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COMPATIBILITY TESTING OF VITRIFIED WASTE FORMS

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## COMPATIBILITY TESTING OF VITRIFIED WASTE FORMS\*

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### INTRODUCTION

This paper describes an experimental program to evaluate candidate metals for use in the fabrication of canisters for long-term storage of vitrified radioactive wastes. The long term compatibility of the candidate metal both with the contained vitrified radioactive waste and with the external environments expected in possible final storage locations will be determined. These tests involve heating combinations of waste forms and canister metals in intimate contact for up to 50,000 hours to accelerate any reactions that occur.

Savannah River Laboratory (SRL) has previous experience with evaluating the compatibility of metals with radioactive waste fixed in cement and in glass and with salt from Carlsbad, NM.<sup>1</sup> More extensive compatibility tests of vitrified waste forms are necessary now to support the Defense Waste Solidification program, where one of the candidate processes is to immobilize waste in vitrified form.<sup>2</sup> The wastes presently exist as liquid, precipitated salts, and insoluble sludge in large carbon steel tanks. The reference vitrification process being developed consists of removing the wastes from the tanks, separating the majority of the radioactivity from the dissolved salts, and incorporating this radioactivity and the sludge into a glass form. The glass would be cast from a continuous melter into metal canisters. The alternative process involves melting the glass in the canister itself and allowing it to cool in place (in-can melting). The canister of glass will be seal-welded and placed inside another seal-welded canister (outer canister). Final storage locations in stable geological formations, such as salt deposits in New Mexico, are being considered.<sup>3-8</sup>

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## BACKGROUND

The Savannah River Laboratory began studies of the compatibility of candidate canister materials with solidified waste in 1974.<sup>1</sup> These studies, which are reviewed in the following paragraphs, involved long-term heating tests for times up to 40,000 hours to evaluate the compatibility between *Cor-Ten*\* A, Type 304L stainless steel, and *Inconel*\*\* 600 with both vitrified waste, and salt from a potential final storage location. The vitrified waste samples used in these tests contain 35 wt % of one of the simulated sludges described in Table 1 and 65 wt % of Glass Frit No. 18 (Table 2). (Glass Frit No. 21 is being used in compatibility tests described later in this paper.)

### Canister Materials

Three canister materials were chosen for testing based on their oxidation resistance and cost. *Cor-Ten* A is a low alloy steel containing 1% chromium, 0.5% nickel, and 0.35% copper. This alloy costs only a little more than low-carbon steel, but it has better atmospheric corrosion resistance. Type 304L stainless steel, which is more expensive than *Cor-Ten* A, has even better air-oxidation resistance. But Type 304L stainless steel might not be suitable for final storage in salt because of its susceptibility to chloride pitting and/or stress corrosion cracking. *Inconel* 600 has well known resistance to oxidation and creep, but the cost of this material is approximately twenty times that of *Cor-Ten* A.

### Cast Vitrified Waste

Test specimens were fabricated by methods developed in early SRL glass-making technology. Small metal containers were preheated at 500°C and filled with molten vitrified waste which was at 1150°C. The containers were returned to a furnace at 500°C and furnace cooled.

The results of 10,000-hour long-term heating tests show that vitrified waste and the candidate alloys are very compatible.<sup>3</sup> No detectible reactions were found in tests at 100°C, the waste temperature expected during final storage. And, no detectible reactions were found in tests made at 350°C to accelerate any reactions that might occur. The oxide film found on the inner surfaces of the container at both temperatures probably formed when capsules were fabricated and did not change significantly during heating.

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\* Trademark of U. S. Steel Company.

\*\* Trademark of International Nickel Company.

## In-Can Melted Vitrifified Waste

Preliminary in-can melting tests were made on small, mockup melters of *Cor-Ten* A and Type 304L stainless steel. Glass Frit No. 18 and Sludge TNX-3 or simulated Tank 13H sludges were used. The melters were heated to 1150°C for approximately three hours. In these tests, Type 304L stainless steel performed satisfactorily; it was penetrated to 0.02 mm (0.001 in.) by reaction with glass containing simulated Tank 13H sludge, and to 0.05 mm (0.002 in.) with glass containing Sludge TNX-3. *Cor-Ten* A was not satisfactory because it reacted to a depth of 1.4 mm (0.056 in.) with glass containing simulated Tank 13H sludge and to 2.4 mm (0.096 in.) with glass containing Sludge TNX-3.

## Carlsbad Salt

The compatibility between candidate metals with salt from a possible final storage location is being evaluated.<sup>4</sup> Test capsules of *Cor-Ten* A and Type 304L stainless steel were filled with crushed salt taken from Drill Hole AEC-8 in Carlsbad, New Mexico. The capsules were then covered, but not sealed, and placed in holes drilled in a solid piece of this salt. The entire assembly (salt block and capsule) was wrapped in aluminum foil in an attempt to confine any gases, such as hydrogen sulfide,\* that might be present in the salt and could influence the attack of metal. Capsules are being heated at 80°C and also at 225°C for up to 50,000 hours to accelerate any reactions. The maximum temperature of the tests is limited to 225°C to prevent salt decrepitation and expansion that would destroy the test assemblies.

Capsules heated for 1000 hours were easily removed from the holes in the block of salt and the powdered salt was poured from the capsules. A small amount of salt adhered to some places on the inside walls of the capsules. *Cor-Ten* A capsules were slightly discolored from oxidation on both the inside and the outside surfaces. The appearance of Type 304L stainless steel capsules was unchanged. No cracking or corrosion products were found, even where powdered salt adhered to the wall of the capsules.

## COMPATIBILITY TESTS TO SUPPORT THE DEFENSE WASTE SOLIDIFICATION PROJECT

On the basis of the results of previous tests a new series of tests will be started to evaluate the materials being considered for canister construction (Table 3). The long-term compatibility of these materials will be characterized with vitrified waste made with the most recently developed compositions of synthetic sludges and with actual sludges, and the oxidation resistance of the materials in air and their corrosion resistance in salt will be determined. All the vitrified waste in the new tests will be composed of 65 wt % Frit 21 and 35 wt % sludge. Frit 21 (see Table 2) contains 4 wt % Li<sub>2</sub>O to decrease the viscosity of the molten glass.

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\* A faint odor suggestive of hydrogen sulfide was detected when the salt was crushed; but hydrogen sulfide was not confirmed by analyses of the salt.

Both simulated and real sludges will be used. Composite sludge will be used for most tests because a blended sludge composite is proposed in the reference process.<sup>2</sup> Selected specimens containing high-aluminum simulated or equivalent real sludge will also be tested. High-aluminum sludge and composite sludge contains the least amount of total metallic elements. Previous in-can melting tests indicated that container materials were attacked more severely by vitrified sludge having the smallest amount of total metallic elements (Table 4).<sup>9</sup>

### Cast Vitrified Waste

Candidate alloys for canisters for the reference process (continuous melting) are low-carbon steel alloy ASTM A 333 (Grade 6) and Type 304L stainless steel. ASTM A 333 specification covers a low-carbon steel pipe suitable for use at low temperatures because of its low nil-ductility transition temperature (NDTT). This specification could be important if this steel is considered for use as the outer canister<sup>2</sup> and if low-temperature impact resistance is required, as in the case of shipping casks for radioactive material.<sup>10</sup> The bottom and top of the test specimens will be made from alloy ASTM A 516 (Grade 70). This alloy is the plate equivalent of ASTM A 333 pipe. Past experience indicates that low-carbon and low-alloy steels have similar compatibility with vitrified waste. Therefore, the corrosion behavior of these two alloys should be representative also of any other similar steels that might be selected.

Type 304L stainless steel is an attractive candidate for canister construction because of its oxidation resistance in air and demonstrated compatibility with vitrified waste. However, its susceptibility to chloride stress corrosion cracking may make it unacceptable for use as an outer canister (overpack) if final storage is in a salt mine; moreover, the slow cooling rate of a full-size waste canister or the extended heating during storage could cause sensitization of the Type 304L stainless steel, which could increase its susceptibility to chloride stress corrosion cracking.

### Test Conditions\*

Tests will be made at three different temperatures: 100°, 600°, and 800°C, according to the test matrix shown in Table 5. The longest test period will be at 100°C, the expected temperature of the actual waste canisters in storage. Specimens of as-cast glass and specimens of glass that has been heat-treated to cause devitrification will be tested. The compatibility of the canisters and the phases formed by devitrification will also be determined. Devitrification of companion glass specimens heat treated with the test specimens will be confirmed by x-ray diffraction analysis before the long-term heating tests are started.

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\* The temperature of 350°C used in previous tests will not be used in the new tests. This temperature originated from compatibility tests with wastes mixed in cement. It was the highest temperature at which cement specimens could be exposed because at 400°C the portlandite [Ca(OH)<sub>2</sub>] decomposes. This temperature was used in the previous tests for both cement and glass specimens so that the results could be compared.

Specimens of cast glass will be exposed at a higher temperature, 600°C, to accelerate any reactions that occur. Since this temperature is above the glass transition temperature, devitrification is expected to occur.

A small number of specimens will be heated at 800°C for 1000 hours. At this temperature the reaction rate will be greatly accelerated. Extent of reaction is expected to be appreciable.

### Test Specimens

Two types of specimens will be used with cast vitrified waste: 1) unsealed small metal containers and 2) sealed capsules. In both cases, the containers will be preheated to 500°C, then three-quarters-filled with molten glass waste at 1150°C.\* The containers then will be returned to a furnace at 600°C and furnace cooled. This cooling method is an attempt to simulate the slow cooling rate expected in a full-size canister of waste. The small unsealed metal containers will be made from pieces of mill-finished pipe with the bottom made from plate (Figure 1). The top cover plate will not be seal welded. The dimensions of the specimens will be measured before and after heating.

The sealed capsules (Figure 2) will consist of a small metal container similar to the unsealed specimen which will be contained in a sealed capsule made of pipe fittings (Figure 3). The capsules will be sealed by tungsten-inert-gas-welding. The purpose of sealed capsule tests is to evaluate possible effects of radiation-induced chemical changes of the air or gases given off by the vitrified waste in the free space in the canister.

After the test capsule is heated it will be sectioned and examined by optical and scanning electron microscopy. The condition of the portions of the metal not in contact with the vitrified waste but sealed in the vapor space will be especially noted.

### In-Can Melted Vitrified Waste

Compatibility tests of materials that might be used for in-can melting will be performed on ASTM A 333 and ASTM A 516 low-carbon steels, Type 304L stainless steel, and *Inconel* 601. These tests will be made only with simulated sludge having either the composite composition or high-aluminum content.

Small-scale in-can melting tests will be performed using specimens shown in Figure 4. The samples will contain full-penetration welds in the tube wall to evaluate the effects of the waste glass on weld metal. Melts will be made at 1050, 1100, and 1150°C for 3, 5, and 7 hours (Table 6). After the specified heating times, the canisters will be furnace cooled to room temperature over a period of ten to twelve hours. The overall dimensional changes, the changes in wall thickness due to both oxidation and reaction by the glass, any microstructural changes of the metal, and any changes in structure of the vitrified waste will be determined.

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\* Glasses containing simulated sludges will be melted in a crucible inside a furnace. Glasses containing actual sludges will be melted in a Joule heated melter.

Coupons of candidate metals (Figure 5 and Table 7) will also be exposed in ceramic crucibles to molten glass wastes at the same temperatures and times used for in-can melting tests discussed above.

### Carlsbad Salt Test

The compatibility of low-carbon steel ASTM A 516 and Type 304L stainless steel with salt from Drill Hole AEC-8, Carlsbad, New Mexico, will be characterized in sealed capsules. The metal specimens will be partially embedded in a block of salt (Figure 6), and then sealed inside a capsule made of pipe fittings.

Capsules will be heated at 80, 225, and 300°C (Table 8). The first two test temperatures will show differences between the results of the sealed tests and the unsealed tests run previously. Decrepitation of the salt heated at 300°C will occur, but this will not destroy the test assembly, as in the case of unsealed capsules. The salt, the metal specimen, and steam that is released will remain in contact with the specimen inside the capsule.

Heating specimens in sealed capsules is necessary to characterize reactions that may occur between the metals and any volatile components of the salt. Any water or hydrogen sulfide in the salt would have escaped from the first test specimens because they were not sealed. But these reactants will be present if final storage of canisters is in a salt mine.

Corrosion of both ASTM A 333 low-carbon steel and Type 304L stainless steel could occur in salt. If moisture in the salt migrated to the warm canister, caustic conditions might be produced. These conditions might cause caustic stress corrosion cracking of ASTM A 333 alloy steel. Moist chloride could also cause pitting or stress corrosion cracking of the Type 304L stainless steel.

### INITIAL RESULTS OF NEW TEST PROGRAM

Exposure of coupons of candidate metals to molten glass to characterize reactions that will occur during in-can melting has been completed.

### Experimental Procedure

Test assemblies (Figure 5) consisted of metal rods suspended in a crucible of molten vitrified waste by a ceramic top attached to the specimen. The end of the specimen outside the crucible was exposed to air oxidation as the outside of a waste canister would be during in-can melting. The bottom of the specimen was exposed to molten vitrified glass-waste representative of the exposure of the bottom inside of a canister during melting. The portion of the specimen inside the crucible between the molten glass and the ceramic top was exposed to both air oxidation and molten glass. This condition would exist near the top of a canister of glass-waste.

Test assemblies were similar to those used previously by G. G. Wicks (to be published) for compatibility tests of candidate refractories for furnace lining with molten vitrified waste. The metal specimens were sawed from plate. The rolled surfaces were not machined. Care was taken to avoid abraiding the mill-scale oxide films on these surfaces. Specimens were degreased and thickness was measured with a micrometer.

SRP composite sludge (Table 4) and Glass Frit 21 (Table 2) were weighed into batches estimated to fill the porcelain crucible half full of molten vitrified waste of the composition 35 wt % sludge, 65 wt % frit. Since the sludge contained  $U_3O_8$ , all operations where sludge could become airborne were performed inside a hood. Each batch of glass components was mixed in a blender to homogenize and pulverize the frit. This reduced the amount of outgassing during melting by eliminating the large, air-filled pores in the frit material.

Next, the ceramic top was cast around the specimen in the following manner: the specimen was press-fitted into an opening in the bottom of a *Styrofoam*\* cup. *Sauereisen*\*\* Cement No. 31 was poured into the cup to form a top of the desired thickness. After the cement had set, the cup was removed, and the assembly was dried overnight in a vacuum.

Specimens were exposed under the conditions shown in Table 7. The blended glass-mixture was put into a porcelain crucible, heated to 1100°C, and allowed to outgas for 15 to 20 minutes. When all boiling had stopped, the glass was heated to the test temperature. The crucible of glass was then removed from the furnace, the specimen quickly inserted, and the assembly returned to the furnace and heated for the required length of time. A small loss of time at temperature occurred because the specimen, originally at room temperature, cooled the molten glass. This time at reduced temperature was believed to be insignificant, and therefore, was neglected.

After the assembly had been heated for the desired length of time, it was removed from the furnace. The metal specimen with the top attached was quickly lifted from the crucible of glass. There was a tendency for the *Sauereisen* cement to adhere to the crucible, so some of the tops were broken during disassembly. The metal specimen and the crucible of glass were allowed to cool to room temperature on a fire brick. Some spalling of glass and oxide occurred from the specimen during cooling. Spalling was less from *Inconel* 601 specimens than from low-carbon steel or Type 304L stainless steel specimens, probably because less differential thermal expansion occurs between *Inconel* 601 and the glass or because there was a better metal-glass bond in these specimens.

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\* Trademark of Dow Chemical Co.

\*\* Trademark of Sauereisen Cement Co.

The specimens were evaluated by thickness measurements and by microscopic examination of metallographic sections. First, the specimen with the top attached was photographed. *Epo-Kwick*\* resin was then poured over the specimen to hold the oxide, glass, and metal in position. After the resin hardened, the *Sauereisen* top was broken off, and the entire specimen was mounted in *Kold-Weld*\*\*. The specimen was ground and polished to show the effect of the exposure of the rolled surfaces.

The thicknesses of heated specimens were measured with a traveling-stage microscope. The thickness was measured every 0.100 in. along its length. These measurements were grouped into areas according to exposure (to glass, air, and air plus glass). This grouping was based on measurements taken from the overall photograph of the specimen after it was heated. Average dimensional changes for each of the three exposure areas were calculated for each specimen. Each specimen was examined at higher magnifications to measure the depth of any intergranular penetration. This value was added to the dimensional change in each specimen to give the total penetration.

## Discussion of Results

The total penetration vs. time of exposure is plotted in Figure 7. The data in Figure 7 show that extent of penetration was greatest in low-carbon steel, less in *Inconel* 601, and least in Type 304 stainless steel. In all three metals tested, the extent of penetration by molten glass was less than the extent of penetration by air oxidation (Figures 8, 9, and 10). Apparently molten glass protects the metal from oxidation more than the metal oxide film does. Also, the area exposed to both air and molten glass was penetrated more than the area exposed to air alone. There is a simple explanation for this. Metal oxide is relatively soluble in molten glass. If the oxide film was thick enough, the glass tended to be drawn into the metal oxide film and over the specimen, as was most evident in the low-carbon steel specimens (Figure 8). The presence of glass in the metal oxide film evidently reduced the film's ability to protect the metal from oxidation. In the *Inconel* 601 specimen extensive intergranular penetration occurred (Figure 10).

The data plotted in Figure 7, suggest that penetration proceeds according to the parabolic rate law, although the data from some specimens more closely follows the law than do data from other specimens. The data from all areas of the low-carbon steel specimens fell very closely to straight lines on the log plot, indicating a parabolic rate. Data for Type 304L stainless steel specimens showed much scatter, probably because the

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\* Trademark of Buehler Ltd.

\*\* Trademark of Precision Dental Manufacturing Co.

dimensional changes were so small (Figure 9). The positions of the lines indicating a parabolic increase in penetration for this material were therefore estimated. Penetration data for the *Inconel* 601 specimens followed the parabolic rate law more closely than did the data from the Type 304L stainless steel specimens, but not as closely as the data from the low-carbon steel specimens.

No significant effect of the composition of the vitrified waste was found. Most of the specimens were made with glass containing composite vitrified waste. However, a few overlapping tests were made with glass containing high-aluminum sludge. These two glasses were expected to have similar compatibility behavior because they have about the same amount of total metallic elements.

The total penetration in five hours vs.  $1/T$  is plotted in Figure 11. This plot can be used to predict the amount of penetration that could occur at conditions other than those tested.

## SUMMARY

The compatibility of vitrified radioactive waste with candidate canister materials will be evaluated with both cast and in-can melted vitrified waste. Both real and simulated sludges will be used. In addition, the compatibility of these materials with salt from a possible final storage location will be determined. Planned experiments are summarized in Table 5, 6, 7, and 8.

Cast vitrified waste will be tested with ASTM A 333 and ASTM A 516 low-carbon steels and Type 304L stainless steel at 100, 600, and 800°C. Cast vitrified waste that has been devitrified by heat treatment will be tested at 100°C. Two types of test specimens will be used with either simulated or real sludges: 1) unsealed capsules (Figure 1) made of pieces of mill-finished pipe into which vitrified waste is cast, and 2) sealed capsules (Figures 2 and 3) containing a small container of vitrified waste identical to the ones in the unsealed capsule.

In-can melted vitrified waste will be tested for synthetic sludge only with ASTM A 333 and ASTM A 516 low-carbon steels, Type 304L stainless steel and *Inconel* 601. Two types of tests will be carried out: 1) melting vitrified waste in miniature metal canisters and 2) exposure of small (carefully measured) metal coupons to molten glass (Figures 4 and 5).

The air oxidation rates of candidate canister materials will be determined, and specimens will also be exposed to salt from Drill Hole AEC-8 in Carlsbad, New Mexico. Sealed capsules containing an ASTM A 516 low-carbon steel or Type 304L stainless specimen partially embedded in a small block of salt will be heated (Figure 6).

The coupon tests with in-can melted glass have been completed. Type 304L stainless steel performed satisfactorily by exhibiting greater resistance to attack by molten glass-air environments than the other metals tested. The stainless steel was penetrated only 0.13 mm (0.005 in.) when exposed to molten glass containing composite or high-aluminum sludge at 1150°C for 7 hours. The low-carbon steel and *Inconel* 601 tested under identical conditions did not perform satisfactorily. The large amount of penetration [1.65 mm (0.065 in.)] which occurred in low-carbon steel was expected. However, the large amount of penetration [0.68 mm (0.027 in.), mostly on grain boundaries] of the *Inconel* 601 was not expected. Most severe penetration in all metals occurred where both air and glass contacted the surface. The penetration of all metals seems to obey the parabolic rate law.

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## FIGURE CAPTIONS

- FIGURE 1. Unsealed Small Metal Container
- FIGURE 2. Small Metal Container for Sealed Capsule Test
- FIGURE 3. Sealed Capsule Assembly
- FIGURE 4. Test Specimen for Small-Scale In-Can Melting
- FIGURE 5. Coupon Test Specimens
- FIGURE 6. Sealed Carlsbad Salt Assembly
- FIGURE 7. Total Penetration vs. Time
- FIGURE 8. Typical Low-Carbon Steel Specimen
- FIGURE 9. Typical Type 304L Stainless Steel Specimen
- FIGURE 10. Typical *Inconel* 601 Specimen
- FIGURE 11. Total Penetration in Five Hours Exposure vs.  $1/T$

TABLE 1

## Composition of Early SRP Simulated Sludges

<i>Element</i>	<i>Amount in Simulated Sludge, wt %</i>		
	<i>TNX-1</i>	<i>TNX-3</i>	<i>Tank 13H</i>
Fe	32.9	23.4	49.7
Al	15.2	-	5.1
Sr	0.04	0.04	
Hg	-		-
Mn		23.56	18.9
Na	1.08	2.26	
K	0.05	1.09	
Ni	-	-	5.0
Ca			3.4

TABLE 2

## Composition of Glass Frits

<i>Metal Oxide</i>	<i>Amount in Frit, wt %</i>	
	<i>18</i>	<i>21</i>
SiO <sub>2</sub>	52.5	52.5
Na <sub>2</sub> O	22.5	18.5
B <sub>2</sub> O <sub>3</sub>	10.0	10.0
TiO <sub>2</sub>	10.0	10.0
CaO	5.0	5.0
Li <sub>2</sub> O	-	4.0

TABLE 3

## Cost of Candidate Canister Materials

<i>Alloy</i>	<i>Cost, \$<sup>a</sup></i>	<i>Remarks</i>
A 53	89	Mild steel, cheapest material for steel pipe.
A 106	98	Mild steel, better quality than A 53.
USS <i>Cor-Ten</i> (A618-1)	115	Low-alloy steel, increased atmospheric corrosion resistance.
A 333-1 or 6	164	Mild steel, improved low-temperature impact resistance.
Type 304L	554	Stainless steel, good oxidation resistance and demonstrated compatibility with vitrified waste.
<i>Inconel</i> 601	2109	Nickel alloy, excellent oxidation resistance.
<i>Inconel</i> 600	2335	Nickel alloy, excellent oxidation resistance.

a. Approximate cost per meter of 61 cm (24 in.) dia × 1.1 cm (0.438 in.) wall pipe.

TABLE 4

## Composition of SRP Sludges

<i>Element</i>	<i>Amount in Sludge, wt %</i>			
	<i>High Fe</i>	<i>High Al</i>	<i>Av.</i>	<i>Composite</i>
Fe	32.8	3.1	22.0	14.0
Al	2.3	33.5	9.9	15.5
Mn	2.0	2.3	5.4	4.1
U	9.2	0.9	6.7	3.3
Ca	2.3	0.2	1.6	1.5
Ni	<u>6.3</u>	<u>0.5</u>	<u>2.8</u>	<u>1.1</u>
Total Metallic Elements	54.9	40.5	48.4	39.5

TABLE 5

## Test Matrix - Cast Glass

Material	Temp, °C	Glass	Time, hr				
			1,000	5,000	10,000	20,000	50,000
Type 304L Stainless Steel	100	As-Cast			x	x	x
	600	As-Cast	x	x	x <sup>a</sup>	x	x
	800	As-Cast	x <sup>a</sup>				
	100	Devitrified <sup>b</sup>			x	x	x
Alloys A 333 and A 516	100	As-Cast			x	x	x
	600	As-Cast	x	x	x <sup>a</sup>	x	x
	800	As-Cast	x <sup>a</sup>				
	100	Devitrified <sup>b</sup>			x	x	x

a. Additional specimens tested at this condition will contain high-aluminum actual sludge or equivalent.

b. Devitrified by heat treatment prior to exposure.

TABLE 6

## Small-Scale, In-Can Melting Tests

Material	Temp, °C	Time, hr		
		3	5	7
Type 304L Stainless Steel	1050	x	x	x
	1100	x	x <sup>a</sup>	x
	1150	x	x	x
Alloys A 333 and A 516	1050	x	x	x
	1100	x	x <sup>a</sup>	x
	1150	x	x	x
Inconel 601	1050	x	x	x
	1100	x	x <sup>a</sup>	x
	1150	x	x	x

a. Additional specimens tested at this condition will contain high-aluminum synthetic sludge.

TABLE 7

## Test Matrix - Coupon Tests

Material	Temp, °C	Time, hr		
		3	5	7
Type 304L Stainless Steel	1050	x	x	x
	1100	x	x <sup>a</sup>	x
	1150	x	x	x
A 516	1050	x	x	x
	1100	x	x <sup>a</sup>	x
	1150	x	x	x
Inconel 601	1050	x	x	x
	1100	x	x <sup>a</sup>	x
	1150	x	x	x

α. Additional specimens will be tested at this condition with high-aluminum synthetic sludge.

TABLE 8

## Test Matrix - Sealed Carlsbad Salt Test

Material	Temp, °C	Time, hr.				
		1,000	5,000	10,000	20,000	50,000
Type 304L Stainless Steel	80	x	x	x	x	x
	225	x	x	x	x	x
	300	x	x	x	x	x
A 516 Alloy Steel	80	x	x	x	x	x
	225	x	x	x	x	x
	300	x	x	x	x	x

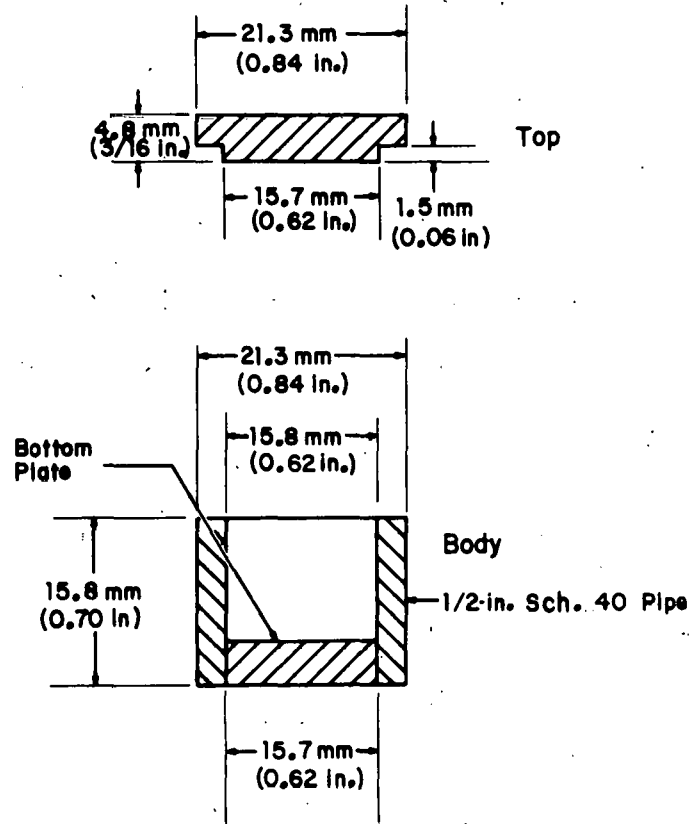


FIGURE 1. Unsealed Small Metal Container

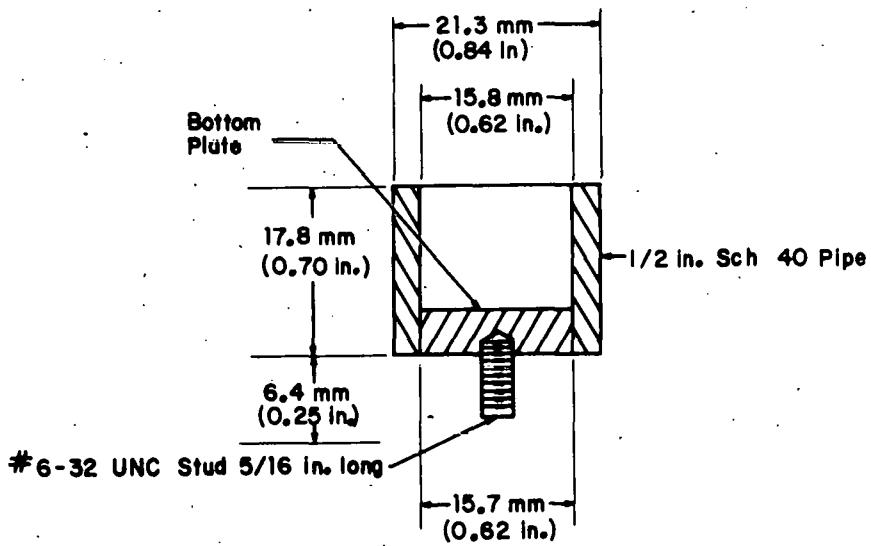


FIGURE 2. Small Metal Container for Sealed Capsule Test

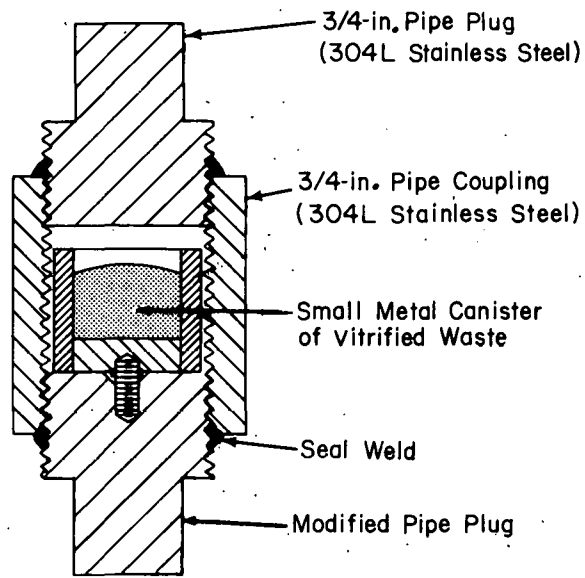


FIGURE 3. Sealed Capsule Assembly

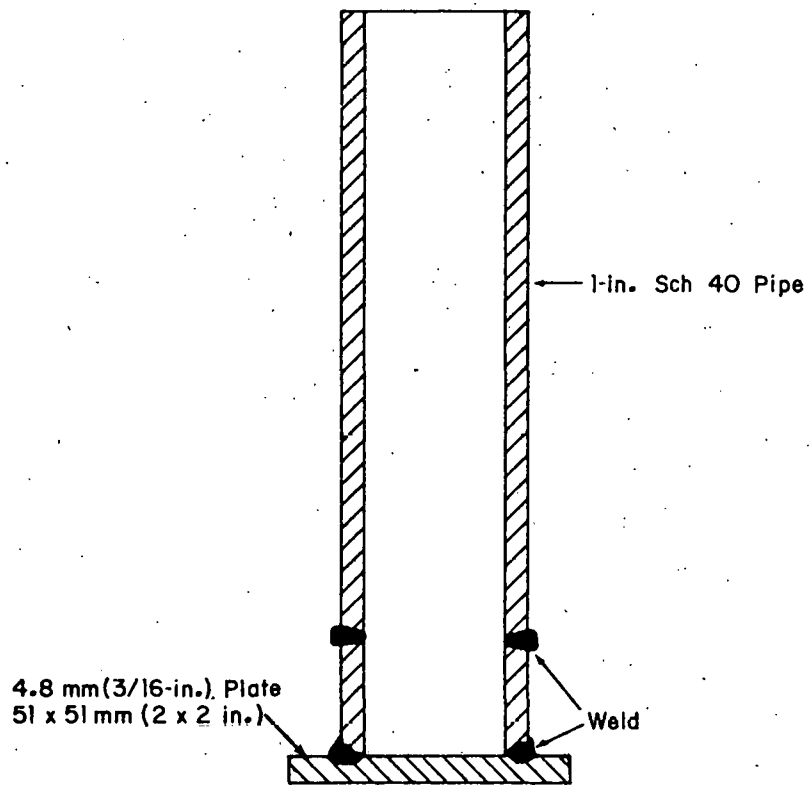


FIGURE 4. Test Specimen for Small-Scale In-Can Melting

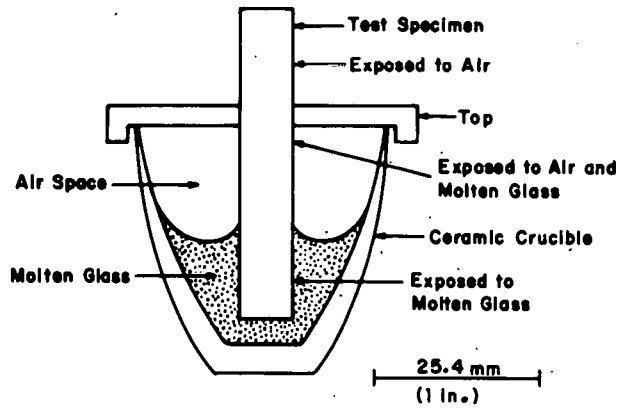


FIGURE 5. Coupon Test Specimens

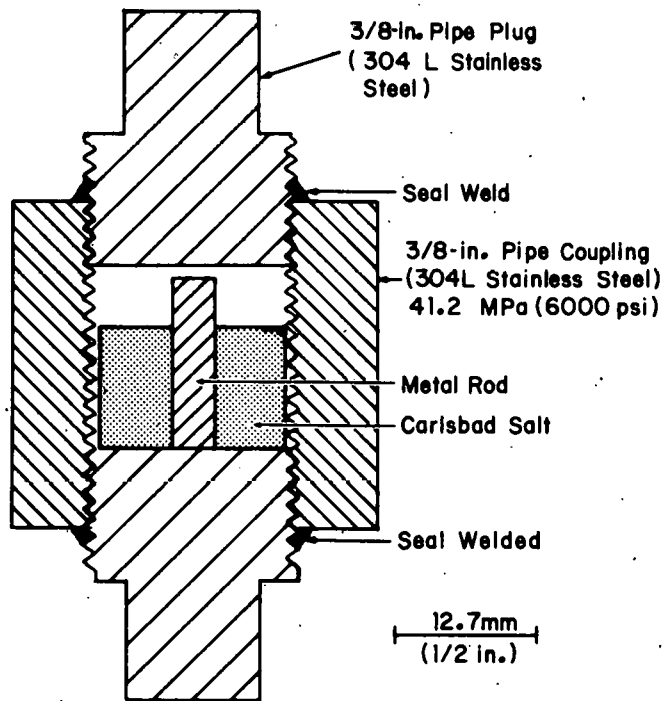


FIGURE 6. Sealed Carlsbad Salt Assembly

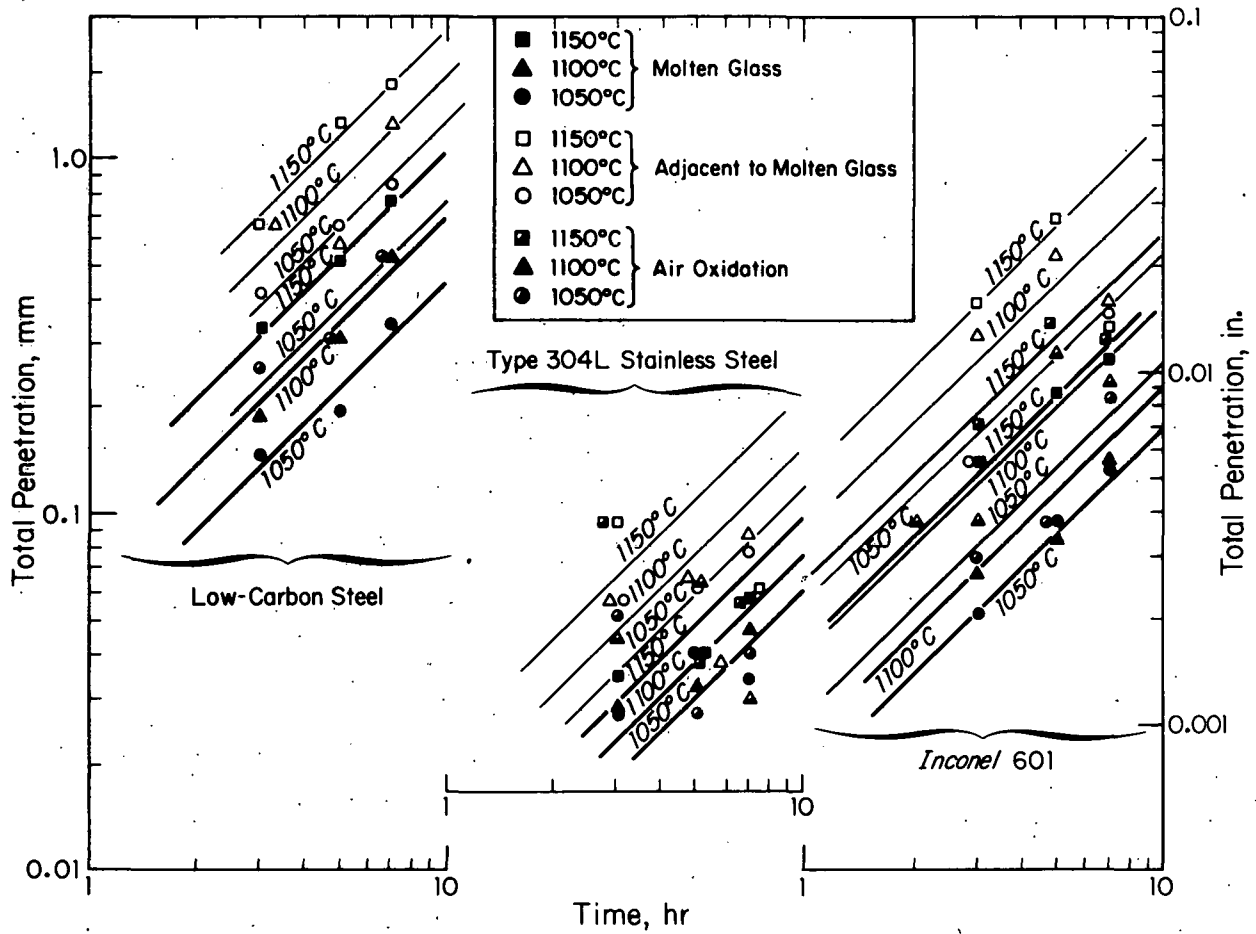


FIGURE 7. Total Penetration vs. Time

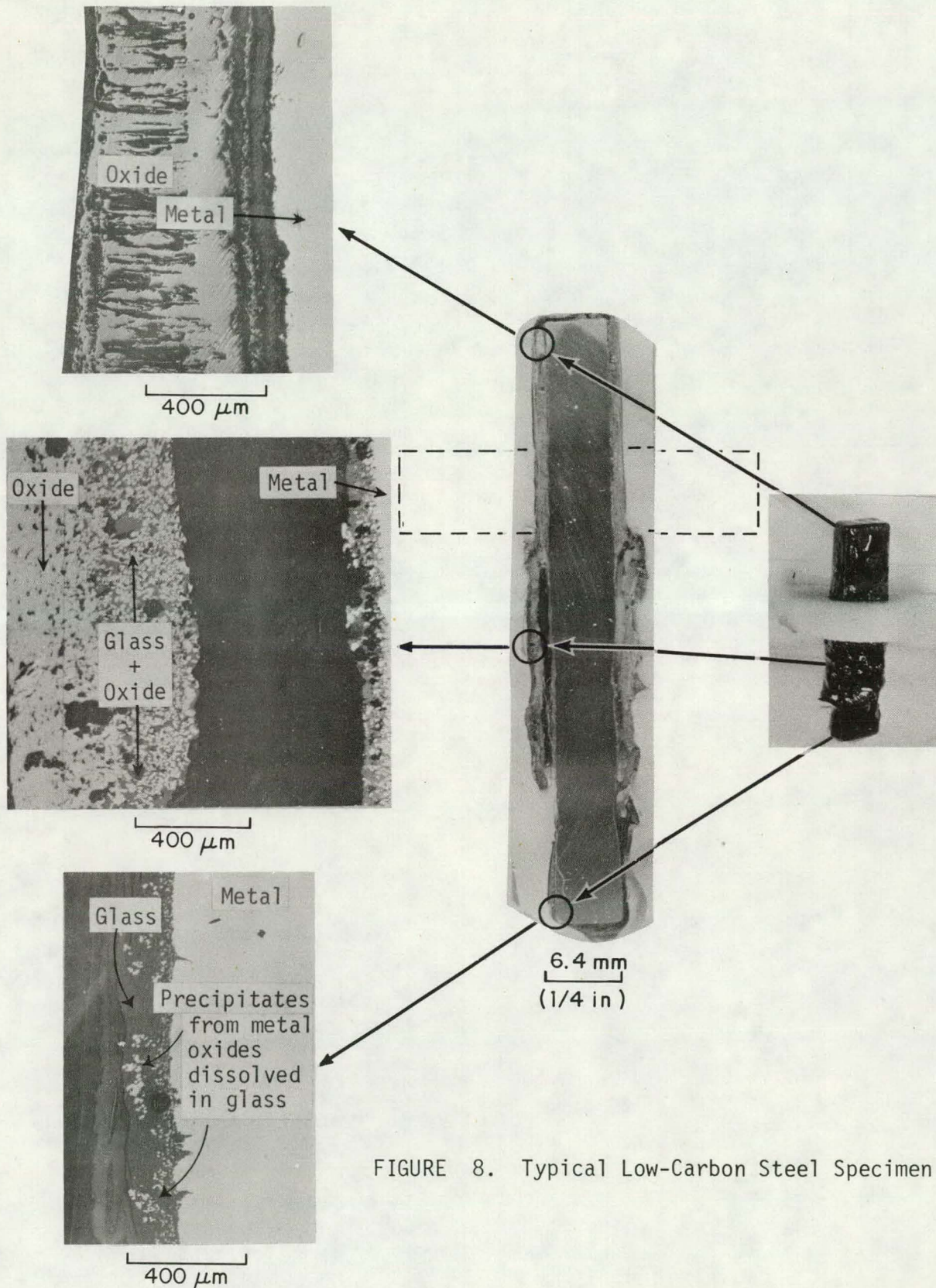


FIGURE 8. Typical Low-Carbon Steel Specimen

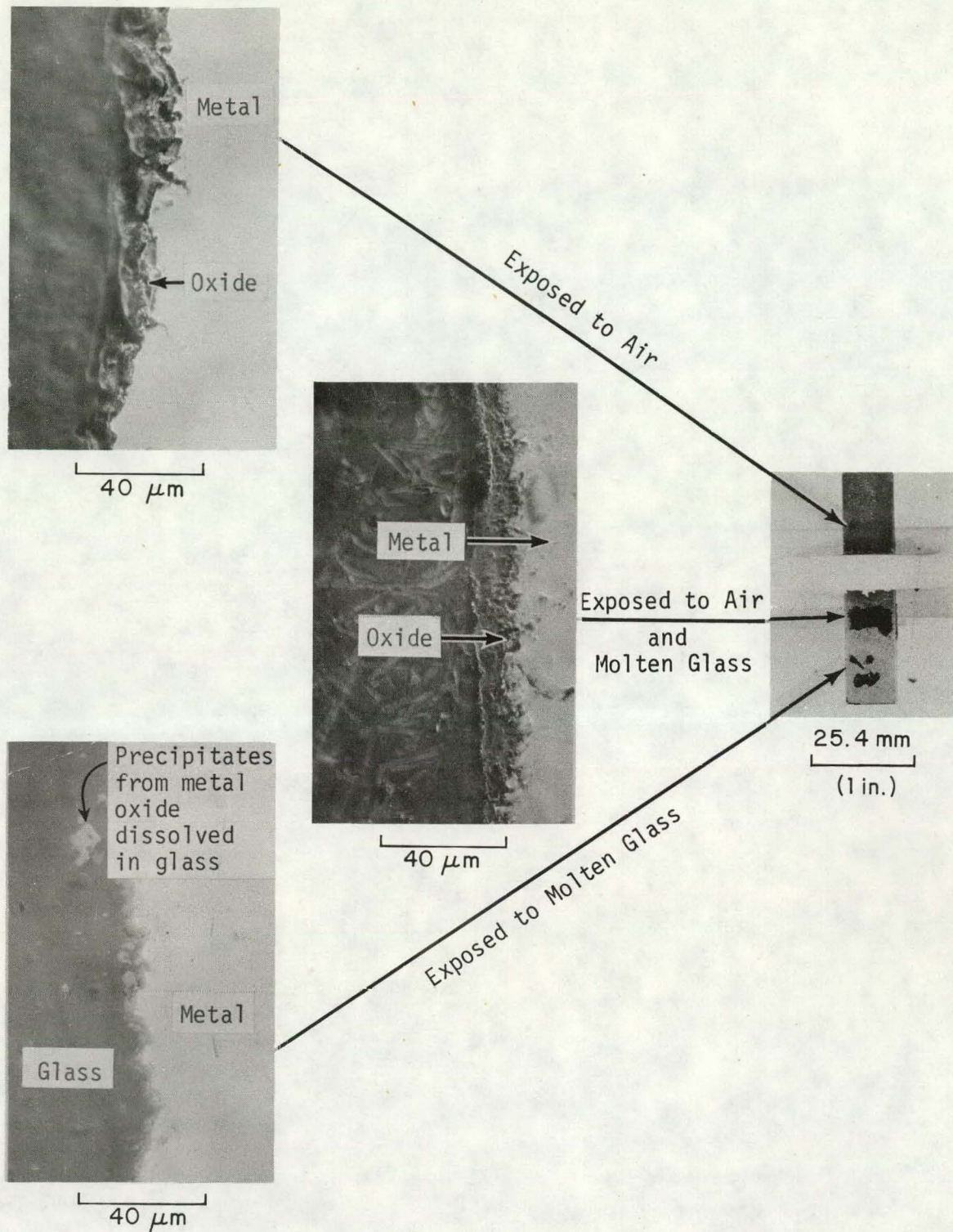


FIGURE 9. Typical Type 304L Stainless Steel Specimen

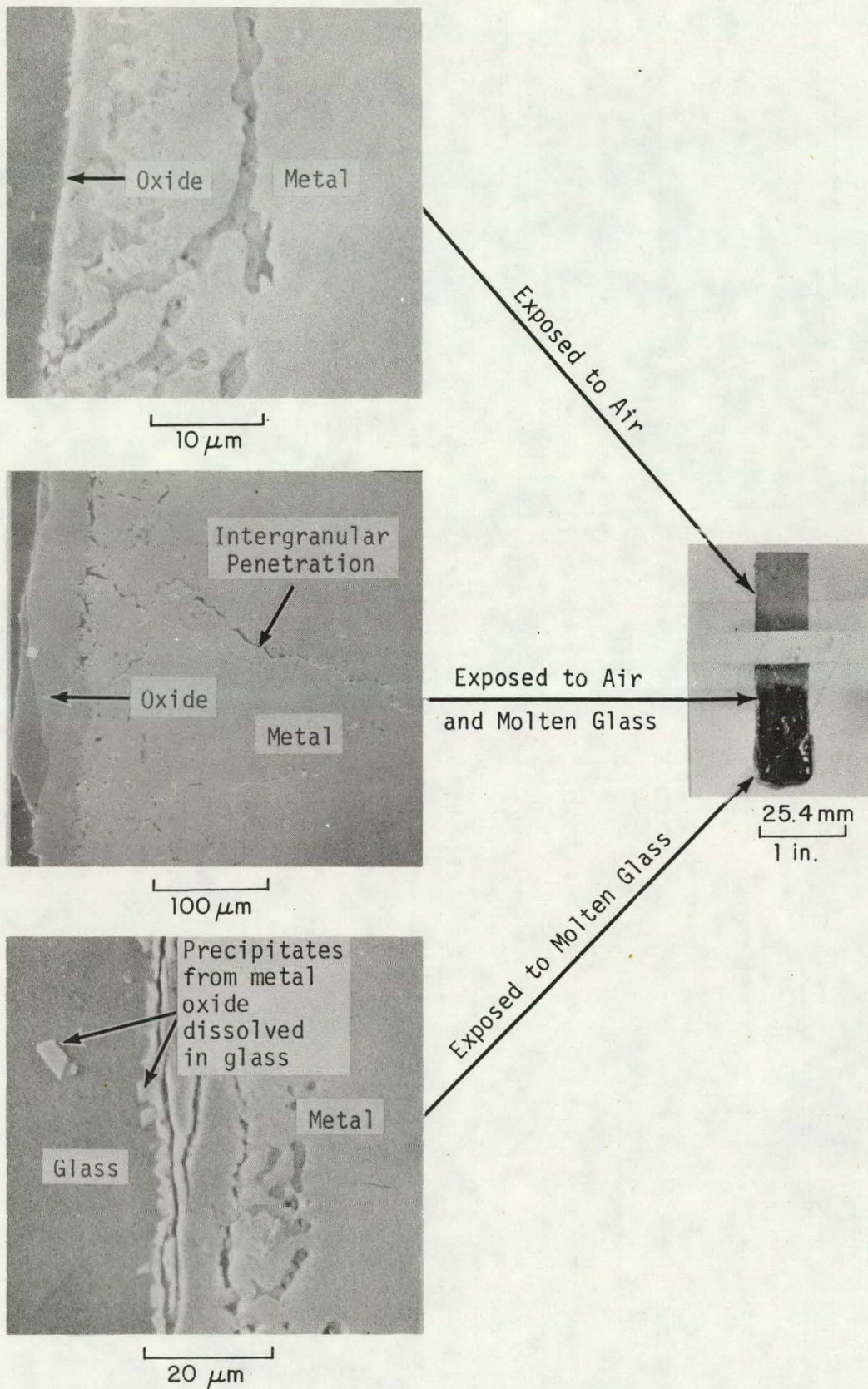


FIGURE 10. Typical *Inconel* 601 Specimen

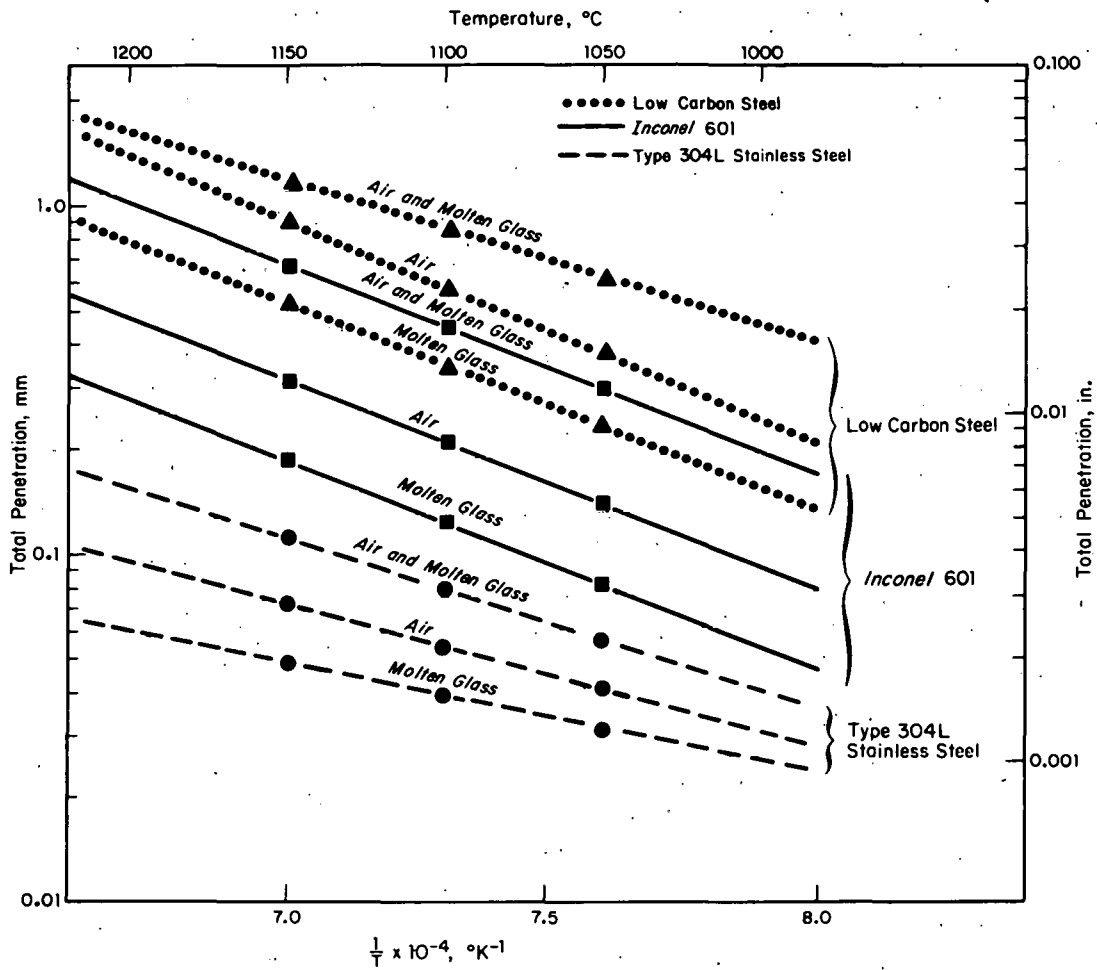


FIGURE 11. Total Penetration in Five Hours Exposure vs. 1/T