**Quarterly Report on the Strontium
Heat Source Development
Program, Advanced Systems and
Materials Production Division for
July - September 1978**

H. T. Fullam

December 1978

**Prepared for the U.S. Department of Energy
under Contract EY-76-C-06-1830**

**Pacific Northwest Laboratory
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QUARTERLY REPORT ON THE STRONTIUM HEAT
SOURCE DEVELOPMENT PROGRAM, ADVANCED
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DIVISION FOR JULY-SEPTEMBER 1978

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Pacific Northwest Laboratory
Richland, Washington 99352

SUMMARY

Metallographic examination of the specimens from the 12,000-hr tests with $^{90}\text{SrF}_2$ was completed this quarter. The 12,000-hr Hastelloy C-276 specimens exhibited substantially greater attack than did the 6000-hr specimens. The 12,000-hr Haynes 25 and TZM specimens, however, showed no more attack than did the 6000-hr specimens.

The 20,000-hr tests with $^{90}\text{SrF}_2$ have also been completed, and arrangements have been made to ship the capsules to Oak Ridge National Laboratory (ORNL) for examination.

Hydrostatic pressure testing of the AISI-1018 steel prototype outer capsules was completed. The 0.4-in. wall capsule collapsed at 11,200 psi, while the 0.5-in. wall capsule collapsed at 15,900 psi. The 0.6-in. wall capsule did not collapse at the maximum test pressure of 20,000 psi, but did exhibit slight ovality after testing. The capsules were subjected to a helium leak check and no leaks were detected. Additional evaluation and destructive examination of the capsules are now underway.

The 5000-hr oxidation tests with Hastelloy S and Hastelloy C-4 were completed. At 600°C oxidation of both alloys continued to adhere to parabolic rate relationships. At 700°C and 800°C, however, oxidation of the two alloys after 5000-hr exposure was less than predicted by the parabolic rate equations that fitted the shorter-term data. Testing of the tensile specimens from the 1000- and 2500-hr oxidation tests indicates that oxidation of the two alloys at 600°C-800°C has no significant effect on their room-temperature tensile properties.

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STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM

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At Hanford, strontium is separated from the high-level waste, converted to the fluoride, and doubly encapsulated in small, high-integrity containers for subsequent long-term storage. The fluoride conversion, encapsulation, and storage take place in the Waste Encapsulation and Storage Facilities (WESF). The encapsulated strontium fluoride represents an economical source of ^{90}Sr if the WESF capsule can be licensed for heat source applications under anticipated use conditions. The objectives of this program are to obtain the data needed to license $^{90}\text{SrF}_2$ heat sources and specifically the WESF $^{90}\text{SrF}_2$ capsules. The information needed for licensing can be divided into three general task areas:

- Task 1 - Chemical and Physical Properties of $^{90}\text{SrF}_2$
- Task 2 - $^{90}\text{SrF}_2$ Compatibility Studies
- Task 3 - Capsule Qualification and Licensing

Efforts are proceeding concurrently on all three tasks to obtain the required information.

TASK 1 - CHEMICAL AND PHYSICAL PROPERTIES OF $^{90}\text{SrF}_2$ (H. T. Fullam)

(No activity this quarter.)

TASK 2 - $^{90}\text{SrF}_2$ COMPATIBILITY STUDIES (H. T. Fullam)

All of the remaining compatibility tests are proceeding on schedule. The 20,000-hr tests with $^{90}\text{SrF}_2$ have been completed, and arrangements have been made to ship the test capsules to Oak Ridge National Laboratory (ORNL) for sectioning and evaluation.

Metallographic examination of the test specimens from the 12,000-hr tests with $^{90}\text{SrF}_2$ has been completed by ORNL. Estimates of metal attack, based on the photomicrographs obtained, are presented in Table 1. The results obtained in the 1000- and 6000-hr tests are also given in Table 1 for comparison purposes. Photomicrographs of the 12,000-hr specimens are presented in Figures 1-3.

The Hastelloy C-276 specimens tested for 12,000 hr suffered considerably more attack than did the 6000-hr specimens, especially at 800° and 1000°C . Both chemical attack and microstructural changes increased sharply with increasing exposure temperature. In addition, the attack mechanisms appeared to vary with temperature. Similar results were observed in the 1000- and 6000-hr tests. Electron microprobe analysis of the 12,000-hr specimens is now underway, and a discussion of attack mechanisms will be provided when the microprobe data are available.

Overall, the Haynes Alloy 25 and TZM specimens exposed to $^{90}\text{SrF}_2$ for 12,000 hr suffered no greater attack than did the 6000-hr specimens. Attack mechanisms and temperature effects were similar to those observed in the shorter-duration tests. Electron microprobe analysis of the specimens is now underway, and discussions of attack mechanism will be given when the microprobe analyses are completed.

TABLE 1. Attack of Metal Specimens Exposed to $^{90}\text{SrF}_2$, Couple S/V = 4.5 cm^{-1}

Material	Temperature °C	Depth of Metal Affected, mils					
		Chemical Attack			Change in Microstructure		
		1,000 hr	6,000 hr	12,000 hr	1,000 hr	6,000 hr	12,000 hr
Hastelloy C-276	600	3	3	5	4	4	7
	600	3	3	4	5	5	10
	800	7	8	12	12	20	25
	800	6	--(a)	11	11	--(a)	18
	1,000	7	8	20	15	24	>60
	1,000	10	12		15	>30	
Haynes 25	600	1	3	4	0	0	4
	600	2	5	3	0	0	4
	800	5	10	5	10	16	8
	800	4	11	7	6	18	10
	1,000	6	12	10	8	18	25
	1,000	7	10		9	10	
TZM	600	1	4	1	0	0	0
	600	2	1	2	0	0	0
	800	2	2	2	0	0	2
	800	2	2	2	0	0	0
	1,000	2	2	3	0	35	0
	1,000	1	2		0	35	

(a) Specimen lost due to furnace malfunction

FIGURE 1. Hastelloy C-276 Specimens Exposed to $^{90}\text{SrF}_2$ for 12,000 hr - Capsule S/V = 4.5 cm^{-1}

FIGURE 2. Haynes Alloy 25 Specimens Exposed to $^{90}\text{SrF}_2$ for 12,000 hr - Capsule S/V = 4.5 cm^{-1}

FIGURE 3. TZM Specimens Exposed to $^{90}\text{SrF}_2$ for 12,000 hr - Capsule S/V = 4.5 cm^{-1}

TASK 3 - CAPSULE QUALIFICATION AND LICENSING (D. G. Atteridge)

Capsule Design

This quarter's work was directed towards experimental verification of the proposed outer capsule design presented in the January-March 1978 Progress Report.⁽¹⁾ A schematic of the prototype capsule can be seen in Figure 4. The required wall and end cap thicknesses were determined through computer simulation modelling, and it was felt that actual proof-of-principle testing of the design was required before the prototype capsules were fabricated from Hastelloy S.

It was decided to use AISI-1018 steel stand-in capsules to simulate the expected properties of the actual capsule material (Hastelloy S) after ten years' exposure at 800°C. The stand-in capsule material was chosen so that it would match the expected room-temperature yield strength of the aged Hastelloy capsule and so that the impact energy of the stand-in capsule could be matched to that of the aged Hastelloy capsule.

The end cap and closure weld design used with the steel capsule was the same as will be used on the prototype capsule. The steel stand-in capsule closure weld was also made by the same automated TIG welding process that will be used in welding the prototype capsules and the actual hot cell capsules. A schematic of the end cap configuration illustrating the closure weld placement is shown in Figure 5. A weld parameter study was conducted to assure a full penetration closure weld in the stand-in capsules, and a photomicrograph of a typical completed closure weld is shown in Figure 6. A photograph of actual end components of a stand-in capsule and a finished closure weld can be seen in Figure 7. A completed capsule and its components are shown in Figure 8. The small tube inserted into the top of the end cap was used for helium leak checking the capsules.

Stand-in capsules with three different wall thicknesses were fabricated in order to allow sufficient evaluation of the hydrostatic collapse model. The wall thicknesses chosen were 0.4 in., 0.5 in., and 0.6 inch. A stand-in capsule wall thickness of 0.5 in. is being used for the drop and the puncture tests. The stand-in capsules were fabricated from heavy-walled tubing having a 2.375-in. inside diameter and a 0.625-in. wall thickness. The inside diameter on all stand-in capsules was left as the original diameter, and the wall thickness was reduced in order to achieve the required wall thicknesses.

Three steel stand-in capsules were used for the hydrostatic pressure test analysis. The three capsules had different wall thicknesses (0.4 in., 0.5 in., and 0.6 in.) and were used to determine the failure mode and failure load as a function of capsule wall thickness. The hydrostatic pressure on the capsules was increased incrementally from 10,000 to 20,000 psi in 1000-psi increments, with a dimensional check being made between pressure increases. The two thinner-walled capsules collapsed prior to reaching 20,000 psi, while the 0.6-in.-wall capsule exhibited slight ovality at 20,000 psi. Photographs of the capsules after testing are shown in Figure 9. A tabulation of the predicted and actual collapse pressures is given in Table 2 as a function of capsule wall thickness.

FIGURE 4. Prototype Outer Capsule Having a 0.45-in. Wall

FIGURE 5. Schematic of Proposed Interlocking Outer Capsule Cap and Cap Weld

FIGURE 6. Photomicrograph of Full Penetration Closure Weld in AISI-1018 Steel Stand-In Outer Capsule (8X)

FIGURE 7. Components for Interlocking End Cap Configuration and Typical Completed Closure Weld - AISI-1018 Steel Capsule

FIGURE 8. Outer Capsule Components and Completed AISI-1018 Steel Outer Capsule Ready for Hydrostatic Pressure Testing

0.4 - IN. WALL
11,200 psi

0.5 - IN. WALL
15,900 psi

0.6 - IN. WALL
20,000 psi

FIGURE 9. Stand-In Steel Capsules After Hydrostatic Pressure Testing

TABLE 2. Predicted and Experimentally Determined Outer Capsule Collapse Pressures

<u>Capsule Wall Thickness, in.</u>	<u>Collapse Pressure, psi</u>	
	<u>Predicted</u>	<u>Experimental</u>
0.4	12,000	11,200
0.5	14,500	15,900
0.6	16,900	>20,000

A fourth capsule, having a wall thickness of 0.5 in., was subjected to the 1000-bar (14,500-psi) hydrostatic pressure required to meet the capsule qualification test requirement. The capsule exhibited slight ovality at the 1000-bar pressure, but did not collapse, as may be seen in Figure 10.

The four capsules that had been pressure tested were subjected to a helium leak check, and no leaks were detected. The leak check system used was capable of detecting leaks of less than 1×10^{-8} cc/sec. Additional evaluation and destructive examination of the capsules is now underway. The experimental results from the pressure tests will be used to fine-tune the analytical tube failure prediction model in order to assure that the final prototype capsule design will meet the hydrostatic pressure test requirement.

Impact and puncture testing of the stand-in capsules to evaluate the end-cap design is underway. The initial tests utilize the steel capsules having a 0.5-in. wall thickness.

FIGURE 10. Outer Capsule with 0.5-in. Wall Thickness After Hydrostatic Pressure Testing at 1000 Bars (14,500 psi)

Oxidation of Hastelloy S and Hastelloy C-4 (H. T. Fullam)

The tests to evaluate the oxidation resistance of Hastelloy S and Hastelloy C-4 are continuing. The tests are being carried out at 600⁰, 700⁰, and 800⁰C in air atmosphere muffle furnaces and are scheduled to last for up to 10,000 hr. Tensile specimens are being tested, in addition to the standard corrosion strips, to determine the effects of air oxidation on the tensile properties of the two alloys. The tests also include control tensile specimens heated in vacuum to provide reference tensile property data. The tensile specimens are 0.113 in. in dia and correspond to ASTM small-size specimen specifications (ASTM-E8-59). They were machined from 1/2-in.-dia bar stock. The Hastelloy S corrosion strips were cut from 1/16-in. plate, and the Hastelloy C-4 strips from 1/8-in. plate.

Both Hastelloy S and Hastelloy C-4 form adherent oxide layers when heated in air at 600⁰- 800⁰C. Results previously reported for tests lasting up to 2500 hr show that oxidation of each alloy adheres to a parabolic rate relationship.⁽²⁾ Results obtained in the 5000-hr test show that oxidation of each alloy at 600⁰C continues to follow the parabolic rate relationship. At 700⁰ and 800⁰C, however, oxidation of the 5000-hr specimens was less than predicted from the parabolic rate equations fitted to the shorter-term data, as shown in Figures 11 and 12. If the 5000-hr results are confirmed by the 7500- and 10,000-hr tests, then oxidation of the two alloys at 700⁰-800⁰C may be a less serious problem than originally anticipated from the initial test results.

The 5000-hr test specimens were subjected to metallographic examination, and the photomicrographs obtained are presented in Figures 13 and 14. At 600⁰C Hastelloy S forms a nonporous adherent oxide layer with little indication of intergranular attack. At 700⁰-800⁰C the oxide layer formed is more porous, and intergranular attack, pitting, and void formation are quite extensive. In addition, the 800⁰C specimens exhibit a relatively wide surface zone in which the normal discrete intergranular precipitates are largely depleted. Microprobe analysis indicates that chromium is a principal component of these precipitates; it also indicates that the chromium apparently diffuses to the surface and is converted to an oxide, since chromium is the major component of the oxide layer.

Hastelloy C-4 forms a relatively uniform adherent oxide scale at 600⁰-700⁰C, with little evidence of grain boundary attack. At 800⁰C the oxide scale layer is relatively porous, and intergranular attack and subsurface void formation are extensive.

At 800⁰C both Hastelloy S and Hastelloy C-4 suffer oxidation damage to a considerably greater depth than is indicated by the weight gain measurements and thickness of the oxide scale. The rate of attack, however, is approximately proportional to the rate of oxide scale formation. This can be seen by referring to Figures 15 and 16, which show Hastelloy S and Hastelloy C-4 specimens after exposure to air at 800⁰C for various times. It is apparent from the micrographs that the depth of metal affected by intergranular attack, void formation, etc., is approximately proportional to the thickness of the oxide scale.

The tensile specimens from the 1000- and 2500-hr oxidation tests were tested at room temperature. The results obtained with the Hastelloy S specimens are summarized in Table 3, while the Hastelloy C-4 results are presented in Table 4. Although some variations are apparent between the test and control specimens, an overall evaluation of the test results

FIGURE 11. Oxidation of Hastelloy S Specimens Exposed to Air in a Muffle Furnace at 600°-800°C.

FIGURE 12. Oxidation of Hastelloy C-4 Specimens Exposed to Air in a Muffle Furnace at 600°-800°C

FIGURE 13. Hastelloy S Specimens Heated in Air for 5000 hr

600°C

700°C

800°C

AS-POLISHED

ETCHED

FIGURE 14. Hastelloy C-4 Specimens Heated in Air for 5000 hr

FIGURE 15. Oxidation of Hastelloy S Specimens in Air at 800⁰

528 hr

2500 hr

5000 hr

AS-POLISHED

ETCHED

FIGURE 16. Oxidation of Hastelloy C-4 Specimens in Air at 800°

TABLE 3. Room-Temperature Tensile Properties of Hastelloy S Specimens^(a) Aged in Air or Vacuum^(b) at 600°, 700° and 800°C

Aging Temp, °C (Sol'n Annealed Alloy) ^(c)	Aging Time, hr	Ultimate Tensile Strength, ksi		Yield Strength, ksi		Elongation, %			
						Uniform		Total	
		Vacuum	Air	Vacuum	Air	Vacuum	Air	Vacuum	Air
	0	(128.3)		(69.9)		(52.3)		(60.4)	
500	1,000	136.0	137.9	73.1	78.4	50.4	50.8	56.0	56.1
600	2,500	147.8	151.2	89.5	86.7	44.3	44.4	48.8	48.3
600	5,000								
600	7,500								
600	10,000								
700	1,000	131.5	127.5	67.2	64.1	53.8	55.4	60.0	61.8
700	2,500	127.8	128.9	61.5	66.3	55.5	47.9	62.6	54.1
700	5,000								
700	7,500								
700	10,000								
800	1,000	132.3	127.7	63.3	61.4	53.2	54.9	59.5	63.6
800	2,500	127.2	126.2	59.1	59.1	54.9	54.4	62.7	62.1
800	5,000								
800	7,500								
800	10,000								

(a) Four tensile specimens heated in air and two tensile specimens heated in vacuum for each set of test conditions.

(b) Tensile specimens heated in evacuated quartz envelopes in the same furnaces as the specimens heated in air.

(c) Solution annealed alloy as received from the vendor.

TABLE 4. Room-Temperature Tensile Properties of Hastelloy C-4 Specimens^(a) Aged in Air or Vacuum^(b) at 600°, 700° and 800°C

Aging Temp, °C	Aging Time, hr	Ultimate Tensile Strength, ksi		Yield Strength, ksi		Elongation, %			
						Uniform		Total	
		Vacuum	Air	Vacuum	Air	Vacuum	Air	Vacuum	Air
(Sol'n Annealed Alloy) ^(c)		(121.0)		(55.0)		(60.9)		(71.5)	
500	2,500	182.3	179.3	103.6	99.1	38.7	42.0	43.6	48.6
600	5,000								
600	7,500								
600	10,000								
700	2,500	125.9	119.0	60.8	54.0	55.3	63.9	61.5	64.1
700	5,000								
700	7,500								
700	10,000								
800	2,500	115.2	116.1	48.5	49.7	63.8	63.2	68.1	72.3
800	5,000								
800	7,500								
800	10,000								

(a) Duplicate specimens tested at each set of conditions.

(b) Specimens heated in evacuated quartz envelopes in the same furnaces as the specimens heated in air.

(c) Solution annealed alloy as received from the vendor.

indicates that oxidation in air at 600⁰-800⁰C has no significant effect upon the room-temperature tensile properties of the two alloys. Room-temperature testing of the tensile specimens from the 5000-hr oxidation tests is now underway, and the results will be presented in the next report.

Seawater Corrosion of Hastelloy S and Hastelloy C-4 (H. T. Fullam)

The tests to evaluate the seawater corrosion resistance of Hastelloy S and Hastelloy C-4 are continuing. The tests are being carried out in flowing natural seawater at ambient temperature, and will last up to 10,000 hr. Tensile and prestressed specimens are being tested, as are standard corrosion coupons. Dimensions of the tensile specimens are the same as the dimensions of the tensile specimens used in the oxidation tests.

The 2500-hr seawater corrosion tests have been completed. Each of the test coupons exhibited some surface discoloration and a very slight weight loss. The results obtained to date with Hastelloy S show erratic behavior, but overall the corrosion by seawater is very low, as shown in Figure 17. Each of the Hastelloy S specimens tested showed a very slight weight loss, except those tested for 1000 hr. Each of the four 1000-hr specimens exhibited a very slight weight gain. No reason for the erratic behavior has been identified.

The Hastelloy C-4 coupons suffered a corrosion rate in seawater that is almost an order of magnitude greater than that of Hastelloy S, as exhibited in Figure 18. Even so, the corrosion rate is still very low.

The Hastelloy S and Hastelloy C-4 coupons exposed to seawater were subjected to metallographic examinations, and the photomicrographs obtained are presented in Figure 19. The micrographs provide no visual evidence of corrosion by the seawater. Microscopic examination of stressed specimens exposed to seawater for 2500 hr provided no evidence of stress corrosion cracking of either alloy.

The Hastelloy S and Hastelloy C-4 tensile specimens exposed to seawater for 1000 and 2500 hr were tested at room temperature. The results obtained are now being evaluated and will be presented in the next report.

FIGURE 17. Corrosion of Hastelloy S in Flowing Natural Seawater at Ambient Temperature

FIGURE 18. Corrosion of Hastelloy C-4 in Flowing Natural Seawater at Ambient Temperature

HASTELLOY S

HASTELLOY C-4

FIGURE 19. Metal Specimens Exposed to Flowing Natural Seawater at Ambient Temperature for 2500 hr

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