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Quarterly Report on the Strontium Heat Source Development Program and the Beneficial Isotopes Utilization Program, Division of Nuclear Research and Applications for January-March 1977

H. T. Fullam

May 1977

Prepared for the Energy Research and Development Administration under Contract EY-76-C-06-1830

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QUARTERLY REPORT ON THE STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM AND THE BENEFICIAL ISOTOPES UTILIZATION PROGRAM, DIVISION OF NUCLEAR RESEARCH AND APPLICATIONS FOR JANUARY-MARCH 1977

H. T. Fullam

April 1977

BATTELLE
Pacific Northwest Laboratories
Richland, Washington 99352

SUMMARY

Metallographic examination of the Haynes Alloy 25 specimens from the 1000 hr tests with WESF $^{90}SrF_2$ has been completed. The specimens exhibited considerably more attack than was observed in the initial screening tests with Haynes Alloy 25 and $^{90}SrF_2$. Attack of the alloy was much greater at 800 and 1000°C than at 600°C, and the attack mechanisms appeared to vary with temperature. Decreasing the S/V ratio of the test couples produced a marked increase in attack at the higher temperatures. The addition of Zr and ZrF_4 to the WESF $^{90}SrF_2$, to simulate decay product buildup, resulted in a marked decrease in attack.

The test couples from the 6000 hr tests with WESF $^{90}SrF_2$ have been sectioned and the 36 metallographic specimens shipped to ORNL for examination.

The first draft of a report detailing the design criteria and qualification requirements for the $^{90}SrF_2$ fueled heat source capsule has been prepared and is now being reviewed.

A draft report on the feasibility of heaters fueled with strontium fluoride capsules for use in cold regions is being reviewed. Similarly, comments are being obtained for the Study of Sludge Handling Alternatives Final Report and the General Application of Radioisotopes in Army Thermal Systems Final Report.

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STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM

H. H. Van Tuyl, Program Manager
H. T. Fullam, Principal Investigator
D. G. Atteridge

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 $^{90}SrF_2$ heat sources and specifically the WESF $^{90}SrF_2$ capsules. The information needed for licensing can be divided into three general task areas:

Task 1 - Chemical and Physical Properties of $^{90}SrF_2$
Task 2 - $^{90}SrF_2$ 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 $^{90}SrF_2$ (H. T. Fullam)

Work has started on the preparation of the $^{90}SrF_2$ data sheet. The data sheet will summarize all currently available data on $^{90}SrF_2$ with special emphasis on the $^{90}SrF_2$ produced at WESF.

TASK 2 - $^{90}SrF_2$ COMPATIBILITY STUDIES (H. T. Fullam)

Long-Term Compatibility Tests

ORNL has completed the metallographic examination of the Haynes Alloy 25 specimens from the 1000 hr tests with WESF $^{90}SrF_2$. Estimates of metal attack based on the micrographs obtained are presented in Table 1. Overall attack of the Haynes Alloy 25 by WESF $^{90}SrF_2$ was greater than would have been predicted based on the initial screening tests with $^{90}SrF_2$.(1) Attack of the Haynes Alloy 25 increased with increasing exposure temperature and the attack mechanisms appeared to change with temperature. At 600°C the test specimens

TABLE 1. Attack of Haynes Alloy 25 Specimens Exposed to WESF $^{90}SrF_2$ for 1000 hr

Temperature, °C	S/V Ratio,[b] cm^{-1}	Depth of Metal Affected,[a] mils: Chemical Attack	Depth of Metal Affected,[a] mils: Change in Microstructure
600	4.5	1	0
600	4.5	2	0
600	2.5	2	0
600[c]	4.5	<<1	0
800	4.5	5	10
800	4.5	4	6
800	2.5	18	10
800[c]	4.5	2	5
1000	4.5	6	8
1000	4.5	7	9
1000	2.5	10	12
1000[c]	4.5	5	3

(a) Attack estimated from specimen micrographs
(b) S/V - the exposed metal surface-to-$^{90}SrF_2$ volume ratio of the test couple.
(c) Zr and ZrF_4 added to the WESF $^{90}SrF_2$ to simulate decay product buildup equivalent to 10 yr ^{90}Sr decay.

suffered limited chemical attack which appeared to consist primarily of general surface dissolution, while there were no significant microstructural changes (see Figure 1-A). Varying the S/V ratio of the test couples had little effect on alloy attack at 600°C.

At 800°C the chemical attack of the specimens was much more extensive and appeared to consist of selective leaching of alloy components and grain boundary attack. The reaction zone contained free grains of unreacted metal (Figure 1-B). Microstructural changes consisted of a broad surface zone in which the normal intragranular precipitates were depleted. Decreasing the S/V ratio of the test couples produced a sharp increase in chemical attack of the Haynes Alloy 25 with extensive grain boundary attack and subsurface void formation (see Figure 2).

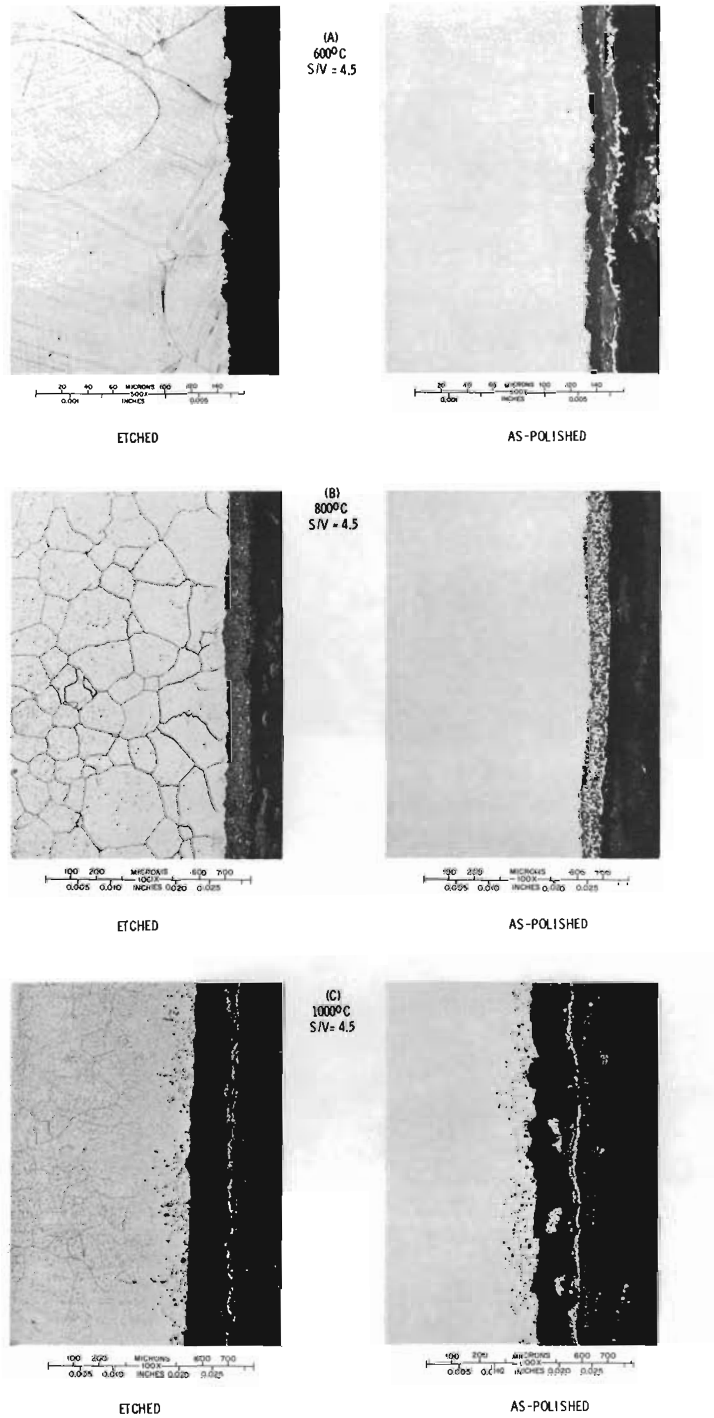
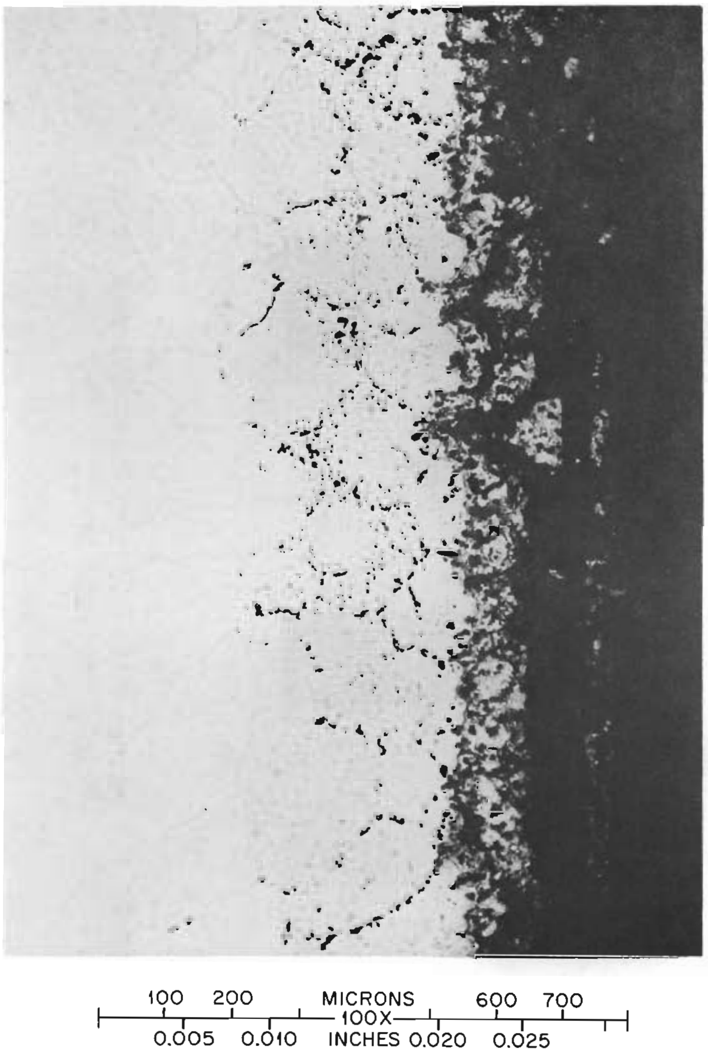


FIGURE 1. Haynes Alloy 25 Specimens Exposed to WESF $^{90}SrF_2$ for 1000 hr



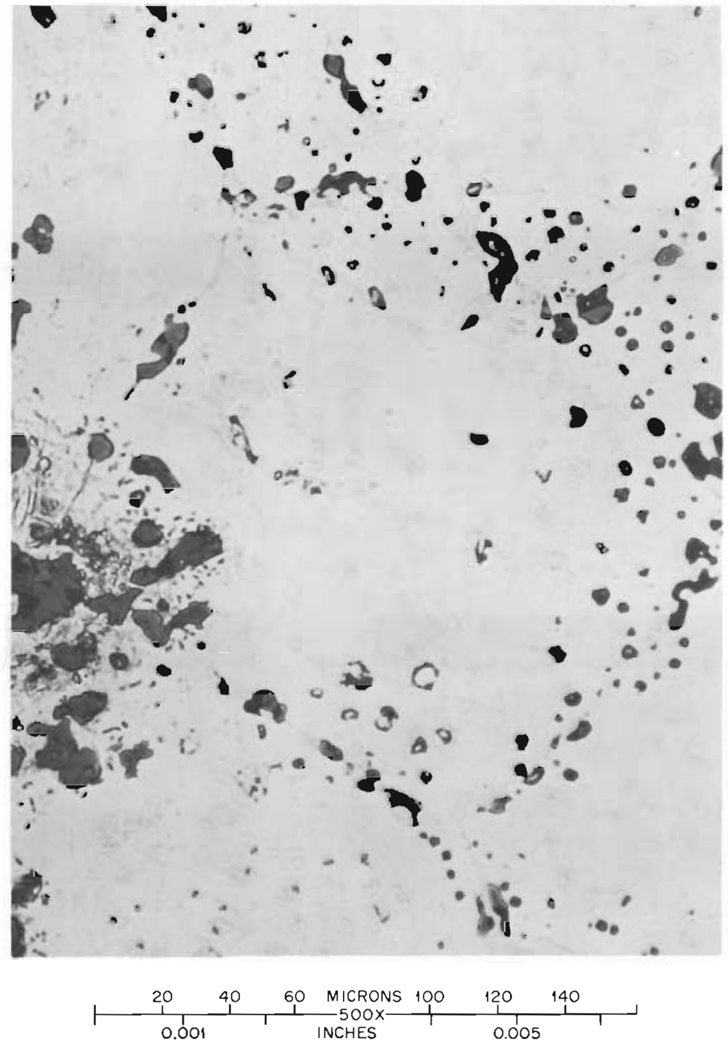


FIGURE 2. Haynes Alloy 25 Specimen Exposed to WESF $^{90}SrF_2$ at 800°C for 1000 hr - Couple S/V = 2.5 cm^{-1}

At 1000°C chemical attack of the Haynes Alloy 25 consisted primarily of extensive subsurface void formation, general surface dissolution with some grain boundary attack (Figure 1-C). Microstructural changes consisted of a surface zone in which normal alloy precipitates were largely depleted. Decreasing the S/V ratio of the test couples produced a small increase in attack of the alloy at 1000°C.

The addition of Zr metal powder and ZrF_4 to the WESF $^{90}SrF_2$, to simulate 10 yr ^{90}Sr decay, resulted in a sharp decrease in Haynes Alloy 25 attack at 600 and 800°C (see Figure 3). At 1000°C alloy attack was also decreased except for subsurface void formation which was only slightly reduced (Figure 3).

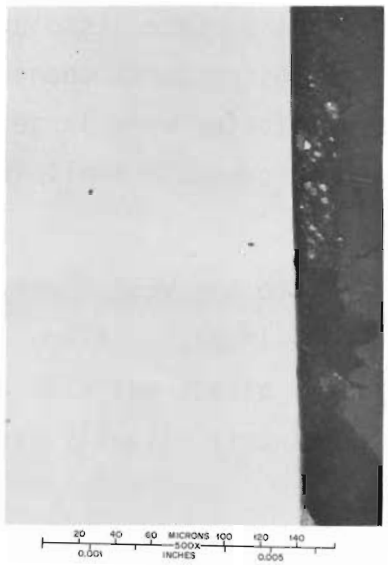
Sectioning of the compatibility couples from the 6000 hr tests with WESF $^{90}SrF_2$ was completed, and the test specimens separated from the $^{90}SrF_2$. The 36 metallographic specimens (12 TZM, 12 Haynes Alloy 25 and 12 Hastelloy C-276) from the sectioned couples were shipped to ORNL for metallographic examination, which is now underway. The tensile and Charpy-V notch specimens from the 6000 hr couples will be shipped to ORNL for examination as soon as the shipping cask is returned to PNL.

WESF $^{90}SrF_2$ Capsule Demonstration Test

ARHCO has completed the sectioning of the full-size WESF capsule, containing WESF produced fuel grade $^{90}SrF_2$, which was held at 800°C for 5000 hr. Several rings were cut from the inner Hastelloy C-276 capsule at various locations along the length of the capsule. Arrangements have been made to ship the rings to PNL where they will be further sectioned to obtain specimens for metallographic examination. The metallographic specimens will be shipped to ORNL for examination.

Supplemental Short-Term Screening Tests

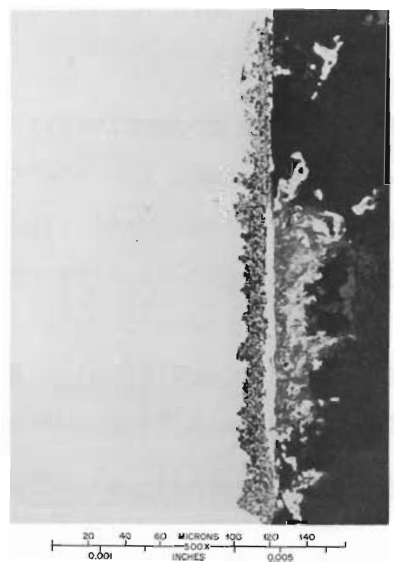
Metallographic examination of all of the specimens from the supplemental short-term screening tests with nonradioactive SrF_2 (see Table 2 for composition) has been completed. Estimates of metal attack based the photomicrographs obtained are presented in Table 3. Preparation of a topical report, which discusses each metal - SrF_2 system in detail, is now underway.



600°C
S/V = 4.5

AS-POLISHED

800°C
S/V = 4.5



AS-POLISHED

1000°C
S/V = 4.5

AS-POLISHED

FIGURE 3. Haynes Alloy 25 Specimens Exposed for 1000 hr to WESF $^{90}SrF_2$ Containing Zr and ZrF_4

TABLE 2. Composition[a] of the Nonradioactive SrF_2 Used in the Supplemental Screening Tests

Component	wt%	Component	wt%
Sr	65.9	Nd	1.0
Al	0.26	Ni	0.02
Ba	0.70	Pb	0.06
Ca	0.21	Si	0.02
Cr	0.050	F	29.5
Fe	0.022	H_2O	<0.01
K	0.010	NO_3^-	<0.01
Mg	0.13	O	<0.01
Na	2.5		

(a) The SrF_2 contains impurities approximating those found in WESF produced $^{90}SrF_2$

TABLE 3. Estimated Attack of Test Specimens Exposed to Nonradioactive SrF_2[a] at 800°C

	Depth of Metal Affected,[b] mils			
	1500 hours		4400 Hours	
Material	Chemical Attack	Change in Microstructure	Chemical Attack	Change in Microstructure
Hastelloy C-276[c]	3	7	3	5
Haynes Alloy 25[c]	2	3	1	2
TZM[c]	1	0	<1	<1
Hastelloy C-4	5	12	4	13
Hastelloy B	4	15	4	11
Hastelloy B-2	10	18	6	18
Hastelloy S	7	15	3	12
Haynes Alloy 556	5	6	5	7
Inconel 617	7	14	8	14
Inconel 671	25	25	20	20
Incoloy 800	8	0	7	0
Rene 41	10	14	11	13
Udimet 700	>25	0	>25	>25
Monel 400	5	8	6	6
Nickel 200	7	10	8	8
Ingot Iron	3	0	4	7
Ductile Cast Iron	CR	CR[d]	CR	CR
316L SS	6	0	22	0
JS 777	6	7	5	6
Copper	>25	0	>25	0
Titanium	>25	(e)	>25	(e)
Hafnalloy 2525	>25	(e)	CR	CR
Molybdenum	2	0	<1	<1
Niobium	3	2	2	2
Ta-10% W	10	0	2	0
Mo-50% Re	2	0	1	0
W-26% Re	2	0	1	0
Rhenium	<1	0	<1	0
Iridium	0	0	<<1	0
Ir-0.3% W	0	0	<<1	0
Platinum	>25	(e)	>25	(e)
Gold	>15	(e)	>15	(e)

(a) The SrF_2 contained impurities approximating those found in the WESF $^{90}SrF_2$

(b) Estimated from photomicrographs

(c) Tested as reference specimens

(d) CR - Complete Reaction

(e) Could not be estimated because of extensive chemical attack

Thermal Gradient Test

In the thermal gradient test a Hastelloy Alloy C-276 capsule containing nonradioactive strontium fluoride was subjected to a temperature gradient of 560°C (320°C to 920°C) for a period of 4400 hr. The capsule was fabricated from 1 1/2-in. Sch 40 pipe and was 26 in. long. The SrF_2 contained impurities similar to those found in WESF produced $^{90}SrF_2$ (see Table 2). At the conclusion of the test the capsule was sectioned; and specimens taken from various locations in the capsule were subjected to metallographic examination to determine the extent of metal-SrF_2 interaction. Estimates of metal attack as a function of temperature, based on the photomicrographs obtained, are presented in Table 4. The micrographs of the test specimens are shown in Figure 4. Electron microprobe analysis of the specimens is underway in an attempt to determine the reaction mechanisms involved. Preliminary results indicate that selective leaching of chromium from the alloy matrix and the reaction of grain boundary precipitates with FeF_3 in the SrF_2 are the principal reaction mechanisms, with grain boundary reactions being more pronounced at the higher temperatures.

TABLE 4. The Effect of Temperature on the Attack of Hastelloy Alloy C-276 by Nonradioactive Strontium Fluoride[a]

Approximate Interface Temperature, °C	Exposure Time, hr	Depth of Metal Affected, mils[b]	
		Chemical Attack	Change in Microstructure
918	4400	8	9
874	4400	7	15
830	4400	6	11
781	4400	4	6
680	4400	2	3
448	4400	<<1	<1

(a) The SrF_2 had the composition shown in Table 2.
(b) Attack estimated from photomicrographs.

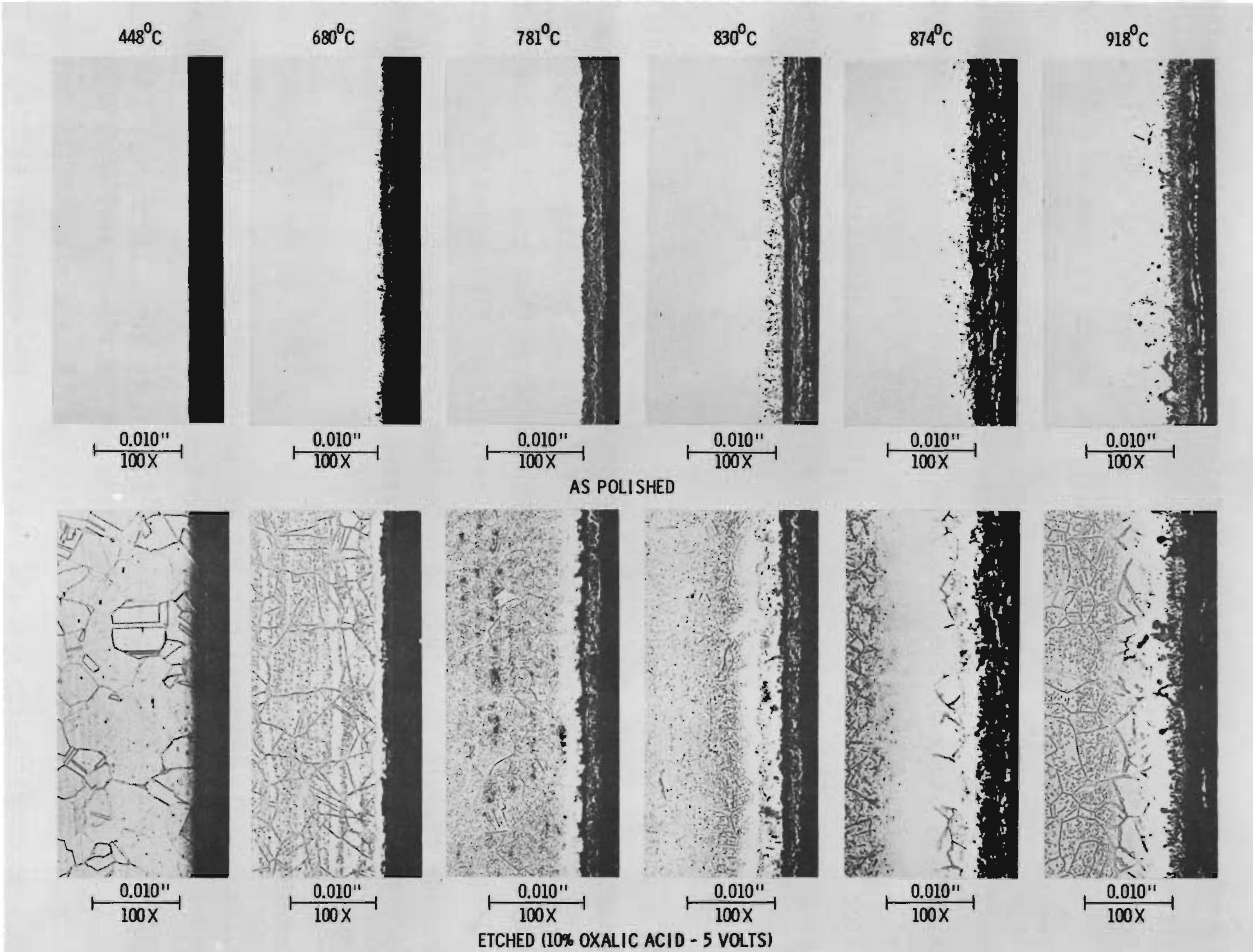


FIGURE 3. Hastelloy C-276 Specimens Exposed to Nonradioactive SrF_2 for 4400 hr

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

Heat Source Capsule Qualification Requirements and Capsule Design Philosophy

A draft report containing PNL's recommended fuel container licensing test matrix and container design philosophy was prepared this quarter. The draft is currently undergoing revision and will be sent to DNRA for comment in the upcoming quarter. The report will be issued as soon as DNRA's comments are incorporated into it.

The licensing test matrix will not include a vibration resistance test, since a cursory study by the Structures and Mechanics Section indicated that there will be no problem as long as the current right circular capsule configuration is maintained. The study included vibration damage from both excitation of any natural frequency of vibration response in the container and movement of the inner container in the outer container.

Capsule Design

No concentrated effort will be made in the capsule design area until the recommendations for the Capsule Design Philosophy have been accepted by DNRA. However, a study to determine the critical design-limiting outer capsule component is continuing, based on the DNRA directive that the final fuel container will retain the right circular cylinder configuration of the existing WESF storage capsule. A study to determine capsule design methods using capsule failure criterion instead of standard elastic strain criterion is also in progress.

Outer Capsule Material Selection

Outer capsule material options are being explored through a continuing literature review and manufacturing contacts as well as the mechanical and corrosion resistant property tests being run at PNL. Cabot Corporation at Kokomo, Indiana has shown continued interest in the general area of radioisotope fuel container materials and has donated a series of its in-house test coupons of Hastelloy C-4 and Hastelloy S for continued testing in PNL's outer capsule material selection program.

The specimens consist of tensile specimens that were aged at various times and temperatures and were then tested at room temperature. The aging temperatures varied from 800 to 1600°F in 200°F increments while the aging times were 1000, 4000, 8000, and 16,000 hr. PNL received one tested specimen for each test condition, and two standard ASTM subsize tensile specimens were subsequently machined out of the broken ends of the original tensile specimens. The gage section of the subsized specimens came from the no-plastic-strain shoulder region of the old specimens, as illustrated in Figure 5. The specimen configuration is the same as that currently used in the compatibility section of this program.

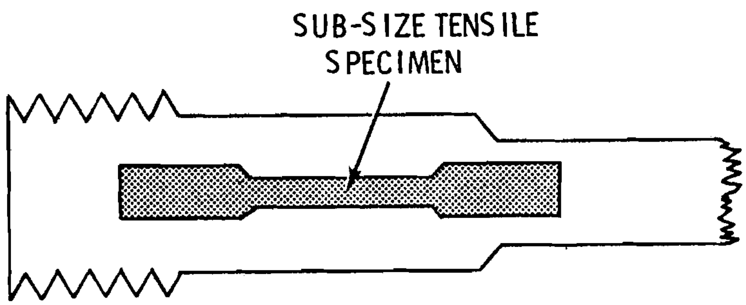


FIGURE 5. Subsized Tensile Specimens to be Machined from Sections of Tested Specimens

It is currently planned that all of the new specimens will be tested at elevated temperatures; one at the aging temperature of the original sample and the other at 800°C, the currently predicted maximum allowable use temperature of the WESF storage capsule inner containment vessel as well as the required fire simulation temperature. The data from these specimens will supplement the Hastelloy C-4 Charpy impact data and the Hastelloy C-4 and Hastelloy S high temperature oxidation resistance and seawater corrosion data being generated in this study.

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

Experiments are underway to determine the effects of thermal aging on the oxidation resistance of Hastelloy C-4 and Hastelloy S. When the solution heat-treated forms of the two alloys are heated at temperatures between approximately 500 and 900°C, they undergo long-range ordering reactions and precipitate formation which affect their mechanical properties.(2) The reactions involved proceed relatively slow and can occur over a several thousand hour period when the alloys are heated. It is anticipated that the microstructural changes which occur when the alloys are heated between 500 and 900°C could affect their resistance to oxidation.

Specimens of the two alloys, which had been held at 600, 700, 800 or 900°C for 1000 hr, were heated in air and the rate of oxidation determined as a function of time. At 1000°C or less the oxidation rates of the thermally aged specimens were similar to those previously reported for the solution heat-treated alloys. At 1100°C, however, there were significant differences in the oxidation rates of the thermally aged and the solution heat-treated specimens. This is suprising since the oxidation temperature of 1100°C is above the standard heat-treating temperature of the two alloys which is 1950°F (1066°C). When heated in air at 1100°C each alloy shows a slight initial weight gain followed by a continuous weight loss due to spalling of the oxide layer. The oxidation rates of the aged and solution heat-treated specimens at 1100°C are shown in Figures 6 and 7. Metallographic examination of test specimens is now underway to determine what microstructural differences might cause the differences in oxidation rates at 1100°C of aged and unages specimens.

Additional oxidation tests are now underway with specimens of Hastelloy S and Hastelloy C-4 which had been aged for 5000 hr at 600, 700, 800, or 900°C.

Prototype Capsule Fabrication and Testing

A capsule containment chamber to be used in conjunction with the helium leak detector purchased for this program has been fabricated and installed on detector cart; Figure 8. The chamber will be used for leak checking the prototype fuel containers both before and after they are subjected to the fuel container licensing test matrix currently being prepared for DNRA.

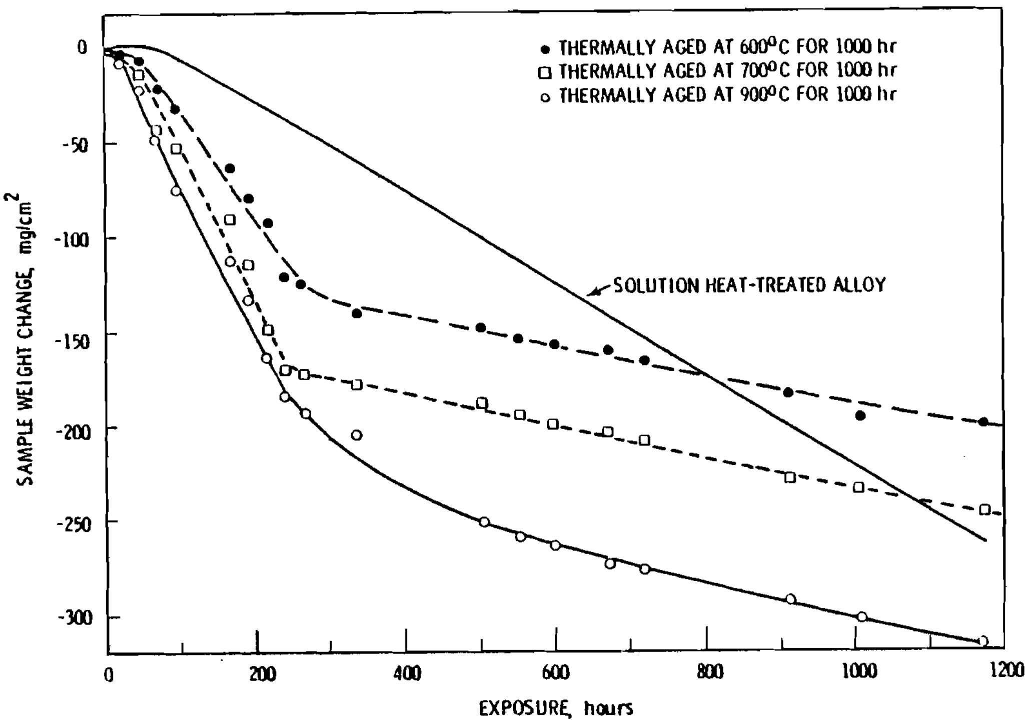


FIGURE 6. The Effect of Thermal Aging on the Oxidation Resistance of Hastelloy C-4 at 1100°C

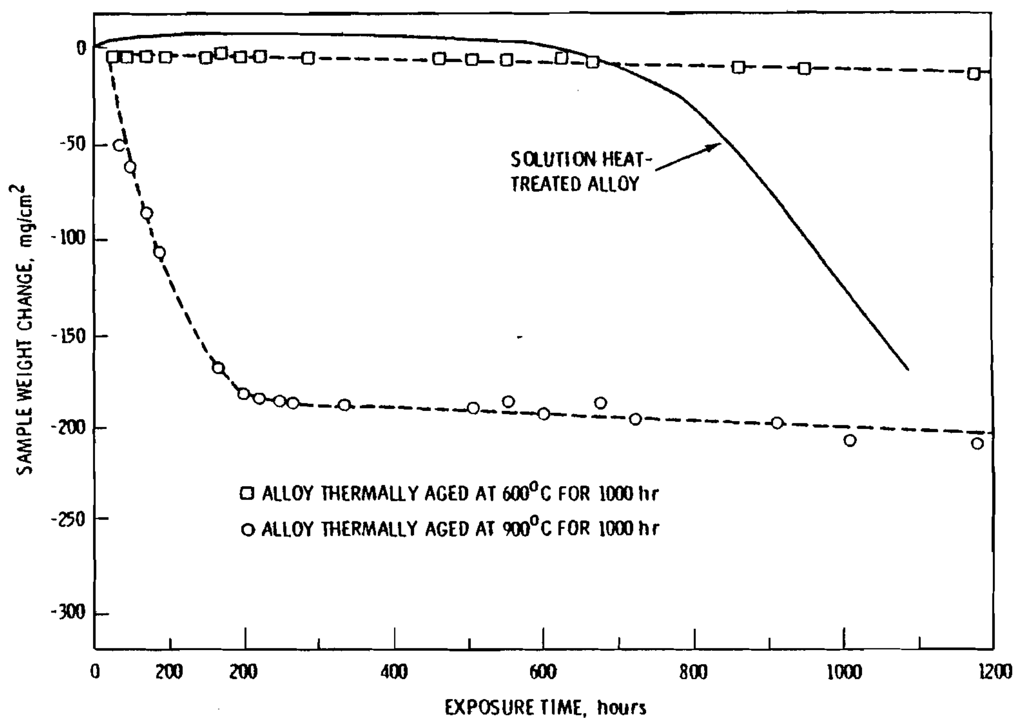


FIGURE 7. The Effect of Thermal Aging on the Oxidation Resistance of Hastelloy S at 1100°C

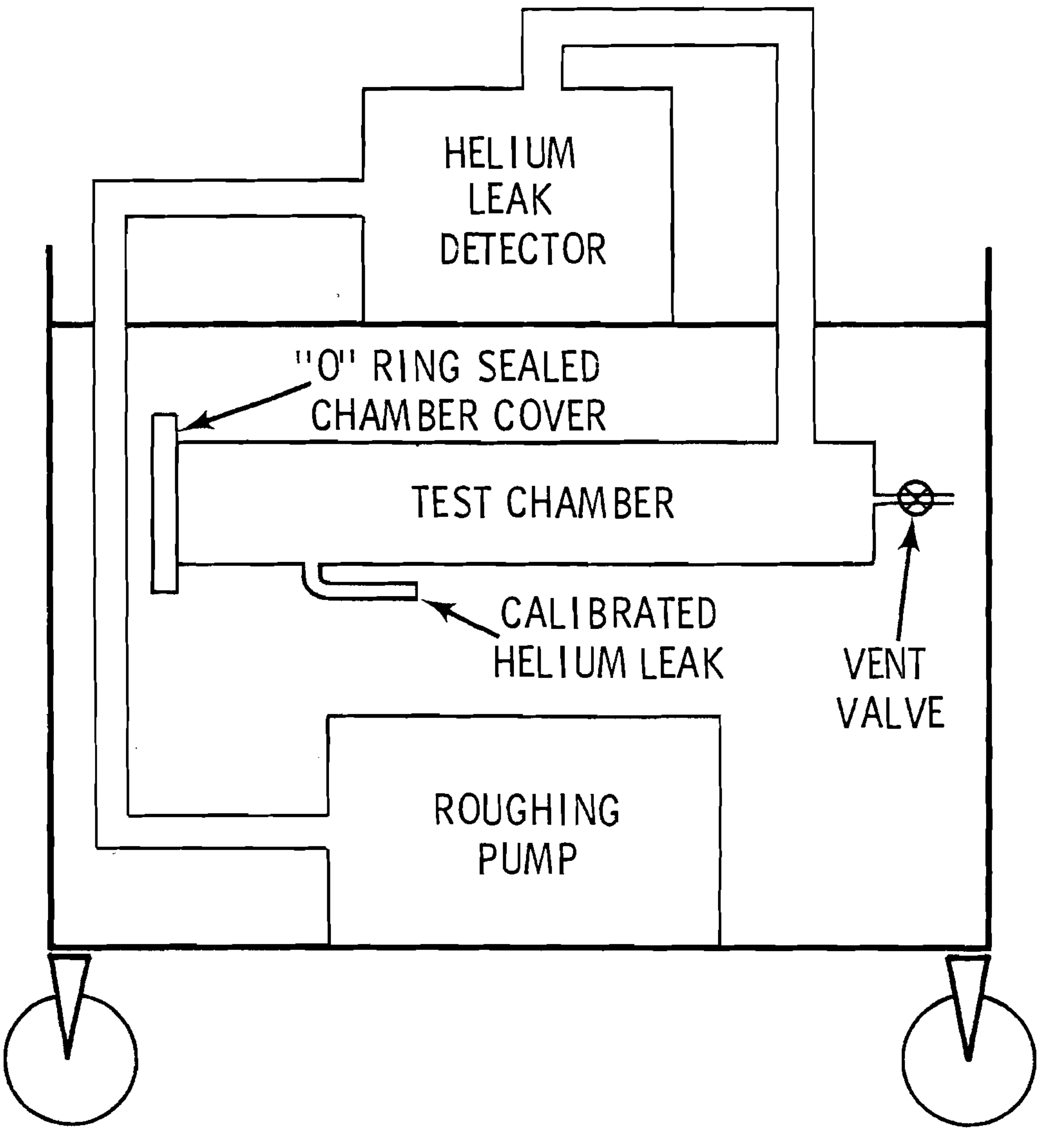


FIGURE 8. Leak Detector and Test Chamber for Testing Full-Size Prototype $^{90}SrF_2$ Capsules

BENEFICIAL ISOTOPES UTILIZATION PROGRAM

J. H. Jarrett, Program Manager

K. M. Harmon, Principal Investigator

The objectives of the program are to identify and develop beneficial uses of nuclear reactor by-products through: 1) estimation of long-term availability and cost of useful isotopes from commercial suppliers; 2) identification and development of beneficial applications of isotopes, including their use in remote regions of the world; 3) identification and evaluation of the actions required to optimize the $^{90}SrF_2$ *and* $^{137}CsCl$ *products from the Hanford Waste Encapsulation and Storage Facility (WESF) for beneficial use; and 4) review and evaluate the use of radioisotopes for fueling thermal systems which might be used by the U.S. Army.*

The program is divided into four tasks:

Task I - Isotopes Availability
Task II - Cold Regions Applications
Task III - WESF Product Utilization
Task IV - General Application of Radioisotopes in Army Thermal Systems

TECHNICAL PROGRESS

TASK I - ISOTOPES AVAILABILITY

Flowsheet Calculations (D. K. Davis)

No reportable progress during this reporting period.

TASK II - COLD REGIONS APPLICATIONS (W. E. Sande and R. L. Aaberg)

A draft report on the economic feasibility of heaters fueled with strontium fluoride capsules for use in cold regions has been written and is being reviewed. New sections are being added to explain isotope systems and to emphasize their safety.

The equivalent cost of fuel oil for an isotope fueled water heater system is estimated to be $9 to 11/gal (about $1/gal not including the cost of the isotope capsules) while the cost of fuel oil for a septic tank heater system is estimated to be $6.00/gal ($2/gal not including isotope costs); development costs are not included in these estimates.

The Federal Aviation Administration has submitted a license application to the NRC for the use of radioisotope thermoelectric generators (RTG's) as aircraft navigational aids. They hope to receive the license in March which would enable installation of the RTG's by next summer. The safety assessment is now being prepared.

TASK III - WESF PRODUCT UTILIZATION

Sandia Support

1. Study of Sludge Handling Alternatives (H. E. McGuire, Jr. and G. W. Dawson)

A final report has been completed and comments are being solicited from appropriate personnel.

2. Odor Evaluation of Treated Sludge (D. B. Cash)

The final report for this study has been completed and distributed. No additional analysis work will be required since Sandia Laboratories indicated that PNL had studied the samples that would have been subjected to the greatest amount of odor variation.

TASK IV - GENERAL APPLICATION OF RADIOISOTOPES IN ARMY THERMAL SYSTEMS (W. E. Sande)

A draft of the final report on the economics of using waste fission product canisters to fuel Army steam plants is currently being reviewed at PNL and by Army staff. It will then be distributed for external review and will be issued in final form in August.

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