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QUARTERLY TECHNICAL PROGRESS REPORT
FUELS, MATERIALS, AND COOLANT
CHEMISTRY PROGRAMS
OCTOBER - DECEMBER 1970

AEC Research and Development Report

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BY 9 H Kahn/amh
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Atoms International
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**QUARTERLY TECHNICAL PROGRESS REPORT
FUELS, MATERIALS, AND COOLANT
CHEMISTRY PROGRAMS
OCTOBER – DECEMBER 1970**

**The preceding Quarterly Progress Report was
AI-AEC-12980**

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CONTENTS

<u>AEC Task No.</u>	<u>Program and Project Title</u>	<u>Page</u>
	<u>Sodium Technology</u>	
1, Phase I	Coolant Related Mechanical Properties	
	A. Mechanical Properties in Dynamic Sodium	5
	B. Mechanical Properties in Static Sodium	25
	C. Interstitial Transport	35
1, Phase II	Sodium Quality Measurement	37
1, Phase III	Special Compatibility (Sodium Frost).	39
1, Phase IV	Radioactivity Transport.	40
1, Phase V	FFTF Component Qualification.	41
	<u>Radiation Effects</u>	
2-A	Irradiation Damage in Cladding and Core Structural Material.	43
	<u>Fuel Cladding and Structural Materials</u>	
2-B	Integral (n, α) Cross-Section Measurements	51
	<u>LMFBR Technology</u>	
3	Fast Reactor Fuels.	54

Program:	Sodium Technology		
AEC Task:	1, Phase I: Coolant Related Mechanical Properties A. Mechanical Properties in Dynamic Sodium		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1970		
General Order:	6507	Subaccount:	39140
		AEC Category:	04-01-61-03.1

Principal Investigator: J. H. Shively

I. PROJECT OBJECTIVES

The objectives of Task A are:

- 1) To determine the effects of mass transport on the mechanical behavior of cladding and structural alloys under LMFBR system conditions.
- 2) To provide data relating the quality of the sodium as indicated by on-line instrumentation and conventional analytical methods to the degradation in biaxial creep and fatigue behavior of cladding and structural alloys.
- 3) To investigate the effects of the sodium system, its operation, and alloy type and composition being studied to the quality of the sodium.
- 4) To provide a basis for extrapolating sodium impurity effects on mechanical behavior in a flowing sodium system under non-isothermal conditions to allowable materials property degradation in large engineering systems.

II. PROGRESS DURING REPORT PERIOD

1. Summary

The second ΔT Leg Experiment was completed this quarter, after 2117 hr. The test involved the following: (1) biaxial stress-rupture specimens of the same lot of previous ΔT Leg Experiments but without wire wrap, (2) five heats of biaxial creep specimens in series, (3) two linear pressure specimens in support of FFTF gas plenum modeling, (4) a series of mass transfer rings for weight loss and carbon/nitrogen measurements, and (5) post-exposure stress-rupture specimens. The test conditions are displayed in Table 1.

TABLE 1
TEST CONDITIONS

Test Temperature	1200°F ± 2.0 F°
Sodium Velocity	8.4 ft/sec ± 0.2 ft/sec
Leg ΔT	50°F ± 2.0 F°
Bulk Sodium Temperature	1150°F ± 2.0 F°
ΔT Axial-Test Section	12°F ± 2.0 F°

TABLE 2
TEST ASSEMBLY INFORMATION

Materials	10 to 15 CW Type 316 stainless steel Annealed Type 304 stainless steel Annealed Type 316 stainless steel
Cladding Dimensions (in.)	0.250 OD by 0.010 wall by 20 long 0.297 OD by 0.010 wall by 4 long
Strain Allowance in Bundle	7.7 ΔD/D
Shroud Material	Annealed Type 316 stainless steel
Pressurization Gas	Helium
Pellet Dimensions (in.)	0.250 by 3.230 long
Pellet Material	Annealed Type 316 stainless steel
End Cap Weld Type	Electron Beam Lap
End Cap Material	Matches Cladding
End Cap Type	Solid Plug

Weight measurements were completed on annealed and cold-worked Type 316 stainless steel rings exposed to flowing sodium in ΔT Leg Experiment No. 2. Preliminary evaluation indicates a lower corrosion rate for annealed stainless steel than that for cold-worked stainless steel. The corrosion rates are less than the values in the literature for Type 316 stainless steel.

Carbon and nitrogen measurements are being made on the cladding used in the second ΔT Leg Experiment.

Creep and rupture strain measurements were completed on the tubes tested in ΔT Leg Experiment No. 2. The results are somewhat surprising in that the creep strains manifested by each of four annealed heats of stainless steel were widely different at the same internal gas pressure. The secondary creep rates varied over an order of magnitude among the annealed heats.

2. Evaluation

a. Second ΔT Leg Test - Description

The test conditions for the second ΔT Leg Experiment in the two-inch flowing sodium loop are presented in Table 1. The geometrical conditions are outlined in Table 2.

The experiment involved a combination of tests to determine the effect of flowing sodium on the mechanical properties of Types 304 and 316 stainless steel. This is accomplished by performing stress-rupture tests, biaxial creep tests, pressure ramp rupture tests, weight loss measurements, and post-exposure stress-rupture tests. The specimen designs are shown in Figure 1. The individual test parameters are presented in Table 3. It should be noted that while the pressures are identical in the series tubes (Figure 1), minor differences in wall thicknesses between lots cause slight differences in the hoop stresses. To prevent early rupture, the stresses were set to give rupture in ~ 2500 hr on the highest stressed specimen in the chain, viz., Heat No. 03574.

Index lines were placed on the end caps of all tubes. Measurements made before exposure will be compared to those after exposure. The rings were weighed prior to exposure. A control sample has been retained to determine the effects of handling and cleaning on the weight loss measurements after exposure.

TABLE 3
SPECIMEN TEST PARAMETERS

Specimen Number	Pressure at Temperature (psi)	Time at Pressure (hr)	Stress for Several Heat Numbers and Materials** (psi)				
			A 20013 304A	B 65808 316A	C 03574 316A	D 20303 316A	E 65808 316 CW
30	1440	1525	19,990	19,910	21,000	19,970	20,070
31	1440	1532	19,990	19,910	21,000	19,970	20,070
32	1440	1433	19,990	19,910	21,000	19,970	20,070
33	1440	1001	19,990	19,910	21,000	19,970	20,070
34	1440	600	19,990	19,910	21,000	19,970	20,070
36	1440	305	19,990	19,910	21,000	19,970	20,070
37	1440	114	19,990	19,910	21,000	19,970	20,070
38	1540	114	21,400	21,300	22,500	21,370	21,470
39	1540	194	21,400	21,300	22,500	21,370	21,470
45	1540	305	21,400	21,300	22,500	21,370	21,470
46	1540	500	21,400	21,300	22,500	21,370	21,470
47	1540	762	21,400	21,300	22,500	21,370	21,470
48	1540	1001	21,400	21,300	22,500	21,370	21,470
49	1540	1200	21,400	21,300	22,500	21,370	21,470
50*	2117	2117					25,148
51*	2117	2117					25,148
8*	2117	1821					25,148
43†	Ramp	2107					Ramp
44§	Ramp	1222					Ramp

*Specimens identical to ΔT Test No. 1 material

†Two specimens of 316 CW, Heat No. 65808, and two specimens 316A, Heat No. 65808, filled with 1 atm argon at room temperature

§Twenty-one mass transfer rings of cold-worked annealed Type 316 stainless steel, Heat No. 65808

**See Subtask B for compositions of three heats of Type 316 stainless steel

PRESSURE
RAMP

MASS TRANSFER RINGS
CW-316 SS AND A-316 SS

NOTE: A, B, C, D AND E REFER TO TABLE 3.

1-14-71

6507-4740A

Figure 1. Mechanical Properties Test Specimens for Loop ΔT

6507-5103

Figure 2. Biaxial Stress-Rupture Behavior of Annealed Type 316 Stainless Steel at 1200°F in Static and Flowing Sodium

6507-5104

Figure 3. Strain-Rate Dependence of Fracture Strain of Annealed Type 316 Stainless Steel

b. Stress Rupture

Previously, it was reported that specimens from Heat No. 03574 had ruptured.⁽⁶⁾ These specimens had the largest initial hoop stress (Table 3). However, visual examination of all the specimens in series revealed that two annealed tubes from Heat No. 65808 had failed instead. A comparison of the stress-rupture life of annealed Type 316 stainless steel from Heat No. 65808 in static sodium vs flowing sodium is presented in Figure 2. This constitutes a minor correction from a similar comparison made last quarter. The data fall at the bottom of the 2σ band. Thus, it was evident, when compared to static sodium, that flowing sodium did not significantly reduce the stress-rupture life of stainless steel.

In an earlier series of tests, it was observed that the biaxial-rupture strain was a factor of two lower in flowing sodium than it was in static sodium. This material was cold-worked Type 316 stainless steel. In the present tests, no difference in effect of flowing sodium, when compared to static sodium, was observed for annealed Type 316 stainless steel from Heat No. 65808. This is illustrated in Figure 3. Clearly, the rupture strain in flowing sodium lies within the scatterband of rupture strain in static sodium. This comparison supports the idea that the stress-rupture behavior in flowing sodium during ΔT Leg Test No. 2 was not significantly different from that observed in previous tests in static sodium.

c. Biaxial Creep

The biaxial creep curves for the four annealed stainless steel heats are presented in Figures 4, 5, and 6. The strain values are presented in Table 4. There are considerable differences in creep behavior between the heats. The small differences in initial stresses, from the same gas pressure, are due to minor variations in the wall thicknesses and diameters between heats. These variations cannot entirely explain the differences in creep behavior. The creep curves for cold-worked Heat No. 65808 are presented in Figure 6. One would have to conclude from this work that "heat-to-heat" variations from alloy chemistry are equally as important as "heat-to-heat" variations from process history. Further work is required to determine whether flowing sodium contributed to the observed differences.

6507-5105

Figure 4. Biaxial Creep of Annealed Heats of Stainless Steels in 1200°F Flowing Sodium

Figure 5. Biaxial Creep of Annealed Heats of Stainless Steels in 1200°F Flowing Sodium

TABLE 4
CREEP AND RUPTURE STRAIN MEASUREMENTS

Specimen Number	Time at Pressure (hr)	Type Test	Strain Rates for Several Heat Numbers and Materials $\epsilon(\%)$				
			20013 304A	65808 316A	03574 316A	20303 316A	65808 316CW
30	1525	R	12.18	24.00	5.61	1.44	0.167
31	1532	R	12.51	23.90	2.28	1.27	0.234
38	1433	C	11.67	21.49	5.07	1.44	0.267
33	1001	C	5.15	7.45	3.67	1.50	0.100
34	600	C	0.98	4.41	3.39	1.34	0.134
36	305	C	0.74	2.67	2.08	1.17	0.134
37	114	C	0.57	2.04	1.58	0.87	0.067
38	114	C	1.01	2.77	2.28	1.50	0.167
39	194	C	1.09	3.53	2.67	1.64	0.167
45	305	C	1.41	4.45	3.02	1.94	0.067
46	500	C	5.02	5.33	3.87	1.84	0.334
47	762	C	5.00	8.43	4.07	2.00	0.367
48	1001	C	5.35	13.06	4.90	1.60	0.200
49	1200	C	12.46	16.76	4.42	1.89	0.333
43	2107	R					5.74
44	1222	R					3.34
8	1831	R					0.97
50	2117	C					1.10
51	2117	C					0.86

C = Creep
R = Rupture

Figure 6. Biaxial Creep of Cold-Worked Type 316 Stainless Steel in 1200°F Flowing Sodium

The extent of primary creep strain reflected in the annealed curves, Figures 4 and 5, should be qualified because the stresses exceeded the yield strength of the material. As a result, a significant fraction of the initial strain is yielding. This point is illustrated in Figure 7. It is interesting to note that the extent of primary creep strain correlates with the yield strengths (0.2% offset) of the three heats. It is also clear that yielding cannot explain all the short-term plastic strain obtained in the creep tests on annealed Type 316 stainless steel. Also, the primary creep strain in the cold-worked stainless steel is $\leq 0.2\%$ strain at stresses below the yield strength.

The secondary creep rate is a function of the alloy heat. As illustrated in Figure 8, the secondary creep rate at a given stress varied almost an order of magnitude between annealed heats. The heat-to-heat variation is almost as large as the effect of 10 to 15% cold-work on Heat No. 65808. Based on these observations, design predictions for a given stainless steel component must account for variations in properties from "heat-to-heat."

d. Ramp Test

The purpose of this test is to study creep-rupture behavior when the stress increases linearly with time. This was prompted by a desire to further investigate the creep response observed in an earlier test.⁽¹⁾ The linear stress ramp

6507-5108

Figure 7. Comparison of Primary Creep Strain With Yielding Strain Predicted From Stress-Strain Curve

6507-5109

Figure 8. Secondary Creep Rate of Several Heats of Stainless Steels at 1200°F

was simulated by a series of small steps. The loading sequences are presented in Table 5 and Figure 9. Initially, the pressure on each specimen was set at 182 psi. Thereafter, every $\sim 168 \text{ hr} \pm 1 \text{ hr}$ (7 days), the internal gas pressures were raised 182 and 364 psi for Specimens Nos. 43 and 44, respectively. Failure was predicted as shown in Table 5 when the pressure equaled 2366 psi and after 2044 and 1192 hr for Specimens Nos. 43 and 44, respectively. The time to rupture was estimated, using the cumulative life fraction rule. This rule states when life fraction equals one, failure is expected. The cumulative life fraction is obtained by adding the fraction of life expended at each stress level. Table 5 illustrates this technique for Specimens 43 and 44.

Since the test was performed in flowing sodium, the stress-rupture data obtained in static sodium had to be corrected for mass transfer. Based on mass transfer results on stainless steel,⁽²⁻⁴⁾ the corrosion rate of 0.7 mils/yr was used. In 2100 hr, this corresponds to ~ 0.2 mils. As a result, the stress was 525 psi higher at the end of the test after that correction. Carbon loss caused a degradation in properties.^(5,6) In order to include this interaction, the actual properties obtained in flowing sodium during the previous ΔT Leg Experiment were used, as shown in Table 5. The observed life agreed quite well with the predictions. Specimen No. 43 lasted 2107 hr vs the predicted 2044 hr, and Specimen No. 44 lasted 1222 vs 1192 hr predicted.

TABLE 5
STRESS RAMP PREDICTION OF ΔT TEST NO. 2

Specimen Number	Hoop Stress (psi)	Time at Stress (hr)	Stress Rupture* Life at Stress (hr)	Fraction of Life Expended	Cumulative Life Fraction
43	2,540	0	1.0×10^9	0	0.000
43	5,080	168	1.0×10^8	1.7×10^{-6}	~ 0.000
43	7,620	168	8.0×10^6	2.1×10^{-5}	~ 0.000
43	10,160	168	2.8×10^5	6.0×10^{-4}	~ 0.000
43	12,700	168	1.7×10^5	1.0×10^{-3}	0.001
43	15,240	168	6.0×10^4	2.8×10^{-3}	0.004
43	17,780	168	2.0×10^4	8.4×10^{-3}	0.012
43	20,320	168	8.4×10^3	2.0×10^{-2}	0.032
43	22,860	168	4.0×10^3	4.2×10^{-2}	0.074
43	25,400	168	1.9×10^3	8.8×10^{-2}	0.162
43	27,940	168	1.0×10^3	1.7×10^{-1}	0.332
43	30,480	168	6.1×10^2	2.8×10^{-1}	0.612
43	33,020	<u>196</u>	4.0×10^2	<u>4.9×10^{-1}</u>	1.002
		2044		1.002	
44	2,540	0	1.0×10^9	0	0.000
44	7,620	168	8.0×10^6	2.1×10^{-5}	~ 0.000
44	12,700	168	1.7×10^5	1.0×10^{-3}	0.001
44	17,780	168	2.0×10^4	8.4×10^{-3}	0.009
44	22,860	168	4.0×10^3	4.2×10^{-2}	0.051
44	27,940	168	1.0×10^3	1.7×10^{-1}	0.221
44	33,020	<u>352</u>	4.0×10^2	<u>8.8×10^{-1}</u>	1.001
		1192		1.001	

*Stress-rupture life is estimated by extrapolating the stress-rupture data obtained in flowing sodium in ΔT Leg Test No. 1.

Figure 9. Pressure Program for Ramp Tests on Cold-Worked Type 316 Stainless Steel in 1200°F Flowing Sodium

TABLE 6
CORROSION OF TYPE 316 STAINLESS STEEL

Specimen Number	Distance From Inlet* (in.)	Weight Loss (mg)	Uncertainty (3 σ)	$\frac{\Delta m}{\text{mass}}$ (%)	Weight Loss/Area (mg/dm ²)	Corrosion Rate (mils/yr)	Material Condition†
A-1	0.75	0.877	0.018	0.0939	19.4	0.0400	Annealed
A-2	1.50	0.851	0.019	0.0916	18.8	0.0386	Annealed
A-3	2.25	0.836	0.019	0.0903	18.5	0.0380	Annealed
A-4	3.00	0.804	0.023	0.0861	17.8	0.0365	Annealed
A-5	3.75	0.775	0.015	0.0828	17.1	0.0351	Annealed
A-6	4.50	0.741	0.017	0.0790	16.4	0.0336	Annealed
C-7	5.25	1.749	0.017	0.1865	38.7	0.0794	Cold-Worked
C-8	6.00	1.616	0.013	0.1723	35.7	0.0733	Cold-Worked
C-9	6.75	1.511	0.015	0.1607	33.4	0.0685	Cold-Worked
C-10	7.50	1.539	0.017	0.1641	34.0	0.0698	Cold-Worked
C-11	8.25	1.480	0.020	0.1576	32.7	0.0671	Cold-Worked
C-12	9.00	1.531	0.015	0.1626	33.8	0.0694	Cold-Worked
C-13	9.75	1.501	0.017	0.1596	33.2	0.0681	Cold-Worked
C-14	10.50	1.464	0.017	0.1561	32.4	0.0665	Cold-Worked
C-15	11.25	1.445	0.018	0.1545	34.2	0.0702	Cold-Worked
C-16	12.00	1.477	0.014	0.1575	32.7	0.0671	Cold-Worked
A-17	12.75	0.533	0.014	0.0569	11.8	0.0242	Annealed
A-18	13.50	0.551	0.020	0.0589	12.2	0.0250	Annealed
A-19	14.25	0.740	0.020	0.0789	16.4	0.0336	Annealed
A-20	15.00	0.546	0.019	0.0586	12.1	0.0248	Annealed
A-21	15.75	0.510	0.011	0.0543	11.3	0.0231	Annealed

*Back edge of ring is 2.05 in. from nose of end cap at inlet.

†Material is from Heat No. 65808.

e. Mass Transfer

The results of weight measurements on rings exposed to flowing sodium are displayed in Table 6. These data were converted to corrosion rates. Figure 10 shows that cold-worked Type 316 stainless steel exhibited significantly higher corrosion rate than annealed Type 316 stainless steel. Also, the corrosion rate decreases with downstream position as reported by others.⁽²⁻⁴⁾ For the 0.3 OD by 0.75-in. long cladding specimens, these weight losses translate into a maximum rate of 0.079 mils/yr. This rate, if constant during the test, is less than literature values at 1200°F.^(2,3) The corrosion rate assumed in the ramp test was approximately nine times this value. The reason for a lower rate is probably associated with the small ΔT for this experimental work.

III. EVALUATION OF EFFORT TO DATE

This task has been successful in measuring some effects of the coolant on the performance of stainless steel. The current effort continues to discover the effects of flowing sodium on several heats of Type 316 stainless steel.

IV. NEXT REPORT PERIOD ACTIVITIES

During this period, activities will be concentrated on completing carbon and nitrogen measurements, and beginning a topical report on ΔT Leg Experiment No. 2.

6507-5111

Figure 10. Corrosion Rate of Annealed and Cold-Worked Type 316 Stainless Steel as a Function of Axial Distance in the ΔT Leg

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TABLE 7
BIAXIAL STRESS-RUPTURE TESTS - 12 SPECIMENS PER RETORT

Retort No.	Test Temperature (°F)	Alloy	Cladding Condition*	Environment	Life Span (hr)	Status		Remarks	Date Start/Completed
						Test	Examination		
17	1200	304SS	Ann	Na	5000-10,000	3/4 complete	-	Extension of data	2-22-68
22	1000	304SS	Ann	Na	5000-10,000	complete	-	Extension of data	2-29-68/7-23-70
25	900	304SS	Ann	Na	1000-10,000	complete	-	Extension of data	10-8-68/8-10-70
26	900	304SS	AR	Na	1000-10,000	complete	-	Extension of data	3-27-69/8-21-70
110	1000	316SS	AR	Na	1000-10,000	3/4 complete	-	Extension of data	4-23-68
113	900	316SS	AR	Na	100-10,000	in progress	-	Extension of data	8-16-68
115	1000	316SS	AR	Na	100-5000	complete	-	Effect of sodium	10-18-68/11-9-70
116	1000	316SS	AR + Aged	Na	100-5000	complete	-	Effect of thermal aging	11-5-68/7-31-70
120	900	316SS	Ann	Na	1000-10,000	1/2 complete	-	Extension of data	12-20-68
126A	1200	316SS	AR	Na	1000	complete	-	Cleaning study	8-20-70/10-8-70
127A	1000	316SS	AR	Na	2000 Soak	on test	-	Cleaning study	10-22-70
130	1200	316SS	AR	Na	200-3000	complete	-	Lot-to-lot study	4-27-70/8-31-70
133	1200	316SS	Ann	Na	200-2000	complete	-	Heat No. 03574	9-15-70/10-27-70
134	1200	316SS	Ann	Na	200-2000	complete	-	Heat No. 20303	9-17-70/12-14-70

*AR = As-received tubing containing 10 to 15% cold-work
Ann = Annealed tubing

Program:	Sodium Technology		
AEC Task:	1, 824 Contract — Phase I: Coolant Related Mechanical Properties B. Mechanical Properties in Static Sodium		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1970		
General Order:	6507	Subaccount:	39130
		AEC Category:	04-01-61-03.1

Principal Investigator: D. F. Atkins

I. PROJECT OBJECTIVES

The objectives are to determine the individual and combined effects of environment (sodium and helium), strain rate (10^{-7} to 10^{-3} /hr) and stress condition (uniaxial and biaxial) on the creep and stress-rupture characteristics of Types 304 and 316 stainless steel cladding alloys.

II. PROGRESS DURING REPORT PERIOD

Long-term biaxial stress-rupture tests were continued in multiple specimen static sodium retorts. Thin-wall tubing of both Types 304 and 316 stainless steel (0.275-ID by 0.010-in. wall) are internally pressurized to produce a 2:1 biaxial stress. The status of the retorts is given in Table 7.

Twelve specimens of Type 316 stainless steel tubing (10 to 15% cold-work) from Retort No. 115 were removed and examined. These specimens had been under biaxial stress-rupture test in helium at 1000°F at stress levels to give rupture over 700 to 15,000 hr. The ruptures were all of the small pin-hole type. In contrast, similar tests in sodium exhibited explosive type ruptures at this temperature. As reported previously, the rupture strength of the tubing in helium was found to be stronger at high stress levels than in the comparison tests in sodium. The results of the present test again show this behavior but at lower stress levels (longer rupture times); the rupture strength in the two environments is the same. This behavior is illustrated in Figure 11.

The rupture strain of the cold-worked Type 316 stainless steel tubing was found to be lower in helium than in sodium at 1000°F. This behavior is depicted in Figure 12. It is of interest to note that at 1400°F, the helium environment produced greater strain for a given stress level. While biaxial tests in both

6507-5112

Figure 11. Biaxial Stress-Rupture Strength of Type 316 Stainless Steel Tubing (10 to 15% Cold-Work) in 1000° F Helium and Sodium

6507-5113

Figure 12. Hoop Stress vs Diametral Strain for Cold-Worked (10 to 15%) Type 316 Stainless Steel Tubing in Sodium and Helium Environments

6507-5114

Figure 13. Heat-to-Heat Study of Annealed Type 316 Stainless Steel Tubing in 1200°F Sodium

environments were not performed at 1200°F, uniaxial stress-rupture tests in helium produced greater strain. A change in rupture mechanism apparently occurs between 1200 and 1000°F.

Thermal aging of cold-worked Type 316 stainless steel was investigated in 1000°F sodium. In this test, twelve specimens were aged for 3000 hr without stress. Subsequent biaxial stress-rupture tests in 1000°F sodium have now been completed. When these results were compared with similar tests on non-aged specimens, it was found that the aging produced a slight increase in rupture strength and a loss in ductility. This behavior is characteristic of aged material where carbide precipitation and possible sigma formation develops during long-term high temperature exposure.

Biaxial stress-rupture tests on two special heats of annealed Type 316 stainless steel tubing were performed in 1200°F sodium. Figure 13 compares the rupture strength of the two heats with the Reference Heat No. 65808 of the same alloy. It is seen that the strength of Heat No. 03574 is identical with that of the Reference Heat. Heat No. 20303 exhibits a stronger behavior than the other two alloys. As tabulated below, the alloy chemistry of these three heats are quite similar in composition. The elemental composition spread among the heats is smaller, compared to the allowable ranges for the alloy. If the heats are

Heat No.	C	Mn	S	P	Si	Ni	Cr	Mo	Cu	Co	N ₂	Cr/Ni
03574	0.07	1.57	0.013	0.011	0.53	13.68	17.34	2.38	0.13	<0.05	0.051	1.38
65808	0.06	1.72	0.007	0.012	0.40	13.30	17.30	2.33	0.06	0.03	0.070	1.41
20303	0.06	1.53	0.013	0.010	0.58	12.95	17.30	2.39	0.05	<0.05	0.056	1.48

TABLE 8

BIAXIAL STRESS-RUPTURE DATA ON ANNEALED TYPE 316 STAINLESS
STEEL - HEAT NO. 03574, 1200°F SODIUM, RETORT NO. 133

Specimen No.	Hoop Stress (psi)	Rupture Time (hr)	Diametral Strain (%)	Average Strain Rate (%/hr)
AK6-1	27,000	275	18.7	6.80×10^{-2}
-2	27,000	246	20.3	8.25×10^{-2}
-3	27,000	275	20.4	7.42×10^{-2}
-4	25,000	435	22.5	5.17×10^{-2}
-5	25,000	435	23.9	5.49×10^{-2}
-6	25,000	435	22.0	5.06×10^{-2}
-7	23,000	999	21.8	2.18×10^{-2}
-8	23,000	903	23.5	2.60×10^{-2}
-9	23,000	903	24.4	2.70×10^{-2}
-10	22,000	200 (creep)	2.4	-
-11	22,000	500 (creep)	4.1	-
-12	22,000	1000 (creep)	9.3	-

Figure 14. Stress vs Rupture Strain for Two Heats of Annealed
Type 316 Stainless Steel in 1200°F Sodium

evaluated on the basis of those elements favoring austenitic formation versus those elements which favor ferrite and sigma, subtle differences in the heats appear. Using the formulas,

$$\text{Ni equivalent (austenite formers)} = \text{Ni} + 30\text{C} + 0.5 \text{ Mn} \quad \dots(1)$$

$$\text{Cr equivalent (ferrite-sigma formers)} = \text{Cr} + 2.5 \text{ Si} + 1.8 \text{ Mo} \quad \dots(2)$$

the calculated Cr/Ni ratios for the heats are listed in the table above. Although the differences are small, the ratios indicate that Heat No. 03574 would have the least tendency to form brittle sigma phase under long-term high-temperature exposure.

Diametral strain measurements were taken on Heat No. 03574 and are listed in Table 8. The values are compared with the Reference Heat in Figure 14. For a given stress level, Heat No. 03574 exhibits significantly larger strain. Since both heats were in the solution annealed condition, the differences in strain are probably related to alloy composition. While strain measurements have not been taken on Heat No. 20303 specimens, the Cr/Ni ratio of 1.48 suggests that this heat will exhibit less ductility than either of the other heats.

Three specimens from each of the two new heats were used to provide biaxial creep data. Diametral strain measurements after 200 hr, 500 hr, and 1000 hr will be compared with similar measurements in the flowing sodium ΔT Test of Task A.

The effects of sodium cleaning and storage on the mechanical properties of Type 316 stainless steel tubing (10 to 15% cold-work) are being studied. Procedures were established to simulate conditions which might be encountered in the handling of irradiation test specimens and subsequent in-cell examination. This work is related to Subtask 5-1.5.4 of the LMFBR Program Plan. The test variables are outlined in Table 9. Two sets of twelve specimens were exposed to high temperature sodium for 2000 hr. One set of specimens was exposed to 1200°F sodium while the second set was exposed to 1000°F sodium. The tubular specimens were sealed so that sodium only contacted the outside surfaces. The internal environment was helium. Four of the twelve specimens in each retort were pressurized with helium to provide a modest hoop stress on the thin-walled tubing.

TABLE 9
TEST MATRIX FOR SODIUM CLEANING
AND STORAGE STUDIES

Specimen No.	Treatment No.			
	1 [*]	2 [†]	3 [§]	4 ^{**}
1	X			
2	X			
3		X		
4		X		
5			X	
6			X	
7				X
8				X
9	0			
10	0			
11		0		
12		0		

X = unstressed specimens during sodium exposure
0 = stressed specimens during sodium exposure

Note: Matrix is the same for both 1000 and 1200°F sodium exposed specimens.

*Treatment 1

- a) React sodium - immersion in butyl alcohol
- b) Ultrasonic rinse - distilled water
- c) Ultrasonic rinse - ethanol (absolute)
- d) Air dry
- e) Handle specimens with clean cotton gloves
- f) Store specimens in laboratory air, 1000 hr

†Treatment 2

Same as Treatment 1, except e) - specimens handled with bare hands by several people

§Treatment 3

Same as Treatment 1, except f) - specimens stored in dry nitrogen, 1000 hr

**Treatment 4

- a) React sodium - immersion in Dowanol
- b) Ultrasonic rinse - distilled water
- c) Ultrasonic rinse - ethanol (absolute)
- d) Air dry
- e) Handle specimens with clean cotton gloves
- f) Store specimens in laboratory air, 1000 hr

This stress, 50% of the stress required for 1% strain in 250,000 hr, was applied to see if there were a stress-induced effect during sodium exposure.

During specimen storage, the laboratory air was monitored continuously for temperature and humidity. The nitrogen environment was characterized as to purity and dew point. No attempt was made on these initial tests to impose stress on the specimens during storage, although residual stresses in the tubing were present due to the cold work and the applied stress during sodium exposure.

Following storage, the specimens were reassembled in static sodium retorts. Biaxial stress-rupture tests were conducted at the same temperature as the original sodium exposure. The stress-rupture life and average diametral strain rate will be the criteria used to evaluate the effect of the cleaning and storage treatments. Base line data under the same sodium exposures (without the cleaning and storage steps) have already been established.

The first set of specimens has completed biaxial stress-rupture test in 1200°F sodium. All specimens were stressed to the same level — 28,000 psi — which should produce rupture in ~900 hr, if they were not affected by cleaning and storage. The specimens failed over the range of 850 to 930 hr, which is well within the experimental scatter band. It was concluded that the 1200°F sodium exposure, cleaning, and storage operations had no effect on the rupture strength of the tubing. Next, strain measurements will be taken on the specimens to evaluate the influence of the cleaning and storage procedures on the rupture ductility of the material.

The second set of specimens was subjected to the same cleaning and storage procedures, following 2000 hr exposure in 1000°F sodium. The specimens are now under biaxial stress-rupture test at 46,000 psi. Assuming that cleaning and storage had not affected the specimens, the rupture life at this stress level is ~2000 hr.

Three biaxial stress-rupture test retorts, Nos. 22, 25, and 26, containing Type 304 stainless steel specimens were opened, and the specimens were examined. The specimen diameters after rupture were measured and diametral strains calculated. The stress-rupture strengths, which extend into the 10,000- to 20,000-hr range are plotted in Figure 15. There seems to be a slight slope change for the annealed tubing at both 900 and 1000°F in the vicinity of 9000 hr.

6507 5116

Figure 15. Biaxial Stress Rupture Strength of Cold Worked and Annealed Type 304 Stainless Steel in Sodium

6507-5117

Figure 16. Diametral Strain Rate of Type 304 Stainless Steel Tubing (Heat 20013) in 900°F Sodium

This is probably caused by carbide and sigma formation. Diametral strain rates at 900°F are plotted in Figure 16. The new data are in good agreement with previously generated data, and extend the curves to lower strain rates.

III. EVALUATION OF EFFORT TO DATE

The static sodium tests have provided the first 10,000- to 20,000-hr biaxial stress-rupture data on annealed and cold-worked austenitic stainless steels. The data at 900 and 1000°F are in the temperature regime of the FFTF and FBR demonstration plants. It was found that at these temperatures, the benefits derived from cold-worked materials are quite stable (particularly for Type 316 stainless steel). However, the creep strain (ductility) of these materials is extremely low (<1%) under the low biaxial stresses.

Because of program redirection, work during this period has been directed toward completion of existing tests. The initiation of new work was stopped pending further program guidance.

IV. NEXT REPORT PERIOD ACTIVITIES

Biaxial stress-rupture testing will continue on the long-term tests of Types 304 and 316 stainless steel tubing.

Testing of the two special heats of Type 316 stainless steel tubing will be completed and the results evaluated with respect to alloy chemistry.

The initial phase of sodium cleaning studies will be completed and the results evaluated.

a STRESS RUPTURE STRENGTH

b STRAIN RATE SENSITIVITY TO BIAXIAL STRESS

6507 5118

Figure 17. Comparison of Biaxial Stress Rupture Properties of Case-Carburized and Noncarburized Type 316 Stainless Steel Tubing (10 to 15% CW) in 1200°F Helium

Program:	Sodium Technology		
AEC Task:	1, 824 Contract - Phase I: Coolant Related Mechanical Properties C. Interstitial Transport		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1970		
General Order:	6507	Subaccount:	39120
		AEC Category:	04-01-61-03.1

Principal Investigator: D. F. Atkins

I. PROJECT OBJECTIVES

The project objectives are to study the influence of interstitial compositional changes (initially carbon) on the engineering design properties of structural alloys to be used in high temperature sodium service. Conditions which produce both surface and bulk modifications to the alloy chemistry will be evaluated.

II. PROGRESS DURING REPORT PERIOD

The bulk carburization retort, containing twelve annealed Type 304 stainless steel specimens, has been on test for over 1900 hr. The specimens are exposed to 1100°F sodium with a carbon source of 2-1/4 Cr - 1 Mo steel. The target exposure time is 3000 hr. Under these conditions, it is calculated that the carbon content of the stainless steel will increase 0.1 to 0.2 wt %. The retort is equipped with a pneumatically operated agitating device to homogenize the sodium. This device has operated without difficulty for 16,800 cycles during 1900-hr of test.

Biaxial stress-rupture tests on two case-carburized specimens of Type 316 stainless steel tubing (10 to 15% cold-work) were completed. The carbon-case on the OD surface of the specimens was applied by exposure to 1200°F sodium. Carbon was introduced into the sodium by the decomposition of methane gas. This procedure produced a thin carburized case (~2 wt % C) on the steel surfaces exposed to sodium.

The biaxial stress-rupture strength of these case-carburized specimens are compared with noncarburized material in Figure 17a. As expected, the rupture strength is increased by the presence of the carbon case. Examination of the ruptured specimens showed that explosive type ruptures occurred. In contrast, noncarburized specimens develop pin-hole type failures at this test temperature (1200°F). The carburized specimens did not exhibit any surface cracking or

crazing, as might be expected of a hard, brittle surface case. Strain measurements taken on the diameter of these specimens gave values of 2.57% at 40,000 psi and 3.64% at 33,000 psi hoop stress. On noncarburized material, the strain was over 6% for this stress range. The average diametral strain rates for the case-carburized specimens are compared with non-carburized material in Figure 17b. The carbon case is shown to reduce the creep rate of the Type 316 stainless steel.

Six 10-ft lengths of low carbon 2-1/4 Cr - 1 Mo tubing were received from Battelle Northwest Laboratories. This tubing was fabricated from three heats of alloy with carbon contents of 0.011, 0.035, and 0.080 wt %. Preliminary examination of the tubing shows the dimensional tolerances to be rather poor with respect to straightness, wall thickness, and ovality. The tubing is in the cold-worked condition (~40%) and will have to be annealed before testing. If funding can be made available, biaxial stress-rupture tests will be performed on this material.

III. EVALUATION OF EFFORT TO DATE

The experimental work on case-carburized stainless steel has yielded preliminary information on the mechanical performance of the material at 1200°F. The carbon surface layer was found to increase the biaxial rupture strength and reduce the rupture strain and creep rate. Low cycle fatigue did not seem to be greatly influenced by the carbon case. It should be emphasized, however, that at lower temperatures the carbon layer may exhibit a brittle behavior and seriously reduce the mechanical performance of the steel. Further testing is required over the expected temperature range of reactor operation.

The initiation of new tests on both case-carburized and bulk-carburized stainless steels has been curtailed as a result of the directed "phase-out" of this task. As it now stands, only tests currently in progress will be carried to completion.

IV. NEXT REPORT PERIOD ACTIVITIES

Exposure of specimens in the bulk carburization retort will be continued.

Program: Sodium Technology		
AEC Task: 1, Phase II: Sodium Quality Measurement		
Project Manager: G. W. Meyers		
Reporting Period: October-December 1970		
General Order: 6507	Subaccount: 39200	AEC Category: 04-01-61-03.1

Principal Investigator: E. W. Murbach

I. PROJECT OBJECTIVES

The objectives of this task are to: (1) apply on-line instrumentation to the experimental systems in support of the mechanical properties work, thereby providing reliable monitoring of sodium quality for correlation with the measured mechanical properties changes; (2) evaluate the currently available instrumentation for its applicability and reliability for continuous operation, and to evaluate advanced instruments as they become available; (3) develop and evaluate advanced purification methods for removing carbon, oxygen, and other deleterious impurities from large sodium systems; and (4) develop methods for controlling the carbon activity in systems which have both carbon sources and carbon sinks.

II. PROGRESS DURING REPORT PERIOD

The work for this phase of the Sodium Technology Program has been terminated in line with redirection of activities. The effort of this report period has been directed toward the documentation of design activities relating to oxygen and carbon meter applications for the 2-in. diameter sodium technology loop. An internal report was completed in which design calculations and drawings were included for future use. Two BNL type carbon meters and six UNC type oxygen meter probes were received. These items had been ordered prior to the redirection of the program and, hence, could not be cancelled. These components have been packaged and placed in storage.

III. EVALUATION OF EFFORT TO DATE

This work has been deferred until the completion of the national meter program. Inasmuch as the effort has stopped, no evaluation is appropriate.

IV. NEXT REPORT PERIOD ACTIVITIES

There will be no further report on this activity.

Program: Sodium Technology		
AEC Task: 1, Phase III: Special Compatibility (Sodium Frost)		
Project Manager: G. W. Meyers		
Reporting Period: October-December 1970		
General Order: 6507	Subaccount: 39300	AEC Category: 04-01-61-03.1

Principal Investigator: L. G. Kellogg, E. Matlin

I. PROJECT OBJECTIVES

The objectives of this work are to evaluate the transport of sodium vapor and its compounds (frost) and their subsequent deposition within the annuli of the IVHM rigid arm and rotating plug. Also, it is an objective to verify the argon purge rate of the reference concept for its effectiveness in suppressing xenon diffusion and sodium-vapor transport into the annuli.

II. PROGRESS DURING REPORT PERIOD

A program letter, dated December 1, 1970, approved an activity in the area of sodium-frost deposition in direct support of the FFTF technology program. The activity authorized has not begun, pending decisions relating to the allocation of equipment funds and funds otherwise uncommitted but part of the Sodium Technology Program. A work plan has been generated and tentatively approved by FFTF.

III. EVALUATION OF EFFORT TO DATE

None.

IV. NEXT REPORT PERIOD ACTIVITIES

The program approval should be forthcoming and work commenced to fulfill technology needs, identified by FFTF.

Program: Sodium Technology		
AEC Task: 1, Phase IV: Radioactivity Transport		
Project Manager: G. W. Meyers		
Reporting Period: October-December 1970		
General Order: 6507	Subaccount: 39410	AEC Category: 04-60-10-01.1

Principal Investigators: M. Silberberg, J. Guon, and G. Zwetzig

I. PROJECT OBJECTIVES

The objective of this work is to develop sufficient understanding of the problems of radioactive material transport to permit realistic evaluation of radiation problems that may affect maintenance or repair of a LMFBR primary system. The work includes studies of the transport of both corrosion and fission products.

II. PROGRESS DURING REPORT PERIOD

Data reduction on an abbreviated manganese test series was continued. This work is ~90% complete. Its completion has been delayed by the priority of the FFTF component test work.

III. EVALUATION OF EFFORT TO DATE

None.

IV. NEXT REPORT PERIOD ACTIVITIES

The reduction of the manganese test data will be completed.

Program:	Sodium Technology		
AEC Task:	1, Phase V: FFTF Component Qualification		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1970		
General Order:	6507	Subaccount:	39700
		AEC Category:	04-01-61-03.1

Principal Investigator: J. L. Ballif, J. Guon

I. PROJECT OBJECTIVES

The general objective of this work is to provide component test and/or ordering information on FFTF components such as vapor traps, freeze traps, and filters. Specifically the objectives are to:

- 1) Determine the basis for, and obtain approval of, the RFP (ordering documents) for FFTF static and dynamic sodium vapor traps, revise and issue the RDT standard for these traps, provide reports on the evaluation of these traps.
- 2) Determine the basis for, and approval of, the RFP (ordering documents) for sodium freeze traps, including a report on the evaluation of the prototype.
- 3) Provide an RFP (ordering document) and/or RDT standard for filters in the FFTF charging facility.

II. PROGRESS DURING REPORT PERIOD

Approval was received December 1, 1970 for this work. Since that time, drawings of the RDT vapor trap, the ANL vapor trap, the Barton vapor trap, and the freeze trap have been completed, checked, and released. The procurement work is 95% complete, and the component fabrication is currently underway. The testing work will be conducted at Santa Susana and will commence as soon as the component fabrication is completed. The test components are being built at AI in order to expedite this schedule.

Experienced personnel have been assigned to writing the RDT standards or the revision of existing ones. A work plan covering the test effort has been sent to RDT and to WADCO. This represents the second iteration after previous discussions with WADCO at their site late in October 1970.

III. EVALUATION OF EFFORT TO DATE

The progress has been excellent thus far. The program letter approval in December pressed the schedule, but the cooperation of all involved has made the progress possible. The fabrication is proceeding very well because of purchased item availability and shop interest in completing a quality job.

IV. NEXT REPORT PERIOD ACTIVITIES

The fabrication of the initial phase components will be completed.

The test will be assembled and test work commenced.

Program:	Radiation Effects		
AEC Task:	2-A, Irradiation Damage in Cladding and Core Structural Material		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1970		
General Order:	6506	Subaccount:	29621, 29622 AEC Category: 04-40-02-01.1

Principal Investigator: D. Kramer

I. PROJECT OBJECTIVES

The objectives of this task are to:

- 1) Study the process of void formation and helium embrittlement in fuel cladding and core structural alloys by means of accelerator irradiations and reactor irradiations.
- 2) Determine the effects of fluence, temperature, helium concentration and microstructure on void formation and helium embrittlement.
- 3) Perform preliminary evaluations of modified and new alloys developed by other laboratories.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1971

Swelling of Type 316 stainless steel at 500°C has been measured as a function of proton-irradiation damage up to 50 displacements per atom.

Six capsules, containing Type 316 stainless steel and a variety of other alloys, were inserted into the EBR-II core in ORNL Subassembly X100.

III. PROGRESS DURING REPORT PERIOD

1. Void Formation in Type 316 Stainless Steel

Examination of Samples 56A, 56B, and 28 continued. In particular, great care was taken in obtaining electron micrographs of Sample 56B where the damage was 50 disp./atom. The previous value for swelling associated with that damage (as reported in the prior Quarterly Report) was in error because of the

Figure 18. Transmission Electron Micrograph of Voids in Sample 56B Where the Damage was 50 Disp./Atom

difficulty in imaging all voids present. Figure 18 shows one of the latest electron micrographs of Sample 56B from which swelling data are being obtained.

Although the final value for the swelling of Sample 56B at 50 disp./atom is not yet available, it is likely to be $\sim 20\%$. Table 10 presents the void data obtained to date for Samples 56A and 56B, and for Sample 28 which received a low proton fluence.

The swelling associated with 0.5 disp./atom in Sample 28 is less than expected from an extrapolation of previous results. This sample was subjected to the least damage of all the samples (6×10^{17} p/cm²), and we are observing a very early stage in the void formation process. This is particularly evident from the variations in void density that were found from area to area.

Swelling as a function of damage is given in Figure 19. A straight line is a good fit to the data points on a fully logarithmic plot; there is no deviation from linearity at high degrees of damage. The empirical equation relating swelling to the damage (disp./atom) is as follows:

Figure 19. Swelling at 500°C as a Function of Damage for Proton-Irradiated Type 316 Stainless Steel Containing Helium

Figure 20. Void Diameter as a Function of Damage at 500°C for Proton-Irradiated Type 316 Stainless Steel Containing Helium

TABLE 10
VOID FORMATION IN TYPE 316 STAINLESS STEEL
PROTON IRRADIATED AT 500° C*

Sample No.	Damage (disp. /atom)	Void Size (Å)	Void Density (/cm ³)	Volume Increase (%)
28	0.5	50	2×10^{15}	0.02
56A	4.9	230	4×10^{15}	3.4
56A	16	260	7×10^{15}	6.0
56B	17	140	2×10^{15}	0.3 [†]
56B	38	360	4×10^{15}	13
56B	50	-	-	20 [§]

*Samples contained 2 to 9 appm He

†Anomalously low value, unexplained

§Revised preliminary value

$$\Delta V/V = 0.22 N^{1.13}$$

where $\Delta V/V$ is the percent volume change, and N is the number of displacements per atom.

The exponent on N is in agreement with results of company-sponsored theoretical work of H. Wiedersich. According to that work, an exponent of about unity is expected when void nucleation occurs primarily early in the irradiation. It is assumed that (1) the probability that defects are annihilated at voids is much less than for annihilation at dislocations, and (2) the dislocations are mainly loops which have nucleated and grown by the agglomeration of interstitials during irradiation.

Further evidence for early void nucleation is shown in Figure 20, where void diameters are plotted as a function of disp./atom. The minimum, average and maximum void diameters increase with damage indicating the growth of voids continues during irradiation, but not nucleation.

The fifth series of proton irradiations was performed, and Table 11 lists the samples and the irradiation conditions. Sample 59 received a fluence high enough to produce 100 disp./atom. Samples of Type 316 + Ti were received from ORNL and helium injected just in time to be proton irradiated.

TABLE 11
SAMPLES OF TYPE 316 STAINLESS STEEL IN THE FIFTH SERIES
OF PROTON IRRADIATIONS

Irradia- tion Number	Sample Number	Treatment	He Concentra- tion (appm)	Tempera- ture (°C)	Fluence (p/cm ² x 10 ¹⁹)
5-1	65	6	0.11	500	1.2
5-2	29	6	28	500	1.2
5-3	79	6	5 (nominal)	400	1.2
5-4	69	6	5 (nominal)	600	1.2
5-5	59	6	5.8	500	13
5-6	99T	a	5 (nominal)	600	1.2
5-7	102-T-20	b	5 (nominal)	600	1.2

Treatment 6: 1 hr 982°C + 8 hr 760°C
a: 1 hr 1050°C
b: 1 hr 1050°C + 20% CR

TABLE 12
HELIUM INJECTED SAMPLES

Alloy	No. of Samples	Nominal He Concentration (appm)
316	15	5
316	5	0.5
316	5	15
316 + Ti	10	5

A series of helium injections was performed on samples to be proton irradiated in the future. These are shown in Table 12.

2. Creep Tests of Type 316 Stainless Steel

Creep testing of samples with and without helium was resumed. A computer program was developed that reduces the data and plots the creep curves. Several interrupted tests have been performed, and they are listed in Table 13, along with all creep tests that have been done to date.

TABLE 13
SUMMARY OF CREEP TESTS AT 700°C AT A STRESS
OF 10.0 kg/mm²

Sample No.	Helium (appm)	Time (hr)	Creep Strain (%)				Secondary Strain Rate (/hr)
			Primary	Secondary	Tertiary	Total	
C-5	0	645	3.0	11	38	52	4.4×10^{-4}
C-6	0	561	3.5	11	17	32	4.2×10^{-4}
C-19	0	120 [†]	3.0	3.3	None	6.6	-
C-20	0	120 [†]	3.0	3.4	None	6.8	-
(19-8)	30 [*]	148	§	§	§	10	§
(19-9)	30 [*]	218	§	§	§	14	§
(19-16)	30 [*]	70 [†]	-	§	§	5.0	§
(19-17)	30 [*]	70 [†]	-	§	§	4.7	§
(19-20)	30 [*]	120 [†]	1.0	§	§	7.4	-
(19-21)	30 [*]	120 [†]	2.0	§	§	6.7	-

*Nominal value pending analysis

†Creep test was stopped at indicated time

§Data not available yet

Metallographic examination of Sample 19-16 revealed small voids in the grain boundaries. Helium bubbles were observed in the matrix and in grain boundaries where the largest were ~ 400 Å in diameter. More grain-boundary voids were seen in Sample 19-20, although the distribution and size of the helium bubbles was about the same. No dislocation-cell structure was present in either sample.

3. Stainless Steels From Other Laboratories

Type 316 stainless steel, from PNL and ORNL, was fabricated into 0.008-in. thick strips. These strips will be used for punching out small tensile samples. Some Type 316 + 0.2 Ti was received from ORNL which is also ready for punching. Additional fabrication is in progress to produce 0.0005-in. foils for proton irradiation.

4. EBR-II Irradiations

Subassembly X100, containing six capsules from AI, was placed in the EBR-II core during the mid-cycle shutdown of Run 46.

IV. EVALUATION OF EFFORT TO DATE

Proton irradiations of Type 316 stainless steel have continued to produce valuable results. Swelling increases about linearly with irradiation damage up to the highest amount of swelling observed to date, $\sim 20\%$ volume increase. It is difficult to determine such a large volume increase from electron micrographs and we may reach the limit of this technique in samples with greater amounts of damage.

The creep machines are now functioning and data are being reduced and plotted by computer. This effort will yield significant information on the effect of helium on creep during the next quarter.

V. NEXT REPORT PERIOD ACTIVITIES

- 1) A sample of Type 316 stainless steel that was irradiated to produce 100 displacements per atom will be examined.
- 2) Samples of Type 316 stainless steel that were proton irradiated at other temperatures will be examined.
- 3) Interrupted Creep tests on Type 316 stainless steel containing helium will continue at 700°C , as will metallographic examination.

Program: Fuel Cladding and Structural Materials		
AEC Task: 2-B, Integral (n, α) Cross Section Measurements		
Project Manager: G. W. Meyers		
Reporting Period: October-December 1970		
General Order: 6506	Subaccount: 29623	AEC Category: 04-40-02-01.1

Principal Investigator: H. Farrar, IV

I. PROJECT OBJECTIVES

The objectives of this task are to: (1) determine which elements in commonly used alloys are contributing to the high production of helium in fast neutron irradiations so that these might either be eliminated or their use minimized, (2) determine the spectrum integrated (n, α) cross sections of 47 elements in various fast reactor neutron spectra, (3) make estimates of the differential (n, α) cross sections of the same elements by correlating the integral cross sections in the different neutron energy spectra by using unfolding techniques, and (4) use this information to predict with some confidence what helium production might be expected from any present or new alloy contemplated for fast reactor use.

II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1971

Approximately 150 specimens of several elements and compounds irradiated at five locations in EBR-II during Run 31F have been prepared for mass spectrometric helium analysis and 32 additional determinations have been made. Some preliminary experiments have been conducted, using an available ruby laser, to vaporize high-melting-point-temperature specimens. However, it appears that much more energy is required for typical samples than this laser can deliver, and so preparations are being made to try a more powerful laser. Other techniques are also being studied.

Plans have been made to include eight capsules containing pure elements, alloys, and compounds in the 1971 dosimetry test irradiation in EBR-II. In preparation for this, 41 well-characterized alloys and new batches of elements

and compounds with the highest purities available were ordered and received. All specimens have been prepared for loading into the capsules.

III. PROGRESS DURING REPORT PERIOD

Plans were made in cooperation with WADCO for including capsules containing pure elements and compounds in the 1971 EBR-II dosimetry test in eight separate locations, Row 4 (center, +15 in., +30 in.), Row 6 (center), Row 7 (center), and Row 8 (center-inside, center-outside, +7 in.). Fabrication of capsules and subcapsules to contain the helium generation specimens has been completed, and new supplies of elements and compounds with the highest purities available have been obtained. Recent unexpected findings in the (n, α) cross section of nickel, and a particular interest in the (n, α) cross sections of Cu and B have resulted in our obtaining and including separated isotopes of these elements.

Helium results from the earlier EBR-II dosimetry test experiment (Run 31F) have shown that impurities play an important role in helium production. Many of the impurity elements such as N, P, and S cannot be irradiated conveniently in pure elemental form, and so are being included either as compounds such as GaP, PbS, etc., or as impurities in well-characterized alloys. Thirteen alloys obtained from WADCO and 17 NBS standard reference materials, all with accurately known impurity concentrations, have been selected, along with specimens of several alloys of LMFBR interest (Type 304, Type 316, Type 316 + Ti, Type 321, IN-718, 12R72HV, 18-8 Ti, and T^{111}). It is anticipated that (n, α) reactions with Ni, Fe, and B will produce most of the helium in many of these specimens, but precise measurements of the helium concentrations will be correlated with the known impurities to give values (or at least upper limits) of several important integrated (n, α) cross sections. The data will also provide generally useful helium generation rates for all these alloys in the eight different fast neutron spectra.

IV. EVALUATION OF EFFORT TO DATE

The helium measurements in irradiated Al, Si, Cr, Fe, Ni, and Type 304 stainless steel for the five widely differing EBR-II locations have given rather

precise values of the integrated (n, α) cross sections of these elements. Comparison with the steel results, moreover, has shown that up to 70% of the helium generated in the stainless steel cannot be attributed to the major steel components, and must therefore have come from (n, α) reactions with trace constituents.

The next most important elements and compounds to be studied: B, Al_2O_3 , TiN, HfN, TaN, SiC, TiC, and ZrC all have melting points well above 2000°C and, therefore, require a higher temperature method of helium extraction. New methods are being tried, and it is hoped that one can be devised that displays a 100% helium recovery which might then replace the existing system for all specimens. Other important elements and compounds with lower melting points, such as Be, V, Mn, Co, MnF_2 , have been prepared for analysis with the existing method of helium extraction.

In the 1971 EBR-II dosimetry test, advantage is being taken of the results from Run 31F. Not only will the elements and compounds have much higher purity, but also some important separated isotopes are being included along with a large number of well-characterized alloys. The eight locations have been chosen from earlier results to give a maximum expected variation in neutron spectrum shape.

V. NEXT REPORT PERIOD ACTIVITIES

During the next report period, efforts will be concentrated on:

- 1) Completing the preparation and identifications of pure elements, separated isotopes, compounds and alloys, wrap them in gold foil and load them with monitor wires into 56 subcapsules and 8 capsules, and ship to WADCO.
- 2) Preparing a file on all known purity information on the substances included in this irradiation.

Program:	LMFBR Technology		
AEC Task:	3, Fast Reactor Fuels		
Project Manager:	G. W. Meyers		
Reporting Period:	October-December 1970		
General Order:	6515	Subaccount:	28210
		AEC Category:	04-40-02-01.1

Principal Investigator: G. Ervin, Jr.

I. PROJECT OBJECTIVES

The objectives will be to:

- 1) Determine selected microstructure attributes and mechanical and physical properties of mixed carbide fuels, both to establish the potential for these materials as nuclear fuels and to provide a basis for interpreting and understanding irradiation behavior in terms of atomistic transport phenomena.
- 2) Develop, where necessary, preparative techniques for polycrystalline mixed carbide materials of controlled structure and stoichiometry.

II. PROGRESS DURING REPORT PERIOD

Work on closing out the task has included equipment disposal and writing reports. A list of equipment to be declared excess was prepared, and these items were disconnected and removed from the laboratory. The remaining items are needed on other programs, and appropriate documents were submitted to justify their transfer to these programs.

A manuscript, "Uranium and Plutonium Carbides With Tungsten Additive," was completed and sent to the Journal of the American Ceramic Society. It contains the material preparation portion of the work on the NRX-9-101 experiment which was presented at the Meeting of the Nuclear Division of the American Ceramic Society a year ago in Seattle. A paper describing the irradiation experiment itself was accepted for publication in the Journal and is now scheduled to appear in the January issue.

A topical report, AI-AEC-12983, "Tungsten-Modified Carbide," has been printed. The report describes the development of a method of synthesis of (U, Pu)C containing tungsten in solid solution. The successful method was the carbothermic reduction of a mixture of the oxides, UO_2 , PuO_2 , and WO_3 , followed by high temperature annealing.

Another topical report, AI-AEC-12984, "Helium Injection as a Technique for Studying Rare Gas Behavior in Carbide Fuel," has been edited and is being printed. The report describes experiments with particle-containing UC-W specimens which indicated attachment of helium bubbles to particles as well as substantial bubble migration during high temperature annealing.

III. EVALUATION OF EFFORT TO DATE

A method of preparing tungsten-modified mixed carbide (i.e., carbothermic reduction of a mixture of UO_2 , PuO_2 , and WO_3) was developed that appears promising for the eventual preparation of irradiation test samples. Further intermediate temperature annealing studies are desirable to find conditions for converting the solid solutions to structures containing a high density of small particles; and more analyses are needed to verify composition.

The helium injection work has made some progress in demonstrating qualitatively that helium bubbles migrate appreciable distances (mils) in a thermal gradient in the absence of an irradiation environment. The work has been slowed by problems arising in developing dependable electron microscope procedures for observing bubbles; but the basic techniques for transmission micrographs exist.

Both phases of the project have reached only an interim stage at which the path toward meaningful results has been delineated, but further work is needed to follow that path and produce the results.

IV. NEXT REPORT PERIOD ACTIVITIES

The project is now terminated, and no further activities will be reported.