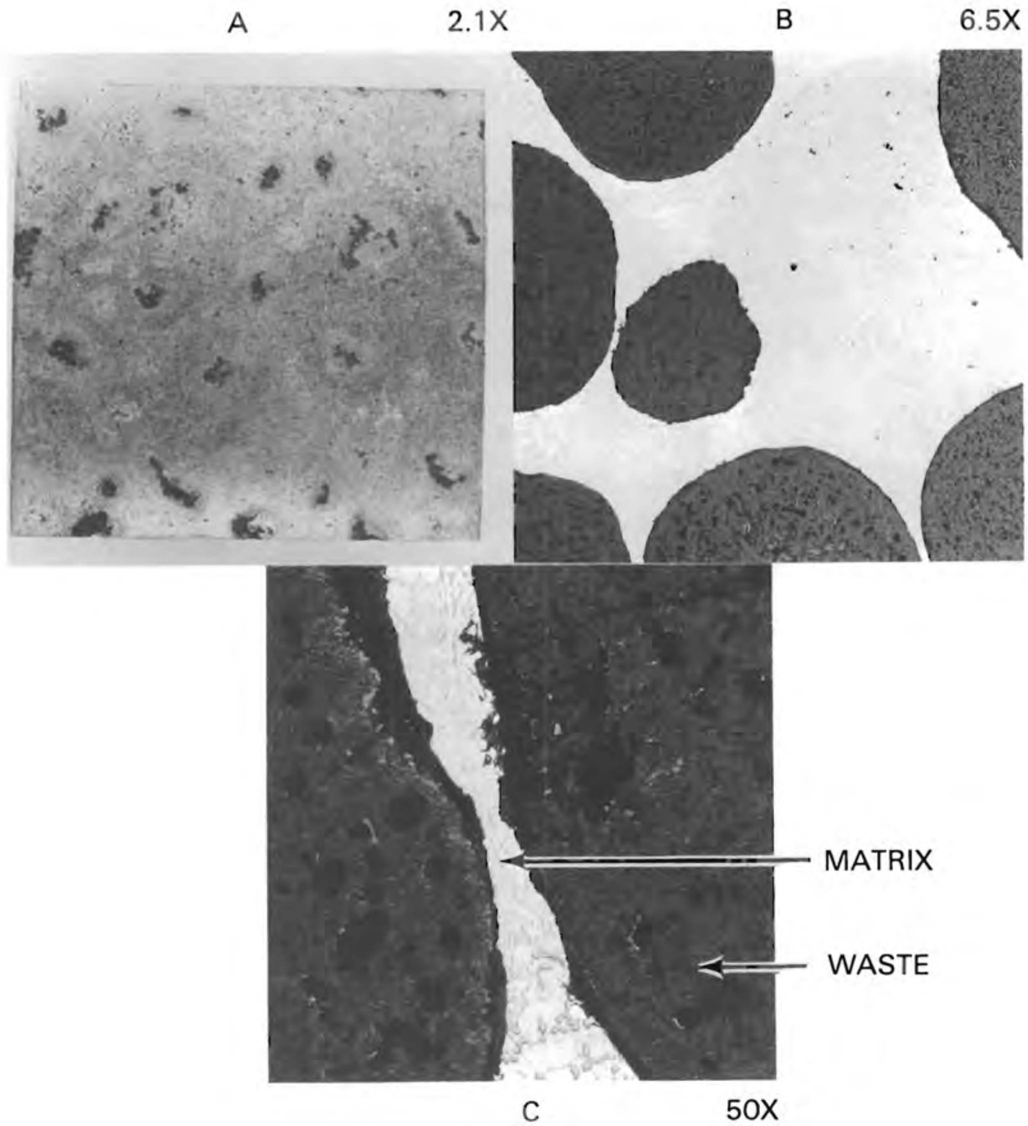
When Figures 20 through 23 are compared, it appears that vacuum-casting Al-12 wt% Si alloy with ceramic pellets offers the greatest degree of infiltration or encapsulation, although this has not been quantitatively determined.



WASTE FORM: BOROSILICATE GLASS MARBLES
MATRIX: LEAD-3 WT% ANTIMONY ALLOY
HEAT NUMBER: 16

FIGURE 22. Gravity-Cast Lead-Shot Feed

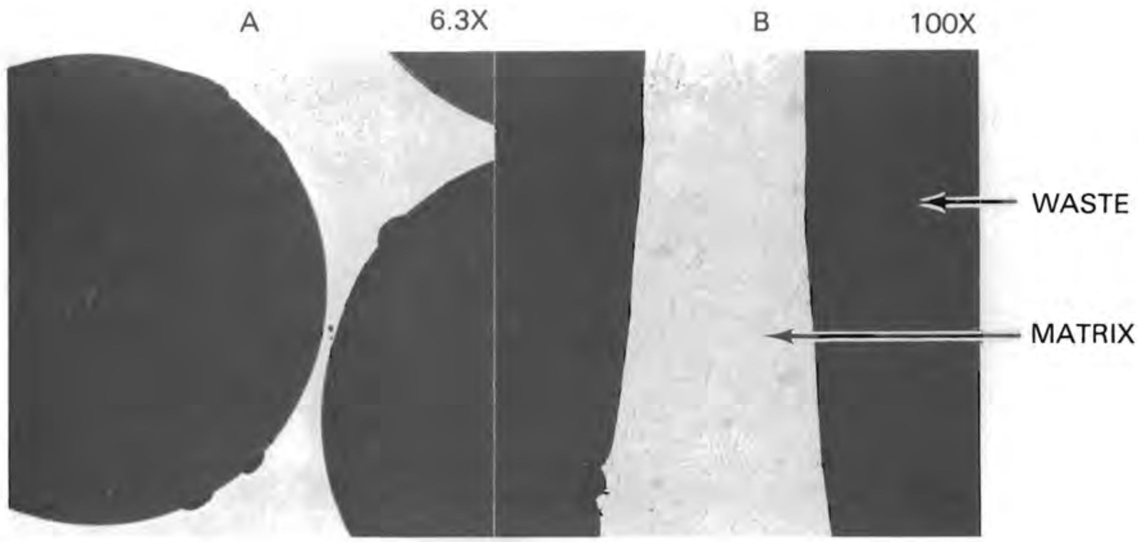
Figures 21 and 22 indicate that the Pb-3 wt% Sb alloy and the Pb-10 wt% Sn alloy have about the same degree of infiltration when each are cast by the gravity technique.



WASTE FORM: CERAMIC PELLETS
MATRIX: ALUMINUM 12 WT% SILICON ALLOY
HEAT NUMBER: 14

FIGURE 23. Vacuum-Cast Aluminum-Silicon Matrix

It is planned to perform an impact test on the full-scale canister (40 cm dia x 213 cm long) (Figure 11) and to evaluate it and its contents. After completion of the impact test, cross-sectional cuts will be made in this



WASTE FORM: SIMULATED WASTE-GLASS MARBLES
MATRIX: ALUMINUM 12 WT% SILICON ALLOY
HEAT NUMBER: 8

FIGURE 24. Vacuum-Immersion Infiltrated Capsule--Marbles

canister to allow evaluation of the metal-matrix infiltration of the marble stack. Also, it is planned to remove small sections of the test product for detailed examination and evaluation of the metal matrix and waste forms, and of their integrity and leach resistance.

DISCUSSION

The vacuum-casting method forces the matrix metal deeper into the interstices of the waste-particle stack than does gravity casting. This increases the contact area of the waste particles in relation to the matrix metal, which probably results in a higher heat-carrying capacity of the infiltrated waste form and which may increase its leach resistance. The integrity of the matrix-metal annulus surrounding the inner can of encapsulated waste remains the same with either vacuum or gravity casting.

Borosilicate glass marbles with a softening point of $\sim$500 to 550^{o}C can be successfully infiltrated with the Al-12 wt% Si alloy at 700^{o}C by vacuum-immersion techniques, if the marble preheat temperature is limited to 400^{o}C. However, during the tests reported here, an occasional marble-to-marble weld was noted. If large-scale work is anticipated using these materials, tests should be performed to determine the optimum preheat time, temperature and loading geometry to eliminate or minimize marble agglomeration during preheat.

The vacuum casting of the Al-12 wt% Si alloy was successful with the 2- to 5-mm (Heat Number 13) and 4- to 9-mm (Heat Number 14)-dia ceramic pellets. Capsule-immersion techniques were successful with 2- to 4-mm-dia ceramic pellets using vacuum but failed with 4- to 5-mm-dia pellets using gravity (Heat Number 8). Therefore, the lower ceramic particle-size limit for successful vacuum infiltration is $<$2 to 4 mm dia; for gravity casting, this limit is between 4 and 10 mm dia. While there are indications that the vacuum-cast Al-12 wt% Si alloy may have the greatest infiltration characteristics, its selection and use as a matrix material will depend on its ability to contain the radionuclides.

Gravity casting is the simplest process to perform remotely. In either gravity casting or vacuum casting, the matrix metal could be melted outside the cell and brought into the cell in pouring ladles, or piped into the cell. The ICE version of gravity casting eliminates the need to bring molten metal into the remote cell.

For vacuum pouring, this investigation used a fusion disk that was melted by the incoming molten metal. Alternative methods should also be considered. These could include a freeze plug that would be melted by an auxiliary heater, or a valve, either of which would be directly coupled to a bottom-pouring ladle.

The tests showed how much time is required to preheat, by conduction, the glass marbles or the ceramic pellets and their containers. In a process involving actual HLW, the preheating requirement would be reduced or even eliminated due to the decay heat of the waste forms.

The ICE tests indicated that the lead-fill rate was very conservative and that this could be increased with either large (8-mm) or small (2-mm)-dia lead shot. Although up to 2.5 cm of dross was on the top of the test canisters, this did not appear to affect the melting or flowing of the lead shot.

The laboratory- and engineering-scale demonstrations confirmed that a water quench on the bottom of the canister is an effective method to cool the mass and to control shrinkage voids. Preliminary results indicated that forced-air cooling was satisfactory on the full-scale tests.

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APPENDIX

CASTING MATERIALS AND COMPOSITION OF SIMULATED WASTE FORMS

CASTING MATERIALS AND COMPOSITION OF SIMULATED WASTE FORMS

TABLE A.1. Melt Stock

A. Lead

- Pure lead pig.
- Pure lead pig corroding grade in compliance with ASTM B 29-55.
- 90 wt% Pb-10 wt% Sn solder alloy pig, Grade 10B in compliance with ASTM B32-76.
- Lead Shot Number 4: 3.3 mm (0.13 in.) dia chilled in accordance with American standards for chilled shot and containing these elements in wt%:

Elements	Weight Percent
Sb	2.05
Sn	0.20
As	0.11
Cu	0.033
Fe	<0.001
Ni	<0.001
Bi	0.013
Ag	0.002
Zn	<0.001

B. Aluminum-12 Wt% Silicon

- Aluminum-12 wt% silicon: die-cast alloy pig, Grade A 413.2 in compliance with ANSI/ASTM B 179-78 and containing these elements in wt%:

Elements	Weight Percent
Cu	0.02
Fe	0.30
Mg	0.02
Mn	0.01
Si	12.0
Zn	0.03
Cr	0.02
Ni	0.01
Ti	0.01

TABLE A.2. Composition of Simulated Waste-Glass Marbles

Composition: Oxide Form	Composition: wt%	Density as Determined by Water Immersion
Al_2O_3	7.52	2.82 g/c^3
B_2O_3	9.22	
BaO	0.83	
CaO	4.01	
Ce_2O_3	0.83	
Dy_2O_3	0.16	
Fe_2O_3	5.48	
La_2O_3	0.52	
Li_2O	5.76	
MgO	0.66	
MnO_2	1.85	
MoO_3	1.19	
Na_2O	9.21	
Nd_2O_3	0.47	
NiO	0.57	
SiO_2	39.2	
SrO	0.73	
TiO_2	0.19	
ZnO	7.08	
Co_2O_3	0.08	
Cr_2O_3	0.12	
CuO	0.03	
Gd_2O_3	0.08	

TABLE A.3. Simulated-Waste Ceramic Pellets

Composition		Density as Determined by Water Immersion
Oxide Form	wt%	
AgO_2	0.12	3.57 g/c^3
BaO	2.14	
CdO	0.14	
Cr_2O_3	0.47	
K_2O	1.56	
Fe_2O_3	4.72	
MoO_3	8.71	
Na_2O	0.16	
NiO	1.43	
P_2O_5	3.75	
SrO	1.45	
ZrO_2	6.75	
CoO	0.39	
CeO_2	5.98	
M_2O_3	37.58[a]	
Al_2O_3	3.99[b]	
CaO	1.83[b]	
SiO_2	15.52[b]	
SrO	3.31[b]	

(a) Other rare earths
(b) Supercalcine additives

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