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Compatibility Testing of the WESF $^{90}\text{SrF}_2$ Storage Capsule at 800°C

H. T. Fullam

June 1980

**Prepared for
the Advanced Nuclear Systems and Projects
Division
U.S. Department of Energy
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**Pacific Northwest Laboratory
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COMPATIBILITY TESTING OF THE WESF
 $^{90}\text{SrF}_2$ STORAGE CAPSULE AT 800°C

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SUMMARY

A program is currently under way at Pacific Northwest Laboratory to develop the information needed to permit the licensing of $^{90}\text{SrF}_2$ as a radioisotope heat-source fuel for terrestrial application. A major portion of the effort is directed at determining the compatibility of $^{90}\text{SrF}_2$ with containment materials at anticipated heat-source operating temperatures. One phase of the compatibility studies involved the testing of two WESF $^{90}\text{SrF}_2$ storage capsules at approximately 800°C . The first capsule was tested for 5000 h and the second for 26,200 h.

The capsules were placed in individual insulated containers and allowed to self-heat to the test temperature. The tests were conducted in a hot cell at WESF. Heat losses from the insulated containers were uneven, and as a result a temperature gradient existed between the center and ends of each capsule. In the 5000-h test the metal/ $^{90}\text{SrF}_2$ interface temperature varied from about 730° to 821°C ; in the 26,200-h test the temperature varied from about 750° to 831°C .

Metallographic examination of sections of metal taken from the Hastelloy C-276 capsules showed that chemical attack of the metal was not significantly greater in the 26,200-h test than in the 5000-h test. Similar results have been observed in other compatibility tests involving Hastelloy C-276 and $^{90}\text{SrF}_2$. The data indicate that chemical attack of the Hastelloy C-276 is due to impurities in the strontium fluoride, and that once the impurities are consumed the rate of attack drops to a very low level.

Micrographs of the test sections showed that the extent and type of chemical attack varied along the length of the capsule. The type of chemical attack also varied between the two test capsules, although the extent of the attack was about the same. The variation in the extent and type of chemical attack along the capsule length appears to be due to differences in alloy microstructure rather than to temperature differences.

Overall, chemical attack of the Hastelloy C-276 capsules was much less than would have been predicted from the results obtained in the other compatibility tests with $^{90}\text{SrF}_2$.

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COMPATIBILITY TESTING OF THE WESF $^{90}\text{SrF}_2$
STORAGE CAPSULE AT 800°C

INTRODUCTION

As part of the Department of Energy's waste management program at Hanford, ^{90}Sr is recovered from the high-level fuel-reprocessing wastes and is stored as strontium fluoride in small, double-walled metal containers in a water basin on the Hanford Reservation. The $^{90}\text{SrF}_2$ preparation, encapsulation and storage take place in the Waste Encapsulation and Storage Facility (WESF), which is operated for the Department of Energy by Rockwell International.

Each storage capsule contains approximately 3 kg of strontium fluoride and, depending upon the ^{90}Sr content, can have a heat output of up to 1 kW. This stored $^{90}\text{SrF}_2$ represents an economical, readily available source of radioisotope heat for terrestrial heat-source applications if the $^{90}\text{SrF}_2$ fuel form can be licensed for use by the cognizant regulatory agencies.

A program has been under way at the Pacific Northwest Laboratory (PNL) for several years to develop the information and technology needed to permit the licensing of $^{90}\text{SrF}_2$ as a radioisotope heat-source fuel for terrestrial applications. The program is currently funded by the Advanced Nuclear Systems and Projects Division of the Department of Energy. A major portion of the program is directed toward evaluating the compatibility of the $^{90}\text{SrF}_2$ fuel form with containment materials at anticipated heat-source operating temperatures. Extensive compatibility testing of the WESF-produced $^{90}\text{SrF}_2$ with selected containment materials done at between 600° and 1000°C is in progress.^(a) One phase of the compatibility studies involved the demonstration testing of two full-size WESF $^{90}\text{SrF}_2$ capsules at approximately 800°C . The tests lasted 5000 and 26,200 h. The capsule was sectioned at the conclusion of each test and samples of the Hastelloy® C-276 inner capsule were subjected to metallographic examination to determine the extent of attack by the fluoride. This report summarizes the results of the study.

(a) Results of the PNL $^{90}\text{SrF}_2$ compatibility studies are produced in the series of monthly and quarterly progress reports having document numbers BNWL-1845-1 through BNWL-1845-36 and PNL-1845-37 through PNL-1845-46.

® Stellite Division of Cabot Corp., Kokomo, Indiana.

WESF $^{90}\text{SrF}_2$ STORAGE CAPSULE

The WESF $^{90}\text{SrF}_2$ storage capsule is a double-walled capsule consisting of a Hastelloy C-276 inner container and a 316L stainless steel outer container. Each container is sealed by tungsten inert-gas (TIG) welding. A sketch of the capsule is shown in Figure 1. The exposed metal surface-to- SrF_2 volume ratio (S/V) for the capsule is approximately 0.86 cm^{-1} .

The strontium fluoride is loaded into the Hastelloy C-276 inner capsule by cold step-pressing. The bulk density of the fluoride in the capsule is about 65% to 70% of the theoretical density, and each capsule contains approximately 3 kg of SrF_2 . The heat output of the capsule will depend on its ^{90}Sr content, which decreases with time due to radioactive decay. The ^{90}Sr isotopic content of the strontium recovered from the fuel-reprocessing wastes can vary over a wide range, depending upon the ages and sources of the wastes. Only those capsules having a high ^{90}Sr content are considered for possible heat-source service; the maximum heat output of the WESF capsule at the time of encapsulation is about 960 W. This corresponds to a maximum capsule power density of approximately 1 W/cm^3 .

FIGURE 1. Sketch of the WESF $^{90}\text{SrF}_2$ Storage Capsule

EXPERIMENTAL

The capsule demonstration tests were carried out inside a shielded hot cell at WESF. Each of the two $^{90}\text{SrF}_2$ storage capsules were placed in individual insulated containers and allowed to self-heat to a $^{90}\text{SrF}_2$ /Hastelloy C-276 interface temperature of approximately 800°C . One capsule was held at temperature for 5000 h, while the second was held at temperature for 26,200 h (~ 3 years). This second test was originally scheduled to last for only 1 year; however, extended operating problems at WESF prevented sectioning of the capsule at the end of the 1-year test. Therefore, the capsule was held at temperature until it could be sectioned, which resulted in the 26,200-h test period.

Figure 2 shows a sketch of the insulated container used to hold one of the test capsules. The container consisted of two concentric 304L stainless steel tubes with stainless steel end plates. Stainless steel spacers were used to center the inner tube in the outer tube and also to center the $^{90}\text{SrF}_2$ capsule within the inner tube. A mineral-blanket insulation was packed in the ends of the tube to reduce heat loss through the ends of the container. Asbestos blanket insulation was wrapped around the outer tube to reduce the radial heat loss. The two containers were suspended in a horizontal position from supports fastened to the hot-cell wall.

Three calibrated, sheathed chromel-alumel thermocouples that entered the container through one of the end plates were used to monitor the surface temperature of the heat source outer

FIGURE 2. Container Holding the WESF $^{90}\text{SrF}_2$ Capsule During the Demonstration Test

capsule at various locations. The thermocouples were made movable so that the surface temperature could be measured along the length of the capsule. A calibrated digital multimeter was used to monitor the output of the thermocouples. The interface temperature between the $^{90}\text{SrF}_2$ and the Hastelloy C-276 inner capsule was calculated from the measured outer capsule surface temperature.

It was the original intent that the demonstration tests be carried out at a Hastelloy C-276/ $^{90}\text{SrF}_2$ interface temperature of 800°C . However, tests with an electrically heated dummy capsule showed a significant temperature gradient existed between the center and ends of the capsule because of uneven heat losses from the insulated container. At a power input of 1000 W to the dummy capsule the maximum interface temperature at the center of the capsule was estimated to be 816°C , with a gradient of about 30°C between the center and end of the capsule. Therefore, the design of the insulated container was assumed to be adequate for the tests.

At the conclusion of a test the container was opened and the $^{90}\text{SrF}_2$ storage capsule removed. Once the storage capsule had cooled, the 316L stainless steel outer capsule was cut open and the Hastelloy C-276 inner capsule removed. The inner capsule was then sectioned at several locations to obtain samples of the Hastelloy C-276 for analysis. Figure 3 shows the locations where the sections were removed from the capsule. The Hastelloy C-276 sections were shipped to Oak Ridge National Laboratory for metallographic examination.

The two $^{90}\text{SrF}_2$ capsules used in the tests were standard WESF production capsules. The ^{90}Sr isotopic content of the strontium fluoride in the two capsules at the beginning of the tests was approximately 52% and the initial heat output of each was about 950 W. When the 5000-h test was completed, the calculated heat output of the test capsule was about 940 W. At the conclusion of the 3-year test the heat output of the capsule was about 885 W.

A chemical analysis was not obtained on the strontium fluoride in each test capsule. An analysis was obtained on the batch of strontium fluoride that was used in filling the capsules. The results of the batch analysis are given in Table 1, and they show that the fluoride

FIGURE 3. Sketch Showing Locations Where Sections Were Cut from the Hastelloy C-276 Inner Capsule After Testing

TABLE 1. Analysis of the $^{90}\text{SrF}_2$ in the Test Capsules(a,b)

Element	wt%	Element	wt%	Element	wt%
Sr	66.6	Cr	0.25	Ni	0.22
Al	0.2	Fe	0.40	Pb	0.025
Ba	0.35	Mn	0.01	RE ^(c)	1.2
Ca	0.43	Na	1.9	Zr	1.2
Cd	<0.02				

(a) Analysis of the batch of SrF_2 used to fill the test capsules.(b) ^{90}Sr Isotopic Content - 52%.

(c) RE - Rare Earths.

contains about 6 wt% impurities--primarily metal fluorides. There is some slight inhomogeneity in each batch of SrF_2 , principally in the iron, chromium, and nickel contents. The inhomogeneity is due to corrosion products from the metal equipment used in preparing the fluoride--mainly the Inconel X boats in which the fluoride is fired prior to encapsulation. Therefore, the values given in Table 1 are only estimates of the iron, chromium and nickel contents of the fluoride in the individual test capsules.

The Hastelloy C-276 used for the inner capsule of the WESF $^{90}\text{SrF}_2$ storage capsule is a high-nickel-base alloy produced by the Stellite Division of the Cabot Corporation. The alloy is a modification of Hastelloy C, in which the minor-element chemistry is controlled to reduce precipitate formation in the weld-heat affected zone. The chemical composition of Hastelloy C-276 is given in Table 2.

TABLE 2. Typical Composition and Properties of Hastelloy C-276

Element	wt%	Element	wt%	Element	wt%
C	0.02	Mn	1.0 ^(a)	S	0.03 ^(a)
Co	2.5 ^(a)	Mo	15.0 to 17.0	Si	0.05 ^(a)
Cr	14.5 to 16.5	Ni	Balance	V	0.35 ^(a)
Fe	4.0 to 7.0	P	0.03 ^(a)	W	3.0 to 4.5

Typical room-temperature tensile data for a 3/4-in. plate heat-treated at 2100°F and water-quenched:

ultimate tensile strength = 783 MPa
yield strength at 0.2% offset = 359 MPa
elongation in 2 in. = 70%.

(a) Maximum

RESULTS AND DISCUSSION

In the demonstration tests with the WESF $^{90}\text{SrF}_2$ capsule it was not possible to directly measure the temperature at the $^{90}\text{SrF}_2$ /Hastelloy C-276 interface. Instead, the surface temperature of the 316L stainless steel outer capsule was measured using the three movable thermocouples. The Hastelloy C-276/ $^{90}\text{SrF}_2$ interface temperature profile was then calculated from the outer capsule's surface-temperature data using standard heat-transfer techniques. The calculated interface temperature profiles for the two test capsules are given in Figures 4 and 5. The interface temperatures at the locations where the sections were removed from the capsules are also shown in the figures.

FIGURE 4. Temperature Profile for the WESF $^{90}\text{SrF}_2$ Capsule Tested for 5000 h and the Locations at Which the Capsule Was Sectioned

FIGURE 5. Temperature Profile for the WESF $^{90}\text{SrF}_2$ Capsule Tested for 26,200 h and the Locations at Which the Capsule Was Sectioned

It was found that the temperature at any given capsule location fluctuated by as much as $\pm 6^{\circ}\text{C}$ during the course of the test. These fluctuations did not follow any set pattern, and were random in nature. In the 5000-h test there was no discernible decrease in the interface temperature profile with time, even though the heat output of the capsule had decreased by about 1% by the end of the test. In the 3-year test it appeared that the interface temperature profile had decreased by an average of about 12°C during the test, while the heat output of the capsule decreased about 7%. The interface temperature profiles given in Figures 4 and 5 represent best estimates of the average test temperatures.

Evaluation of the sections taken from the Hastelloy C-276 inner capsules was limited to metallographic examination. The extent of the Hastelloy C-276 attack by the fluoride was estimated from the photomicrographs obtained. Thickness measurements on the test sections eliminated the possibility of gross attack, which might not be apparent from the photomicrographs.

Earlier compatibility studies at PNL involving Hastelloy C-276 and strontium fluoride have shown that the alloy suffers two distinctly different types of attack, designated as a) chemical attack and b) changes in microstructure. Chemical attack of the Hastelloy C-276 is defined as attack that can be directly attributed to reaction of the alloy with the strontium fluoride and/or contained impurities. Chemical attack can include such mechanisms as surface dissolution, selective leaching of alloy components, intergranular attack, pitting, and subsurface void formation.

Changes in microstructure are defined as those instances where the Hastelloy C-276 exhibits areas in which the microstructure differs significantly from that of the bulk alloy. The microstructural changes usually consist of one or more of the following: 1) the disappearances of normal alloy precipitates, 2) the formation of abnormal precipitates, and/or 3) marked changes in grain size. Photomicrographs of alloy test specimens provide no direct evidence that any of the microstructure changes that may occur are due to fluoride attack. However, since the changes are not observed in Hastelloy C-276 specimens held at the same temperature without fluoride present, these changes must result, at least indirectly, from chemical attack by the fluoride.

It can be assumed that any chemical attack of the Hastelloy C-276 by the strontium fluoride will be deleterious to the structural properties of the inner capsule. This may not necessarily be the case, however, with changes in the microstructure of the alloy. For example, the disappearance of normal alloy precipitates could result in an increase in the impact strength and ductibility of the affected area.

The only way the effects of microstructural changes on capsule properties can be accurately assessed is by a detailed study to measure the physical properties of Hastelloy C-276 specimens exposed to the fluoride over the temperature range of interest. This has not been done, and one must therefore assume that the microstructural changes will be harmful to the capsule properties.

The extent of Hastelloy C-276 attack by the strontium fluoride was estimated from photomicrographs of the test sections and the results obtained are presented in Table 3.

TABLE 3. Attack of the Hastelloy C-276 by $^{90}\text{SrF}_2$ in the WESF Capsule Demonstration Tests

Test Duration, h	Section No.	Interface Temperature, °C	Depth of Hastelloy C-276 Affected, ^(a) μm	
			Chemical Attack	Change in Microstructure
5,000	1	762	80	0
	2	821	230	0
	3	813	210	0
	4	790	280	0
26,200	1	778	130	0
	2	831	180	310
	3	824	310	560
	4	800	230	510

(a) Estimated from photomicrographs.

Micrographs of the sections from the capsule tested for 5000 h are shown in Figure 6 and micrographs of the sections from the capsule tested for 3 years are presented in Figure 7.

Although no physical property measurements were obtained on the ring sections removed from the test capsules, one piece of qualitative information was obtained. The ring sections removed from the capsules were partially flattened to facilitate cask shipment. No problems were encountered with the sections from the 5000-h test. When the ring sections from the 3-year test were flattened, one of the sections shattered, apparently because of the reduced ductility of the Hastelloy C-276. It was not determined whether the reduced ductility resulted from thermal aging reactions that occurred in the alloy or was due to attack by the fluoride.

In evaluating the data presented in Table 3, one important fact becomes apparent. Chemical attack of the Hastelloy C-276 was not appreciably greater in the 3-year test than in the 5000-h test. Similar results were observed in extensive strontium fluoride compatibility studies carried out at PNL (Fullam 1975, Fullam 1977a, Fullam 1979). These tests, involving both nonradioactive SrF_2 and WESF-produced $^{90}\text{SrF}_2$, were carried out at 600°, 800°, 1000° and 1100°C and lasted for up to 30,000 h. The available compatibility data shows that chemical attack of the Hastelloy C-276 and most other metals tested is due primarily to impurities in the strontium fluoride, and that the impurities are consumed in a relatively short period of time. Once the impurities in the fluoride have reacted, the rate of chemical attack drops to a very low level. Although the reaction mechanisms have not been completely determined, the data indicate that iron and chromium fluorides in the strontium fluoride are the principal impurities that produce the attack of the Hastelloy C-276 (Fullam 1977a, Fullam 1977b, Fullam 1977c). This is a critical factor, because iron and chromium are two of the major contaminants that are introduced into the WESF $^{90}\text{SrF}_2$ by corrosion of the processing equipment.

FIGURE 6. Photomicrographs of the Sections from the Hastelloy C-276 Inner Capsule Tested for 5000 h

SECTION #1, 778°C

SECTION #2, 831°C

SECTION #3, 824°C

SECTION #4, 800°C

AS-POLISHED

ETCHED HCl-HNO₃

FIGURE 7. Photomicrographs of the Sections from the Hastelloy C-276 Inner Capsule Tested for 26,200 h

The data presented in Table 3 also show that in each test the extent of chemical attack varied along the length of the capsule. The variation in attack cannot be directly attributed to temperature differences, because the most extensive chemical attack occurred at intermediate interface temperatures. Examination of the micrographs shown in Figures 6 and 7 indicates that the type of chemical attack also varied somewhat along the length of the capsule. In addition, the chemical attack that occurred in the 5000-h test appeared to be quite different from that which occurred in the 3-year test, even though the capsules' interface-temperature profiles were very similar. The chemical attack of the capsule tested for 3 years was quite similar to that observed in other compatibility tests carried out at 800°C involving Hastelloy C-276 and $^{90}\text{SrF}_2$ (Fullam 1978). The chemical attack of the capsule tested for 5000 h was more similar to that observed in other tests carried out at 600°C (Fullam 1978).

Detailed analyses were not performed on the test sections. However, electron microprobe analysis of Hastelloy C-276 samples exposed to nonradioactive SrF_2 that contained impurities similar to those found in the WESF $^{90}\text{SrF}_2$ show that the extent and type of chemical attack is influenced by the microstructure of the alloy. The data indicates that chemical attack involves two principal mechanisms: 1) selective leaching of chromium from the alloy matrix, and 2) reaction of alloy precipitates with iron- and chromium-fluoride impurities in the SrF_2 . Reaction with the alloy precipitates becomes more pronounced as precipitate formation increases. Leonard (1969) reports that when Hastelloy C-276 is aged for long periods of time at 750° to 850°C an Ni_7Mo_6 -type phase is the principal precipitate formed. The PNL data indicates that it is probably this precipitate which reacts with the impurities in the SrF_2 .

The micrographs of the sections from the two test capsules show that there are marked differences in the Hastelloy C-276 microstructure along the length of a capsule, even though the temperature gradient between the test sections in each case is at most 50° to 60°C. The micrographs also showed that precipitate formation was much heavier in the Hastelloy C-276 sections from the 3-year test than in the sections from the 5000-h test. These differences in the Hastelloy C-276 microstructure probably account for the differences in the extent and type of chemical attack observed in each capsule and between capsules.

One unexpected result was the absence of changes in microstructure due to fluoride attack in the capsule tested for 5000 h and in one section from the capsule tested for 3 years. In all of the other PNL compatibility tests involving Hastelloy C-276 and strontium fluoride the alloy suffered extensive microstructural changes. It was not determined why the changes in microstructure did not occur as expected. In the 3-year test, three of the Hastelloy C-276 sections exhibited extensive changes in microstructure. These changes consisted of a narrow surface layer largely depleted of normal alloy precipitation, and a much broader inner zone containing very heavy precipitation not found in the body of the alloy. The microstructural changes observed in the 3-year test were very similar to those observed in other tests carried out at 800°C involving Hastelloy C-276.

Since attack of the Hastelloy C-276 is due primarily to impurities in the strontium fluoride, the extent of metal attack should depend on the exposed metal surface-to-SrF₂ volume ratio (S/V) of the capsule. The smaller the S/V value for the capsule, the greater the metal attack should be. Earlier tests at PNL involving both nonradioactive SrF₂ and WESF-produced ⁹⁰SrF₂ shows this to be the case, as can be seen by referring to the data presented in Table 4.

Based on the results given in Table 4, one would expect much greater metal attack in the demonstration tests than was actually observed, since the S/V ratio of the WESF ⁹⁰SrF₂ storage capsules is about 0.86 cm⁻¹. Reasons for the lower than expected metal attack in the tests with the full-sized WESF ⁹⁰SrF₂ capsules are not obvious from the data available. The most likely explanation is that the actual iron and chromium concentrations in the strontium fluoride in the two capsules were lower than the batch analysis indicated. The results obtained in the demonstration tests are encouraging, inasmuch as they indicate that attack of Hastelloy C-276 at 800°C in the WESF ⁹⁰SrF₂ storage capsule may be much lower than the results of the other PNL compatibility studies have indicated.

TABLE 4. Effect of the S/V Ratio on the Attack of Hastelloy C-276 Exposed to Strontium Fluoride at 800°C (Fullam 1977d, Fullam 1978)

Exposure Time, h	Capsule S/V, (a) cm ⁻¹	Depth of Metal Affected, μm			
		Chemical Attack		Change in Microstructure	
		Nonradioactive SrF ₂ (b)	⁹⁰ SrF ₂ (c)	Nonradioactive SrF ₂	⁹⁰ SrF ₂
1000	4.5	25	180	75	310
	2.5	25	760	50	>900
	0.9	250		380	
6000	4.5	50	290	75	510
	2.5	50	610	50	>760
	0.9	250		380	

(a) Metal surface-to-SrF₂ volume of the test capsule.

(b) Nonradioactive SrF₂ containing impurities similar to those found in WESF-produced ⁹⁰SrF₂.

(c) WESF-produced ⁹⁰SrF₂.

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