

ATTACK OF HIGH-STRENGTH, OXIDATION-RESISTANT ALLOYS  
DURING IN-CAN MELTING OF SIMULATED WASTE GLASSES

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To be presented by  
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Proposed for Presentation at  
Corrosion/80 NACE Meeting,  
Chicago, Illinois,  
March 6-10, 1980

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INTRODUCTION

This paper describes the results of tests that evaluated the compatibility of high-strength, oxidation-resistant alloys with melts of Savannah River Plant (SRP) waste and glass-forming additives under in-can melting conditions. They were carried out to evaluate high temperature alternatives to Type 304L stainless steel for use as in-can melting canisters. The compatibility of candidate canister alloys with simulated waste glass during both waste-form fabrication and during conditions expected in long-term storage is being characterized.<sup>1,2</sup>

Two processes for incorporating high-level radioactive waste into glass waste forms are being developed at Savannah River Laboratory (SRL). In the primary process, continuous melting, glass frit and calcined sludge are melted in a joule-heated melter and the glass is then cast into the canister. In the backup process, in-can melting, frit and calcined sludge are melted in the canister and allowed to solidify in place.

Reactions between simulated waste glass and canister alloy during in-can melting have a greater effect on the canister than reactions for continuous melting, because in-can melted waste forms are at higher temperatures for a longer time. Reactions under these conditions must be characterized in order to select the canister alloy and establish the fabrication parameters that will ensure the production of high-integrity waste forms.

\* The information contained in this article was developed during the course of work under Contract No. DE-AC09-76SR00001 with the U. S. Department of Energy.

## SUMMARY

The resistance of candidate canister alloys to penetration under the most severe conditions expected during in-can melting was directly proportional to the chromium content of the alloy, and inversely proportional to the  $\text{Na}_2\text{O}$  content of the glass melt. Specimens were exposed for 24 hours, which is the time required for in-can melting full-size waste-glass forms based on tests carried out at Pacific Northwest Laboratories (PNL) and at SRL.

The penetration resistance to Frit 211 (Table 1) at  $1150^\circ\text{C}$  for 24 hours of most alloys tested was satisfactory. The amount of penetration would not affect the integrity of the waste form.

*Inconel*\* 625, *Hastelloy*\*\* X, and *Inconel* 601 were penetrated <20 mils. This was considered excellent.

*Incoloy*\* 801, Type 310 stainless steel, Type 304L stainless steel, *Inconel* 600, and Type 347 stainless steel were penetrated <40 mils. This was considered good.

*Hastelloy* C-4 was penetrated >100 mils by a glass composed of 65 wt % Frit 21 and 35 wt % composite sludge (with uranium) at  $1150^\circ\text{C}$  for only 7 hours. This amount of penetration of an in-can melting canister would not be satisfactory.

## BACKGROUND

One series of coupon tests was completed previously.<sup>1</sup> In these tests three alloys (low-carbon steel, Type 304L stainless steel, and *Inconel* 601) were exposed to Frit 21 base-glass containing composite or high-aluminum sludge (Table 2) at up to  $1150^\circ\text{C}$  for up to 7 hours.

Alloys were selected for testing because of their oxidation resistance, cost, and high-temperature strength. Type 304L stainless steel has good compatibility with glass-waste and may have sufficient strength if the in-can melting temperature is  $\leq 1050^\circ\text{C}$ . *Inconel* 601 was chosen as a high-temperature alternative to Type 304L stainless steel because it is more resistant to oxidation in air and because it has more strength at elevated temperatures ( $>1050^\circ\text{C}$ ).

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Type 304L stainless steel exhibited good resistance to attack by being penetrated only 5 mils.<sup>1</sup> But, low-carbon steel and *Inconel* 601 were not as resistant to attack. Penetration of low-carbon steel of 65 mils was not surprising. But, penetration of *Inconel* 601 of 27 mils (mostly intergranular) was not expected.

Additional tests were carried out to find a high-temperature alternative to Type 304L stainless steel. The creep resistance and oxidation resistance of Type 304L stainless steel may be inadequate if glass-waste compositions require temperatures above 1050°C to produce high-integrity waste forms. The intergranular penetration of *Inconel* 601 under these conditions, revealed in the first test, made the recommendation of this alloy questionable.

## DISCUSSION

### Alloys Tested

Alloys were selected for testing based on their resistance to oxidation, strength, fabricability, and cost. Compositions and properties of the alloys are given in Tables 3 and 4.

#### *Austenitic Stainless Steel*

Austenitic stainless steels exhibit strengths at elevated temperatures superior to those shown by martensitic and ferritic stainless steels.<sup>3</sup> The compatibility of Type 304L stainless steel with glass-waste has been shown in previous tests.<sup>1</sup> Two additional austenitic stainless steels, Type 310 and Type 347, were also chosen for testing because they have greater high-temperature strength than Type 304L. Type 310 stainless steel, however, contains 0.25 wt % carbon which will cause sensitization in applications which require cooling. This problem would have to be addressed if Type 310 stainless steel were chosen for canister fabrication.

Type 347 stainless steel is a niobium-containing, stabilized grade of stainless steel recommended for corrosion-resistant parts requiring welding. It is also recommended for parts which are intermittently heated and cooled to temperatures between 800 and 1600°C to avoid intergranular embrittlement. It has slightly greater creep resistance at elevated temperatures than Type 304L, and its oxidation resistance is as good or better.

### *Cr-Ni-Fe Alloys*

Higher chromium and nickel contents than austenitic stainless steel give Cr-Ni-Fe alloys greater high-temperature oxidation resistance. The availability of *Incoloy* 801, which was included in these tests, is limited. Its composition is similar to the composition of *Inconel* 800. Its behavior in these tests is expected to be representative of Cr-Ni-Fe alloys. At in-can melting temperatures, these more expensive alloys have only slightly better creep resistance than austenitic stainless steels.

### *Ni-Cr Alloys*

Reduction of the iron content gives *Inconel* 600, 601, and 625 greater high-temperature oxidation resistance than the Cr-Ni-Fe alloys. At in-can melting temperatures, the creep resistance of these more expensive alloys is only slightly better than austenitic stainless steels.

### *Ni-Base Superalloys*

The high-temperature oxidation resistance of *Hastelloy* C-4 and *Hastelloy* X is excellent. But, the creep resistance of these more expensive alloys is only slightly better than austenitic stainless steels. *Hastelloy* C-4 has been recommended for other components of the melter system.

### **Glass Compositions**

Two basic types of glass were used in these tests: (1) 65 wt % glass frit (Table 1) combined with 35 wt % simulated sludge (Table 2), and (2) pure glass frit.

### *Glass Frit Combined with Sludge*

This glass was composed of glass frit thoroughly mixed with sludge before melting. Two types of sludge were used: (1) simulated composite sludge (with uranium), and (2) simulated composite sludge (without uranium). These sludges are identical except for the uranium content. No significant difference in the results of either is expected. Use of the sludge without uranium facilitated specimen preparation and evaluation.

### *Pure Glass Frit*

The penetration of candidate canister alloys by pure glass frit during in-can melting was characterized. Experimental production of full-size in-can melted glass-waste forms at PNL showed that the penetration of canister alloys by pure glass frit is much more severe than the penetration by mixtures of frit and sludge.

### *Description of Tests*

Coupon tests and thermocouple tests were used to characterize the attack of candidate canister alloys by molten glass during in-can melting.

#### *Coupon Tests*

Test assemblies for coupon tests (Figure 1) consist of metal rods suspended in a crucible of molten glass-waste by a ceramic top attached to the specimen. The crucible is partially filled with a frit-sludge mixture at room temperature, the specimen is put in place, and the assembly is put in a furnace equilibrated at the test temperature. When the desired time expires, the assembly is removed from the furnace and the specimen is removed from the melt.

Thickness measurements and microscopic examination of the specimen are carried out to determine the extent of attack. First, the specimen with the top attached is photographed. Epoxy resin is then poured over the specimen to hold the oxide, glass, and metal in position. After the resin hardens, the ceramic top is broken off, and the entire specimen is mounted in plastic. The specimen is then ground and polished to show the effect of the exposure of the rolled surfaces.

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



The attack of the canister by three types of exposure can be characterized by these coupon tests. The end of the specimen outside the crucible is exposed to air oxidation as the outside of a waste canister will be during in-can melting. The bottom of the specimen is exposed to molten vitrified glass-waste representative of the exposure of the bottom inside a canister during melting. The portion of the specimen inside the crucible between the molten glass and the ceramic top is exposed to both air oxidation and molten glass. This condition would exist near the top of a canister of glass-waste.

### *Thermocouple Tests*

Test specimens for thermocouple tests consist of clad thermocouples, 1/8-inch-diameter rods, or small strips cut from plate. Replicate specimens are suspended over the side of small canisters made of 1-inch-diameter Type 304L stainless steel pipe, or Type 304L stainless steel pipe caps. The canisters are then filled with pure frit or a frit-sludge mixture at room temperature (Figure 2). Next the canisters are put into a muffle furnace equilibrated at temperature. When the desired time expires, the canister is removed from the furnace and a replicate specimen is removed. The canister is then returned to the furnace.

The thermocouple test is used to evaluate the attack of the canister alloy only at the glass-metal interface. Thickness measurements and microscopic examination of metallographic sections are carried out to determine the extent of attack. First, epoxy resin is poured over the specimen to hold the surface films and adhering glass in position. After the resin hardens, the section of the specimen containing the glass-metal interface (the point of maximum penetration) is removed with an abrasive cutoff wheel. This section is mounted longitudinally to show the effect of exposure on the rolled surfaces. It is prepared for examination on the metallograph. The point of minimum thickness is measured on a traveling-stage microscope. The type of attack is characterized by metallograph and scanning electron microscope (SEM) examination.

### *Test Matrix*

Specimens were exposed to molten glass as shown in the test matrix in Figure 3. The tests were carried out in the following order:

- (1) Coupon tests of Type 347 stainless steel, *Incoloy* 801, *Inconel* 625, and *Hastelloy* C-4 were carried out to find a high-temperature alternative to Type 304L stainless steel.

- (2) Thermocouple tests were carried out to determine the relative corrosiveness of the pure frits and frit-sludge mixtures under consideration at SRL.
- (3) Thermocouple tests were carried out to determine the penetration of the candidate alloys at the most severe conditions (at the highest temperature with the most corrosive frit) expected during waste-form fabrication by in-can melting.

## RESULTS

### *Durability of Candidate Alloys*

The maximum penetration of all alloys tested are listed in Table 5 and plotted in Figure 4 in the order of decreasing resistance to penetration by molten Frit 211 at 1150°C, the most corrosive frit composition available, at the highest temperature tested.

These results show that the resistance to penetration of alloys tested is proportional to the chromium content of the alloys. The metallographic examination of the coupon test specimens showed that chromium oxide was more resistant to dissolution by molten glass than any other oxide formed on the specimen surface. All other oxides, such as Fe or Ni, dissolved in the glass and were found in spinels that precipitated during cooling. Other oxides such as Mo were dissolved and found in the glass matrix. The oxide film remaining on the surface of the alloy exposed to molten glass contained only Cr.

These results are in agreement with similar tests by Wicks to determine the resistance of candidate refractories and candidate electrode alloys to penetration by molten glass in the joule-heated melter.<sup>4</sup> In these tests the refractories with the highest chromium oxide content were the most resistant to penetration. And the resistance to penetration of candidate electrode alloys was proportional to their chromium content.

### *Corrosiveness of Glass Melts*

The amount of attack of canister alloys by the pure frit under in-can melting conditions is directly proportional to the Na<sub>2</sub>O content of the frit (Tables 3 and 6 and Figure 5). The ratio between the amount of penetration of Type 304L stainless steel and the Na<sub>2</sub>O content of Frits 411, 21, and 18 is almost constant. There are differences in the composition of Frits 411, 21, and 18 other than the Na<sub>2</sub>O content. But, the Na<sub>2</sub>O content is the

only component that is consistently different in the three frits. The Na<sub>2</sub>O content of Frit 18 is almost double the Na<sub>2</sub>O content of Frit 411. The Na<sub>2</sub>O content of Frit 21 is intermediate.

#### *Coupon Tests*

Coupon tests of Type 347 stainless steel, *Incoloy* 801, *Inconel* 625, and *Hastelloy* C-4 were carried out to find a high-temperature alternative to Type 304L stainless steel. The appearance and maximum penetration of Type 347, *Incoloy* 801, *Inconel* 625, and *Hastelloy* C-4 specimens exposed to 65 wt % Frit 21 and 35 wt % composite sludge without uranium for up to 7 hours at up to 1100°C is shown in Figure 6.

The maximum penetration of each specimen occurred in the part exposed to both glass and air at the glass-metal interface. This phenomenon was seen in previous tests at SRL.<sup>1</sup> Metal oxide is relatively soluble in molten glass. At the glass metal interface there is a portion of the oxide above the glass that contacts the glass. Molten glass is drawn up into this oxide film by capillary action. Evidently the presence of the glass in the metal oxide reduces the film's ability to protect the metal from further oxidation.

Each specimen was examined on the metallograph and the SEM to characterize the attack. The results of these examinations are discussed in the following paragraphs.

#### *Type 347 Stainless Steel Tests (Figure 7)*

*Air.* The portion of the specimen exposed to air was covered with an oxide film composed of Cr, Fe, and Ni oxide. No selective penetration was observed of this portion of the specimen.

*Glass and Air.* The portion of the specimen exposed to glass and air was covered with types of material. There was a layer of Cr oxide adjacent to the metal. Adjacent to this layer was a layer composed of the components of both Cr, Fe, and Ni oxide and glass. There was only a small amount of selective penetration.

*Glass.* The glass had spalled off the metal. There was, however, evidence of a Cr oxide layer adjacent to the metal. There was only a small amount of intergranular penetration.

#### *Incoloy 801 Tests (Figure 8)*

*Air.* The outside film covering the portion of the *Incoloy* 801 specimen exposed to air was composed of several layers. The

layer adjacent to the metal was composed of only Cr oxide. Adjacent to this layer was a layer composed of a mixture of Cr, Fe, and Ni oxide.

*Glass and Air.* This portion of the specimen was covered with an oxide film composed of only Cr oxide. This indicates that the oxide layer composed of Cr, Fe, and Ni oxides on the specimen had dissolved in the glass-waste. There was selective penetration of the specimen under the oxide film.

*Glass.* This portion of the specimen was also covered by an oxide film composed of only Cr oxide. There were spinels in the glass-waste adjacent to the specimen. These spinels contained Cr, Fe, and Ni from the oxide layer that dissolved in the glass-waste.

#### *Inconel 625 Tests (Figure 9)*

*Air.* The portion of the specimen exposed to air was covered with a Cr-Ni oxide.

*Glass and Air.* The portion of the specimen exposed to glass and air was covered with a material made up of Cr, Ni oxide, and glass.

*Glass.* All the glass had spalled off this portion of the specimen. There was a Cr oxide film adjacent to the metal. And there was a small amount of intergranular penetration in the metal.

#### *Hastelloy C-4 Tests (Figure 10)*

*Air.* The oxide layer covering the end of the specimen exposed to air was composed of many small particles. Most of these particles fell off the specimen when it was removed from the crucible of molten glass. A band of particles remained adjacent to the metal. X-ray energy spectrometry (XES) analysis showed that this material was composed of Mo, Cr, and Ni.

*Air and Glass.* There was severe attack in the area of the specimen exposed to air and glass. Many large pits were formed. A band of Mo, Cr, and Ni oxide existed adjacent to the metal. A fine particulate Mo-Ni oxide was trapped in the pits.

The appearance of this portion of the specimen is consistent with Kofstad's description of an alloy undergoing catastrophic oxidation.<sup>5</sup> This phenomenon is accompanied by the formation of liquid oxide phases such as  $\text{MoO}_3$  (m.p.  $795^\circ\text{C}$ ).

*Glass.* A layer of Cr oxide remained on the surface of the metal. There were Cr, Fe, and Ni spinels in the glass adjacent to the metal. These spinels formed because of localized supersaturation of Cr, Fe, and Ni caused by dissolution of these elements from the oxide films into the glass. The Mo from the dissolved oxide films was incorporated in the glass matrix rather than into the spinels. There are small areas in the glass matrix adjacent to the metal that contain Mo while the bulk of the glass matrix contains no Mo. There was no Mo in the glass frit or the sludge.

#### *Thermocouple Tests*

##### *Tests at 1050°C*

Thermocouple tests were carried out at 1050°C to determine the relative corrosiveness of the pure glass frits and frit-sludge mixtures under consideration at SRL. The penetration of Type 304L stainless steel by glass frit combined with sludge and by several compositions of pure frit was determined as shown by the test matrix in Figure 3. Test specimens were heated for up to 24 hours because experimental production of full-size waste-forms at PNL showed that this was the required heating time.

*Penetration.* Maximum penetration of all specimens occurred just above the glass surface where the specimen was exposed to both air oxidation and glass. This phenomenon has been seen in previous tests at SRL.<sup>1</sup> The penetration of the specimens is given in Table 7 and plotted against time in Figure 11.

During in-can melting, pure Frit 411 penetrated Type 304L stainless steel approximately an order of magnitude faster than a mixture of Frit 411 and Savannah River Plant (SRP) simulated composite-sludge, based on penetration of specimens exposed to molten glass for 24 hours. Dimensional changes of specimens with the longer exposure times is believed to be more reliable than dimensional changes of specimens exposed for shorter times. Penetration by pure Frit 411 was 13.5 mils in 24 hours. In companion specimens, penetration by a mixture of Frit 411 and SRP simulated composite-sludge was less than 2 mils. Penetration of the Type 304L stainless steel by the pure Frit 411 increased with time at a linear rate (Figure 11).

Penetration of Type 304L stainless steel by pure Frit 21 and pure Frit 18 also proceeded at a linear rate. But, penetration of Type 304L stainless steel by these frit compositions was faster than the penetration by pure Frit 411.

A Type 304L stainless steel specimen exposed to pure Frit 411 at 1050°C for 24 hours was examined on the SEM (Figure 12). This specimen was taken from near the point of maximum penetration. The metal oxide layer on the specimen had been completely impregnated with glass. The point of maximum penetration of the test specimen would have been just above where this metallography specimen was taken. At that point the metal oxide layer would have been only partially impregnated with glass. The presence of glass in the metal oxide layer reduces its effectiveness to protect the metal from further oxidation.

The glass impregnating the metal oxide layer was enriched in chromium. And the glass adjacent to the metal oxide layer contained many large particles. Metal oxide is relatively soluble in molten glass. The chromium in the glass impregnating the metal oxide layer could be from metal oxides that dissolved in the glass from the metal oxide layer. There is no chromium in the glass frit or the sludge. The precipitates that formed during cooling due to the localized concentration of metallic elements from the metal oxide dissolved in the glass. These particles are common in glass-waste containing large amounts of metallic elements.<sup>6</sup> They have a distorted iron oxide structure and effect the leachability of the glass-waste.

#### *Tests at 1150°C*

Thermocouple tests were carried out at 1150°C to expose the candidate alloys to the most severe conditions expected during waste-form fabrication by in-can melting. In these tests, the alloys were exposed to pure Frit 211 which has the highest Na<sub>2</sub>O content (20.6 wt %) of any glass frit available. Specimens were exposed for 24 hours which is the time required for in-can melting full-size waste glass forms based on tests carried out at PNL.

Maximum penetration of all specimens occurred at the glass surface where the specimen was exposed to both air oxidation and glass. This phenomenon has been seen in previous tests at SRL.<sup>1</sup> The penetration of the specimen is given in Table 8.

## REFERENCES

1. W. N. Rankin. "Compatibility Testing of Vitrified Waste Forms." DP-MS-115 (Rev. 2/15/78), Presented at Corrosion/78 Meeting, Houston, Texas (March 6-10, 1978).
2. C. L. Angerman and W. N. Rankin. "Durability of Containers for Storing Solidified Radioactive Waste." *Materials Performance*, Vol. 17, No. 4, pp. 9-18 (April 1978).
3. *Tool Manufacturing Engineers Handbook*. Third Edition, Society of Manufacturing Engineers, Dearborn, MI (1976).
4. G. G. Wicks. "Compatibility Tests of Materials for the Joule-Heated Ceramic Melter for Production of Defense Glass Waste Products." DP-MS-78-90X, Presented at the International Symposium on Ceramics in Nuclear Waste Management, April 30-May 3, 1979, Cincinnati, OH.
5. P. Kofstad. *High-Temperature Oxidation of Metals*. John Wiley and Sons, Inc., New York, NY, p. 292 (1966).
6. W. N. Rankin and J. A. Kelley. "Microstructure and Leachability of Vitrified Radioactive Waste." *Nuclear Technology*, Vol. 41, No. 3, pp. 373-380 (1978).

TABLE 1

## Composition of Glass Frits

Metal Oxide	<u>Amount in Frit, wt %</u>			
	18	211	21	411
SiO <sub>2</sub>	52.5	58.3	52.5	58.3
Na <sub>2</sub> O	22.5	20.6	18.5	12.5
B <sub>2</sub> O <sub>3</sub>	10.0	11.1	10.0	11.1
TiO <sub>2</sub>	10.0	-	10.0	-
CaO	5.0	5.6	5.0	5.6
Li <sub>2</sub> O	-	4.4	4.0	12.5

TABLE 2

## Composition of SRP Simulated Sludges

Element	<u>Amount in Sludge, wt %</u>	
	<u>Composite</u> (With Uranium)	<u>Composite</u> (Without Uranium)
Fe	14.0	14.0
Al	15.5	15.5
Mn	4.1	4.1
U	3.3	-
Ca	1.5	1.5
Ni	<u>1.1</u>	<u>1.1</u>
Total Metallic Elements	39.5	36.2



TABLE 3

## Alloys Tested for In-Can Melting

<i>Alloy Composition (wt % of element)</i>	<i>Type 304L SS</i>	<i>Type 310 SS</i>	<i>Type 347 SS</i>	<i>Incoloy<sup>a</sup> 801</i>	<i>Inconel<sup>a</sup> 600</i>	<i>Inconel<sup>a</sup> 601</i>	<i>Inconel<sup>a</sup> 625</i>	<i>Hastelloy<sup>b</sup> C-4</i>	<i>Hastelloy<sup>b</sup> X</i>
<i>Element</i>									
Fe	68	51	68	44	7	14	3	-	19
Ni	10	21	11	33	72	61	62	68	45
Cr	19	25	18	20	16	23	21	16	22
Mo	-	-	-	-	-	-	8	15	9
Cb + Ta	-	-	Trace	-	<3	-	4	-	-

a. Trademark of the International Nickel Company.

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TABLE 4

## Properties of Candidate Alloys for In-Can Melting Canisters

<i>Material</i>	<i>Penetration by Oxidation in 1000 hr at 1100°C, mils<sup>a</sup></i>	<i>Rupture Strength at 1090°C, psi<sup>a</sup></i>	<i>Cost, \$ per lb<sup>a</sup></i>
Austenitic Stainless Steel (Types 347 and 304L)	100	1000 (Type 347) 700 (Type 304L)	1.50
Cr-Ni-Fe Alloy (Incoloy 801)	5.5		5.00
Ni-Cr Alloy (Inconel 625)	2.6	1800	5.50
Ni-Base Superalloys (Hastelloy C-4)	2.4	1600	11.72

<sup>a</sup>. Representative of type of material. Not necessarily for specific alloy listed.

TABLE 5

## Penetration of Canister Alloys by Pure Frit 211 at 1150°C

<i>Alloy</i>	<i>Penetration, mils</i>	<i>Wt % Cr</i>	<i>Test Condition</i>	<i>Remarks</i>
<i>Inconel 625</i>	16.9	21	25 hr at 1150°C	
<i>Hastelloy X</i>	19.8	22		
<i>Inconel 601</i>	19.0	23		Little selective penetration as found in previous test. <sup>1</sup>
<i>Incoloy 801</i>	>19.0	20		Specimen completely penetrated.
Type 310 Stainless Steel	25.2	25		
Type 304L Stainless Steel	25.9	19		
<i>Inconel 600</i>	25.9	16		
Type 347 Stainless Steel	38.3	18		
<i>Hastelloy C-4</i>	110	16	7 hr at 1150°C <sup>a</sup>	15 wt % Mo reduces resistance to penetration.

a. *Hastelloy C-4* exposed to 65 wt % Frit 21 and 35 wt % composite sludge (with uranium).

TABLE 6

## Penetration of Type 304L Stainless Steel by Pure Frit

<i>Identification</i>	<i>Wt % Na<sub>2</sub>O</i>	<i>Penetration in 24 Hours at 1050°C, mils</i>
Frit 411	12.5	13.5
Frit 21	18.5	16.5
Frit 211	20.6	25.9
Frit 18	22.5	26.0

TABLE 7

Penetration of Type 304L Stainless Steel  
During In-Can Melting

<i>Time at 1050°C, hours →</i>	<i>1</i>	<i>2</i>	<i>5</i>	<i>10</i>	<i>24</i>
<i>Glass Composition</i>	<i>Penetration, mils</i>				
Pure Frit 411	1.3	1.8	3.9	4.5	13.5
75 wt % Frit 411, 25 wt % SRP Simulated Composite Sludge	1.1	0.9	0.6	0.8	1.7
Pure Frit 21	1.2	0.9	3.0	7.1	16.2
Pure Frit 18	0.3	2.6	1.7	12.3	26.7
Pure Frit 211	-	2.1	5.7	8.7	25.9

TABLE 8

Penetration of Canister Alloys by Pure Frit 411  
in 24 Hours at 1150°C, mils

<i>Alloy</i>	$\frac{\Delta \text{ Thickness}^a}{2}$	+ <i>Intergranular Penetration</i>	= <i>Total</i>
<i>Inconel</i> 625	13.9	3.0	16.9
<i>Hastelloy</i> X	16.8	3.0	19.8
<i>Inconel</i> 601	18.0	1.0	19.0
<i>Incoloy</i> 801	13.2	>6.0	>19.0 <sup>b</sup>
Type 310 Stainless Steel	23.2	2.0	25.2
Type 304L Stainless Steel	16.6	1.0	17.6
<i>Inconel</i> 600	23.9	2.0	25.9
Type 347 Stainless Steel	37.3	1.0	38.3

a. Thickness before heating - Thickness after heating =  $\Delta$  Thickness.

$\frac{\Delta \text{ Thickness}}{2}$  is penetration of one side of specimen.

b. The *Inconel* 801 specimen was completely penetrated.

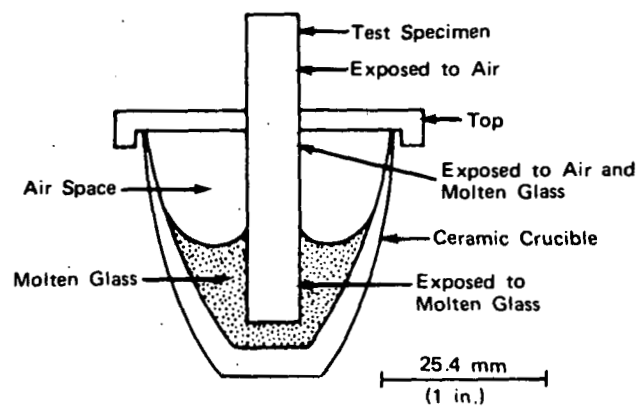


FIGURE 1. Coupon Test Assembly

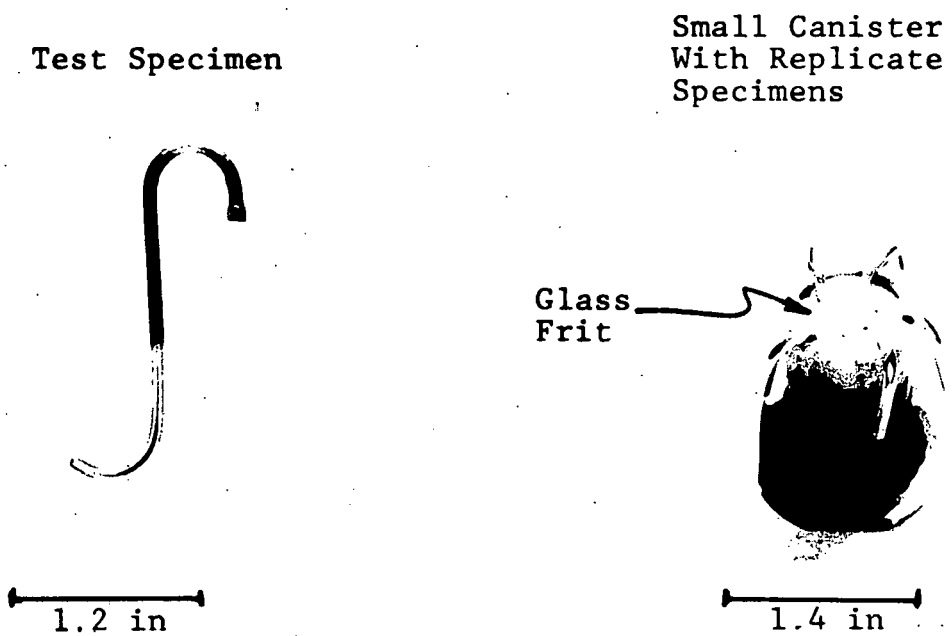


FIGURE 2. Thermocouple Test Assembly

TYPE TEST	GLASS COMPOSITION	TEST CONDITIONS	TYPE 304L SS	TYPE 310 SS	TYPE 347 SS	INCOLOY 801	INCONEL 600	INCONEL 601	INCONEL 625	HASTELLOY C-4	HASTELLOY X	TESTING ORDER
COUPON	65 wt % Frit 21 35 wt % Composite Sludge (with uranium)	1050, 1100 & 1150°C for 3, 5 & 7 hours			X	X			X	X		TEST 1
THERMO- COUPLE	Pure Frit 18	1050°C for 2, 5, 10 & 24 hours	X									
	Pure Frit 211	1050°C for 2, 5, 10 & 24 hours	X									
	Pure Frit 21	1050°C for 2, 5, 10 & 24 hours	X									
	Pure Frit 411	1050°C for 2, 5, 10 & 24 hours	X									TEST 2
	75 wt % Frit 411 25 wt % Composite Sludge (without uranium)	1050°C for 2, 5, 10 & 24 hours	X									
	Pure Frit 211	1150°C for 24 hours	X	X	X	X	X	X	X		X	TEST 3

FIGURE 3. Test Matrix



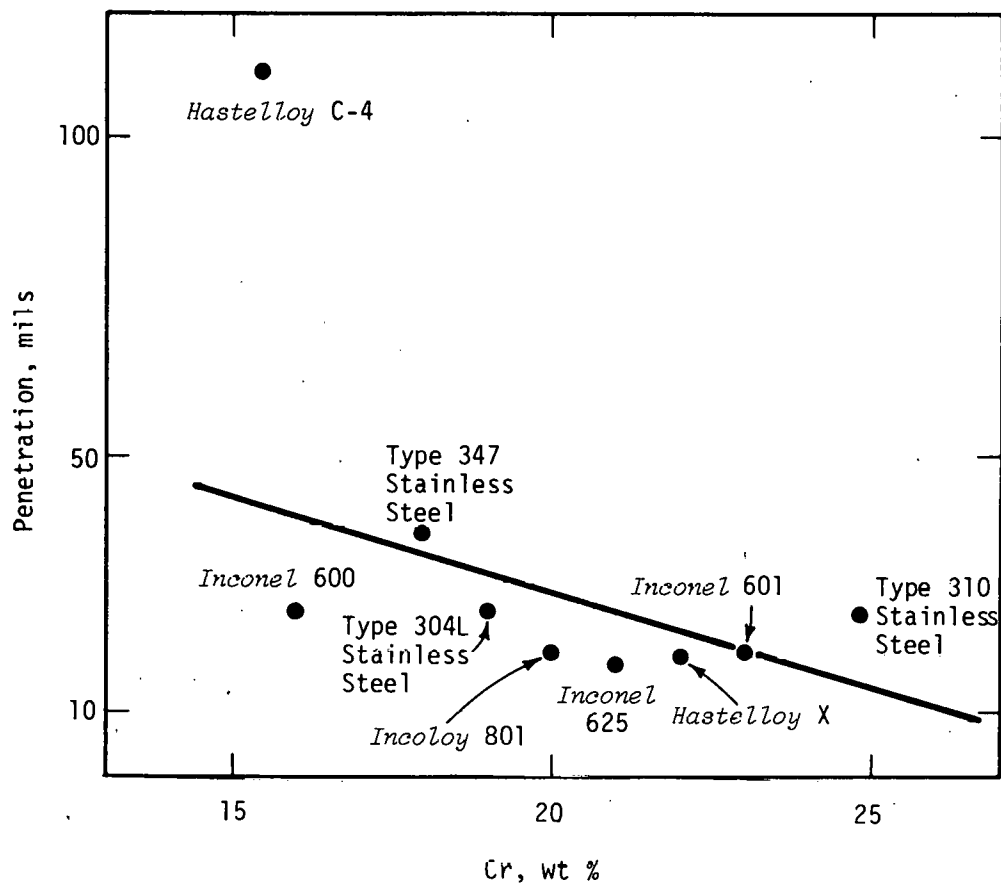


FIGURE 4. Penetration of Canister Alloys by Pure Frit 211 at 1150°C vs. Chromium Content of the Alloys

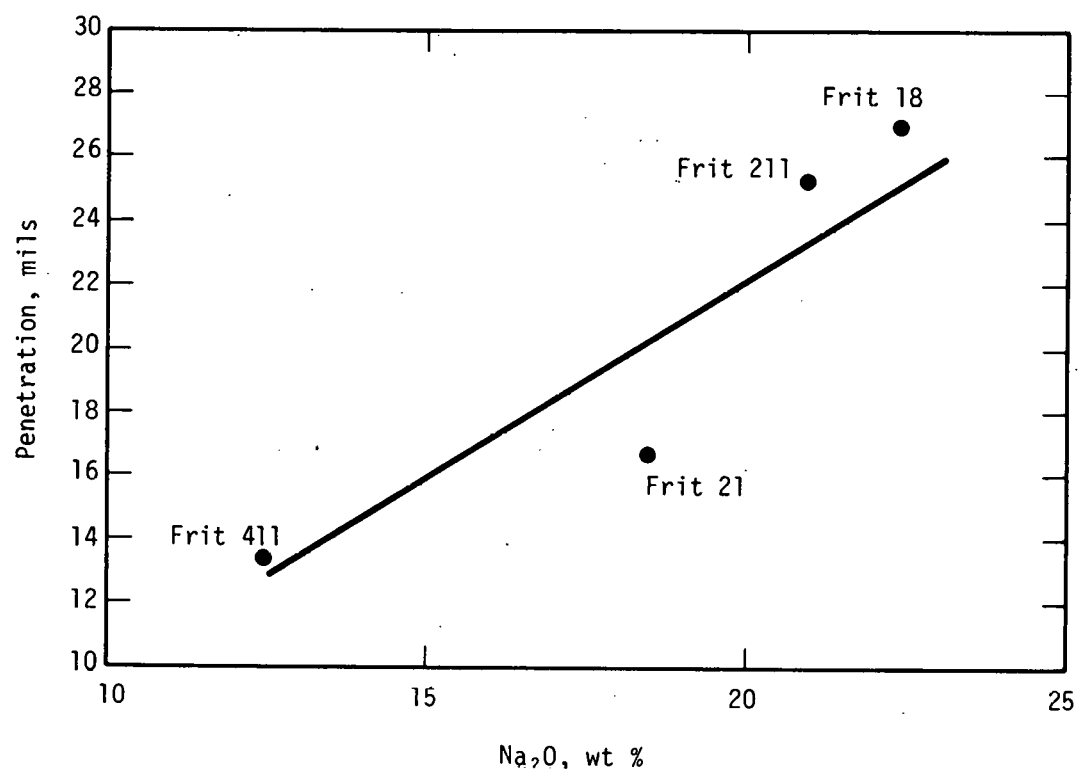


FIGURE 5. Penetration of Type 304L Stainless Steel by Pure Frit in 24 Hours at 1050°C

Type 347 Stainless Steel

"Incoloy" 801

"Inconel" 625

"Hastelloy" C-4



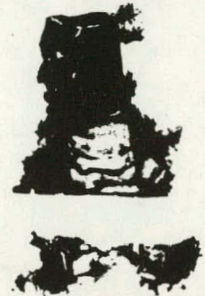
5.6 mils



6.9 mils



9.8 mils



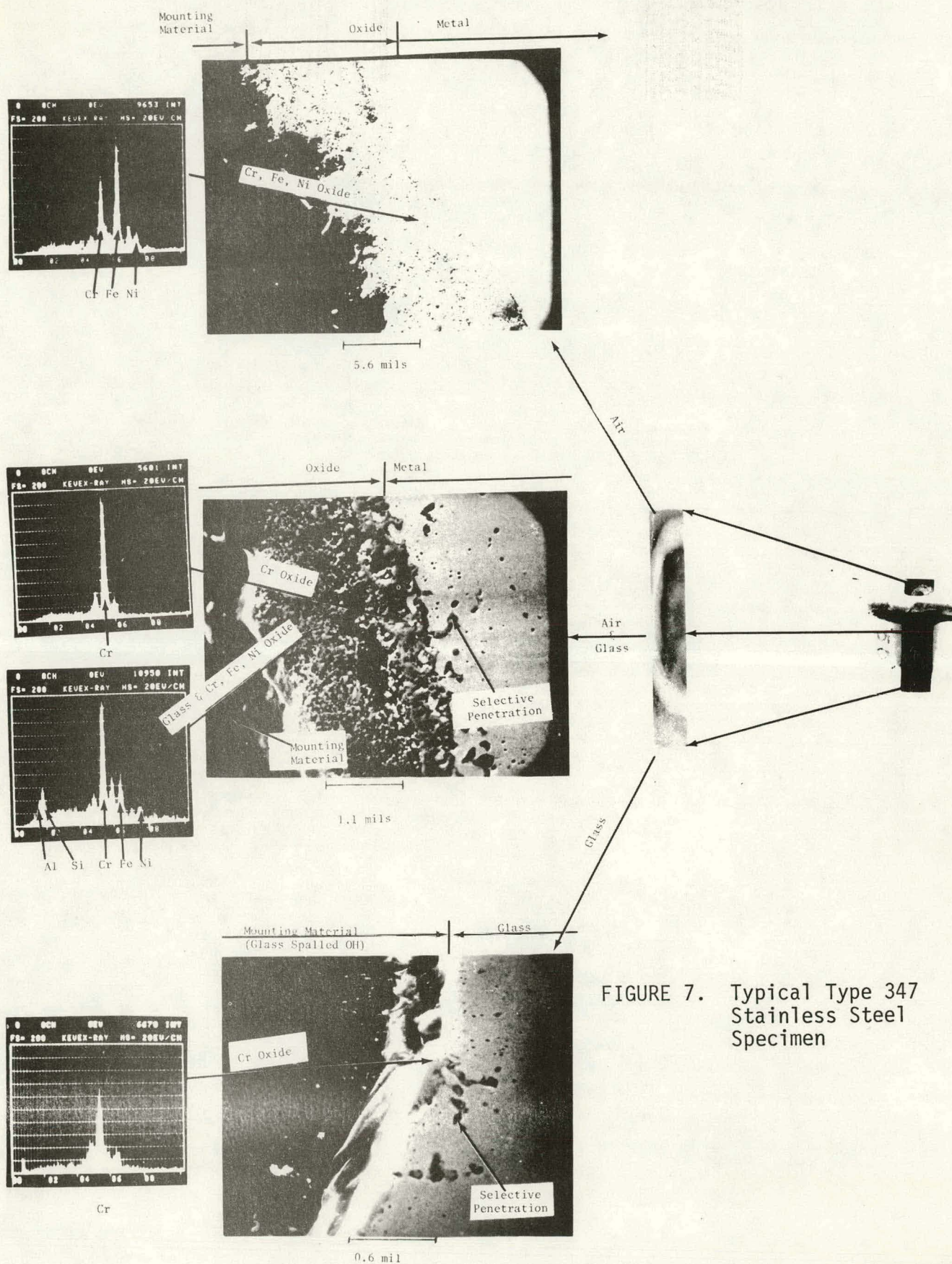
110 mils

Maximum Penetration

\* Specimens exposed to 65 wt % Frit 21 and  
35 wt % composite sludge (with uranium)  
for up to 1150°C.

FIGURE 6. Appearance of Typical Coupon Test Specimens







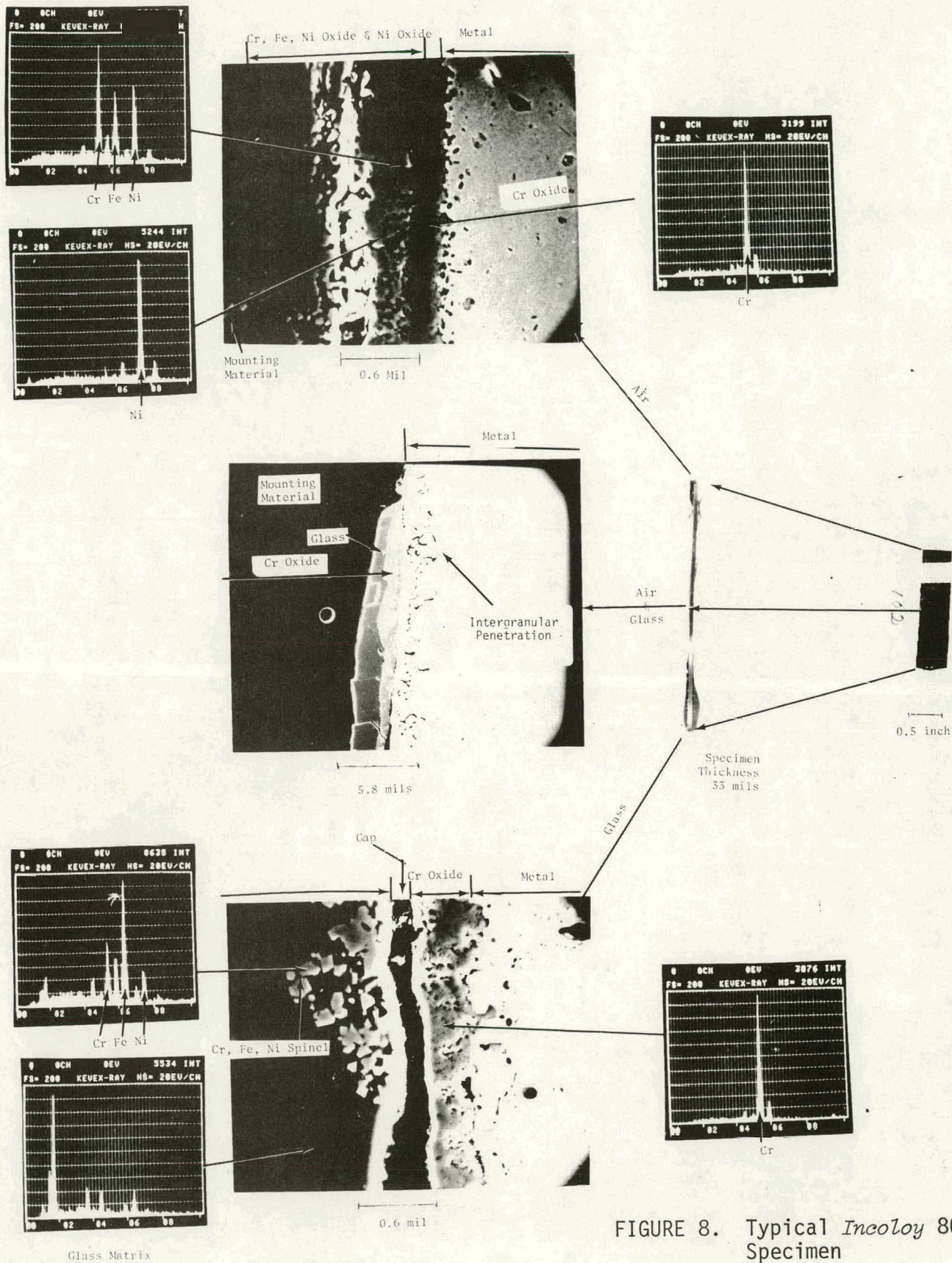


FIGURE 8. Typical *Incoloy* 801 Specimen



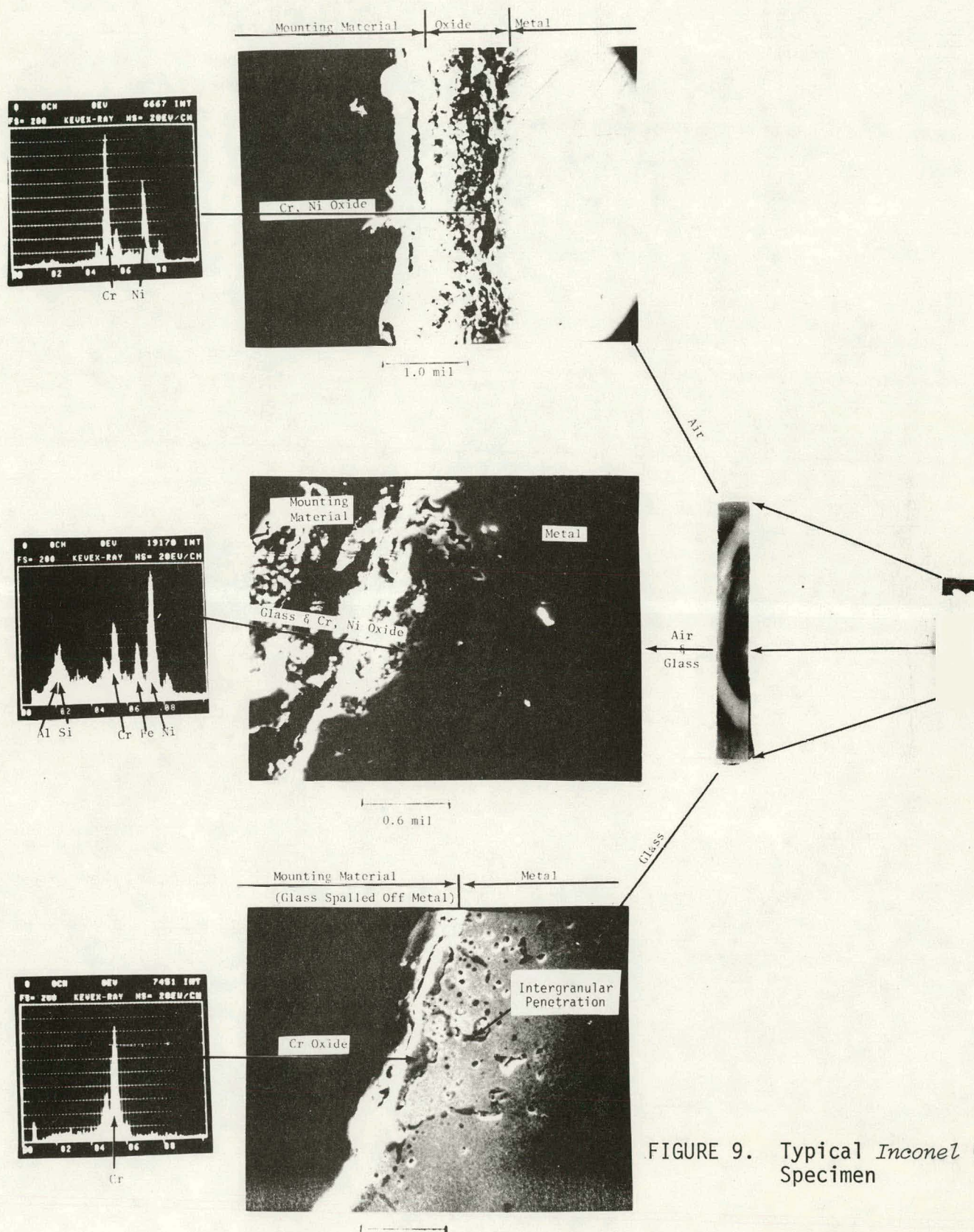
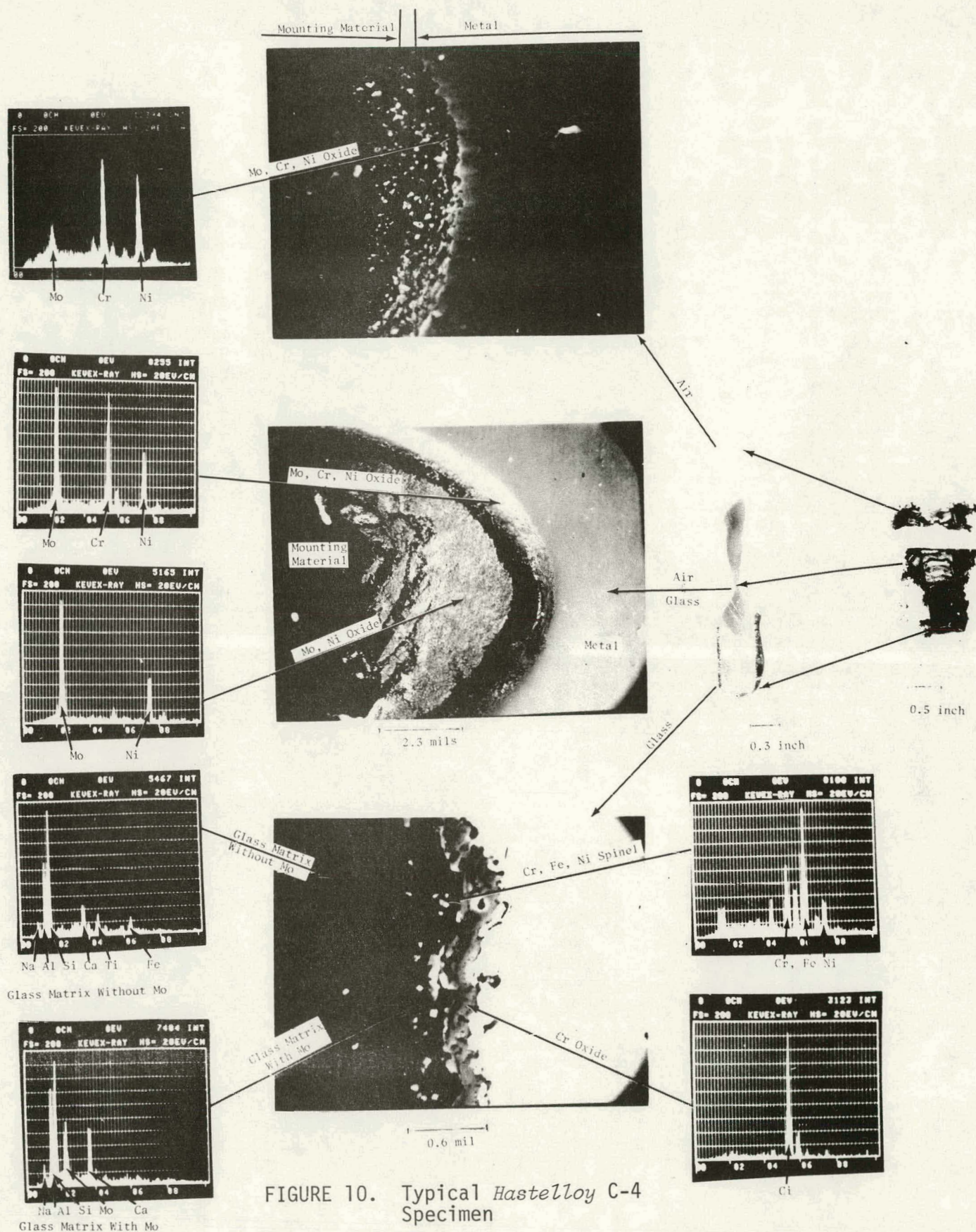


FIGURE 9. Typical *Inconel* 625 Specimen







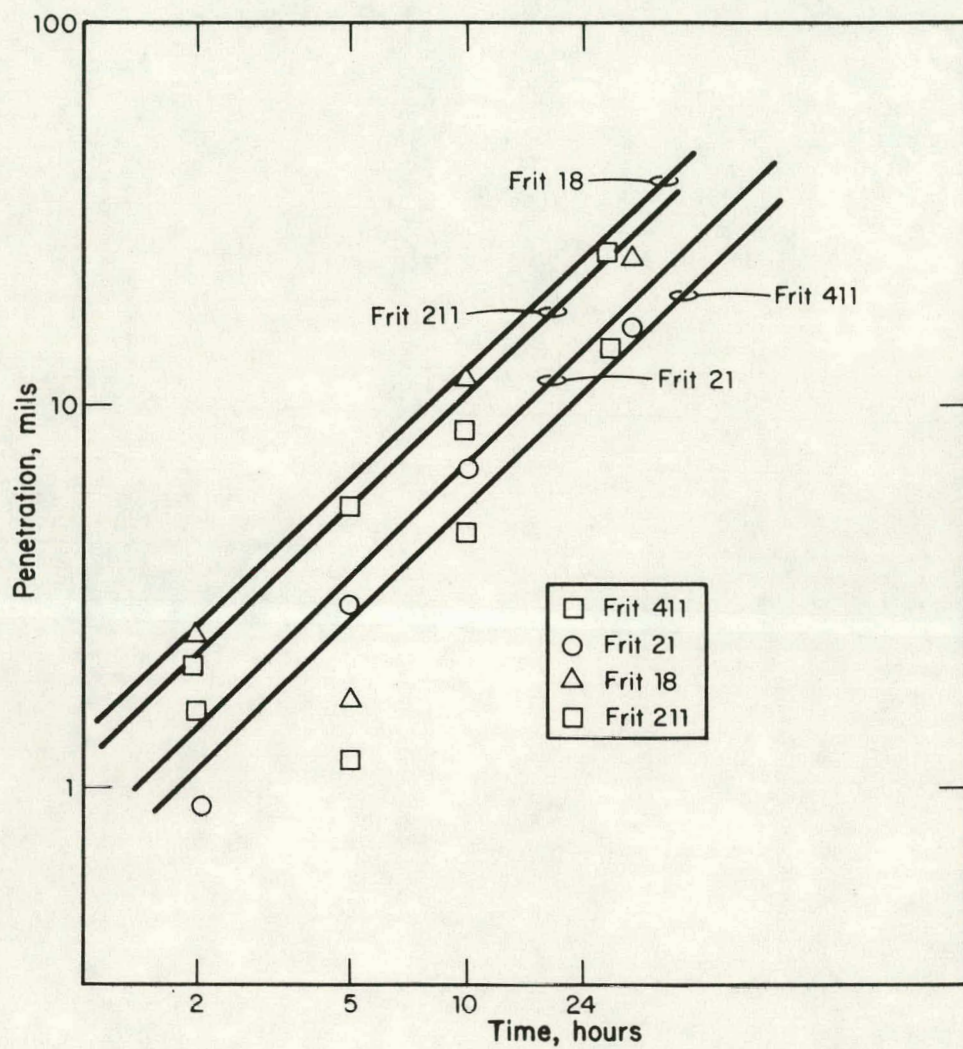
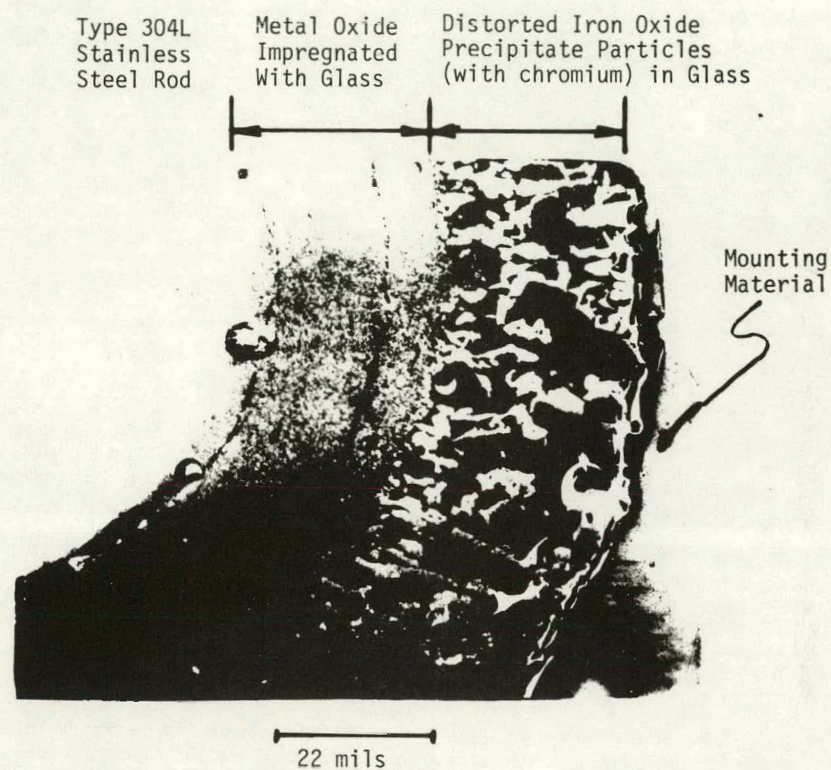


FIGURE 11. Penetration of Type 304L Stainless Steel by Pure Frit at 1050°C





\*Total penetration 13.5 mils

FIGURE 12. Transverse Section Through Type 304L Stainless Steel Rod Exposed to Pure Frit 411 at 1050°C for 24 Hours