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Compatibility of Strontium Space Fuel Forms

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June 1966

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Prepared by:

Frank J. Miraglia, Jr.

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For
Division of Isotopes Development
USAEC
AT(30-1)-3062

MARTIN MARIETTA CORPORATION
NUCLEAR DIVISION

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ABSTRACT

The compatibility of strontium fluoride and strontium metal, both of which are candidate space fuels, with various encapsulating materials is presented. A summary of the procedures used in preparing, testing and analyzing the specimens and a presentation of the results obtained is given. For the 19 month test period at 925°C, it has been concluded that Haynes 25 is the material most compatible with the strontium fluoride fuel and the decay products of Sr-90, ZrO₂ and ZrF₄. The presence of Zr metal in the fluoride fuel matrix significantly affected the compatibility of the metals tested. With Zr metal present, Hastelloy X proved the better fuel containment material in a 4 month test at 925°C.

Short term 'scoping' tests of the compatibility of strontium metal with various materials at low (700°C) and high (925°C) temperature indicated that molybdenum and stainless steel 321 were the most compatible materials with the strontium metal.

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I. Introduction

An important property of an isotopic fuel is its compatibility with various encapsulating materials. During the course of the Sr-90 Fuel Development Program, several compatibility studies were conducted.

Early short-term tests in which various metals were held in contact with pelletized and powdered SrF_2 at 1000°C for 200 hours indicated that Rene-41 and 321 stainless steel were less resistant to attack than the Haynes and Hastelloy alloys¹. Therefore, a long-term study of the compatibility of SrF_2 with various Haynes and Hastelloy alloys was initiated in September of 1964 and terminated in April 1966. This study investigated the compatibility of new and aged fuel with Haynes-25, Haynes-25 with a tantalum liner, and alloys of Hastelloy C, X and N. Fuel age was simulated as follows:

1. New fuel was simulated by the preparation of SrF_2 from a strontium nitrate solution containing 1 wt% Ca^* and 0.5 wt% Fe (expected impurity levels in Sr-90 feed material).
2. Aged fuel was simulated in two ways:
 - a) By the addition of 2 1/2 wt% Zr as ZrO_2 which represents fuel prepared from aged feed material (~ 2 years old).
 - b) By the addition of 5 wt% Zr as ZrF_4 which is the expected level in a four-year old fuel capsule.

The stable element zirconium-90 is the end product of the strontium-90 decay chain. In the closed system of a fuel capsule, the postulated ZrF_4 production reaction¹ is:

The test program, initiated in September 1964, investigated the effect of ZrF_4 on the compatibility of the candidate encapsulating alloys. In October 1965, the program was extended to include the investigation of the compatibility of $\text{SrF}_2 + \text{Zr}$ metal. The addition of 5 wt% Zr metal to SrF_2 was used to simulate the fuel in an aged capsule.

Recently, interest in the use of strontium metal as a potential space fuel has increased. Therefore, two scoping-type compatibility programs using strontium metal

*NOTE: All wt% of impurities given in this report are based on impurity to total Sr ratio.

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were initiated in September 1965 and terminated in March 1966. The scoping-type test was one of short duration (~1000 hrs) and tested many materials at one time. Not all of the materials which showed poor compatibility, with this type of testing, should be eliminated from consideration, since their properties may be adversely affected by the presence of the other materials. However, this type of testing does allow many materials to be tested in a short period of time and does indicate which materials are worthy of further consideration for testing in a long-term program.

This report includes a summary of the procedures used in preparing, testing and analyzing the specimens and presents the results obtained from each of the studies.

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II. Procedures

A. Specimen Preparation

1. Space Fuel Capsules:

For the program initiated in September 1964, forty-five test capsules, nine of each Hastelloy alloy and 18 of the Haynes 25 alloy, were fabricated conforming to Figure 1. The internal diameter of nine Haynes 25 capsules was enlarged to accommodate a 5 mil tantalum liner. The capsules were fabricated from bar stock and end caps of each like material, punched from 1/16" plate, were welded to one end. Each capsule was leak checked and vapor degreased before loading with the simulated fuels.

Twenty-five capsules, five of each material, were fueled as shown in Figure 2. The twenty remaining capsules, four of each material, were fueled with the $\text{SrF}_2 + 5 \text{ wt\% Zr as ZrF}_4$. The space fuel was step-pressed into each test capsule at 50 TSI to a density of 85 to 90% of theoretical. End caps of each like material were welded onto each open end.

Since the test furnace did not have provisions for maintaining an inert atmosphere, the test specimens were initially sealed in evacuated Vycor tubes to prevent oxidation of the capsules' outer surfaces. The temperature of the test, 925°C, approached the Vycor tubes temperature limit² for continuous service. When the first series of test specimens was sampled, the Vycor containers for the test specimens were cracked, deformed and discolored. Some oxidation of the outer surfaces of the test specimens had occurred. The test furnace was shut down; an inspection of the remaining Vycor containers showed them to be cracked, deformed and/or discolored. A thin white-gray coating, identified as SiO_2 by x-ray diffraction, was found on the inner and outer surfaces of the Vycor containers. Therefore, it was concluded that devitrification² of the glass had occurred because of the high operating temperature.

The remaining test specimens were removed from the furnace and sealed in stainless steel 321 containers. The containers were degassed, evacuated, backfilled at room temperature with 0.25 atm. of argon and welded shut. Stainless steel 321 showed good oxidation resistance up to 1700°F (925°C)³.

Three of the four stainless steel 321 sample cans contained two series of capsules; a series comprised of one capsule of each encapsulating material. One series was fueled as shown in Figure 2 and the other with the $\text{SrF}_2 + 5 \text{ wt\% Zr as ZrF}_4$. The fourth sample can contained the one remaining series of capsules.

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FIGURE 1. Typical Capsule Configuration for Space Fuel Compatibility Specimens

- Notes: 1. An end cap of like material to be welded to open end after fueling.
2. I.D. is made ~10 mils larger in the Haynes 25 capsules accomodating Ta liners.

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FIGURE 2. Typical Fuel Loading of 25 Space Fuel Compatibility Capsules

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The sample cans were then placed into a Marshall furnace and the temperature brought to 1700°F (925°C). This temperature simulated the operating conditions of a Snap 17 fuel capsule. The temperature of the test furnace was monitored continuously. The furnace operated at the test temperature for the entire test period; only two instances of heater element failure were experienced.

In September 1965, twenty-four additional capsules were fabricated, six each of the Hastelloy alloys and Haynes-25, conforming to Figure 1. A mixture of $\text{SrF}_2 + 5 \text{ wt}\% \text{ Zr}$ as the metal was step-pressed into each capsule to $\sim 80\%$ of theoretical density. The capsules were sealed in sample cans of stainless steel 321 and placed into the test furnace on 11 October 1965.

2. Strontium Metal Test Capsules

Experiments were initiated to study the compatibility of strontium metal with various encapsulating materials. Test capsules were exposed to different temperature environments for a period of at least 1000 hours. The test program, using commercial grade strontium metal, was run at two different temperatures:

- (a) 700°C (1300°F) - At this temperature, the fuel was in a solid form. This temperature simulated the operating conditions of a generator using PbTe thermoelectric elements.
- (b) 925°C (1700°F) - At this temperature, the fuel was in a molten form. This temperature simulated the operating conditions of a generator using GeSi thermoelectric elements.

Materials tested for compatibility with the strontium metal included:

Haynes 25
Hastelloy N
Molybdenum
Wrought Iron
Tantalum
Copper
Hastelloy C
Hastelloy X
Nickel
Titanium
Manganese (5% Cu)

Six containment capsules were fabricated from stainless steel 321. Three of the containment capsules were fueled with metal strontium wafers (Figure 3). Coupons of each candidate metal (3/4" x 3/4") were sandwiched between each 1/4"

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FIGURE 3. Typical Fuel Loading Configuration for Low Temperature Strontium Metal Compatibility Tests

- Notes:
1. All loading and welding done in an inert atmosphere glovebox.
 2. All test coupons were 3/4" x 3/4".

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thick strontium metal wafer (0.65" O.D.). Pressure was applied to the loaded capsules to insure contact between the metal surfaces. These capsules were heated to 700°C. The three remaining containment capsules each contained a stainless steel coupon holder with the test metals and were filled with molten strontium metal (Figure 4). These capsules were heated to 925°C. The test capsules for the 925°C tests were overpacked in stainless steel tubes to provide secondary containment and oxidation protection.

One containment capsule at each temperature was removed from the furnace after ~200, ~600 and ~1000 hours exposure to the test temperature.

B. Metallographic Procedures

All the test specimens were examined optically utilizing a Bausch and Lomb Balphot Metallograph, an American Optical Model 2400 Metallograph and a 'zoomer' type microscope for quick routine appraisals (Figure 5). In Addition, numerous specialized table microscopes were used, along with photographic equipment for obtaining a permanent record of the observations made on the specimens. The procedures used for preparing and observing the specimens are outlined below.

1. Sectioning and Mounting: All specimens were wet sectioned and mounted in bakelite to facilitate handling. A typical sectioning of specimens is shown in Figure 6.

a. Metallographic Preparation for 321 Stainless Steel, Iron, Manganese-Copper, Haynes-25, Hastelloy C, Hastelloy N, Hastelloy X, and Molybdenum

- Grinding: (1) Coarse grinding on a wet 180 grit SiC belt.
(2) Fine grinding on wet SiC grinding papers, 220, 320, 400, 600 grits.
- Polishing: (1) Rough polishing, using a 3 micron diamond paste on a wheel covered with a metcloth and ultrasene lubrication.
(2) Semi-final polishing, using a 1/2 micron diamond paste on a wheel covered with a Selvyt cloth and ultrasene lubrication.
(3) Final polishing, using a Linde-B slurry on a Gamal cloth. The mounts were weighted and run on a Syntron Vibratory polisher set for 40% capacity for approximately 40 minutes.

b. Metallographic Preparation of Titanium and Tantalum

- Grinding: (1) Course grinding on a wet 180 grit SiC belt.

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FIGURE 4. Typical Fuel Loading Configuration for High Temperature Strontium Metal Compatibility Tests

- Notes:
1. All loading and welding done in an inert atmosphere glovebox.
 2. All test coupons are $3/4" \times 3/4"$.
 3. Molten strontium metal added to the capsule body using an induction furnace with the capsule body as the susceptor.

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FIGURE 5. Metallographic Equipment

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FIGURE 6. TYPICAL SECTIONING OF TEST SPECIMENS

SPACE FUEL CORROSION STUDY

#109
HAYNES #25

#110
HAYNES #25

#111
HASTELLOY "C"

#115
HASTELLOY "N"

#117
HASTELLOY "N"

#108
HAYNES #25

#114
HAYNES #25

#112
HASTELLOY "C"

#113
HASTELLOY "X"

#116
HASTELLOY "N"

TWO FUELS

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- (2) Fine grinding on wet SiC grinding papers, 220, 320, 400, 600 grits. Best results were obtained using slightly worn papers that had been dressed with a wax to keep the embedded particles to a minimum.

- Polishing:
- (1) Rough polishing using a 8 micron diamond paste on a wheel covered with a metcloth and ultrasene lubrication.
 - (2) Semi-final polishing - same as set up #1, but using 3 micron diamond paste.
 - (3) Final polishing - Linde B slurry on a Gamal cloth. The mounts were weighted and run on a Synthron Vibratory polisher set for 40% capacity.

Chemical
Polish:

- (1) Tantalum

50 cc Lactic Acid
30 cc Nitric Acid
2 cc Hydrofluoric Acid
- (2) Titanium

2 cc Hydrofluoric Acid
6 cc Nitric Acid
92 cc Water

2. Observation: All specimens were examined in an 'as polished' condition. Subsequent corrosion penetration measurements and photomicrographs were taken. The specimens were then observed in an etched condition.

3. Etching: The following etchants were used:

321 Stainless Steel

30 cc Glycerine
20 cc Hydrofluoric Acid
10 cc Nitric Acid

Iron

2 cc Nitric Acid
98 cc Ethyl Alcohol

*Alternate chemical polishing and rough polishing may be necessary if the specimens are heavily embedded with SiC particles.

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Haynes-25 and Hastelloy C, N, & X

60 cc Hydrochloric Acid
10 cc Hydrofluoric Acid
Dropwise - 30% Hydrogen Peroxide

Tantalum

25 cc Hydrofluoric Acid
25 cc Nitric Acid
25 cc 30% Hydrogen Peroxide

Titanium

2 cc Hydrofluoric Acid
3 cc Nitric Acid
95 cc Water

Molybdenum

10 g Potassium Ferricyanide
10 g Sodium Hydroxide
100 cc Water

C. Other Analytical Methods Used in Corrosion-Compatibility Studies

In addition to metallography, a number of other analytical disciplines were applied to the analysis and interpretation of compatibility data. The most widely applicable methods were x-ray diffraction and emission spectroscopy.

1. X-Ray Diffraction (XRD)

The basic equipment used was a General Electric XRD-3 goniometer with updated electronic support equipment (Fig. 7). A No. 2SPG power supply unit furnishes filament bias and plate voltages for the ratemeter and preamplifier as well as high voltage for the counter tube and power for the scaler and recorder. The transients or variations in the power supply to the No. 2SPG unit are controlled by the modified Stabeline Voltage regulator in the XRD equipment. The No. 2SPG scaler in conjunction with the SPG Electronic Time Register provides count rate information.

2. Emission Spectroscopy

The basic instrument used is the Baird-Atomic Spectroscopy (Figure 8) embodying a modified Eagle mounting with a concave grating of 3-meter focal length. Gratings of 15,000 and 30,000 grooves-per-inch are available to give resolutions of 5.56 A/mm and 2.73 A/mm, respectively, in the first order. The instrument has

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FIGURE 7. X-Ray Diffraction Equipment

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high trace element sensitivity. Support equipment includes controlled photographic development equipment to assure high uniformity in the plates and a Jarrell-Ash photoelectric densitometer for interpretation of the developed plates.

These complimentary methods and the correlation of the results with the metallographic examination leads to a better understanding of the reaction systems involved in a particular study.

The metallographic microscope ascertains and measures the depth of penetration of foreign phases. In addition, the metallographs may yield significant insights as to the path or mode of penetration of a foreign phase.

In the study of foreign phases, x-ray diffraction methods may identify the foreign phase or phases and give valuable information as to the extent of the phases. In many cases, these techniques can be applied to undisturbed surfaces direct from the experimental environment. Modifications of the Debye-Schener powder and layer techniques are useful in this approach to surface corrosion studies.

Once the surfaces have been scanned by x-rays on the diffraction goniometer emission spectrographic techniques are employed to definitely identify the metallic elements on the surface as well as to estimate their quantitative distribution.

By combining the results of all techniques, a comprehensive understanding of the corrosion reactions and the results of these reactions on the surfaces of the materials under study may be gained.

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III. Results of the Strontium Fuel Form Compatibility Studies

A. Space Fuel Compatibility Studies (September 1964)

Since the initiation of the test program in September 1964, samples were taken after approximately 1, 3, 6, 18 and 19 months exposure at 925°C of the encapsulation metals with the simulated space fuels (described in II-A-1). The following observations were made during each sampling:

a. First sampling - the initial Vycor overpacks were deteriorated (see II.A.1) and the outer surfaces of some test specimens were slightly oxidized. All remaining samples were then overpacked in stainless steel 321 cans.

b. Second and Third samplings - the outer surfaces of the test specimens were free from oxide films which indicated that the stainless steel overpacks provided adequate protection.

c. Fourth and Fifth samplings - the stainless steel overpack cans were severely oxidized and breached; as a result, oxidation of the test specimens (on the O.D.) was also severe.

1. Metallographic Results

Conventional metallographic examinations of all the test specimens were made, yielding maximum and average penetration data (Table I). Differences in the extent of penetration were expected and represented experimental variations. The results of these examinations are discussed in Section IV and photomicrographs of the test specimens are given in the Appendix A.

2. XRD Results

X-ray diffraction studies of the behavior of the fuel-encapsulating materials were conducted with each of the test specimens with close attention being paid to the correlation with the metallographic observations. Throughout the testing period of 19 months, the dominant pattern of the metals remained unchanged and no evidence of the appearance of a new phase structure within the encapsulating material was observed. In most cases, with the exception of the tantalum liners, there was little evidence of the formation, in any great degree, of any 'corrosion phase.' Most of the test specimens indicated the formation of a 'reaction phase'; however, they were usually of a low level and had not attained a sufficient concentration so as to be identified. The test specimens containing the tantalum liners showed complete penetration by all of the fuel forms and the formation of TaF_3 was identified. X-ray diffraction analyses of the fuel forms indicated complete stability of the phases over

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TABLE I
Metallographic Results From Compatibility Test Specimens
Test Temperature 925°C (1700°F)

A. Compatibility with simulated SrF₂ fuel (1 wt% Ca, 0.5 wt% Fe)

Encapsulating Material	Initial Nominal ¹ Wall Thickness (in)	EXPOSURE TIME (mo)*														
		~1 mo.			~2.8			~6.1			~18			~19		
		Specimen Avg Wall Thick. (in)	Penetration		Specimen Avg Wall Thick. (in)	Penetration		Specimen Avg Wall Thick. (in)	Penetration		Specimen ² Avg. Wall Thick. (in)	Penetration		Specimen ² Avg Wall Thick. (in)	Penetration	
			Maximum (in)	Average (in)		Maximum (in)	Average (in)		Maximum (in)	Average (in)		Maximum (in)	Average (in)		Maximum (in)	Average (in)
Haynes 25	0.1185	0.1183	0.0011	0.00029	0.1195	0.00083	0.00023	0.1178	0.00153	0.00088	0.114	0.0006	nil	0.1215	0.0015	0.0008
Ta liner ³ in Haynes 25	0.115	0.1158	0.00057	0.00025	0.1162	Complete Penetration		0.1155	Complete Penetration		0.107	Complete Penetration		0.1083	Complete Penetration	
Hastelloy C	0.1185	0.1181	0.00226	0.00117	0.1179	0.0015	0.00058	0.1180	0.0023	0.0015	0.117	0.0022	0.0008	0.1170	0.001	0.0004
Hastelloy N	0.0815	0.0816	0.0021	0.00098	0.0827	0.0011	0.00071	0.0813	0.0021	0.0015	0.077	0.003	0.0005	0.086	0.002	0.001
Hastelloy X	0.1185	0.1185	0.0012	0.00068	0.1187	0.00187	0.00090	0.1180	0.0027	0.0011	0.0115	0.0024	0.0016	0.1285	0.0075	0.0014
B. Compatibility with fuel from simulated aged feed material (simulated SrF ₂ fuel + 2 1/2 wt% Zr as ZrO ₂).																
Haynes 25	0.1185	0.1182	0.0013	0.00051	0.1195	0.00075	0.00014	0.1175	0.00065	0.00014	0.1115	0.002	0.0005	0.120	0.0005	nil
Ta liner ³ in Haynes 25	0.115	0.1162	0.00087	0.00025	0.1162	Complete Penetration		0.1148	Complete Penetration		0.1095	Complete Penetration		0.109	Complete Penetration	
Hastelloy C	0.1185	0.1180	0.00145	0.00065	0.1173	0.0012	0.00056	0.1183	0.0015	0.0005	0.1155	0.0012	0.0008	0.1195	0.0007	0.0003
Hastelloy N	0.0815	0.0811	0.0023	0.00054	0.0822	0.0014	0.00068	0.0821	0.0028	0.00085	0.071	0.003	0.001	0.083	0.0028	0.0011
Hastelloy X	0.1185	0.1186	0.00156	0.00032	0.1175	0.00061	0.00015	0.1180	0.0015	0.0006	0.117	0.0024	0.0003	0.127	0.0055	0.0011
C. Compatibility with simulated fuel of an aged fuel capsule (simulated SrF ₂ + 5 wt% Zr as ZrF ₄).																
Haynes 25	0.1185	0.1194	0.0013	0.00041	0.1193	0.00099	0.00024	0.1181	0.0017	0.0005	0.115	0.0015	0.0006			
Ta liner ³ in Haynes 25	0.115	--	Complete attack		0.1158	Complete Penetration		0.1137	Complete Penetration		0.1015	Complete Penetration				
Hastelloy C	0.1185	0.1184	0.0027	0.00063	0.1181	0.0041	0.00044	0.1175	0.0028	0.0005	0.1155	0.0012	0.0003			
Hastelloy N	0.0815	0.0816	0.0021	0.0013	0.0815	0.0012	0.00054	0.0811	0.0024	0.0010	0.074	0.002	0.0008			
Hastelloy X	0.1185	0.1177	0.0028	0.0019	0.1177	0.0026	0.0013	0.1180	0.0051	0.0025	0.116	0.0032	0.002			

*Based on 720 hours/month.

- NOTES:
1. Nominal thickness of initial specimens (tolerances $\pm$ 0.002 inches).
 2. Average wall thickness does not account for the corrosion on the O.D. due to the severe oxidation of test specimens.
 3. Tantalum lines capsules were heavily corroded on Ta portion only; there was a diffusion zone between the Ta and Haynes 25.

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the 19 month test period. Diffusion of the containment materials into the fuel matrix was not found in any of the test specimens. The x-ray diffraction data agreed, in all respects, with the optical examination and measurements made on the test specimens.

B. Space Fuel Compatibility Studies (October 1965)

On October 11, 1965, six stainless steel cans were placed into the test furnace. Each stainless steel can contained four 1/2" O.D. x 1" capsules: one of Haynes-25 and one each of Hastelloy C, N and X. Each test capsule was fueled with a mixture of standard SrF_2 + 5 wt% Zr metal based on total Sr, which was pressed to 80% of theoretical density.

The test furnace was operated at 925°C. A visual check of the test cans in the furnace was made periodically (~every 30 days). Upon sampling of the first test can (~125 days exposure) on February 17, 1966, it was noticed that the six container stainless steel cans had been breached. All the test cans were removed from the furnace at this time. The failure of one capsule in a test can apparently started a chain reaction and all but one test can was attacked from both inside and outside.

Of the 24 capsules, initially placed in the furnace, the following were recovered.

<u>Capsule Material</u>	<u>Number Recovered</u>	<u>No. Severely Damaged</u>	<u>Number Destroyed</u>
Haynes-25	1	-	5
Hastelloy C	1	4	1
Hastelloy N	1	3	2
Hastelloy X	6	-	-

Metallography, emission spectroscopy and x-ray diffraction were used in analyzing the test specimens in an attempt to determine the cause of capsule failure and to extract the maximum amount of data from this test. The result of these analyses follows:

1. Metallographic Results

The results of the metallographic examination of the test specimens are shown in Table II. Photomicrographs are given in Appendix A.

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TABLE II

Results of Metallographic Examination of SrF₂ Test Capsules

Total Exposure: ~125 days at 925°C

Fuel: SrF₂ + 5 wt% Zr as the metal

<u>Test Material</u>	<u>Test Can #</u>	<u>Max. Penetration (In)</u>	<u>Avg. Penetration (In)</u>	<u>Remarks</u>
Hastelloy X	1	nil	nil	
	2	0.0155	0.010	XRD indicated oxides present. Possible leakage.
	3	nil	nil	
	4	nil	nil	
	5	0.0003	nil	Heavily corroded on O.D.
	6	nil	nil	
Hastelloy C	1	0.0015		Pit type corrosion, present in isolated spots.
	2	0.014	0.011	Heavily corroded on O.D.
	3	0.018	0.013	
	5	0.009	0.006	Heavily corroded on O.D.
	6	0.011	0.006	Heavily corroded on O.D.
Hastelloy N	1	0.003	--	Pit type corrosion present in isolated spots.
	2	0.010	0.004	Heavily corroded on O.D.
	4	0.010	0.006	Heavily corroded on O.D.
Haynes 25	1	nil	nil	--

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2. XRD Results

The x-ray diffraction and emission spectroscopy results have been correlated. Figure numbers refer to Appendix A.

(a) Hastelloy X from

- (1) Test Can 1: This material showed no penetration or evidence of reaction (Fig. 6a).
- (2) Test Can 2: This specimen showed reaction and penetration (average 0.010") (Fig. 6b). The x-ray diffraction pattern indicated the presence of mixed oxides. This is the only Hastelloy X specimen to show evidence of attack and the presence of the oxide phases is an indication of a leak in the capsule.
- (3) Test Can 3: This specimen showed no evidence of attack (Fig. 6c). X-ray diffraction patterns indicated that no phase changes had occurred on the surface or in the fuel matrix.
- (4) Test Can 4: X-ray diffraction showed no foreign phase formation of the surface. The fuel matrix did not undergo any phase transformation. No penetration of the surface was detected (Fig. 6d).
- (5) Test Can 5: A slight penetration (maximum 0.0003") into the surface was found. (Fig. 6e.) The x-ray diffraction pattern of the surface gave anomalous results, indicating a possible phase change. This capsule was heavily corroded from the outside which may account for a possible phase change in the metal structure. X-ray diffraction patterns of the fuel matrix indicated no phase changes.
- (6) Test Can 6: No evidence of penetration was detected in this specimen (Fig. 6f). X-ray diffraction patterns of the fuel matrix showed no phase changes.

(b) Hastelloy C from

- (1) Test Can 1: Specimen showed pit type corrosion with a maximum penetration of 0.0015" (Fig. 6g).
- (2) Test Can 2: Specimen showed a maximum penetration of 0.014" (Fig. 6h). X-ray diffraction patterns indicated the presence of an unidentified second phase.

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- (3) Test Can 3: The specimen showed general attack with maximum penetration of 0.018" (Fig. 6i). The x-ray diffraction pattern of the fuel matrix showed the presence of an unidentified second phase.
- (4) Test Can 4: Destroyed
- (5) Test Can 5: This specimen showed attack with a maximum penetration of 0.009" (Fig. 6j). The x-ray diffraction pattern of the fuel matrix indicated a second phase which was identified as SrZrO_3 (evidence of oxygen penetration into the test specimen).
- (6) Test Can 6: The specimen showed penetration and reaction (Fig. 6k). X-ray diffraction patterns indicated the presence of a new phase on the surface. The results were obscured by outside surface corrosion.

(c) Hastelloy N from

- (1) Test Can 1: The specimen showed pit-type corrosion with a maximum penetration of 0.003" (Fig. 6L). X-ray diffraction patterns indicated a phase change at the surface of the test material. No changes in the fuel matrix were noted.
- (2) Test Can 2: A maximum penetration of 0.010" was noted (Fig. 6m). X-ray diffraction patterns of the surface indicated a second unidentified phase. No changes in the fuel matrix were noted.
- (3) Test Can 3: Destroyed.
- (4) Test Can 4: Corrosion with a maximum penetration of 0.010" was noted (Fig. 6n). X-ray diffraction patterns of the surface indicated the presence of a NiO phase and a third unidentified phase. No changes in the fuel matrix were noted.
- (5) Test Can 5: Destroyed.
- (6) Test Can 6: Destroyed.

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(d) Haynes 25 from

- (1) Test Can 1: The specimen showed no penetration or corrosion (Fig. 6o). X-ray diffraction patterns of the fuel matrix indicated the presence of a weak second phase, tentatively identified as SrZrO_3 (evidence of leakage).
- (2) Test Cans 2-6: Destroyed.

C. Strontium Metal Compatibility Studies

Six containment capsules were fabricated from stainless steel 321 to protect the test specimens. These containment capsules were fueled as described in Section IIA-2.

One containment capsule at each temperature was removed from the test furnace after approximately 200, 600 and 1000 hours exposure to the test temperature. The following observations were made during each sampling:

(a) 700°C Test (All Samplings)

1. The interior surface of the stainless steel 321 test can was clear, which indicated that the argon atmosphere within the capsule was clean.
2. The 1/4" thick strontium metal wafers had melted although the test temperature was $\sim 70^\circ\text{C}$ below the melting point. This was a possible indication of an alloying affect with one of the test metals which formed a low melting eutectic mixture.
3. The test coupons were removed by dissolving the Sr^0 metal. Only 8-9 of the 11 test coupons were recovered, each of which was examined by metallographic and x-ray diffraction techniques.
4. The test coupons of Cu and Ni had apparently dissolved in the Sr metal.
5. The manganese (95% Mn-5% Cu) test coupon was extremely brittle at the first sampling after 260 hours. The manganese test coupon had apparently dissolved in the strontium metal during the second and third sampling periods.

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(b) 925°C Test (All Samplings)

1. The surface of the inner stainless steel 321 can was clear, which indicated the argon atmosphere between the inner test can and the protection overpack was "clean."
2. The test coupons were removed by dissolving the strontium metal in water. Only 4 to 5 of the 8 test coupons were recovered, each of which was examined by metallographic and x-ray diffraction techniques.
3. The test coupons of Hastelloy C, Hastelloy N and titanium had apparently dissolved in the strontium metal. The tantalum test coupon had apparently dissolved in the strontium metal during the third test period.

1. Metallographic Results

Conventional metallographic examinations of all the test specimens were made (Table III and IV). The results of these examinations are discussed in Section IV, and photomicrographs of the test specimens are given in Appendix A.

2. XRD Results

X-ray diffraction analyses of all the test coupons, along with supporting emission spectroscopy, was performed and the observations from these analyses are presented in Appendix B.

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TABLE III

Results of Metallographic Examination of Test Coupons Exposed to Strontium Metal at 700°C

Exposure Time (Hours)	260			764			1434		
Test Material	Total Thickness (in)	Max Penetration (in)	Avg Penetration (in)	Total Thickness (in)	Max Penetration (in)	Avg Penetration (in)	Total Thickness (in)	Max Penetration (in)	Avg Penetration (in)
S.S. 321	0.0672	0.0008	0.0004	0.0680	0.0011		0.066	0.0015	0.0015
Tantalum	0.031	0.0007(DZ)*		0.0299	0.0002		0.033	0.005	0.001(DZ)
Titanium	0.0308	0.0014(DZ)*		0.0383	0.014(DZ)		0.039	2nd phase penetration in isolated areas	
Molybdenum	0.031	0.0015	0.0003	0.0303	0.0005	0.0003	0.031	nil	0.0035**
Manganese	----- Complete Penetration -----								
Copper									
Wrought Iron	0.0341	0.003	0.0017	0.0353	0.002		0.0355	0.0075	0.005**
Hastelloy X	0.060	0.0005		0.0616	0.008(DZ)		0.061	0.003	--
Hastelloy N	0.063	0.025		0.0625	0.020		0.063	Massive Attack	
Hastelloy C	0.0604	0.003		0.0607	0.009		0.0605	0.0075	Specimen ends dissolved away
Haynes 25	0.063	0.0009		0.0632	0.0009		0.063	0.004	0.001
Copper	-----	Dissolved	-----						
Nickel	-----	Dissolved	-----						

*DZ = Diffusion Zone

** = Plate

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TABLE IV

Results of Metallographic Examination of Test Coupons Exposed to Strontium Metal at 925°C

Exposure Time (Hours)				816			2036		
Test Material	Total Thickness (in)	Max Penetration (in)	Avg Penetration (in)	Total Thickness (in)	Max Penetration (in)	Avg Penetration (in)	Total Thickness (in)	Max Penetration (in)	Avg Penetration (in)
S.S. 321	--	--	--	0.0645	--	0.0005	0.065	nil	nil
Tantalum	0.030	0.002	0.0003	0.029	0.004	0.002	Dissolved		
Titantium		Dissolved			Dissolved		Dissolved		
Molybdenum	0.031	0.0001	0.00003	0.0315	0.0008	0.0005	0.0318	nil	nil
Wrought iron*	0.033	0.004	0.0025	0.036	Complete penetration along grain boundaries		0.0373	0.004	Complete penetration along grain boundaries
Hastelloy X	0.0608	0.034	0.022	0.062	Complete attack			Complete attack	
Hastelloy C	--	Dissolved	--	--	Dissolved	--	--	Dissolved	--
Hastelloy N	--	Dissolved	--	--	Dissolved	--	--	Dissolved	--
Haynes 25	0.0644	0.0072	0.0045	0.062	0.016	0.009	0.058	0.010	0.0025

*Diffusion zone along grain boundaries.

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IV. Discussion of Results, Conclusions and Recommendations

A. Space Fuel Compatibility Studies (September 1964)

From the data presented in Table I and correlation with the XRD analyses, the following observations can be made:

a) The average wall thickness of the test specimens for the first three samplings, in which the oxidation protection of the specimens prevented oxidation of the outer surfaces, was always within the tolerances for wall thickness of the initial specimens.

b) X-ray diffraction analyses of the fuel forms indicated complete stability over the 19 month test period. Diffusion of the containment materials into the fuel matrices was not found in any of the test specimens.

These observations indicate that the penetration data given in Table I are actual penetrations into the initial test specimen wall and that 'dissolution' of the encapsulating materials into the fuel forms did not occur over the ~19 month test period. Therefore, from the data in Table I, the following conclusions can be made:

1. The tantalum liners are not compatible with any of the three simulated fuel forms.
2. After ~19 months exposure, Haynes 25 showed the most resistance to attack by the three simulated fuel forms.
3. Slight differences can be seen in the performance of the Hastelloy series of encapsulating materials. After 19 months of testing, Hastelloy C was most compatible and Hastelloy X was the least compatible with the three simulated fuel forms.

In summary, the compatibility of the encapsulating alloys with the three simulated fuel forms of strontium fluoride in order of decreasing compatibility is:

Haynes 25
Hastelloy C
Hastelloy N
Hastelloy X
Tantalum (incompatible)

B. Space Fuel Compatibility Studies (October 1965)

The addition of zirconium metal to the space fuel matrix had an appreciable

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effect on the compatibility performance of the encapsulating materials. This was dramatically illustrated by the severe damage to greater than 50% of the test specimens. The data presented in Section II, Part B was analyzed and the following mechanism is postulated as the probable cause of capsule failure:

1. A capsule of Hastelloy C or N was breached by the fuel matrix ($\text{SrF}_2 + 5 \text{ wt\% Zr}$) or by a leak in the container. Hastelloy C and N were the only test materials in Test Can #1, which was essentially intact at sampling, that indicated corrosion of the interior surfaces (Figs. 6g and 6L in Appendix A).
2. The fuel matrix from the breached fuel capsule then attacked the stainless steel 321 overpack admitting O_2 into the system.
3. The presence of oxygen and the fuel mixture provided a highly corrosive environment and initiated a 'chain reaction' in which the other fuel capsules and adjacent test cans were destroyed or severely attacked.

Based on this mechanism, the following conclusions and recommendations are made:

1. Of the materials tested, Hastelloy X appears to provide the best containment for the fuel matrix ($\text{SrF}_2 + 5 \text{ wt\% Zr}$). All six test capsules of Hastelloy X survived exposure to the highly corrosive environment of SrF_2 , Zr metal and air at high temperature (925°C) Figs. 6a thru 6f in Appendix A.
2. Haynes 25 is worthy of further study. The Haynes 25 capsule which was recovered intact showed little or no corrosion of the interior surfaces. (Fig. 6a in Appendix A.)
3. The presence of oxygen and the fuel mixture provided a highly corrosive environment and initiated a 'chain reaction' in which the other fuel capsules and adjacent test cans were destroyed or severely attacked.
4. Hastelloy C and N are not candidate containment materials for the fuel matrix tested. The test capsules of Hastelloy C and N which were recovered intact indicated the presence of pit type corrosion (Figs. 6g and 6L) in Appendix A.

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5. It is recommended that both Hastelloy X and Haynes 25 be tested further to establish their compatibility with the space fuel and its stable decay product of Zr metal.
6. It is further recommended that each test material be tested in separate furnaces to preclude the occurrence of a 'chain reaction' as postulated above.

C. Strontium Metal Compatibility Studies

It should be emphasized that the studies performed were only of a 'scoping' type in nature. Many materials were tested at one time and for fairly short time periods. The compatibility of some materials may have been adversely affected by the presence of the other test elements. With this fact in mind, the results presented in Section 3, Part C were evaluated and the following conclusions and recommendations are made:

1. For the 700°C Test

- (a) Materials not compatible with Sr metal for containment purposes at 700°C over long periods:
 - (1) pure metallic Cu
 - (2) metallic Ni
 - (3) Mn-5% Cu alloy
 - (4) Hastelloy C
 - (5) Hastelloy X (questionable due to presence of Cu on the surface). See Appendix B.
 - (6) Hastelloy N
- (b) Materials showing good compatibility with Sr metal over the 1434 hr. test period:
 - (1) Molybdenum
 - (2) S.S. 321
 - (3) Haynes 25

It is recommended that longer tests be run with the above materials, in which the Sr metal is encapsulated in each candidate material.

- (c) Materials worthy of further study (See Appendix B):
 - (1) Wrought iron
 - (2) Titanium
 - (3) Tantalum
 - (4) Hastelloy X

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The true compatibility of these four materials with Sr may have been hidden due to the effects of the other test materials which were present. It is recommended that further testing include the above materials. The Sr metal should be encapsulated in the presence of one test material, thereby eliminating the effect of other elements on the test.

2. For the 925°C Test

- (a) Materials not compatible with Sr metal for containment purposes at 925°C for extended periods:
 - (1) Hastelloy C
 - (2) Hastelloy N
 - (3) Hastelloy X
 - (4) Titanium
- (b) Materials showing good compatibility with strontium metal at 925°C for the 2000 hour test period:
 - (1) Molybdenum
 - (2) S.S. 321

It is recommended that longer tests be run with the above materials, in which the Sr metal is encapsulated in each candidate material.

- (c) Materials worthy of further study (See Appendix B).
 - (1) Wrought iron
 - (2) Tantalum
 - (3) Haynes 25

The true compatibility of these materials with Sr may have been hidden due to the effects of the other test materials which were present. It is recommended that further testing include the above materials. The Sr metal should be encapsulated in the presence of one test material, thereby eliminating the effect of other elements on the test.

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1. MND-3062-2, Properties and Selection of a Strontium Space Fuel, February 1964.
2. E. L. Wheeler, Scientific Glassblowing, Interscience Publishers, Inc., N. Y., 1958.
3. T. Lyman, Metals Handbook, Volume 1, 8th Edition, American Society of Metals, Novelty, Ohio, 1961.

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APPENDIX A

Photomicrographs of Test Specimens

Photomicrographs of all the test specimens are included in this appendix. Unless otherwise noted, all photos were taken in the as polished condition and at 250X magnification.

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FIGURE 1

Compatibility of Haynes 25 With Simulated Space Fuel Forms

A. With standard space fuel

a. As received

b. One month exposure

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FIGURE 1 (Cont)

c. 2.8 months exposure

d. 6 months exposure

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FIGURE 1 (Cont)

e. 18 months exposure

f. 19 months exposure

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FIGURE 1 (Cont)

B. With standard space fuel + 2 1/2 wt% Zr as ZrO_2

a. One month exposure

b. 2.8 months exposure

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FIGURE 1 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 1 (Cont)

e. 19 months exposure

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FIGURE 1 (Cont)

C. With standard space fuel + 5 wt% Zr as ZrF_4

a. One month exposure

b. 2.8 months exposure

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FIGURE 1 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 2

Compatibility of Tantalum With Simulated Space Fuels

A. With standard space fuels

a. As received

b. One month exposure

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FIGURE 2 (Cont)

c. 2.8 months exposure

d. 6 months exposure

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FIGURE 2 (Cont)

B. With standard space fuel + 2 1/2 wt% Zr as ZrO_2

a. One month exposure

b. 2.8 months exposure

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FIGURE 2 (Cont)

c. 6 months exposure

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FIGURE 2 (Cont)

C. With standard space fuel + 5 wt% Zr as ZrF_4

a. One month exposure

b. 2.8 months exposure

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FIGURE 2 (Cont)

c. 6 months exposure

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FIGURE 3

Compatibility of Hastelloy C With Simulated Space Fuels

A. With standard space fuel

a. As received

b. One month exposure

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FIGURE 3 (Cont)

c. 2.8 months exposure

d. 6 months exposure

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FIGURE 3 (Cont)

e. 18 months exposure

f. 19 months exposure

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FIGURE 3 (Cont)

B. With standard space fuel + 2 1/2 wt% Zr as ZrO_2

a. One month exposure

b. 2.8 months exposure

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FIGURE 3 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 3 (Cont)

e. 19 months exposure

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FIGURE 3 (Cont)

C. With standard space fuel + 5 wt% Zr as ZrF_4

a. One month exposure

b. 2.8 months exposure

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FIGURE 3 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 4

Compatibility of Hastelloy N With Simulated Space Fuels

A. With standard space fuel

a. As received

b. One month exposure

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FIGURE 4 (Cont)

c. 2.8 months exposure

d. 6 months exposure

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FIGURE 4 (Cont)

e. 18 months exposure

f. 19 months exposure

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FIGURE 4 (Cont)

B. With standard space fuel + 2 1/2 wt% Zr as ZrO₂

a. One month exposure

b. 2.8 months exposure

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FIGURE 4 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 4 (Cont)

e. 19 months exposure

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FIGURE 4 (Cont)

C. With standard space fuel + 5 wt% Zr as ZrF_4

a. One month exposure

b. 2.8 months exposure

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FIGURE 4 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 5

Compatibility of Hastelloy X With Simulated Space Fuels

A. With standard space fuel

a. As received

b. One month exposure

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FIGURE 5 (Cont)

c. 2.8 months exposure

d. 6 months exposure

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FIGURE 5 (Cont)

e. 18 months exposure

f. 19 months exposure

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FIGURE 5 (Cont)

B. With standard space fuels + 2 1/2 wt% Zr as ZrO₂

a. One month exposure

b. 2.8 months exposure

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FIGURE 5 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 5 (Cont)

e. 19 months exposure

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~~CONFIDENTIAL~~

FIGURE 5 (Cont)

C. With standard space fuel + 5 wt% Zr as ZrF_4

a. One month exposure

b. 2.8 months exposure

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FIGURE 5 (Cont)

c. 6 months exposure

d. 18 months exposure

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FIGURE 6

Compatibility of Strontium Fluoride (With Zirconium Metal Additive)
With Various Encapsulating Alloys
Total exposure 125 days
all photos in as polished condition.
Magn. 125X

a. Hastelloy X
Test can 1

b. Hastelloy X
Test can 2

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FIGURE 6 (Cont)

c. Hastelloy X
Test can 3

d. Hastelloy X
Test can 4

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FIGURE 6 (Cont)

e. Hastelloy X
Test can 5

f. Hastelloy X
Test can 6

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FIGURE 6 (Cont)

g. Hastelloy C
Test can 1

h. Hastelloy C
Test can 2

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FIGURE 6 (Cont)

i. Hastelloy C
Test can 3

j. Hastelloy C
Test can 5

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FIGURE 6 (Cont)

k. Hastelloy C
Test can 6

l. Hastelloy N
Test can 1

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FIGURE 6 (Cont)

m. Hastelloy N
Test can 2

n. Hastelloy N
Test can 4

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FIGURE 6 (Cont)

- o. Haynes 25
Test can 1

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FIGURE 7

Compatibility of Various Materials With Strontium Metal
At 700°F

Magn. 125X

A. Molybdenum

a. 260 hrs. exposure

b. 764 hrs. exposure

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FIGURE 7 (Cont)

c. 1434 hrs. exposure

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FIGURE 7 (Cont)

B. Stainless Steel #321

a. 764 hrs. exposure

b. 1434 hrs. exposure

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FIGURE 7 (Cont)

C. Haynes 25

a. 746 hrs. exposure

b. 1434 hrs. exposure

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FIGURE 7 (Cont)

D. Hastelloy C

a. 764 hrs. exposure

b. 1434 hrs. exposure

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FIGURE 7 (Cont)

E. Hastelloy X

a. 764 hrs. exposure

b. 1434 hrs. exposure

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FIGURE 7 (Cont)

F. Hastelloy N

a. 260 hrs. exposure

b. 764 hrs. exposure

c. 1434 hrs. exposure

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FIGURE 7 (Cont)

G. Wrought Iron

a. 260 hrs. exposure

b. 764 hrs. exposure

c. 1434 hrs. exposure

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FIGURE 7 (Cont)

H. Titanium

a. 260 hrs. exposure

b. 764 hrs. exposure

c. 1434 hrs. exposure

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FIGURE 8

Compatibility of Various Materials With Strontium Metal at 925°C

Magn. 125X

A. Molybdenum

a. 288 hrs. exposure

b. 816 hrs. exposure

c. 2036 hrs. exposure

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FIGURE 8 (Cont)

B. Stainless Steel #321

a. 816 hrs. exposure

b. 2036 hrs. exposure

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FIGURE 8 (Cont)

C. Haynes 25

a. 288 hrs. exposure

b. 816 hrs. exposure

c. 2036 hrs. exposure

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FIGURE 8 (Cont)

D. Hastelloy X

a. 288 hrs. exposure

b. 860 hrs. exposure

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FIGURE 8 (Cont)

E. Wrought Iron

a. 288 hrs. exposure

b. 816 hrs. exposure

c. 2036 hrs. exposure

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FIGURE 8 (Cont)

F. Tantalum

a. 288 hrs. exposure

b. 816 hrs. exposure

c. 2036 hrs. exposure

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APPENDIX B

Tabulation of XRD Analyses of Test Coupons from Strontium Metal Compatibility Tests

A. 700°C Test

1. 260 hours exposure

(a) Hastelloy X coupon - Showed the least surface change both from x-ray phase study as well as metallographic study.

(b) Hastelloy C coupon - Showed a small surface phase not found on the original material. This has a very low concentration. Metallography showed no penetration of the coupon beyond the surface.

(c) Hastelloy N coupon - Not only exhibited a second surface phase as the result of the test, but metallographic examination revealed some definite penetration by a second phase along the grain boundaries. (Fig. 7-F-a.)

(d) Haynes 25 coupon - Exhibited no new phase formation on the surface as the result of the test. Metallography indicated a very slight penetration by an unknown phase.

(e) Wrought iron coupon - Exhibited no phase formation as a result of the test. Metallographic examination showed a definite penetration by a foreign phase, perhaps a temperature sensitive diffusion reaction. (Fig. 7-G-a.)

(f) Molybdenum coupon - X-ray diffraction indicated a heavy new phase on the surface of the molybdenum. This phase was not identifiable. Emission spectroscopy confirmed the existence of a coating on the molybdenum containing no molybdenum but Ni, Cu, and Sr. Metallography showed no evidence of penetration of the molybdenum below this coating. Either the molybdenum is not attacked by the Sr or this coating formation protected it. (Fig. 7-A-c.)

(g) Tantalum coupon - Was coated with a foreign mixture of Cu, Ni, and Sr. Beneath this coating no evidence of attack of the tantalum was observed.

(h) Titanium coupon - Was like the molybdenum and tantalum, coated with a foreign mixture of Cu, Ni, and Sr. Beneath the coating there was evidence of attack and penetration. (Fig. 7-H-a.)

(i) Manganese - 5% Cu coupon - A new alloy phase formed on the surface of the manganese-5% copper coupon. The parent coupon penetrated, embrittled and cracked in places. No compatibility was exhibited.

NOTE: Figure numbers refer to Appendix A.

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2. 764 hours exposure

- (a) Titanium coupon - Evidence of heavy attack and penetration followed by cracking (Fig. 7-H-b).
- (b) Hastelloy N coupon - Massive penetration leading to attack (Fig. 7-F-b).
- (c) Hastelloy C coupon - Massive penetration and attack (Fig. 7-D-a).
- (d) Hastelloy X coupon - Significant penetration along grain boundaries (Fig. 7-E-a).
- (e) Tantalum coupon - No penetration but a slight foreign phase on surface.
- (f) Molybdenum coupon - Slight foreign phase on surface (Fig. 7-A-b).

3. 1434 hours exposure

- (a) Hastelloy N coupon - Showed massive attack, complete penetration at grain boundaries, ends of test coupon were completely eroded away (Fig. 7-F-c).
- (b) Wrought iron coupon - The surface of this coupon had become coated with Al (impurity in Sr metal) and Ni (dissolved test coupon) and showed FeAl, Fe₃Al patterns on x-ray diffraction. The Sr content of this coating was low. This surface reaction and formation of a protective layer leaves the question of the true compatibility of wrought iron with Sr unanswered. The surface phase may be the compatible phase (Fig. 7-G-c).
- (c) Hastelloy X coupon - This material showed some penetration of a surface phase. Sr and Cu were in high concentration on the surface (Fig. 7-E-b) which indicated possible corrosion due to Cu and not necessarily due to Sr.
- (d) Molybdenum coupon - Coatings of Ni, Mn, Al and Cu alloys with varying quantities of Sr on the surface of the Mo are not considered to deny that this element exhibits excellent compatibility with Sr, since no evidence of reaction or penetration has been found (Fig. 7-A-c).
- (e) Hastelloy C coupon - Ends were completely eroded away on the test coupon of this material (Fig. 7-D-b).
- (f) Tantalum coupon - This material had a 0.004" plating on the surface composed of Ni, Al, Cu, Sr and Mn. Under the plated deposit, was a surface with a diffusion zone of 0.001". The diffusing elements were not identified, therefore, the question of the true compatibility of Ta with Sr is doubtful.

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(g) Haynes 25 coupon - No compound formation with the surface contaminating Cu, Sr and Al was detected. This material showed good compatibility in spite of the slight penetration detected (Fig. 7-C-b).

(h) Titanium coupon - This material showed complete penetration by a second phase (Fig. 7-H-C). A 0.014 plating was found on the surface containing Sr, Ti, Ni, Cu and Al which prevented any conclusion, as to the compatibility with Sr, to be reached.

B. 925°C Test

1. 288 hours exposure

(a) Wrought iron coupon - The metallographic examination showed an intense grain boundary diffusion pattern (Fig. 8-E-a). Grain growth was evident. Spectroscopy showed surface layer to be high in Sr, Ni and Mg (impurity in Sr) content.

(b) Hastelloy X coupon - Spectroscopy indicated high Sr on surface, therefore, attack can be attributed to Sr. Surface phase was not identified by XRD, however, it indicated that surface contained two or more phases. The depth of penetration indicated that Hastelloy X would not contain Sr at 925°C for long periods of time (Fig. 8-D-a).

(c) Molybdenum coupon - Metallography indicated practically no penetration (Fig. 8-A-a). Emission spectroscopy showed that surface was heavily coated with Sr. This pointed to Mo as one of the most promising containment materials for Sr at 925°C.

(d) Haynes 25 coupon - Penetration into this material was less than that into Hastelloy X, but more than that into Mo (Fig. 8-C-a). Surface studies indicated that the Sr content of the surface was high; therefore, the attack can be attributed to Sr. Haynes 25, therefore, would provide short term containment of Sr at 925°C.

(e) Tantalum coupon - Metallographic examination indicated a uniform surface attack (Fig. 8-F-a). The test material became quite brittle. Spectroscopy showed the surface to be heavily coated with Sr, therefore, the attack can be attributed to Sr. Based on the indicated attack and embrittlement of the test coupon, Ta can be considered as a doubtful containment material for Sr at 925°C.

2. 816 hours exposure

(a) Tantalum coupon - The average penetration into the tantalum

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was 0.002". Physically, the Ta had become extremely brittle. Emission spectroscopy showed intimate contact and high surface density of Sr on the Ta. The phases formed have not been identified (Fig. 8-F-b).

(b) Haynes 25 coupon - The average penetration into the Haynes 25 was 0.009". The metallograph showed grain growth and dissolution of the surface (Fig. 8-C-b).

(c) Wrought iron coupon - The metallograph showed complete penetration by a second phase along the grain boundaries. The 0.0025" diffusion zone at the surface was found to be high in strontium content, but also contained nickel, chromium and magnesium (Fig. 8-E-b).

(d) Molybdenum coupon - The average penetration into the Mo was 0.0005". The metallograph showed no diffusion zone, but a coating layer. The coating consisted principally of Sr, Cr and Mg (Fig. 8-A-b).

(e) Hastelloy X coupon - The metallograph clearly showed massive attack with complete penetration (Fig. 8-D-b).

(f) S.S. 321 coupon - The configuration prevented x-ray diffraction of surfaces (Fig. 8-B-a). The metallographical studies indicated about the same average penetration experienced by the molybdenum.

3. 2036 hours exposure

(a) Haynes 25 coupon - In general, the material showed extensive surface reaction, with a maximum penetration of 0.010" (Fig. 8-C-c). Unidentified phases on the surface were high in Sr, Ca and Mg.

(b) Wrought iron coupon - The specimen showed complete penetration along grain boundaries (Fig. 8-E-c). The surface consisted of two phases, the Fe-austenite phase and an unidentified phase.

(c) Molybdenum coupon - The specimen showed no penetration of the surface. The heavy coating of the surface was found to be high in Sr, Mg and Ca, with traces of Cr, Fe and Ni. The crack in the specimen is not attributed to corrosion but to capsule fabrication (Fig. 8-A-c).

(d) Hastelloy X coupon - The test specimen was heavily attacked. The powder residue was not amenable to metallographic or XRD analyses.

(e) Stainless Steel 321 coupon - The configuration prevented x-ray diffraction of the surfaces. Metallographic examination indicated no attack or penetration of the surface (Fig. 8-B-b).

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