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Nuclear Materials & Propulsion Operation

**HIGH-TEMPERATURE MATERIALS PROGRAM
PROGRESS REPORT No.49, Part A**

July 28,1965

GENERAL ELECTRIC

ATOMIC PRODUCTS DIVISION

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PROGRESS REPORT No. 49, Part A**

July 28, 1965

United States Atomic Energy Commission Contract No. AT (40-1)-2847

**NUCLEAR MATERIALS and PROPULSION OPERATION
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CONVERSION TABLE

To Convert From	To	Multiply By
Atmospheres	Pounds/inch ²	14.7
Calories (mean)	Btu (mean)	0.00397
Calories/gram-°C	Btu/pound-°F	1.0
Calories/sec-cm-°C	Btu/hr-ft-°F	241.8
Calories/sec-cm ²	Btu/hr-ft ²	1.32 x 10 ⁴
Calories/sec-cm ² -°C	Btu/hr-ft ² -°F	7370
Centimeters	Feet	0.03281
	Inches	0.3937
Cubic centimeters	Cubic feet	3.531 x 10 ⁻⁵
	Cubic inches	0.06103
Grams	Pounds	0.002205
Grams/cm ³	Pounds/ft ³	62.43
Grams/cm ²	psi	0.01422
Kilograms	Pounds	2.205
Kilograms/cm ²	Atmospheres	0.9678
	Pounds/ft ²	2048
	Pounds/inch ²	14.22
Kilograms/mm ²	Pounds/inch ²	1422.32
Kilowatts	Btu/sec	0.948
Liters	Cubic feet	0.0353
Meters	Inches	39.37
Millimeters of mercury	Atmospheres	0.001316
Square centimeters	Square feet	0.001076
	Square inches	0.155
Torr	mm of Hg	1.0
	Atmospheres	0.001316
Watts/cm-°C	Btu/hr-ft-°F	57.8
Watt-seconds	Btu	0.000948
Watts/cm ² -°C	Btu/hr-ft ²	3170
Watts/cm ² -°C	Btu/hr-ft ² -°F	1760
Centimeters/sec	Feet/sec	0.03281
Meters/sec	Feet/sec	3.281

1. INTRODUCTION AND SUMMARY

Introduction

This report is the unclassified portion of the forty-ninth in a series of monthly reports of the work in process on materials development for the Atomic Energy Commission under Contract AT(40-1)-2847.

Included is a summary of the work from April 15, 1965 to June 15, 1965, on four of the fourteen specific development programs in process. Two of the remaining programs are reported in the classified portion of this report, GEMP-49, Part B. The other eight programs, involving the development of ceramic fuel materials, are reported in alternate months.

Summary

OXIDATION-RESISTANT FUEL ELEMENT MATERIALS RESEARCH (57001)

Laboratory testing of Fe-Cr-Al-Y-clad fueled specimens has exceeded 7000 hours at 950°C and 1100°C in air with continuing indication of excellent stability. A new series of tests at 750°C was started and a series of enriched samples was in preparation which will be tested at 750°, 950°, 1100°, and 1250°C.

The first in-pile test of a 2541-clad Fe - 40UO₂ specimen completed 1007 hours at 1100°C in air. Steady-state fission product release during this time did not exceed 10⁻⁷.

Weight gain data on a series of eight alloys were obtained at 900°C and 1100°C for times up to 8000 hours in air. The 2541 and 1541 alloys continued to show lower oxidation rates than any commercial alloy.

Pre-oxidation at 980°C of Fe-Cr-Al-Y alloys of low chromium content continued to show promise of enhancing corrosion resistance in steam. Up to 4 percent strain in pre-oxidized specimens can be tolerated without reducing the steam corrosion resistance of these alloys.

HIGH-TEMPERATURE REACTOR MATERIALS RESEARCH (57003)

Additional creep-rupture tests of arc-cast tungsten were performed to provide stress-rupture and creep curves from 1600° to 2800°C for time periods ranging from approximately 5 to 300 hours. Tests at 2000°C and 2200°C with special pre-annealed specimens indicate that a change in the creep mechanism for tungsten occurs between 2000°C and 2200°C.

First stage creep analyses of the arc-cast tungsten data from 1600° to 2800°C led to the general equation

$$\epsilon = \bar{d} \bar{g} \bar{c} T \sigma^{\bar{c}} t^m + \epsilon_0$$

where $\bar{d}$, $\bar{g}$, $\bar{c}$, m and ϵ_0 are constants.

Creep-rupture tests of arc-cast W - 25Re at 2000°C revealed that this material has a greater rupture life and is more creep resistant than arc-cast tungsten for stresses greater than 1.0 kg/mm². The creep curves for the arc-cast W - 25Re at 2000°C exhibited no primary or secondary creep.

W-Re-Mo alloys ranging from W - 26Re to W - 29Re, with 0, 10, and 20 percent molybdenum, creep-rupture tested at 1600°C and 4.22 kg/mm², exhibited a grain refinement as the rhenium content was increased and an increase in grain size as the molybdenum was increased. The variations in the creep-rupture properties for the alloys are not related to the structure. Creep-rupture data for the W-Re-Mo alloys tested at 2200°C and 0.56 kg/mm² reveal that W - 27Re - 20Mo has the greatest rupture life and is the most creep resistant.

Additional creep data for arc-cast molybdenum were obtained with the electro-optical extensometer in tests from 1600° to 2200°C. First-stage creep analyses gave results similar to those obtained for arc-cast tungsten.

Metallurgical evaluations revealed that W - 25Re - 30Mo alloy is resistant to excess grain growth whether thermal-cycled between 260°C and 2150°C, or held isothermally at 2200°C. Both W - 25Re - 30Mo and W - 30Re - 30Mo alloys withstood 90-degree room-temperature bend tests after the above treatments.

Because of widespread interest in methods for improving molybdenum and in alloys containing high concentrations of molybdenum, the purification technology developed for W-Re alloys was applied to unalloyed molybdenum and to molybdenum alloys with additions of 1, 3, or 5 atomic percent rhenium. These alloys resisted room-temperature embrittlement when heated to 2200°C. Weldability was better than with the commercial powder metallurgy (sintered) molybdenum sheet, but slightly poorer than with the arc-cast product.

Recently, emphasis was placed on trial production runs of W - 25Re - 30Mo (NMP-256) as plates, sheet, and small-seamless tubing. Sheets larger than any heretofore produced were obtained without difficulty, confirming the soundness of sheet-rolling procedures. Procedures for producing seamless tubing by extruding, hot-working, and predraw cleanup were tentatively identified using carbide "plug" mandrels and sacrificial core fillers.

EFFECT OF RADIATION ON HIGH-TEMPERATURE METALS AND ALLOYS (57004)

Creep-rupture testing of irradiated W - 25Re specimens at 800°C and 900°C indicates that a significant change in flow properties may be occurring at some temperature between these two temperature levels. The mechanism appears to be an interaction of dislocations with the irradiation-induced defects which is much stronger at the 800°C test temperature than at the 900°C level rather than the possibility of the annealing out of those defects at the higher temperature.

Measurement of the recovery of irradiation-induced resistivity yielded good results for specimens tested at elevated temperature in isothermal anneals.

Hot-hardness measurements of worked and of neutron-irradiated molybdenum specimens show very similar recovery behavior. In particular, the apparent small increase in the hardness value at temperatures of about 150°C ($0.15 T_m$) are observed to occur in both types of specimens.

Creep-rupture testing of irradiated Hastelloy N specimens at 815°C shows about a factor of 3.5 reduction in the time to rupture when tested at relatively high stresses. There is a tendency for some recovery to occur when testing at the lower stresses, resulting in about a factor of 1.6 reduction compared to the control data. The times to rupture approach those of the control specimens in a manner observed for irradiated Hastelloy X specimens tested at a slightly higher temperature (870°C).

HIGH-TEMPERATURE THERMOCOUPLE AND ELECTRICAL MATERIALS RESEARCH (57014)

The thermoelectric properties as a function of temperature for Cr, Nb - 10Mo, Nb - 20Mo, and Nb - 40Mo were measured to 1800°C. The alloys of Nb - 20Mo and Nb - 40Mo showed good potential as negative thermoelement materials. BeO, MgO, and HfO₂ were evaluated for use as thermocouple electrical insulators in systems for measuring temperatures up to 2450°C in graphite and hydrogen environments. Molybdenum, rhenium, and Mo - 50Re were evaluated for use as high-temperature thermocouple sheath materials in a graphite - hydrogen environment. These tests showed rhenium to be the best potential sheathing material. Of the three insulator materials tested, HfO₂ exhibited the best stability.

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2. OXIDATION-RESISTANT FUEL ELEMENT MATERIALS RESEARCH

(57001)

The objective of this program is to develop fuel element materials for use at high temperatures in air, steam, or oxidizing coolants which are superior to presently available fuel materials in terms of high-temperature capabilities, long-time service life, and containment of fission products.

FUEL ELEMENT MATERIALS EVALUATIONS

The Fe-Cr-Al-Y alloys have proven to be more resistant to high-temperature oxidation and steam corrosion than any known commercial alloys that may be suitable fuel element cladding. The current efforts in this program are to evaluate the compatibility of the Fe-Cr-Al-Y alloys with various forms of UO_2 by testing specimens of these fuel element materials at high temperatures for times up to 10,000 hours. The capability of the Fe-Cr-Al-Y alloys to retain fission products is being determined in a series of in-pile tests conducted in the LITR at temperatures of 950° to 1100°C in flowing air.

Fueled Capsule Test Program

A total of 60 fuel element specimens was on test at the end of this report period. Most of these contained a dispersion-type fueled core of Fe - 40UO_2 or Cr - 50UO_2 ¹ encapsulated in either the 2541 or the 1541 alloy.² The specimens are cylindrical capsules 1.05 cm in diameter by 2.54 cm in length with a cladding thickness of 0.5 mm. The cladding is bonded to the core matrix material by hot-gas pressure autoclaving at 1250°C under 700 kg/cm^2 helium pressure. After a preliminary proof-test, the specimens are subjected to long-time tests with goals of 10,000 hours at 750° , 950° , and 1100°C in air. The tests are interrupted periodically for visual inspection of the specimens and for X-ray fluorescence analysis of the cladding surface for possible uranium diffusion. Sufficient specimens of one type are started to permit removal of one specimen after 1000, 3000, 5000, and 7500 hours for destructive analysis of the materials to determine their stability at high temperatures.

The status of this long-time test program is summarized in Table 2.1. Test times in excess of 7000 hours at 950°C and 1100°C have shown little effect on the 2541-clad Fe- UO_2 specimens containing agglomerated UO_2 particles.

It was reported previously³ that studies of various fuel forms were being conducted to achieve a better dispersion structure and a more stable form of UO_2 . One of these fuel forms was plasma-arc spheroidized UO_2 prepared at GE-NMPO from agglomerated fuel particles. Six specimens containing this fuel form (Nos. FSU-10, -11, -12, -15, -16, and -17, Table 2.1) were tested at 1100°C for 2500 to 2900 hours and showed relative uranium

¹Cladding compositions are given in weight percent and core compositions in volume percent.

²2541 alloy composition is Fe - 25Cr - 4Al - 1Y and 1541 alloy composition is Fe - 15Cr - 4Al - 1Y.

³"High-Temperature Materials Program Progress Report No. 43, Part A," GE-NMPO, GEMP-43A, January 29, 1965, p. 14.

TABLE 2.1

STATUS OF Fe-Cr-Al-Y-CLAD FUELED SPECIMENS UNDERGOING THERMAL STABILITY TESTS IN AIR

Cladding	Core Composition, vol %	Type Fuel ^a	Specimen Number	Test Temperature, °C	Current Test Time, hr	Uranium Diffusion To Surface, ^b %	Status ^c
2541	Fe - 50UO ₂	KM-A	FAU-17	950	6885	0.04	
			FAU-36	950	6885	<0.02	
			FAU-38	950	6885	<0.02	
			FAU-16	1100	7090	0.04	
			FAU-37	1100	5985	0.11	
1541	Fe - 50UO ₂	KM-A	FAU-18	1100	4996	<0.02	Completed
2541	Fe - 40UO ₂	AR	FU-29	950	6885	<0.02	
			FU-30	1100	5984	<0.02	
			FU-32	1100	5098	<0.02	Completed
2541	Fe - 40UO ₂	GE-A	FAU-52	750	195	-	
			FAU-53	750	195	-	
			FAU-54	750	195	-	
			FAU-55	950	195	-	
			FAU-56	950	195	-	
			FAU-57	950	195	-	
			FAU-58	1100	195	-	
			FAU-60	1100	195	-	
			FAU-45	1100	2510	<0.02	
			FAU-46	1100	2950	<0.02	Completed
2541	Fe - 40UO ₂	KM-A	FAU-19	950	2500	<0.02	Completed
			FAU-21	950	2845	d	Blistered
			FAU-27	950	4035	d	
			FAU-28	950	4035	d	
			FAU-22	1100	5755	d	
			FAU-25	1100	3001	<0.02	Completed
			FAU-29	1100	2950	0.12	
			FAU-49	1100	2510	<0.02	Completed
2541	Fe - 40UO ₂	S-KM	FSU-25	750	195	-	
			FSU-26	750	195	-	
			FSU-27	750	195	-	
			FSU-28	750	195	-	
			FSU-29	750	195	-	
			FSU-9	950	2510	<0.02	
			FSU-13	950	2940	d	
			FSU-22	950	195	-	
			FSU-23	950	195	-	
			FSU-24	950	195	-	
			FSU-10	1100	2510	0.60	
			FSU-11	1100	2890	0.66	
			FSU-12	1100	2890	1.45	Completed
1541	Fe - 40UO ₂	S-KM	FSU-19	950	2940	d	
			FSU-20	950	2940	d	
			FSU-21	950	2510	<0.02	
			FSU-15	1100	2510	3.10	
			FSU-16	1100	2890	3.80	
			FSU-17	1100	2890	0.74	
2541	Fe - 25UO ₂	AR	FU-52	1100	4996	<0.02	Completed
2541	Cr - 50UO ₂	AR	CU-26	950	7785	0.03	
			CU-25	1100	4996	<0.02	Completed
2541	Cr - 50UO ₂	KM-A	CAU-27	1100	4915	0.73	
			CAU-28	1100	5985	0.26	

TABLE 2.1 (Cont.)

STATUS OF Fe-Cr-Al-Y-CLAD FUELED SPECIMENS UNDERGOING THERMAL STABILITY TESTS IN AIR

Cladding	Core Composition, vol %	Type Fuel ^a	Specimen Number	Test Temperature, °C	Current Test Time, hr	Uranium Diffusion To Surface, ^b %	Status ^c
1541	Cr-50(UO ₂ -11ThO ₂)	GE-A	CUT-23	950	7005	< 0.02	Completed
			CUT-24	1100	4915	0.19	
2541	(Fe-25Cr)-50UO ₂	KM-A	FCAU-4	950	7785	< 0.02	
			FCAU-3	1100	6980	0.05	
2541	(Fe-25Cr)-40UO ₂	GE-A	FCAU-6	750	195	-	
			FCAU-7	750	195	-	
			FCAU-8	750	195	-	
			FCAU-9	950	195	-	
			FCAU-10	950	195	-	
			FCAU-12	950	195	-	
			FCAU-13	1100	195	-	
2541	(Fe-25Cr)-40UO ₂	S-KM	FCSU-1	750	195	-	
			FCSU-4	950	195	-	
			FCSU-5	950	195	-	
			FCSU-6	950	195	-	
			FCSU-7	1100	195	-	
			FCSU-8	1100	195	-	
			FCSU-14	1100	195	-	
1541	Fe - 40UN	AR	FUN-8	950	6885	< 0.02	Completed
2541	Cr - 40UN	AR	CUN-5	950	8425	< 0.02	
1541	Cr - 50UN	AR	CUN-7	1100	4996	7.30	
2541	(Fe-25Cr)-40UN	AR	FCUN-1	950	8425	< 0.02	
			FCUN-3	1100	4915	4.30	
1541	(Fe-25Cr)-40UN	AR	FCUN-5	950	6885	< 0.02	

^aAR - Ceramic grade UO₂ or UN of -325 mesh particle size.

GE-A - UO₂ agglomerated at GE-NMPO, -100/+200 mesh.

KM-A - Commercial agglomerated UO₂, -100/+200 mesh.

S-NM - Commercial spheroidized UO₂.

S-KM - UO₂ plasma spheroidized at GE-NMPO, -140/+200 mesh.

^bAnalysis of cladding surface for uranium by X-ray fluorescence; presence of uranium in amounts less than 0.02 percent cannot be detected.

^cUnless otherwise noted, specimens are continuing on test satisfactorily.

^dSurface analyses not completed.

intensities as high as 3.6 percent which is considerably higher than values measured for similar specimens containing other fuel forms. Specimens containing the spheroidized UO₂ tested at 950°C did not show uranium diffusion through the cladding.⁴ The plasma-arc spheroidized UO₂ was of stoichiometric composition and did not contain free uranium initially. Additional specimens of 2541-clad Fe - 40UO₂ and Fe - 25Cr - 40UO₂ containing the spheroidized fuel were placed on test at 750°, 950°, and 1100°C; they had accumulated 195 hours at the end of the report period.

Five fuel element specimens were removed from the test program for destructive analysis after 2500 to 5000 hours of test time at 950°C or 1100°C. The compositions, test conditions, and results of metallographic examination of these specimens are shown in Table 2.2. None of these specimens had more than 15 microns of cladding surface oxide and all had some oxide at the core - cladding interface.

⁴Presence of uranium in amounts less than 0.02 percent cannot be detected by X-ray fluorescence technique.

TABLE 2.2

SUMMARY OF DATA ON FUEL ELEMENT SPECIMENS ANALYZED METALLOGRAPHICALLY

Specimen Number	Core Composition, vol %	Type Fuel ^a	Cladding	Test Temperature, °C	Exposure, hr	Surface Oxide, microns	Core - Cladding Interface Oxide	
							Nature of Oxide ^b	Thickness, microns
FAU-19	Fe - 40UO ₂	KM-A	2541	950	2500	6	VI	1.0
FAU-49	Fe - 40UO ₂	KM-A	2541 ^c	1100	2500	10	I	5.8
FAU-25	Fe - 40UO ₂	KM-A	2541	1100	3000	8	SC	3.0
FAU-45	Fe - 40UO ₂	GE-A	2541	1100	2500	9	I	3.5
FAU-18	Fe - 50UO ₂	KM-A	1541	1100	5000	15	C	9.0

^aKM-A - commercial agglomerated UO₂, -100/+200 mesh.

GE-A - UO₂ agglomerated at GE-NMPO, -100/+200 mesh.

^bI - Intermittent, VI - Very Intermittent, C - Continuous, SC - Semi-Continuous.

^cCapsule cladding was commercially drawn tubing; all other specimens had cladding machined from swaged rod.

Photomicrographs of three of the specimens tested for 2500 hours are shown for comparison in Figure 2.1. Two of these specimens, FAU 10 and FAU 40, contained commercially agglomerated UO₂ and the other, FAU-45, contained UO₂ agglomerated at GE-NMPO. The cladding on specimens FAU-19 and FAU-45 was tubing machined from swaged rod, while the cladding on specimen FAU-49 was commercially drawn tubing of the same 2541 alloy. The specimen tested at 950°C showed a very intermittent oxide formation at the core - cladding interface with a small amount of grain boundary oxidation and YFe₉ phase in the interface area. The porosity of the fuel particles is characteristic of commercially agglomerated UO₂.

The two specimens tested at 1100°C had intermittent oxide barriers and heavier intergranular oxidation than the specimen tested at 950°C. An Al₂O₃ layer partially surrounds many of the UO₂ particles in the specimens tested at 1100°C. The cladding of commercially drawn tubing on specimen FAU-49 had more grain boundary oxidation at the interface since the heavily worked alloy initially had a finer grain size than the rod material used for cladding on specimen FAU-45. No other significant difference was observed between the two types of cladding nor was there any noticeable difference in the stability of the two fuel forms.

Metallographic examination of specimen FAU-25 after 3000 hours at 1100°C revealed a structure similar to the specimens tested for 2500 hours except the oxide barrier at the interface was semi-continuous. The specimen tested for 5000 hours (FAU-18) had a continuous Al₂O₃ interface barrier and more pronounced intergranular oxidation of the cladding than the specimens shown in Figure 2.1. However, this specimen was clad with the 1541 alloy which may have somewhat less oxidation resistance at 1100°C than the 2541 alloy.

A series of 16 specimens was prepared containing enriched UO₂ either alone or dispersed in an iron matrix. These were clad with drawn tubing of 1541 alloy and are being tested at temperatures of 750° to 1250°C in air. Periodic monitoring for uranium diffusion will be performed using both alpha counting techniques and X-ray fluorescence analyses. From these data, a correlation will be made which will aid in interpreting the data obtained in the past by X-ray fluorescence analysis of depleted UO₂ specimens.

In-Pile Testing

A series of in-pile tests of the Fe-Cr-Al-Y-clad fuel elements is scheduled to demonstrate fission product retention capability as a function of temperature and burnup capa-

Specimen FAU-19 tested at 950°C (Neg. 5949)

Specimen FAU-49 tested at 1100°C (Neg. 5947)

Specimen FAU-45 tested at 1100°C (Neg. 5946)

Fig. 2.1—Photomicrographs of 2541-alloy-clad $\text{Fe}-40\text{UO}_2$ specimens showing condition of interface areas after 2500 hours at 950°C or 1100°C in air (250X)

bilities at various temperatures. These irradiation tests will be conducted at the Oak Ridge National Laboratory using both the LITR and the ORR.

LTFC-1 - The first in-pile test of a 2541-clad Fe - 40UO₂ specimen (test LTFC-1), described in the previous report,⁵ completed its test schedule in the C-48 facility of the LITR. It was irradiated for a total of 1069 hours, 1007 hours at maximum indicated temperature of approximately 1100°C and the initial 62 hours at a maximum of 575°C, in a flowing air atmosphere (680 liters per minute). The specimen was subjected to 12 thermal cycles caused by reactor shutdowns prior to termination of the experiment. The burnup is calculated to be about 0.7 percent as a result of 4.8×10^{19} fissions per cubic centimeter of fueled core. The heat flux generated was about 0.5 kilowatts per cubic centimeter. Disassembly of the cartridge at GE-NMPO is underway.

Thirteen carbon traps were taken to sample the effluent gas stream for released gaseous fission products. The results of the trap analyses are shown in Table 2.3 along with test temperatures and times of reactor shutdown. The data for Xe¹³³ are less reliable than those for the other isotopes because of the low activity levels found in the traps. The fission gas release data for trap C-1, taken at the lower temperature, appears to be somewhat high suggesting that steady-state conditions had not yet been achieved. The level of steady-state release after this time appears to remain low ($R/B = 1$ to 5×10^{-7}) and essentially constant.

TABLE 2.3
FISSION PRODUCT RELEASE FROM TEST LTFC-1

Trap Number	Test Temperature, °C	Test Time, hr	Fractional Release (R/B) ^a				
			Kr ⁸⁷	Kr ⁸⁸	Kr ^{85m}	Xe ¹³⁵	Xe ¹³³
C-1	582	41.9	5×10^{-7}	6×10^{-7}	1×10^{-6}	9×10^{-7}	1×10^{-6}
C-2	1092	134.3	1×10^{-7}	1×10^{-7}	3×10^{-7}	3×10^{-7}	3×10^{-7}
C-3	1092	157.7	1×10^{-7}	2×10^{-7}	3×10^{-7}	3×10^{-7}	4×10^{-7}
Shutdown		158.7					
C-4	1092	181.8	7×10^{-8}	1×10^{-7}	3×10^{-7}	2×10^{-7}	3×10^{-7}
C-5	1092	202.6	2×10^{-7}	2×10^{-7}	4×10^{-7}	2×10^{-7}	4×10^{-7}
Shutdown		290.1					
C-6	1092	299.2	6×10^{-8}	8×10^{-8}	2×10^{-7}	1×10^{-7}	2×10^{-6}
Shutdown		319.2					
C-7	1100	345.5	1×10^{-7}	2×10^{-7}	2×10^{-7}	1×10^{-7}	1×10^{-6}
Shutdown		345.9					
Shutdown		346.5					
C-8	1115	389.8	7×10^{-8}	7×10^{-8}	2×10^{-7}	2×10^{-7}	6×10^{-7}
Shutdown		481.4					
Shutdown		484.7					
C-9	1120	519.5	9×10^{-8}	1×10^{-7}	3×10^{-7}	3×10^{-7}	5×10^{-7}
Shutdown		616.4					
C-10	1095	652.6	9×10^{-8}	1×10^{-7}	3×10^{-7}	3×10^{-7}	3×10^{-7}
Shutdown		662.9					
Shutdown		744.7					
C-11	1097	788.2	1×10^{-7}	2×10^{-7}	3×10^{-7}	4×10^{-7}	4×10^{-7}
Shutdown		811.9					
Shutdown		882.8					
C-12	1100	953.9	1×10^{-7}	2×10^{-7}	2×10^{-7}	3×10^{-7}	3×10^{-7}
C-13	1095	1050.5	1×10^{-7}	2×10^{-7}	3×10^{-7}	4×10^{-7}	9×10^{-7}
Shutdown		1069.4					
End of Test							

^aFractional release is defined as the ratio of the rate of release of atoms from the specimen to the equilibrium production rate.

⁵"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, pp. 16-18.

Traps C-6 and C-7 were taken during or shortly after thermal cycles and indicate increased release levels due to transient conditions. Examination of the steady-state release with respect to half-lives of the various isotopes suggests that release probably resulted from a combination of diffusion and recoil from fuel near the surface.

LTFC-2 - The next in-pile test of a 1541-alloy-clad Fe - 40UO₂ specimen will be irradiated for 1000 hours at 950°C in flowing air. This test is scheduled to be inserted in the LITR in August. All cartridge components have been fabricated, the test specimen has been proof-tested, and assembly will be completed early in the next report period.

Laboratory Testing - Two specimens of the in-pile test design⁶ containing the same amount of enriched UO₂ as the specimen in in-pile test LTFC-1 were tested in the laboratory at 950°C and 1100°C in air. These specimens were analyzed periodically for surface alpha activity using a proportional counter. Each specimen was counted for 10 minutes, rotated 180 degrees, and recounted. Based on a surface area over the fueled core of 16.7 cm², the average alpha count per unit area per minute was calculated. The data accumulated for 1700 hours of test time are shown in Figure 2.2. The somewhat erratic behavior of the alpha count data of the 2541-clad Fe-UO₂ specimen tested at 1100°C suggests either a non-uniform rate of diffusion or variations in the surface oxide. There is no correlation between this alpha count data and the fission product release data for LTFC-1 shown in Table 2.3. Metallographic examination of this specimen in comparison with the irradiated in-pile specimen may provide some explanation for this high alpha count data.

Fig. 2.2 - Alpha surface activity as a function of test time at 950°C or 1100°C for Fe-Cr-Al-Y-clad Fe - 40UO₂ enriched specimens of in-pile test design

The second specimen, clad with 1541 alloy and tested at 950°C, showed very little surface activity up to 1100 hours when a sharp increase occurred. No leak in the cladding was detected and no visible cause was found. The specimen will be destructively analyzed after 2000 hours at temperature and examination for possible breakdown of the Al₂O₃ barrier will be made.

ALLOY DEVELOPMENT

The objective of this portion of the task is to improve the structural properties of the Fe-Cr-Al-Y alloys without sacrificing oxidation or corrosion resistance.

⁶Ibid., p. 18.

Fabrication and Preparation

During this report period a 90-kg heat of 1541 alloy was melted and subsequently extruded to 2.85-cm and 3.18-cm bar stock.

Approximately 60 feet of commercially drawn tubing has been inspected by eddy current, ultrasonic, and black light techniques. Numerous longitudinal seams on the outside diameter of the tubing were noted, the origin of which has not been determined.

A group of 14 alloys was prepared as 80-gram arc-melted ingots. The alloys were designed for preliminary evaluation of the effects of aluminum and yttrium contents on the ductility and oxidation resistance of Fe-Cr-Al-Y alloys containing 13 to 15 percent chromium. The nominal compositions of these 14 alloys are listed in Table 2.4. As indicated previously, reduction of the yttrium content from 1 percent to about 0.2 percent in an Fe-25Cr-4Al alloy resulted in improved weldability, impact properties, and oxidation resistance at temperatures below 1100°C. The optimum yttrium content for the Fe-15Cr-4Al alloys will be determined. Also a reduction of the chromium content and an increase in aluminum content should result in improved stability with respect to freedom from α' embrittlement.

TABLE 2.4
COMPOSITIONS OF Fe-Cr-Al-Y ALLOYS
PREPARED FOR STUDY OF OPTIMUM
ALUMINUM AND YTTRIUM CONTENTS

Heat No.	Nominal Composition, wt %		
	Cr	Al	Y
F-1	15	4	0.1
F-2	15	4	0.2
F-3	15	4	0.5
F-4	15	4	1.0
F-5	15	5	0.1
F-6	15	5	0.5
F-7	15	6	0.2
F-8	15	7	0.1
F-9	15	7	0.5
F-10	15	4	0.0
F-11	15	6	0.0
F-12	13	4	0.5
F-13	13	5	0.5
F-14	13	6	0.5

Property Measurements

The effect of temperature on the dynamic elastic constants of the 2541 alloy was determined utilizing an ultrasonic technique. The results are shown in Figure 2.3. The elastic modulus decreases to a value of about 7×10^5 kg/cm² at 1000°C. Variation in Poisson's ratio are large, probably because of small errors in the measurement of the elastic (E) and shear (G) moduli. Poisson's ratio was calculated from E and G using the relationship:

$$\mu = \frac{E}{2G} - 1$$

AIR OXIDATION AND STEAM CORROSION STUDIES

Air Oxidation

Air oxidation tests of a series of Fe-Cr-Al-Y alloy compositions were continued at 450°, 550°, 730°, 900°, and 1100°C. Weight change data for times up to 8000 hours at

Fig. 2.3 – Dynamic elastic constants of 2541 alloy

900°C and 1100°C are shown in Figure 2.4. All the alloys containing 1 percent yttrium exhibit comparable weight gains, except for the 1041 alloy (Fe – 10Cr – 4Al – 1Y) which exhibited a sharp increase in weight after 4000 hours at 1100°C.

The alloys containing 0.35 and 0.5 nominal percent yttrium continue to exhibit low weight gains (heat Nos. 13 and 10, respectively, Figure 2.4). Metallographic examination of these alloys after 4000 hours at 900°C and 1100°C indicates that the lower weight gains are a result of less internal oxide formation. As the yttrium content is reduced, the grain size is increased and less intergranular oxidation of yttrium occurs. Intergranular oxidation of yttrium occurs primarily during initial testing. The oxidation of YFe_9 particles at the surface also occurs during initial testing. Both of these factors influence the initial oxidation kinetics; however, after extended oxidation times, the oxidation kinetics for alloys with both low- and high-yttrium contents should be equal with the rate controlling step being the diffusion of oxygen across the $\alpha - \text{Al}_2\text{O}_3$ surface oxide. This is demonstrated in Figure 2.5 where the square of the weight gain is plotted as a function of time. The data after long times approximate straight lines with equal slopes. This indicates that oxidation of alloys with both low- and high-yttrium contents is controlled by a single diffusion process after long times. Extrapolation of the data back to zero time provides a measure of the oxidation of surface YFe_9 particle, intergranular Y_2O_3 formation, and possibly other effects of yttrium.

The absence of intergranular oxides in the alloys with low-yttrium content is evident in Figures 2.6 and 2.7. A peculiar effect was noted for the 253 (0.5) alloy (MS-10) when tested at 1100°C. As shown in Figure 2.7, the yttrium-rich phase has been internally oxidized, indicating that the aluminum content has been depleted by surface Al_2O_3 formation. This may result in a matrix containing enough oxygen to oxidize the yttrium.

As pointed out in the "Alloy Development" section, 13 to 15 percent chromium alloys with low-yttrium contents will be evaluated. The results obtained for the 25 percent

Fig. 2.4—Weight gain as a function of test time at 900°C and 1100°C in static air for Fe-Cr-Al-Y alloys for times up to 8000 hours

Fig. 2.5—Square of the weight gain as a function of time for three alloys showing parabolic behavior during oxidation at 1100°C

chromium alloys indicate that 0.1 to 0.2 percent yttrium is sufficient to maintain excellent oxidation resistance for long times at temperatures to at least 1100°C.

Steam Corrosion Studies

Steam corrosion tests of Fe-Cr-Al-Y alloys are being conducted at 550°C and 730°C in low-pressure steam generated from de-ionized water. Several commercial and experimental alloys are being tested at 730°C and 3 atmospheres pressure. The current weight gain data are presented in Table 2.5. The Fe-Cr-Al-Y alloys continue to exhibit lower weight gains than the nickel-base alloys and Type 446 stainless steel.

Microstructures of the Inconel 800 alloy and 80Ni – 20Cr alloy after long-time steam corrosion testing at 730°C are shown in Figures 2.8 and 2.9. Both of these alloys show irregular oxide formation. Type 446 stainless steel showed a uniform heavy surface oxide with negligible penetration, whereas the 1541 and 2541 alloys showed thin surface oxides with occasional deeper penetrations.

TABLE 2.5
WEIGHT GAIN DATA FOR ALLOYS TESTED AT 730°C IN STEAM

Alloy	Heat No.	Surface ^a Preparation	No. Of Specimens	Time, hr	Weight Gain (Range), mg/dm ²	Mean Corrosion Rate, mg/dm ² – 1000 hr
2541 + 1Nb	M-233	VB	6	3500	72 – 116	27.7
1541	M-231	VB	2	5160	77 – 136	20.5
1541	M-231	VB	4	3900	59 – 119	20.8
1541	MS-38	VB	4	3900	81 – 97	22.3
2541	MS-36	VB	4	3900	83 – 106	24.1
1041	M-239	POx	3	2000	27 – 31	14.5
Inconel 600	-	BV	1	5160	126	24.4
Inconel 600	-	Pk	2	5160	268 – 677	91.5
Incoloy 800	-	Pk	1	5160	211	40.9
Incoloy 800	-	VB	1	5160	134	25.9
80Ni – 20Cr	-	VB	4	2550	104 – 130	46.3
446 SS	-	VB	6	4400	81 – 175	27.0
Incoloy 625	-	VB	2	1000	117 – 118	118.0

^aVB – Vapor-blast, POx – 980°C treatment in air, Pk – Acid-pickled.

Fig. 2.6 – Photomicrograph of 254(0.35) alloy (MS-13) oxidized for 4000 hours at 900°C showing only slight intergranular formation of Y_2O_3 (Neg. 5905, as-polished, 500X)

Fig. 2.7 – Photomicrograph of 253(0.5) alloy (MS-10) oxidized for 4000 hours at 1100°C showing adherent oxide and oxidation of YFe_9 phase (Neg. 5906, as-polished, 250X)

Fig. 2.8 – Photomicrograph of Inconel 800 alloy after steam corrosion for 5160 hours at 730°C (Neg. 5843, as-polished, 250X)

Fig. 2.9 – Photomicrograph of 80Ni – 20Cr alloy after steam corrosion for 2550 hours at 730°C (Neg. 5844, as-polished, 250X)

As reported previously,⁷ pre-oxidation in air greatly improves the steam corrosion resistance of Fe-Cr-Al-Y alloys at lower temperatures. The 1041 alloy exhibited poor steam corrosion resistance at 550°C and 730°C when not pre-oxidized. Specimens of the 1041 alloy, pre-oxidized at 980°C then plastically strained 1 to 2 percent, have withstood 2000 hours at 730°C in steam, exhibiting corrosion resistance equal to an unstrained specimen.

During this report period, specimens of the 1041 alloy were pre-oxidized, strained by roll-forming to various radii, then tested in steam at 550°C. The weight change data are presented in Table 2.6. The approximate strain was calculated from the radius of curvature of the specimen. Short pre-oxidation times at 980°C are effective in improving steam corrosion resistance only on unstrained specimens. After long pre-oxidation times, it appears that strains up to about 4 percent may be tolerated. Metallographic examination indicates that rapid corrosion takes place in cracks in the pre-formed surface oxide.

TABLE 2.6
WEIGHT GAIN DATA FOR PRE-OXIDIZED AND STRAINED
1041 ALLOY TESTED IN STEAM AT 550°C

Calculated Plastic Strain, %	Pre-Oxidation Time At 980°C, hr	Weight Gain, mg/cm ²	
		200 hours	400 hours
0	1	0.01	0.01
	20	-0.01	0.03
	100	-0.03	0.00
1	1	0.07	0.14
	20	0.09	0.08
	100	0.00	0.00
2	1	0.15	0.17
	20	0.06	0.08
	100	0.05	0.13
3	1	0.13	0.12
	20	0.03	0.06
	100	0.03	0.12
4	1	0.15	0.27
	20	0.12	0.12
	100	0.02	0.08
5	1	0.68	-
	20	0.30	-
	100	0.32	-

WORK PLANNED FOR NEXT PERIOD

Laboratory testing of Fe-Cr-Al-Y-clad fueled specimens for times up to 10,000 hours at 750°C, 950°C, and 1100°C in air will be continued. Specimens containing enriched UO₂ will be prepared and introduced into the test program to provide a more sensitive measure of uranium diffusion.

The specimen from in-pile test LTFC-1 will be evaluated destructively in the hot cell. A second test cartridge will be prepared containing a 1541-clad Fe - 40UO₂ specimen.

The long-time corrosion testing of Fe-Cr-Al-Y alloys and selected commercial alloys will continue in both air and superheated steam. Further studies of the effects of yttrium and aluminum contents on the oxidation resistance and ductility of the alloys will be carried out.

⁷Ibid., p. 22.

3. HIGH-TEMPERATURE REACTOR MATERIALS RESEARCH

(57003)

The purpose of this program is to develop and evaluate methods of preparing and joining refractory metals and alloys and other high-temperature materials to be used for fueled and non-fueled high-temperature (1000° to 3000°C) reactor structural applications.

MECHANICAL PROPERTY EVALUATIONS

Creep-Rupture Studies

Tungsten - The previous progress report¹ presented the results of an extensive study of the creep-rupture characteristics of arc-cast tungsten, designated W(3), tested at 1600° to 3000°C. During this report period, additional tests of the same material were performed at temperatures of 1600°, 1800°, 2000°, and 2200°C to more completely define the creep-rupture characteristics in this temperature range. All the results accumulated to date are given in Table 3.1 and Figures 3.1 and 3.2. A majority of the previous and present tests were performed on specimens pre-annealed at test temperature for 2 hours to stabilize the structure. At test temperatures of 2000°C and 2200°C structural changes apparently occurred during the progress of the test, as indicated by the unusual creep curves obtained at 2000°C and by the nonlinearity of the stress-rupture and linear creep rate curves at 2000°C and 2200°C (Figures 3.1 and 3.2). At 2000°C, the creep curves contained two linear portions having different slopes with a transition between them approximating third-stage creep behavior.

Additional tests of W(3) material pre-annealed at 2400°C for 1 hour were performed at 2000°C and 2200°C (Figures 3.3 and 3.4). The test results revealed a normal creep curve but the creep rates and rupture times differed from those obtained with specimens annealed at the test temperatures. The results also indicate a possible change in the creep mechanism in the 2000° to 2200°C region. Figures 3.5 and 3.6 compare the stress-rupture and linear creep rate results obtained at 2000°C and 2200°C to data obtained with specimens annealed at test temperatures. The slope from the 2200°C tests, for both stress-rupture and creep rates is similar to the results obtained at 2400°C and 2600°C; the slope obtained at 2000°C is similar to the results obtained at 1600°C and 1800°C. It is this change in slope between 2000°C and 2200°C which indicates the occurrence of a change in the mechanism for creep in this temperature region. On the basis of the creep rate curve for 2800°C (Figure 3.2) an additional change in the creep mechanism may be occurring.

Creep-rupture data for a number of different sources of tungsten were evaluated. One source supplied arc-cast tungsten, W(3), while the others supplied wrought powder metallurgy material, designated W(1), W(2), and W(4). Figure 3.7 presents a summary of the stress-rupture data obtained in hydrogen for each of the materials. As was the case for molybdenum, different creep-rupture results were obtained at the same conditions for

¹"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, pp. 27-46.

TABLE 3:1

CREEP-RUPTURE RESULTS FOR ARC-CAST TUNGSTEN, W(3), TESTED AT 1600° TO 3000°C IN HYDROGEN^a

Specimen Number	Test Conditions		Time To Indicated Elongation, hr								Rupture Elongation, %	Linear Creep Rate, min ⁻¹
	Temperature, °C	Stress, kg/mm ²	0.2%	0.5%	1%	2%	3%	5%	10%	Rupture		
47	1600	3.37	0.08	0.80	2.90	11.6	21.9	45.5	76.8	137 ^b	—	1.4 x 10 ⁻⁵
71 ^f	1600	3.37	—	0.35	2.10	8.90	18.2	40.2	64.0	160	89	1.5 x 10 ⁻⁵
48	1600	4.22	—	0.08	0.15	1.40	3.35	9.70	16.0	30.9	76	5.1 x 10 ⁻⁵
70 ^f	1600	4.92	—	0.01	0.13	0.70	1.67	4.20	7.85	14.7	65	1.3 x 10 ⁻⁴
69 ^f	1600	5.62	—	0.01	0.07	0.32	0.71	1.83	3.75	6.45	73	2.9 x 10 ⁻⁴
52 ^f	1800	2.11	0.05	0.75	3.10	11.5	21.7	46.0	92.2	150	62	1.4 x 10 ⁻⁵
51 ^f	1800	2.81	—	0.03	0.37	2.10	4.45	9.90	14.7	28.4	73	6.1 x 10 ⁻⁵
50 ^f	1800	3.37	—	0.03	0.13	0.58	1.25	2.85	4.85	7.60	60	1.9 x 10 ⁻⁴
45	2000	1.05	0.12	1.70	6.30	16.5	25.2	45.3	101	256	65	1.5 x 10 ⁻⁵
39	2000	1.41	—	0.80	2.50	6.10	8.25	11.0	16.2	63.1	94	4.6 x 10 ⁻⁵
46	2000	1.41 ^c	—	0.17	0.65	2.80	6.10	14.4	35.1	100	92	3.9 x 10 ⁻⁵
58 ^f	2000	1.76	—	—	0.25	1.40	2.93	4.95	6.35	17.6	60	9.8 x 10 ⁻⁵
53 ^f	2000	2.11	—	—	0.08	0.50	1.15	2.63	3.85	6.70	70	2.3 x 10 ⁻⁴
57 ^f	2000	2.11 ^c	—	—	—	0.01	0.06	0.28	1.73	6.50	60	5.4 x 10 ⁻⁴
16	2200	0.70	0.12	1.33	5.00	14.5	25.0	46.5	95.0	237	125	1.5 x 10 ⁻⁵
72 ^f	2200	0.70 ^c	0.01	0.29	1.73	6.33	12.2	25.0	57.5	182	91	2.6 x 10 ⁻⁵
15	2200	0.84	0.01	0.41	1.90	6.45	11.8	22.3	48.0	119	86	3.1 x 10 ⁻⁵
14	2200	1.05	—	0.05	0.35	1.58	3.30	6.60	14.8	40.7	106	9.6 x 10 ⁻⁵
40	2200	1.26	—	—	0.10	0.52	1.18	2.60	—	3.35 ^b	—	2.3 x 10 ⁻⁴
13	2200	1.41	—	—	0.02	0.20	0.51	1.30	3.30	7.72	74	4.0 x 10 ⁻⁴
60 ^f	2200	1.41 ^c	—	—	—	0.05	0.18	0.63	2.00	6.77	95	6.3 x 10 ⁻⁴
59 ^f	2200	2.11 ^c	—	—	—	—	0.01	0.05	0.25	0.91	76	4.4 x 10 ⁻³
22	2400	0.46	0.05	0.50	2.12	6.00	9.80	18.5	40.0	146	102	3.9 x 10 ⁻⁵
24	2400	0.56	—	0.17	0.80	2.70	5.00	9.75	21.3	61.4	120	7.0 x 10 ⁻⁵
21	2400	0.70	—	0.02	0.18	0.78	1.52	3.18	7.40	17.5	83	2.0 x 10 ⁻⁴
17	2400	0.84	—	0.004	0.05	0.28	0.60	1.40	3.42	9.25	73	4.0 x 10 ⁻⁴
38	2400	1.05	—	—	0.01	0.08	0.20	0.50	1.30	3.09	86	1.0 x 10 ⁻³
27	2600	0.28	0.12	0.71	2.30	6.30	10.5	19.2	41.0	123	84	3.8 x 10 ⁻⁵
26	2600	0.35	0.03	0.17	0.64	1.97	3.45	6.60	14.0	43.4	87	1.1 x 10 ⁻⁴
33	2600	0.46	—	0.04	0.20	0.71	1.32	2.62	5.60	15.0	104	2.6 x 10 ⁻⁴
25	2600 ^e	0.56	—	—	0.02	0.12	0.28	0.67	1.62	4.77 ^d	64	8.5 x 10 ⁻⁴
19	2600	0.84	—	—	—	0.004	0.02	0.08	0.25	0.78	78	5.0 x 10 ⁻³
34	2800	0.18	0.33	1.48	4.00	12.0	22.0	45.0	79.0	144	40	1.5 x 10 ⁻⁵
36	2800	0.21	0.06	0.33	1.18	3.40	6.30	13.4	28.8	51.9	46	4.9 x 10 ⁻⁵
29	2800	0.28	—	0.03	0.12	0.55	1.14	2.50	5.60	14.0	74	2.4 x 10 ⁻⁴
28	2800	0.35	—	—	0.01	0.10	0.28	0.68	1.61	4.30	64	8.3 x 10 ⁻⁴
49	3000	0.21	—	0.01	0.03	0.10	0.22	0.61	1.82	2.80	22	6.7 x 10 ⁻⁴

^aAnnealed for 2 hours at test temperature in H₂ prior to test.^bTest terminated, no rupture.^cAnnealed for 1 hour at 2400°C in H₂ prior to test.^dSpecimen rotated at 4.1 hours, affecting rupture time.^eTested in argon.^fNew data.

different sources of powder metallurgy material. Essentially identical results were obtained from creep-rupture tests at 2200°C in hydrogen using arc-cast molybdenum from four different vendors. The stress-rupture results for the arc-cast tungsten, given in Figure 3.7, therefore, are considered to be more representative of the behavior of tungsten than the powder metallurgy data given in the same figure.

The first-stage creep data obtained for the arc-cast tungsten, W(3), at temperatures from 1600° to 3000°C were analyzed using the parabolic creep law,

$$\epsilon = b t^m + \epsilon_0 \quad (1)$$

where ϵ and ϵ_0 are strain, t is time in minutes and b and m are constants. In this analysis ϵ_0 was computed from:

$$\epsilon_0 = \frac{\epsilon_2^2 - \epsilon_1 \epsilon_4}{2\epsilon_2 - \epsilon_1 - \epsilon_4} \quad (2)$$

Fig. 3.1 – Stress-rupture curves for arc-cast tungsten sample W(2) tested at various temperatures in hydrogen

Fig. 3.2 – Creep rate curves for arc-cast tungsten sample W(3) tested at various temperatures in hydrogen

Fig. 3.3 - Effect of annealing at 2400°C on elongation of arc-cast tungsten tested at 2000°C in hydrogen

Fig. 3.4 - Effect of annealing at 2400°C on elongation of arc-cast tungsten tested at 2200°C in hydrogen

Fig. 3.5 – Effect of annealing at 2400°C on stress-rupture of arc-cast tungsten

Fig. 3.6 – Effect of annealing at 2400°C on linear creep rate of arc-cast tungsten

Fig. 3.7 – Results of stress-rupture tests in hydrogen for tungsten from several vendors

where the subscript notations denote the strain corresponding to the times $t_0 = 0$, $t_1 =$ one unit of time, $t_2 =$ two units, and $t_4 =$ four units. These values of time are selected for convenience in the computations.

When t is equal to 1, $b = \epsilon - \epsilon_0$. Taking the logarithm of the parabolic creep law and rearranging:

$$\log b + m \log t = \log (\epsilon - \epsilon_0) \quad (3)$$

and

$$m = \frac{\log (\epsilon - \epsilon_0) - \log b}{\log t} \quad (4)$$

When $t = 4$,

$$m = 1.66085 \log \frac{(\epsilon_4 - \epsilon_0)}{(\epsilon_1 - \epsilon_0)} \quad (5)$$

The unit of time, t_1 , is selected so that t_4 ($t_4 = 4t_1$) is between 50 to 80 percent of the time to the end of first-stage creep. After the values of ϵ_0 and m have been computed, the value of b can be obtained by solving the parabolic creep equation. For the calculation of the b values in Table 3.2, time values corresponding to t_1 were consistently employed.

Values of ϵ_0 , m and b for the arc-cast tungsten tests are presented in Table 3.2. Values of b , when plotted versus stress, are linear at constant temperature on logarithmic coordinates as shown in Figure 3.8. This yields a relationship of the type:

$$b = \bar{a} \sigma^{\bar{c}} \quad (6)$$

where $\bar{a}$ and $\bar{c}$ are constants and σ is stress in kg/mm^2 . The data points in Figure 3.8 suggest that the curves for the seven temperatures are parallel indicating $\bar{c}$ to be independent

TABLE 3.2
CONSTANTS OBTAINED IN FIRST-STAGE CREEP ANALYSIS
OF DATA FOR ARC-CAST TUNGSTEN, W(3)

$\epsilon = bt^m + \epsilon_0$, t in minutes					
Specimen Number	Temperature, °C	Stress, kg/mm ²	ϵ_0	m	b
47	1600	3.37	0.0018	0.80	0.00017
71	1600	3.37	0.0026	0.61	0.00047
48	1600	4.22	0.0029	0.57	0.0017
70	1600	4.92	0.0054	0.62	0.0021
69	1600	5.62	0.0042	0.58	0.0039
52	1800	2.11	0.0024	0.66	0.00029
51	1800	2.81	0.0059	0.70	0.00093
50	1800	3.37	0.0023	0.62	0.0025
45	2000	1.05	0.0014	0.61	0.00039
39	2000	1.41	0.0014	0.82	0.00028
46 ^a	2000	1.41	0.0015	0.54	0.0013
58	2000	1.76	0.0025	0.55	0.0023
53	2000	2.11	0.0043	0.57	0.0039
57 ^a	2000	2.11	0.0072	0.38	0.018
16	2200	0.70	0.0021	0.77	0.00016
72 ^a	2200	0.70	0.0025	0.66	0.00047
15	2200	0.84	0.0031	0.75	0.0011
14	2200	1.05	0.0042	0.67	0.0011
40	2200	1.26	0.0061	0.71	0.0024
13	2200	1.41	0.0082	0.67	0.0043
60 ^a	2200	1.41	0.0089	0.50	0.010
59 ^a	2200	2.11	0.026	0.75	0.029
22	2400	0.46	0.0012	0.67	0.00040
24	2400	0.56	0.0031	0.72	0.00062
21	2400	0.70	0.0052	0.74	0.0016
17	2400	0.84	0.0062	0.72	0.0062
38	2400	1.05	0.0097	0.71	0.0072
27	2600	0.28	0.0016	0.77	0.00025
26	2600	0.35	0.0020	0.73	0.00070
33	2600	0.46	0.0034	0.72	0.0017
35	2600	0.46	0.0024	0.65	0.0020
25	2600	0.56	0.0084	0.77	0.0050
19	2600	0.84	0.018	0.79	0.031
34	2800	0.18	0.00029	0.67	0.00025
36	2800	0.21	0.0017	0.72	0.00047
29	2800	0.28	0.0037	0.64	0.0025
28	2800	0.35	0.010	0.77	0.0068
49	3000	0.21	0.0043	0.64	0.0055

^aAnnealed at 2400°C for 1 hour prior to test.

of temperature with a value of 4.32 as calculated from the slopes. Plotting $\bar{a}$ versus temperature, as shown in Figure 3.9, yields a straight line on semilogarithmic coordinates with the relationship of the form:

$$\bar{a} = \bar{d} e^{\bar{g}T} \quad (7)$$

where $\bar{d}$ and $\bar{g}$ are constants and T is the temperature in °K.

This correlation leads to the following equation for the calculation of first-stage strain at various times for arc-cast tungsten tested at temperatures from 1600° to 2800°C:

$$\epsilon = \bar{d} e^{\bar{g}T} \sigma^{\bar{c}} t^m + \epsilon_0 \quad (8)$$

Fig. 3.8 - Plot of "b" versus stress showing constant slope "c" for arc-cast tungsten from 1600° to 2800°C

Fig. 3.9 - Plot of $\bar{a}$ versus temperature showing linearity for arc-cast tungsten from 1600° to 2800°C

Substituting the constants obtained in Figures 3.8 and 3.9 gives:

$$\epsilon = 2.8 \times 10^{-15} e^{1.07 \times 10^{-2} T} \sigma^{4.32} t^m + \epsilon_0 \quad (9)$$

where:

$m = 0.64$ from 1600° to 2000°C

$m = 0.72$ from 2200° to 2800°C

ϵ = strain, cm/cm

T = temperature, $^\circ\text{K}$

σ = stress, kg/mm^2

t = time, minutes

Because attempts to correlate values of ϵ_0 have not been successful, specific values for a given temperature and stress must be employed with the above expression. Additional work is in progress to develop a general equation to express the first-stage creep behavior for arc-cast tungsten.

W - 25Re - Creep-rupture tests of W - 25Re² were initiated in support of the Vendor Evaluation Program. Sheets of 0.05-cm-thick W - 25Re were purchased from several vendors to evaluate variations in quality and to compare mechanical properties at elevated temperatures. The vendor sources are designated as EV-1, etc. The material from EV-1 and EV-3 was arc-cast and the remainder was fabricated by the powder metallurgy technique.

To date, six creep-rupture tests have been performed at 2000°C in hydrogen using arc-cast material from vendor source EV-1. The results are given in Table 3.3, which also presents the times required to obtain various elongations. Figure 3.10 shows the test specimens after test.

TABLE 3.3
CREEP-RUPTURE RESULTS FOR ARC-CAST W - 25Re SHEET TESTED AT 2000°C IN HYDROGEN

Specimen Number	Annealing Conditions		Test Stress, kg/mm^2	Time To Indicated Elongation, hr								Rupture Elongation, %
	Temperature, $^\circ\text{C}$	Time, hr		0.2%	0.5%	1%	2%	3%	5%	10%	Rupture	
EV-1B-1	2000	2	1.05	3.6	10.8	22.0	39.5	53.8	77.2	121	266	96
EV-1B-2	2000	2	1.41	2.0	5.0	8.5	15.1	20.1	28.6	46.9	107	103
EV-1B-6	2400	1	1.41	1.4	2.2	3.7	6.2	8.4	12.5	22.1	85.1	110
EV-1B-3	2000	2	2.11	0.6	1.2	1.9	3.2	4.4	6.3	9.8	21.9	86
EV-1B-5	2400	1	2.11	0.3	0.5	0.8	1.2	1.7	2.5	4.4	18.1	90
EV-1B-4	2000	2	3.52	—	0.1	0.2	0.3	0.5	0.7	1.1	2.40	81

^a0.05 by 0.64 by 2.54 cm gage section.

In Figure 3.11, the stress-rupture results for arc-cast W - 25Re are compared to previously obtained results for arc-cast tungsten tested at 2000°C . At the higher stresses, W - 25Re has a greater rupture life than pure tungsten. At $1.05 \text{ kg}/\text{mm}^2$, the rupture life for the two materials is essentially identical. If the same trends persist, tungsten should be stronger than W - 25Re at stresses below $1.05 \text{ kg}/\text{mm}^2$ at 2000°C .

Because of the unusual creep curves obtained for tungsten at 2000°C when pre-annealed at 2000°C for 2 hours, specimens of both materials were pre-annealed at 2400°C for 1 hour prior to testing at 2000°C and stresses of 1.41 and $2.11 \text{ kg}/\text{mm}^2$. This annealing stabilizes the structure of the material and eliminates any changes in the creep curve caused by structural changes occurring during the test. Such structural changes apparently caused the unusual creep curve for tungsten at 2000°C .

As shown in Figure 3.12, the high-temperature annealing caused the tungsten to exhibit the conventional creep curve. Also, the tungsten was more creep-resistant than the

²All compositions are in atomic percent unless otherwise noted.

Stress, kg/mm ²			
3.52	2.11	1.41	1.05
Time to rupture, hours			
2.40	21.9	107	266
Elongation at rupture, percent			
81	86	103	96

Notes:

1. Arc-cast W-25Re sheet was 0.05 cm thick.
2. Specimens annealed at 2000°C in H₂ for 2 hours before test

Fig. 3.10 – Arc-cast W-25Re sheet creep-rupture tested at 2000°C in hydrogen
(Neg. P65-6-24B)

Fig. 3.11 – Stress-rupture results for arc-cast W and arc-cast W-25Re tested at 2000°C in hydrogen

Fig. 3.12 - Creep curves for arc-cast W-25Re and pure tungsten tested at 2000°C and 1.41 kg/mm² in hydrogen; material annealed at 2400°C for 1 hour prior to test

Fig. 3.13 - Creep curves for arc-cast W-25Re and pure tungsten tested at 2000°C and 1.41 kg/mm² in hydrogen; material annealed at 2000°C for 2 hours prior to test

W - 25Re for identical stress conditions at 2000°C. As shown in Figure 3.13, the reverse was the case when the specimens were pre-annealed for 2 hours at 2000°C. Figure 3.14 shows the decrease in the creep resistance of the W - 25Re pre-annealed at 2400°C. Significantly, no first- or second-stages of creep occurred in arc-cast W - 25Re tested at 2000°C. The creep curves tend to start with a third-stage type of creep and deformation continues until rupture occurs. The arc-cast W - 25Re is quite ductile at 2000°C with elongations at rupture ranging from 81 to 110 percent for the 2.54-cm gage length.

Fig. 3.14 - Creep curves for arc-cast W - 25Re tested at 2000°C and 1.41 kg/mm² in hydrogen

Molybdenum - During this report period, additional creep testing was conducted on sheets of arc-cast molybdenum designated M(6), at temperatures from 1600°C to 2200°C. As described in the previous progress report³ an electro-optical extensometer (Optron)⁴ is used for measurement of instantaneous strain. The test results obtained to date, including those from the previous progress report, are summarized in Table 3.4. For the specimens tested during this report period (the last nine in Table 3.4), a different method was used to determine the exponential value, m , for the parabolic creep equation, $\epsilon = bt^m + \epsilon_0$. Previously, the method presented by Hazlett and Parker⁵ was used to obtain m values. Recently, a more direct procedure, based on the more precise method proposed by de Lacombe,⁶ was employed. This method obtains the m value directly as opposed to the trial and error approach in the Hazlett and Parker analysis.

Tests were performed at 1600°C, 1800°C, 2000°C, and 2200°C and sufficient data accumulated to plot stress versus linear creep rate as shown in Figure 3.15. The time to reach a specified strain versus stress is shown in Figure 3.16. While the data available for the

³"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, p. 56.

⁴Registered trade name of Optron Corporation, Santa Barbara, California.

⁵T. H. Hazlett and E. R. Parker, "Nature of the Creep Curve," *J. Metals*, Vol. 5, 1953, p. 318.

⁶J. de Lacombe, "A Method of Representing Creep Curves," *Rev. Metal.*, Vol. 36, 1939, p. 178.

TABLE 3.4

CREEP DATA FOR ARC-CAST MOLYBDENUM² FROM 1600° TO 2200°C OBTAINED WITH THE OPTRON

Specimen No. For M(6) Material	Test Temperature, °C	Stress, kg/mm ²	Test Time, hr	Test Atmosphere	Room- Temperature Elongation, %	Elongation Per Optron, %	Optron Elongation Error, %	Second-Stage Creep Rate, min ⁻¹	Total Train Second-Stage Creep Rate, min ⁻¹	ϵ vs t^m	$\log(\epsilon - \epsilon_0)$ vs $\log t$	ϵ_0	b	Duration Of First-Stage Creep, min
36	2200	0.32	4.28	Ar	51.8	rupture	—	1.0×10^{-3}	1.2×10^{-3}	c	c	c	c	13
17	2200	0.39	2.03	Ar	48.7	rupture	—	2.3×10^{-3}	2.6×10^{-3}	0.65	0.65	0.0005	0.0076	8
45 ^d	2200	0.32	0.88	Ar	6.20	6.96	+12.0	1.1×10^{-3}	—	0.65	0.60	0.0009	0.0049	18
1-3	2200	0.32	0.63	Ar	4.60	4.55	-1.0	9.9×10^{-4}	—	0.60	0.62	0.0008	0.0048	13
1-1	2200	0.32	0.62	H ₂	3.60	3.54	-1.7	7.5×10^{-4}	9.5×10^{-4}	0.65	0.65	0.0005	0.0030	12
44	2200	0.30	0.58	H ₂	b	2.60	—	5.2×10^{-4}	—	0.65	0.65	0.0013	0.0024	19
1-2	2200	0.32	0.63	H ₂	3.62	3.63	+0.3	7.8×10^{-4}	9.8×10^{-4}	0.65	0.64	0.0013	0.0030	13
2-1	2200	0.39	0.32	H ₂	4.83	4.75	-1.7	1.9×10^{-3}	2.8×10^{-3}	0.65	0.64	0.0028	0.0062	9
2-2	2200	0.20	1.20	H ₂	0.98	0.92	-5.0	Not run to second stage	—	0.80	0.80	0.00055	0.00028	>72
2-8	2200	0.25	4.75	H ₂	8.18	8.25	+0.9	2.5×10^{-4}	—	0.75	0.75	0.0017	0.0010	85
2-11	2000	0.70	0.77	H ₂	b	9.60	—	1.7×10^{-3}	—	0.60	0.57	0.0025	0.0069	8
2-9	2000	0.50	3.70	H ₂	9.50	9.11	-4.1	3.7×10^{-4}	4.8×10^{-4}	0.75	0.79	0.0014	0.0013	70
2-4	1800	1.20	0.65	He	8.42	8.40	+0.2	1.8×10^{-3}	2.6×10^{-3}	0.70	0.71	0.0030	0.0056	13
2-3	2000	0.32	20.83	He	9.55	9.70	-1.5	7.0×10^{-5}	8.3×10^{-5}	0.60	0.60	0.0000	0.00064	110
2-7	2200	0.25	3.75	He	8.8	9.3	+4.9	3.6×10^{-4}	4.4×10^{-4}	0.77	—	0.00085	0.0013	64
2-15	1800	1.2	0.77	He	b	9.2	—	1.5×10^{-3}	2.2×10^{-3}	0.69	—	0.0052	0.0048	11
2-14	1800	0.84	4.66	He	7.8	7.6	-3.3	2.4×10^{-4}	3.3×10^{-4}	0.79	—	0.0011	0.00084	120
2-17	2200	0.32	1	He	7.0	7.6	+8.9	9.8×10^{-4}	1.3×10^{-3}	0.7	—	0.003	0.0036	19
2-20 ^d	1600	2.1	2.1	He	b	5.6	—	1.3×10^{-3}	1.6×10^{-3}	0.62	—	0.0074	0.0056	12
2-6	1600	1.62	4.58	H ₂	8.7	8.6	-1	2.7×10^{-4}	3.8×10^{-4}	0.70	—	0.0007	0.0014	65
2-18	1600	1.2	22.1	H ₂	9.4	9.04	-4	5.8×10^{-5}	8.8×10^{-5}	0.615	—	0.0013	0.00076	120
2-13	1800	0.53	24.8	H ₂	7.3	7	-4	4.2×10^{-5}	5.1×10^{-5}	0.47	—	0.00016	0.00099	80
2-19	2200	0.14	44.5	H ₂	b	10.9	—	2.7×10^{-5}	3.6×10^{-5}	—	—	—	—	—

^aAll samples annealed for 2 hours at test temperature before test.^bTarget wire broken upon removal of sample from furnace.^cTracker unlocked on sample loading — sample removed, first stage data not obtained.^dCyclic temperature control.

Fig. 3.15 – Creep rate curves for arc-cast molybdenum, sample M(6), tested in hydrogen at various temperatures

1600°C, 1800°C, and 2000°C curves are limited, the data appear reasonable when compared to the results at 2200°C for which more data were accumulated. Logarithmic plots of strain as total strain and $(\epsilon - \epsilon_0)$ versus time at three different stresses at 1600°C are shown in Figures 3.17 and 3.18. The straightness of the $\epsilon - \epsilon_0$ plot extending to the initiation of second-stage creep verifies that the creep data are well represented by the parabolic first-stage creep law.

As was the case with arc-cast tungsten, a correlation exists between stress and b values for molybdenum. A plot of b versus stress yields a family of curves as shown in Figure 3.19, indicating a constant $\bar{c}$ independent of temperature for the expression $b = \bar{a} \sigma^{\bar{c}}$. The data for $\bar{a}$ obtained from Figure 3.19 are plotted versus temperature in Figure 3.20. As with tungsten, a relationship exists in the form:

$$\bar{a} = \bar{d} e^{\bar{g}T} \quad (10)$$

This relationship allows the general first-stage creep equation:

$$\epsilon = bt^m + \epsilon_0 \quad (11)$$

to be rewritten as

$$\epsilon = \bar{d} e^{\bar{g}T} \sigma^{\bar{c}} t^m + \epsilon_0 \quad (12)$$

where:

ϵ = strain, cm/cm
 T = temperature, °K
 σ = stress, kg/mm²

Fig. 3.16 – Creep data for arc-cast molybdenum tested in hydrogen at various temperatures

t = time, min.

$\bar{c} = 4.1$

$\bar{d} = 3.913 \times 10^{-14}$

$\bar{g} = 0.012035$

m and ϵ_0 are constants tabulated in Table 3.4.

A number of the most recent molybdenum tests in which the Optron was used for measurement of strain were duplicates of tests performed using the twin optical extensometer. All Optron data agree with the data previously obtained with the twin optical extensometer. Since the operation of the Optron has now become routine and its accuracy has been verified, a new program has been initiated to determine the effects of various heat treatments on the first-stage creep behavior.

Creep Analysis Techniques

In the preceding report,⁷ arc-cast tungsten creep data at 2400°C in hydrogen at various stresses were analyzed in terms of several different empirical creep equations. In general, a fairly linear relation was obtained when strain was plotted as a function of time raised to

⁷ "High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, pp. 46-48.

Fig. 3.17 – Actual creep curves for arc-cast molybdenum tested at 1600°C in hydrogen

Fig. 3.18 – Adjusted creep curves for arc-cast molybdenum tested at 1600°C in hydrogen

Fig. 3.20 - Plot of $\bar{a}$ versus temperature showing linearity for arc-cast molybdenum tested at 1600° to 2200°C

the 0.6 power. Slope and intercept calculations led to the evaluation of the creep equation constants for use in the familiar parabolic form:

$$\epsilon = bt^{0.6} + \epsilon_0 \quad (13)$$

Other constants were obtained assuming the exponent on time to be 0.5; however, when the higher exponent was employed, linearity was found to extend over greater portions of the first stage of creep. In addition, the Andrade one-third creep law, written in terms of strain, gave an excellent representation of the data over the entire range of first-stage creep behavior. A special least-squares computer program was developed for use in this particular case. Finally an equation of the form

$$\epsilon = f + gt - he^{-pt} \quad (14)$$

where f , g , h , and p are constants, was employed to enable first- and second-stage creep behavior to be described in a single equation.

Analyses of the type described above were continued during this report period, using arc-cast tungsten data obtained at other temperatures. These studies are not complete and hence no additional creep equation constants can be reported. However, a study was made of various other empirical equations to determine how accurate such equations are in providing a representation of experimental data. In this analysis, the Andrade⁸ data for lead at 14.5°C and a constant stress of 1.67 kg/mm² were selected and the sum of the squares of the residuals was used to provide the measure of how well a given equation represented the data. Residuals were employed in the usual manner to express the difference between a given experimental strain value and the value of strain calculated by the equation under study. The results obtained are presented in Table 3.5 where the values for the sum of the squares of the residuals are arranged in decreasing order.

TABLE 3.5
A COMPARISON OF THE DEGREE OF REPRESENTATION OF THE
ANDRADE DATA FOR LEAD AT 14.5°C AFFORDED BY
VARIOUS EQUATION TYPES

Equation	$\epsilon(R)^2$
$\epsilon = 40.176 + 1.739 \times 10^{-2}t - 1.468 \times 10^{-5}t^2$	0.97
$\epsilon = 40.01 + 2.642 \times 10^{-2}t - 6.282 \times 10^{-5}t^2 + 6.06 \times 10^{-8}t^3$	0.40
$\epsilon = 39.723 + 7.002 \times 10^{-3}t + 0.316 \ln t$	0.27
$\epsilon = 39.616 + 6.832 \times 10^{-3}t + 0.347 \ln t$	0.21
$\epsilon = 37.941 (1 + 0.044t^{1/6})e^{1.177 \times 10^{-4}t}$	0.055
$\epsilon = 39.55 + 0.251t^{1/2}$	0.027
$\epsilon = 39.549 + 0.250t^{1/2} + 8.105 \times 10^{-5}t$	0.021
$\epsilon = 39.335 + 0.376t^{1/3} + 0.0454t^{2/3}$	0.0082
$\epsilon = 39.13 (1 + 0.01457t^{1/3})e^{6.95 \times 10^{-5}t}$	0.0035 ^a
$\epsilon = 39.465 + 0.295t^{1/2} - 4.827 \times 10^{-3}t + 1.38 \times 10^{-4}t^{3/2}$	0.0031
$\epsilon = 39.149 + 3.16 \sinh (0.177t^{1/3})$	0.0027
$\epsilon = 39.148 (1 + 0.0143t^{1/3})e^{7.26 \times 10^{-5}t}$	0.0024
$\epsilon = 39.205 + 0.508t^{1/3} + 1.071 \times 10^{-2}t^{2/3} + 2.56 \times 10^{-3}t$	0.0007

^aOriginal equation proposed by Andrade to represent these data.

⁸E. N. daC. Andrade, "On the Viscous Flow in Metals and Allied Phenomena," *Proceedings of the Royal Society*, Vol. 84, 1910, p. 1.

Obviously, the poorest representation of these data is that corresponding to a second-degree polynomial in time, while the best results are afforded by a third-degree polynomial in time raised to the one-third power.

Some of the results obtained in this study are shown graphically in Figure 3.21. In plot "A" the specially developed least-squares computer program was employed to obtain the best values for the Andrade creep equation constants. The results were:

$$l = 39.148 (1 + 0.0143t^{1/3})e^{7.26 \times 10^{-5}t} \quad (15)$$

Fig. 3.21 - A comparison of the degree of fit afforded by various equation types when applied to the Andrade data for lead at 14.5°C and 1.67 kg/mm²

compared to the original equation:

$$\ell = 39.13 (1 + 0.01457t^{1/3})e^{6.95 \times 10^{-5}t} \quad (16)$$

reported by Andrade. A calculation of the sum of the squares of the residuals gave values of 0.0024 and 0.0035, respectively. Therefore, the least-squares analysis employed in the present study is seen to identify equation constants which provide a better representation of the experimental data than the equation developed in the original study. Obviously, because of the nature of the least-squares procedure, the equation identified in this manner leads to the best representation obtainable for this equation type.

Since, as mentioned previously, a plot of length (or strain) versus $t^{1/2}$ is very nearly linear, an acceptable representation might evolve from a polynomial expression written in terms of $t^{1/2}$. A least-squares analysis for a second- and third-degree polynomial of this form led to the following results:

$$\ell = 39.549 + 0.250t^{1/2} + 8.105 \times 10^{-5}t \quad (17)$$

and

$$\ell = 39.465 + 0.295t^{1/2} - 4.827 \times 10^{-3}t + 1.38 \times 10^{-4}t^{3/2} \quad (18)$$

for which the sums of the squares of the residuals were 0.021 and 0.0031, respectively. Obviously, these forms lead to very good representations. Figure 3.21c is a plot of the third-degree polynomial.

Since it might appear from the shape of the curve in Figure 3.21a that polynomial expressions yield an adequate representation, this equation type was investigated briefly. It was soon found, however, that the results were completely unsatisfactory. A least-squares computer program was employed to obtain second- and third-degree polynomials with the following results:

$$\ell = 40.176 + 1.739 \times 10^{-2}t - 1.468 \times 10^{-5}t^2 \quad (19)$$

and

$$\ell = 40.01 + 2.642 \times 10^{-2}t - 6.282 \times 10^{-5}t^2 + 6.06 \times 10^{-8}t^3 \quad (20)$$

Curves representing these equations are shown in Figure 3.21b and are seen to provide completely unsatisfactory representations of the experimental points. Indeed, in the case of the third-degree equation, a point of inflection is involved which is obviously completely contrary to the general form of the standard deformation curve. Also, the values for the sum of the squares of the residuals are higher than for any other equation employed in this study.

Another polynomial expression was suggested from a study of the Andrade equation. Since the value of k is usually very small, it is possible to make use of the relation:

$$e^{kt} = 1 + kt \quad (21)$$

for small values of kt . When this is done, the equation can then be expanded to yield:

$$\ell = \ell_0 + \beta \ell_0 t^{1/3} + k \ell_0 t + \beta k \ell_0 t^{4/3} \quad (22)$$

suggesting the use of a polynomial expression in $t^{1/3}$. After re-defining the time variable as $t^{1/3}$, the data were submitted to the same least-squares analysis program as used for the equations in t to yield polynomials in $t^{1/3}$. The results were as follows:

$$\ell = 39.335 + 0.3761t^{1/3} + 0.045t^{2/3} \quad (23)$$

and

$$\ell = 39.205 + 0.508t^{1/3} + 1.071 \times 10^{-2}t^{2/3} + 2.56 \times 10^{-3}t \quad (24)$$

As shown in Figure 3.21d, this equation provides an excellent fit for the experimental data. As a matter of fact, the sum of the squares of the residuals is 0.0007 and hence is smaller than any of the other values obtained in this study. It follows, therefore, that this equation type allows a better representation of the experimental data analyzed in this study than that afforded by the Andrade equation. Such behavior might be expected due to the fact that the third-degree equation contains one more constant than the Andrade equation; nevertheless, further analysis of the behavior of this polynomial in $t^{1/3}$ is very interesting. For example, it does not exhibit the inflection phenomenon observed in Figure 3.21b for the third-stage polynomial in t . Also, the derivative of this equation, which gives the creep rate as a function of time, possesses the desired form for expressing first- and second-stage creep behavior. In the range of small time values, the creep rate decreases with time and gradually assumes a constant value at large time values.

The second-degree polynomial in $t^{1/3}$ also gives a fairly good fit although, as expected, it is slightly inferior to that given by the third-degree equation. The curve representing the second-degree equation was not plotted in Figure 3.21d since it would not be too different from that for the equation plotted. The sum of the squares of the residuals corresponding to the second-degree equation was found to be 0.0082 and hence not too inferior to the third-degree equation. Based on this analysis, equations of the type representing second- and third-degree polynomials in $t^{1/3}$ should be given careful consideration in any attempt to develop an accurate representation of creep data.

More recently, a somewhat different creep equation forms was employed and while results are available for only one set of experimental data, some degree of success seems to have been achieved. In creep analyses involving length (or strain) versus $t^{1/3}$, plots of the deviations from linearity appeared to exhibit the form of a modified hyperbolic sine function. For this reason, a special least-squares computer program was developed to permit identification of the constants in an equation of the form:

$$\ell = \ell_0 + A \sinh(Bt^{1/3}) \quad (25)$$

where A , B , and ℓ_0 are constants. Applied to the same data used in the results shown in Table 3.5, the following equation was obtained:

$$\ell = 39.149 + 3.16 \sinh(0.177t^{1/3}) \quad (26)$$

This relation, plotted in Figure 3.22, indicates that an excellent representation of the experimental data is obtained. Since the sum of the squares of the residuals in this case is found to be 0.0027, it ranks with the more effective equation types given in Table 3.5. This appears to be the first use of a hyperbolic sine function to express the time dependency of first-stage deformation behavior. A very interesting aspect of this equation follows from one of the characteristics of hyperbolic sine functions. For small values of the argument, the value of the hyperbolic sine is equal to the value of the argument itself. Therefore for small time values the above equation can be written:

$$\begin{aligned} \ell &= 39.149 + 3.16 (0.177t^{1/3}) \\ \ell &= 39.149 + 0.56t^{1/3} \end{aligned}$$

This is consistent with the fact that in the region of small time values the data actually exhibit a decided linearity in $t^{1/3}$. It is also very interesting that for small time values, when the exponential term can be assumed equal to unity, the least-squares Andrade equation reduces to:

$$\ell = 39.148 + 0.56t^{1/3} \quad (27)$$

The agreement here is exceptionally good.

Fig. 3.22 – Representation of first-stage creep data by a hyperbolic sine function

W-Re-Mo ALLOY DEVELOPMENT

In this program, 20 W-Re and W-Re-Mo alloys, with melting points above 2760°C, are being evaluated. The W – 25Re – 30Mo and W – 30Re – 30Mo alloys were selected for additional testing including radiation effects.

The W-Re-Mo alloys are resistant to grain coarsening at high temperatures. As shown in Figures 3.23 and 3.24, the W – 25Re – 30Mo alloy showed no grain coarsening after 30 temperature cycles between 260°C and 2150°C with a total of 100 hours at 2150°C or after 100 hours at 2200°C. Similar results were obtained with W – 30Re – 30Mo.

The room-temperature bend ductility of the W – 25Re – 30Mo and W – 30Re – 30Mo alloys was measured after both cyclic and isothermal heat treatments. As shown in Table 3.6, both alloys retained good ductility (greater than 90-degree bend at room temperature) following either cyclic or isothermal heating. Included is the effect of various hydrogen treatments used for removing the contamination resulting from the hot-rolling. The current preferred treatments (e and f in Table 3.6) did not influence the final bend test results although a now-discarded "duplex" treatment (d in Table 3.6) appeared to be associated with poor ductility in W – 25Re – 30Mo alloy. Both alloys withstood 90-degree bend tests after heating to 100 hours at 2600°C.

In the bend ductility test series for W-Re and W-Re-Mo alloys, a number of specimens were bend-tested at room temperature after 110 hours at 2600°C in hydrogen (Table 3.7).

Fig. 3.23 – Cross section showing fine grains of a W – 25Re – 30Mo sheet after 30 heating cycles between 260°C and 2150°C in hydrogen. A total of 100 hours was sustained at 2150°C.
(Neg. 5972, Etchant, 200X)

Fig. 3.24 – Cross section showing fine grains of a W – 25Re – 30Mo sheet after isothermally heating 100 hours at 2200°C in hydrogen
(Neg. 5971, Etchant, 200X)

As anticipated, all of the alloys tested exhibited good ductility. Alloys with high Re-Mo content withstood 90-degree bends.

Creep-rupture evaluations of W-Re-Mo alloys fabricated at GE-NMPO into 0.05-cm-thick sheet were continued during this period. The previous report⁹ gave the test results

⁹“High-Temperature Materials Program Progress Report No. 47, Part A,” GE-NMPO, GEMP-47A, May 28, 1965, Table 3.11, p. 79.

TABLE 3.6

ROOM TEMPERATURE 4T^a BEND TEST RESULTS OF W - 25Re - 30Mo AND W - 30Re - 30Mo FOLLOWING CYCLIC AND ISOTHERMAL HEAT TREATMENTS IN HYDROGEN

Alloy	Decontaminating Process	As Stress-Relieved ^b	Isothermal Treatment						Cyclic Treatment ^c	
			2200°C			2600°C			3 Cycles	30 Cycles
			2 hr	24 hr	100 hr	2 hr	24 hr	100 hr		
W - 25Re - 30Mo	20 hr at 1420°C ^d	>90°	30°	40°	80°	40°	50°	50°	>60°	80°
	32 hr at 1420°C ^e	>90°	25°	>90°	>90°	>90°	>90°	>90°	>90°	>90°
	2 hr at 2640°C ^f	>90°	>45°	>90°	>90°	>90°	>90°	>90°	>90°	>90°
W - 30Re - 30Mo	32 hr at 1420°C ^e	>90°	>90°	>90°	>90°	>90°	>90°	>90°	>90°	>90°
	2 hr at 2640°C ^f	>90°	>90°	>90°	>90°	>90°	>90°	>90°	>90°	>90°

^aAll sheet 0.0508 cm thick.

^bCold-rolled plus 20 minute stress-relief treatment at 1400°C in hydrogen.

^cHeated from 260°C to 2150°C in 20 min; held 3 hr and 20 min at 2150°C, cooled in 1 hr to 260°C, held at 260°C for 1 hr.

^dHeat treated at 2640°C in hydrogen followed by 20 hours at 1420°C in hydrogen.

^eUsual purification treatment applied after hot-rolling sheet in air.

^fHigh-temperature purification treatment - not fully evaluated for influence on final sheet properties.

TABLE 3.7

ROOM-TEMPERATURE 4T BEND TESTS FOR VARIOUS W-Re AND W-Re-Mo ALLOYS AFTER 110 HOURS AT 2600°C IN HYDROGEN

Assigned Laboratory Number	Nominal Composition	Room-Temperature Bend-Angle To Fracture
EV-5a	W - 25Re	40°
363	W - 25Re - 10Mo	30°
364	W - 25Re - 20Mo	50°
365	W - 25Re - 30Mo	90°
362	W - 30Re	45°
360	W - 30Re - 10Mo	90°
386	W - 30Re - 20Mo	90°
382	W - 30Re - 30Mo	90°

obtained at 1600°C and 4.22 kg/mm² for specimens of W - 26Re, W - 27Re, W - 28Re, and W - 29Re, some with molybdenum additions of 10 or 20 percent. During this report period, these test specimens were examined metallographically. Figure 3.25 presents photomicrographs of the specimens taken from the 2.54-cm gage section after testing. A summary of the observations are as follows:

- For a constant molybdenum content and increasing amounts of rhenium:
 - Grain size decreases from approximately 20 to 10 microns in specimens with no molybdenum added, and from approximately 35 to 20 microns in those with 20 percent molybdenum added
 - Intergranular voids become smaller and more numerous
 - Sigma particles are evenly dispersed but increase in quantity and become stringered
 - DPH hardness values increase from 475 to 575 in specimens with no molybdenum, and from 415 to 500 in those with 20 percent molybdenum.
- For a constant rhenium content and increasing amounts of molybdenum:
 - Grain size increases from approximately 20 to 35 microns in specimens with 26 percent rhenium, and from 10 to 20 microns in those with 29 percent rhenium
 - Intergranular voids become larger and decrease in quantity
 - Sigma particles are evenly dispersed but decrease in quantity
 - DPH hardness values decrease from 475 to 415 in specimens with 26 percent rhenium, and from 575 to 500 in those with 29 percent rhenium.

Fig. 3.25 - Photomicrographs of W-Re-Mo alloys (at. %) after creep-rupture testing at 1600°C and 4.22 kg/mm² in hydrogen showing grain size in microns, hardness in DPH, and rupture elongation in percent (250X)

One additional observation was made in connection with grain size and hardness conditions; i. e., elongation at rupture increased as grain size decreased and/or as hardness increased. This is also shown in Figure 3.25.

In some cases, the observed differences in the creep characteristics of the alloys could not be correlated with the microstructure of the specimens after testing. At 1600°C and 4.22 kg/mm², the time to give 2 percent strain for the W - 26Re alloys decreased by 30 percent with the addition of 20 percent molybdenum (9.2 and 6.1 hours, respectively), whereas the time to give 2 percent strain for the W - 28Re alloys was increased substantially by the addition of 20 percent molybdenum (1.6 and 4.3 hours, respectively).

Further creep-rupture evaluations of the same series of alloys, taken from the same 0.05-cm-thick sheets, were performed at 2200°C and 0.56 kg/mm². The results, including the time in hours to various percentages of elongation, are given in Table 3.8. The creep curves are shown in Figures 3.26, 3.27 and 3.28 with each figure presenting data for a constant molybdenum content. On the basis of the linear creep rate and also the time in hours to give 2 percent elongation, the W - 27Re - 20Mo alloy is the most creep-resistant and the W - 29Re - 20Mo, the least resistant ($4.8 \times 10^{-5} \text{ min}^{-1}$, 6.6 hours and $2.1 \times 10^{-4} \text{ min}^{-1}$, 1.5 hours, respectively). The effect of the molybdenum in the W-Re alloys at 2200°C is not easily identified but the trend is toward a decrease in the rupture time as well as in the elongation at rupture as molybdenum content increases. When the rhenium content is held constant, the trend is toward decreased rupture life and decreased elongation at rupture as molybdenum content increases. However, the trend is not consistent in all cases for the time required to give 2 percent strain, as can be seen in Table 3.8.

TABLE 3.8

CREEP-RUPTURE RESULTS^a FOR W-Re-Mo ALLOYS TESTED AT 2200°C AND 0.56 kg/mm² IN HYDROGEN^b

Alloy ^c	Time To Indicated Elongation, hr							Elongation At Rupture, %	Linear Creep Rate, min ⁻¹	DPII ^d Hardness	
	0.5%	1%	2%	3%	5%	10%	Rupture			Before	After
W - 26Re	0.6	1.3	2.8	4.3	7.0	13.8	30.6	45	1.1×10^{-4}	550	440
W - 27Re	0.4	1.0	2.1	3.3	5.6	10.9	24.5	42	1.4×10^{-4}	495	465
W - 28Re	0.4	1.0	2.3	3.5	6.0	11.2	23.9	42	1.4×10^{-4}	570	475
W - 29Re	0.6	1.4	2.8	4.3	7.0	12.7	21.8	32	1.1×10^{-4}	630	400
W - 26Re - 10Mo	1.0	2.0	4.0	6.1	10.0	18.0	32.0	32	8.1×10^{-5}	490	410
W - 27Re - 10Mo	0.6	1.2	2.6	3.8	6.2	11.0	20.5	38	1.3×10^{-4}	525	425
W - 28Re - 10Mo	0.6	1.1	2.2	3.4	5.4	9.5	16.6	40	1.5×10^{-4}	590	450
W - 29Re - 10Mo	0.5	1.2	2.4	3.6	5.8	10.1	16.6	32	1.3×10^{-4}	610	450
W - 26Re - 20Mo	0.9	1.9	3.8	5.6	9.0	15.4	23.0	23	8.8×10^{-5}	450	415
W - 27Re - 20Mo	1.6	3.3	6.6	10.0	15.8	24.8	29.0	15	4.8×10^{-5}	480	410
W - 28Re - 20Mo	0.4	0.9	1.8	2.7	4.2	7.4	12.5	33	1.8×10^{-4}	500	410
W - 29Re - 20Mo	0.3	0.7	1.5	2.3	3.8	6.8	12.6	35	2.1×10^{-4}	525	410

^a0.05 cm sheet, 0.6 x 2.54 cm gage section.

^bAnnealed 2 hours at 2200°C in H₂ before test.

^cAtomic percent.

^d5 kg load.

Several phenomena, noted during the W-Re-Mo alloy development, are scheduled for future study. These are:

1. The addition of molybdenum makes the W-Re alloys ductile at room temperature regardless of their thermal history.
2. Increasing the molybdenum content increases creep-resistance accompanied by a corresponding decrease in elongation, while increasing rhenium content lowers creep resistance.

Fig. 3.26 - Creep curves for W-Re alloys tested at 2200°C and 0.56 kg/mm² in hydrogen

Fig. 3.27 - Creep curves for W-Re-10Mo alloys tested at 2200°C and 0.56 kg/mm² in hydrogen

Fig. 3.28 – Creep curves for W – Re – 20Mo alloys tested at 2200°C and 0.56 kg/mm² in hydrogen

- Maximum creep-rupture resistance at specific temperatures appears to be dependent on alloy composition; e.g., of the alloys tested, the W – 27Re – 20Mo showed the best creep resistance at 2200°C whereas W – 27Re – 10Mo showed the best creep resistance at 1600°C.

Development of Improved Molybdenum and Molybdenum Alloys

Methods for producing improved molybdenum and molybdenum alloys are being investigated. During the early phases of the work, unalloyed molybdenum and molybdenum alloys of low rhenium content will be investigated.

Currently available commercial molybdenum is subject to several limitations: (1) arc-cast molybdenum is generally weldable, but powder metallurgy products are noted for weld porosity and fragility of the weld and (2) results of chemical analyses are reported by vendors as "typical," and not necessarily specific for a given shipment. In addition, the industry requests "exceptions" before bidding on any order specifying low impurity levels of carbon and oxygen.

Processing procedures were previously established for producing high purity W-Re alloys.^{10,11} The utilization of these techniques for improving molybdenum was initiated. In addition, small rhenium additions to molybdenum were evaluated.

Six powder metallurgy compacts, three of unalloyed molybdenum and three of molybdenum with additions of 1, 3, or 5 atomic percent rhenium, were processed as shown in Figure 3.29. The compacts were hot-rolled to 0.0508-cm-thick sheet. The nominal composition of the sheets and the results of room-temperature bend ductility tests are shown in Table 3.9. For comparative purposes, tests were run on commercial unalloyed

¹⁰C. O. Tarr, W. G. Baxter, and J. A. McGurty, "The Development and Evaluation of a Homogeneous W – 25Re Alloy for 2600°C Applications," AIAA 6th Structures to Materials Conference, April 5–7, 1965, pp. 432–436.

¹¹C. O. Tarr, "Processing Procedures for High Purity W – 25Re Alloy Sheet," GE-TM 65-4-18, presented June 18, 1965, AIAA Propulsion Specialists Conference.

Fig. 3.29 – Schematic of processing variables in sheet fabrication of unalloyed molybdenum and alloys of Mo- 1, 2, and 5 atomic percent rhenium

TABLE 3.9
ROOM-TEMPERATURE BEND-TEST RESULTS FOR Mo AND Mo-Re ALLOYS

Laboratory Number	Nominal Composition	Significant Processing Variable ^a	Bending Punch Speeds ^{b,c}		Bend Test Results After 2 hr At Temperature ^{b,c}				
			2.5 cm/min	0.012 cm/min	1500°C	1600°C	1800°C	2000°C	2200°C
1	Unalloyed Mo	None – standard	45°	75°	>45°	60°	60°	40°	>45°
1 ^b	Unalloyed Mo	Purified – single sinter	>90°	>90°	>90°	>90°	80°	>90°	90°
1 ^c	Unalloyed Mo	Purified – double sinter	70°	70°	>45°	>90°	45°	45°	>45°
2	Mo	Purified – double sinter	50°	>90°	90°	60°	>90°	70°	>90°
3	Mo + 2Re ^d	Purified – double sinter	50°	>90°	>90°	>90°	>90°	50°	>90°
4	Mo + 5Re ^d	Purified – double sinter	>90°	>90°	>90°	>90°	>90°	>90°	>90°
D	Unalloyed Mo	Commercial – sintered	>90°	>90°	–	–	>90°	–	>90°
E	Unalloyed Mo	Commercial – arc-cast	50°	>90°	–	–	45°	–	45°

^a All sheet tested was 0.0508 cm thick. No edge or surface cleaning or polishing.

^b Laboratory sheet was stress-relieved at 1400°C in hydrogen prior to the bend test. The bending punch speed of 2.5 cm per minute corresponds to Materials Advisory Board requirements. All other bend tests were conducted with a bending punch speed of 0.012 per minute as more appropriate to recrystallized material. Commercially produced sheet tested as-received from vendor.

^c Angle of bend to fracture using a 4x thickness radius punch is given.

^d Atomic percent.

molybdenum sheet fabricated from the powder metallurgy and the arc-cast processes. The room-temperature bend tests were conducted on sheets heated for 2 hours at 1500°, 1600°, 1800°, 2000°, and 2200°C.

The room-temperature bend ductility after holding at 2200°C was considered quite good for all compositions. Metallographically, both the molybdenum sheet and the sheets of Mo-Re appeared to be single-phase and devoid of any extraneous phases. The chemical determination of impurities in molybdenum alloys made by GE-NMPO has not as yet been completed. However, the impurity level is expected to be higher than desired as indicated by the voids observed in electron beam weld areas. These results are shown in Table 3.10.

TABLE 3.10

RESULTS OF ELECTRON-BEAM WELDING OF MOLYBDENUM TEST SPECIMENS

Laboratory Number	Nominal Composition	Processing Variable	Voids Revealed By X-Ray In 5.0 cm Weld	Room-Temperature Bend-Test Result Of Simulated Weld
1	Unalloyed Mo	None	Many	Not bent (voids)
1a	Unalloyed Mo	Purified	3 (small)	90 degrees (weld break)
1b	Unalloyed Mo	Purified	18 (1 large)	45 degrees (weld break)
2	Mo + 1 at. % Re	Purified	4 (small)	45 degrees (weld break)
3	Mo + 2 at. % Re	Purified	9 (2 large)	90 degrees (weld break)
4	Mo + 5 at. % Re	Purified	20 (small)	90 degrees ^a
D	Unalloyed Mo	Commercial (sintered)	Many	30 degrees
E	Unalloyed Mo	Commercial (arc-cast)	None	90 degrees

^aFractures appeared as two small intergranular cracks crossing from weld to adjacent base metal.

The arc-cast commercial molybdenum was found to be void-free in the weld zone and also withstood a 90-degree bend at room temperature. In contrast, the commercial sintered molybdenum sheet formed many voids in the electron-beam fused metal and the welded area broke after a 30-degree bend. Many large holes and voids also formed in the unpurified laboratory-processed unalloyed specimen (No. 1). Closely paralleling the initial bend ductility results shown in Table 3.9, the purified molybdenum (specimen No. 1a) and the two alloys containing 2 and 5 atomic percent Re (specimens No. 3 and 4, respectively) sustained 90-degree bends before fracturing in the weld. These results are believed to indicate that additional purification is required, even though purified molybdenum sheets were superior to the commercial sintered powder sheet both in weld porosity and weld ductility.

Fabrication of NMP-256 Alloy Into Primary Shapes

A pilot production program to fabricate 0.0254-cm-thick sheet, 0.5-cm-thick plate or discs, and small-diameter seamless tubing of the W - 25Re - 30Mo (NMP-256) alloy is being conducted to provide test assembly material and to establish fabrication procedures.

Over 70 pieces of the alloy have been produced or started in the production trial. Fifty of the sheets in process include longer and wider material than heretofore processed. To obtain the larger sheet size, 325-gram sintered metal powder compacts were cross-rolled to width, and finish hot-rolled, as shown in Figure 3.30. A new furnace was installed to permit rolling to the wider sheet size. The hot-rolled sheets were finished by cold-rolling to 10 by 30 by 0.101 cm.

Thick plates or discs for use as end caps on test assemblies are produced by extruding a solid bar of NMP-256 alloy. Final sizing is by hot-press upsetting to discs as shown in Figure 3.31.

The major development effort in the trial production of primary shapes has been directed to fabricating small seamless tubing. Tube fabrications begin with the extrusion of sintered powdered compacts at 1670°C using a molybdenum container and glass lubrication. Extrusion components are shown in Figure 3.32; the rod containing the extruded tube is shown in Figure 3.33. Size data for 18 complete extrusions produced during this report period are given in Table 3.11. These extrusions were planned mainly to provide the basic sizes for drawing to small tubing with and without intermediate processing steps. The designated use for each extrusion is also included in Table 3.11.

Fig. 3.30 – Six sheets of NMP-256 alloy hot-rolled finished, prior to final finishing by cold-rolling (Neg. P65-5-19)

Fig. 3.31 – Extruded bar of NMP-256 with discs obtained by sectioning the bar and hotpress-forging; discs are machined to test assembly end caps (Negs. P65-5-32 and P64-9-16A)

Four methods of secondary hot-working were evaluated. These included secondary extrusions, hot-drawing, hot-swaging, and hot-rolling. Currently, the preferred method is to hot-roll with the molybdenum sheath and core-filler of the original extrusion retained. The product of extrusion No. D-8 following hot-rolling is shown in Figure 3.34 with the molybdenum outer sheath removed by acid leaching from one tube-blank rod. The hot-rolled tube blank is straightened and worked to acceptable circularity by drawing through a die. Initial hot-drawing operations were successful. Concurrently, studies on lubrication and gripping techniques are being evaluated.

The generally excellent properties of the NMP-256 alloys produced as sheet was attributed, in part, to close structural control in sheet rolling. Therefore, tube drawing

TABLE 3.11
EXTRUSIONS COMPLETED ON PILOT PRODUCTION TRIAL OF NMP-256 ALLOY

Extrusion Number	Product Size, OD, cm	Extrusion Blank Before Extrusion				Final Extrusion		Ratio By Area Of Extrusion Reduction	Extruded Tube	
		Length, cm	OD, cm	ID, cm	Weight, g	Length, cm	OD At Center, cm		Ratio Of ID/OD	Designated Use
B	1.37	12.30	2.84	1.82	776.3	45.7	1.42	4.06:1	0.634	Various ^d
D-1 ^a	1.37	9.49	2.72	2.26	290.6	32.2	1.43	3.71:1	0.806	Various ^d
D-2	1.37	10.05	2.74	2.25	337.7	36.3	1.37	4.00:1	0.798	Hot-roll ^e
D-3	1.30	10.02	2.74	2.25	335.9	27.1	1.36	4.00:1	0.812	Hot-roll ^e
D-4	1.37	10.49	3.02	2.27	522.1	—	1.37 ^c	4.63:1	0.754	Hot-roll to 0.53 cm
D-5	1.37	10.50	2.97	2.26	513.0	—	1.37 ^c	4.48:1	0.756	Hot-roll to 0.53 cm
D-6	0.76	10.53	3.02	2.25	567.5	147.3	0.76 ^c	15.27:1	0.740	Extrude to 0.76 OD
D-7	0.76	10.95	2.97	2.26	531.5	166.6	0.76 ^c	14.83:1	0.740	Extrude to 0.76 OD
D-8	1.37	11.94	4.95	3.43	2011.2	150.1 ^b	1.37 ^c	12.50:1	0.690	Hot-roll to 0.53 cm ^b
D-9	1.37	11.94	4.95	3.43	2011.2	150.1 ^b	1.37 ^c	12.50:1	0.692	Hot-roll to 0.53 cm ^b
D-11	2.54	9.72	5.10	—	2842.0	39.1	2.54 ^c	4.03:1	—	Solid bar ^{f,g}
D-12	0.63	11.44	2.97	—	780.8	127.0	0.63 ^c	13.05:1	—	Solid bar ^h
D-10	0.68	11.74	2.89	1.95	658.2	—	0.63 ^c	—	0.675	Extrude to 0.68 OD
D-17	0.68	11.74	2.89	1.95	658.2	—	0.63 ^c	—	0.675	Extrude to 0.68 OD
D-18	2.54	9.72	5.10	—	2842.0	—	2.54 ^c	4.00:1	—	Solid bar ^{f,g}
D-19	2.54	9.72	5.10	—	2842.0	—	2.54 ^c	4.00:1	—	Solid bar ^{f,g}
D-20	0.68	11.74	2.89	1.95	658.2	—	0.63	—	0.675	Extrude to 0.68 OD
D-13	1.39									W – 25Re thermocouple wire – extrusion to yield 1800 grams of wire ⁱ

^aD-1 contained two sleeves extruded in tandem. D1-A data are given in the table. D1-B was 7.58 cm long – 2.70 cm OD by 2.28 cm ID, weighing 204.3 grams.

^bPreviously extruded blanks almost 5.08 cm in OD supplied for D-8 and D-9, extrusion lengths of 150.1 cm theoretical will hot-roll to 504 cm long each.

^cApproximate diameter.

^dPermits use of ironing type draws on tungsten carbide solid mandrels of 1.20-cm OD with dies of 1.37 cm down to 1.27 cm. Also permits survey of gripping problems in addition to a basic lubricant evaluation.

^eExtrusions D-2 and D-3 were committed to making 0.20-cm OD BY 0.15-cm ID tubing before the heavier walled tube of 0.15-cm ID was requested.

^f2.54-cm OD solid bar as required for hot-forging to thick discs for tube sheets.

^gUsed sintered tungsten end blocks to suppress turbulent metal flow.

^hBlended originally to NMP-306 alloy; rebled to NMP-256 composition. Intended as pull rod and mandrel wires. Starting material purity is questionable.

ⁱMaterial extruded at 1930°C – wire to be drawn from extruded rod by commercial vendor.

Fig. 3.32—Sintered and machined tubular extrusion blank shown with molybdenum extrusion container, core filler, container closure plug and Zr-coated hardened extrusion die (Neg P65-441B)

Fig. 3.33—Extruded molybdenum bars containing tubular NMP-256 alloy tubes. The lower bar contains 140 cm OD tubing made from extrusion shown above. (Neg P65 5 13)

schedules have directly duplicated the reduction – stress relieving factors used in the sheet rolling program. As a result, all drawing operations are conducted at 200°C maximum to prevent absorption of atmospheric or lubricating contaminants by the tube. A total of 121 die stages with matching mandrels were obtained for this work; all tooling is of the carbide type.

WORK PLANNED FOR NEXT PERIOD

1. Analysis of first-stage creep data will be continued to evaluate various correlations for arc-cast tungsten as well as other materials.
2. Metallurgical evaluations of the W-Re-Mo alloys creep-rupture tested at 2200°C will be performed.
3. Creep-rupture comparisons of tungsten sheet from the Navy Sheet Rolling Program as well as other sources will be made at 1600°C .
4. Development of capabilities to allow creep-rupture testing of refractory metals in vacuum at temperatures from 1600° to 2000°C will be initiated.

Fig. 3.34—Tubular extrusion of NMP-256 alloy following hot-rolling; molybdenum of the extrusion container is retained on the outer surface and in the inner core. Hot-rolled tube at bottom was leached to remove molybdenum. (Neg P65-6-2)

5. Trial production effort for producing high-purity, high-quality sheet, plate, rod, and tubing as required to complete test assemblies of new alloys developed under this program will be continued.¹²
6. Creep-rupture evaluations of new W-Re-Mo alloys will be initiated on 18 compositions to confirm and extend the general knowledge of the effect of composition in areas previously found to be a major interest.¹²
7. Purification techniques applied to unalloyed molybdenum and low rhenium content molybdenum alloys will be further evaluated.¹²
8. Alloys with high tungsten and very low rhenium content, processed with and without special purification techniques, will be evaluated for their fabricability and short-time high-temperature properties.¹²
9. Thirty small button-type specimens will be evaluated in continuation of work on the Mo-Cr-Re system.¹²
10. Work will continue on establishing the mechanical and metallurgical quality desired for small seamless W-Re-Mo tubing, including weldability and surface texture.¹²

¹²The alloy development work described under this job will be performed under Task No. 57015 in FY-66.

4. EFFECT OF RADIATION ON HIGH-TEMPERATURE METALS AND ALLOYS

(57004)

The objective of this program is to determine the effect of radiation on the time-, temperature-, and stress-dependent properties of selected high-temperature alloys and refractory metals, to identify the causes of any observed changes in these properties, and to develop remedial measures.

The radiation effects studies are divided into two separate materials categories. The first (Job 57004)¹ considers a family of refractory metals and alloys, and the second (Job 57018), designated as the heat-resistant metals and alloys program, includes the materials A-286, Hastelloy X, Hastelloy N, Fe-Cr-Al-Y alloys, and Type 304 stainless steel.

REFRACTORY-METALS AND ALLOYS PROGRAM

The experimental program to study the effects of neutron irradiation on the creep-rupture, tensile, resistivity, and hardness properties of refractory metals and alloys is continuing. In addition to studies on the above properties, theoretical calculations on the nature of the neutron-induced defects and the direct observations of defects by the use of the transmission electron microscope are also being performed.

Some work on the measurement of the creep-rupture properties of irradiated W - 25Re specimens at temperatures of 800°C and 900°C, the hot-hardness of unirradiated worked molybdenum specimens, and the resistivity recovery studies of irradiated molybdenum specimens annealed in the temperature region of $0.31 T_m^2$ are discussed. A summary of the experimental program, test conditions, and preliminary results is presented in Table 4.1.

Status of Irradiations

The ORR series of dose-dependency irradiations³ is nearing completion. The status of the current irradiations is shown in Table 4.2. Capsule ORM-36 was returned to Evendale and disassembled. Capsule ORM-37 was irradiated and is awaiting return to GE-NMPO. ORM-35 is being irradiated in the ORR-A2 facility.

Capsule ORM-38 containing 16 molybdenum sheet creep-rupture specimens, 15 tungsten tensile specimens, and 9 tungsten and 5 molybdenum resistivity specimens is in the process of assembly. After leak checking, it will be shipped to ORNL for insertion in the ORR and will be irradiated to a fast neutron dose of approximately 5.4×10^{19} nvt.

¹Studies performed under this task will be reported as two separate jobs (57004 and 57018) starting in Fiscal Year 1966. They will appear in the even numbered unclassified progress reports beginning with GEMP-50A.

² T_m is the absolute melting temperature of the metal.

³"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, p. 85.

TABLE 4.1
SUMMARY OF MAJOR EXPERIMENTS IN THE REFRACTORY METALS AND ALLOYS RADIATION EFFECTS PROGRAM

Exp. No.	Objective	Property	Test Specimen		Irradiation Conditions ^c			Post-Irradiation Heat Treatment		Test Conditions	Percent Change In Property ^d			Comments					
			Material ^a	Type	Lot Number ^b	Capsule Number	Temp, °C	nvt, neutrons/cm ² Thermal (E _n ≥1 Mev)	Temp, °C		Time, hr	Creep rate	Rupture life		Elongation				
1	To investigate the effects of neutron irradiation on the creep-rupture and tensile properties. To perform supporting studies on the effects of neutron irradiation on the hardness and resistivity properties.	Creep rupture	W	Rod	MK137(PM)-X	ORM-4	70	2.2 x 10 ¹⁸	5.8 x 10 ¹⁷			T, °C	σ(kg/mm ²)	Creep rate	Rupture life	Elongation	Warm-worked. To be tested. To be tested. To be tested.		
					MK137(PM)-X	ORM-8	70	9.5 x 10 ¹⁹	2.5 x 10 ¹⁹			1100	19.34	- 10.0	15	- 1			
					MK137(PM)-Y	ORM-6	70	2.0 x 10 ¹⁹	3.7 x 10 ¹⁸			1100	20.35	-	17	-			
					MK137(PM)-Y	ORM-8	70	9.5 x 10 ¹⁹	2.5 x 10 ¹⁹			1100	20.35	- 60	160	- 7			
					MK137(PM)-E	ORM-17	70	7.5 x 10 ¹⁹	1.6 x 10 ¹⁹			1100	18.28	- 16	28	-10			
					MK137(PM)-E	ORM-19	70	3.9 x 10 ²⁰	8.2 x 10 ¹⁹			1100	18.28	-76	490	- 6			
					MK137(PM)-Y	ORM-6	70	2.0 x 10 ¹⁹	3.7 x 10 ¹⁸			1400	13.01	-	1	10			
					MK137(PM)-Y	ORM-8	70	9.5 x 10 ¹⁹	2.5 x 10 ¹⁹			1400	13.01	-	9	14			
					MK137(PM)-J	ORM-19	70	3.9 x 10 ²⁰	8.2 x 10 ¹⁹			1400	10.55	- 6	38	19			
					MK137(PM)-J	ORM-19	70	3.9 x 10 ²⁰	8.2 x 10 ¹⁹			1500	8.44	- 8	10	-29			
					MK137(PM)-J	ORM-19	70	3.9 x 10 ²⁰	8.2 x 10 ¹⁹			1700	5.82	- 13	9	-28			
					MK137(PM)-E	33MT-137	1300	2.2 x 10 ²⁰	1.8 x 10 ²⁰			900							
					MK137(PM)-J	ORM-17	70	7.5 x 10 ¹⁹	1.6 x 10 ¹⁹			900	22.64	- 76	280	-26			
					W Sheet PF(PM)	ORM-12	70	9.1 x 10 ¹⁹	3.2 x 10 ¹⁹			1100	37.26	- 30	60	8			
					W - 25Re Sheet RWS9(PM)-4	ORM-20	70	3.5 x 10 ²⁰	8.0 x 10 ¹⁹			1100	43.9	- 52	55	-56			
					Mo Sheet KDM-515A(AC)-8	ORM-23	70	5.2 x 10 ¹⁹	1.4 x 10 ¹⁹			580	21.09	29	- 96	- 4			
					KDM-515A(AC)-2	ORM-13	70	5.5 x 10 ¹⁹	6.9 x 10 ¹⁸			980	10.55	- 2	10	14			
					KDM-515A(AC)-1	ORM-16	70	1.4 x 10 ²⁰	3.7 x 10 ¹⁹			580	21.09	210	- 65	-			
					KDM-515A(AC)-1	ORM-16	70	1.4 x 10 ²⁰	3.7 x 10 ¹⁹			600	21.09	57	- 32	-			
					50Mo-50Re Sheet	ORM-20	70	3.5 x 10 ²⁰	8.0 x 10 ¹⁹										
					Ta Sheet	ORM-23	70	5.2 x 10 ¹⁹	1.4 x 10 ¹⁹										
					Ta - 10W Sheet	ORM-23	70	5.2 x 10 ¹⁹	1.4 x 10 ¹⁹										
		Tensile	W	Rod	MK142(PM)-G	33MT-138	70	3.6 x 10 ²⁰	1.3 x 10 ²⁰			371		Yield Strength (Brittle fracture)	Tensile Strength	Elongation	Testing continuing at higher temperatures. To be tested. To be tested.		
					MK142(PM)-B	ORM-9	70	2.8 x 10 ¹⁹	5.1 x 10 ¹⁸										
					MK142(PM)-C	ORM-10	70	9.3 x 10 ¹⁹	1.7 x 10 ¹⁹										
		Resistivity	W	Rod	MK142(PM)-E	ORM-7	70	2.5 x 10 ¹⁹	5.3 x 10 ¹⁸			-196		Resistivity			Warm-worked.		
					MK142(PM)-D	ORM-11	70	1.2 x 10 ²⁰	3.3 x 10 ¹⁹			-196		105					
					MK142(PM)-F	33MT-113	1300	2.2 x 10 ²⁰	8.0 x 10 ¹⁹			-196		315					
					W - 25Re Rod RWW5(PM)-A	ORM-14	70	6.2 x 10 ¹⁹	1.2 x 10 ¹⁹			-196		39					
			W - 25Re Rod		RWW5(PM)-C	33MT-113	1300	2.2 x 10 ²⁰	8.0 x 10 ¹⁹			-196		10					
					GEH(PM)-B	ORM-14	70	6.2 x 10 ¹⁹	1.2 x 10 ¹⁹			-196		15					
					GEH(PM)-C	ORM-14	70	6.2 x 10 ¹⁹	1.2 x 10 ¹⁹			-196		80					
			Mo-TZM Rod		GEH(PM)-L	33MT-113	700	2.2 x 10 ²⁰	8.0 x 10 ¹⁹			-196		118					
					ZM6003(AC)-5	ORM-18	70	1.1 x 10 ²⁰	3.7 x 10 ¹⁹			-196		2					
					ZM6003(AC)-5	33MT-113	700	2.2 x 10 ²⁰	8.0 x 10 ¹⁹			-196		74					
		Hardness	W	Sheet	411-1	ORM-15	70	1.3 x 10 ²⁰	3.5 x 10 ¹⁹			15	500 grams	DPH Hardness			Single crystal. From isochronal annealing studies. Polycrystal. From isochronal annealing studies.		
												650		8					
												1300		60					
												15	500 grams	5					
												300		18					
												1200		55					
														0					

TABLE 4.1 (Cont.)
SUMMARY OF MAJOR EXPERIMENTS IN THE REFRACTORY METALS AND ALLOYS RADIATION EFFECTS PROGRAM

Exp. No.	Objective	Property	Test Specimen		Irradiation Conditions ^c			Post-Irradiation Heat Treatment		Test Conditions	Percent Change In Property ^d			Comments						
			Material ^a	Type	Lot Number ^b	Capsule Number	Temp, °C	nt, neutrons/cm ² Thermal (E _n ≥ 1 Mev)	Temp, °C		Time, hr	Creep rate	Rupture life		Elongation					
2	To determine the influence of post-irradiation heat treatment on the creep-rupture and tensile properties. To perform supporting post-irradiation annealing studies on the resistivity and hardness properties and to make direct observations on the sub-structure.	Creep rupture	W	Rod	MKI37(PM)-Y	ORM-6	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1100	1	T, °C 1100	20.35	Creep rate - 27	13	Elongation 4	Warm-worked.			
						ORM-8	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1400	1	1100	20.35	16	- 15	-				
						ORM-8	70	9.5 × 10 ¹⁸	2.5 × 10 ¹⁹	1400	1	1100	20.35	- 73	190	-				
						ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1400	1	1100	18.28	- 64	210	- 6				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1650	1	900	22.64	- 74	520	-10				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1200	1	900	22.64	- 91	900	-17				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1300	1	900	22.64	- 61	190	- 7				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1400	1	900	22.64	- 56	160	-				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1700	1	900	22.64	- 46	90	- 7				
						ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1700	43	900	-	- 80	710	4				
						ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1700	1	900	-	- 98	-	-				
						ORM-12	70	1.5 × 10 ²⁰	3.2 × 10 ¹⁹	1100	3	1100	37.26	- 39	51.9	- 4				
						ORM-23	70	5.2 × 10 ¹⁹	1.4 × 10 ¹⁹	757	8	580	21.09	- 27	62.0	-20.8				
						ORM-23	70	5.2 × 10 ¹⁹	1.4 × 10 ¹⁹	779	8	580	21.09	- 28	-	-				
		Tensile	W	Rod	MKI42(PM)-B	ORM-9	70	2.5 × 10 ¹⁹	5.1 × 10 ¹⁸					Yield Strength	Tensile Strength	Elongation	To be tested. To be tested.			
						ORM-10	70	9.3 × 10 ¹⁹	1.7 × 10 ¹⁹											
						Resistivity	W	Rod	MKI42(PM)-D	ORM-7	70	2.5 × 10 ¹⁹	5.3 × 10 ¹⁸	650	1	-196			Resistivity 8	
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	650	1	-196		12		
		ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹					650	1	-196	72							
		ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹					1900	1	-196	1							
		ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹					550	1	-196	1							
		ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹					450	1	-196	14							
		Hardness	W	Sheet	411-1	ORM-15	70	1.3 × 10 ²⁰	3.5 × 10 ¹⁹	650	1	Load 650 500 grams	DPH Hardness 80	Single crystal.						
	3	To investigate the influence of specimen configuration, purity level, structure and substructure on the post-irradiation creep-rupture and tensile properties. To perform supporting studies on the post-irradiation resistivity and hardness properties.	Creep-rupture	W	Rod	MKI37(PM)-Y	ORM-6	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1100	1	T, °C 1100	20.35	Creep rate - 27	13	Elongation 4	Warm-worked.		
							ORM-8	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1400	1	1100	20.35	16	- 15	-			
							ORM-8	70	9.5 × 10 ¹⁸	2.5 × 10 ¹⁹	1400	1	1100	20.35	- 73	190	-			
							ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1400	1	1100	18.28	- 64	210	- 6			
ORM-17							70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1650	1	900	22.64	- 74	520	-10				
ORM-17							70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1200	1	900	22.64	- 91	900	-17				
ORM-17							70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1300	1	900	22.64	- 61	190	- 7				
ORM-17							70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1400	1	900	22.64	- 56	160	-				
Tensile			W	Rod	MKI42(PM)-B	ORM-9	70	2.5 × 10 ¹⁹	5.1 × 10 ¹⁸					Yield Strength	Tensile Strength	Elongation	To be tested. To be tested.			
						ORM-10	70	9.3 × 10 ¹⁹	1.7 × 10 ¹⁹											
						Resistivity	W	Rod	MKI42(PM)-D	ORM-7	70	2.5 × 10 ¹⁹	5.3 × 10 ¹⁸	650	1	-196			Resistivity 8	
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	650	1	-196		12		
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	650	1	-196		72		
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	1900	1	-196		1		
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	550	1	-196		1		
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	450	1	-196		14		
Hardness		W	Sheet	411-1	ORM-15	70	1.3 × 10 ²⁰	3.5 × 10 ¹⁹	650	1	Load 650 500 grams	DPH Hardness 80	Single crystal.							
4		To determine the in-reactor creep properties of refractory metals and alloys.	Creep-rupture	W	Rod	MKI37(PM)-Y	ORM-6	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1100	1	T, °C 1100	20.35	Creep rate - 27	13	Elongation 4	Warm-worked.		
							ORM-8	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1400	1	1100	20.35	16	- 15	-			
							ORM-8	70	9.5 × 10 ¹⁸	2.5 × 10 ¹⁹	1400	1	1100	20.35	- 73	190	-			
							ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1400	1	1100	18.28	- 64	210	- 6			
							ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1650	1	900	22.64	- 74	520	-10			
							ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1200	1	900	22.64	- 91	900	-17			
							ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1300	1	900	22.64	- 61	190	- 7			
							ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1400	1	900	22.64	- 56	160	-			
			Tensile	W	Rod	MKI42(PM)-B	ORM-9	70	2.5 × 10 ¹⁹	5.1 × 10 ¹⁸					Yield Strength	Tensile Strength	Elongation	To be tested. To be tested.		
							ORM-10	70	9.3 × 10 ¹⁹	1.7 × 10 ¹⁹										
	Resistivity						W	Rod	MKI42(PM)-D	ORM-7	70	2.5 × 10 ¹⁹	5.3 × 10 ¹⁸	650	1	-196			Resistivity 8	
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	650	1	-196	12			
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	650	1	-196	72			
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	1900	1	-196	1			
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	550	1	-196	1			
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	450	1	-196	14			
Hardness	W	Sheet	411-1	ORM-15	70	1.3 × 10 ²⁰	3.5 × 10 ¹⁹	650	1	Load 650 500 grams	DPH Hardness 80	Single crystal.								
5	To determine the in-reactor creep properties of refractory metals and alloys.	Creep-rupture	W	Rod	MKI37(PM)-Y	ORM-6	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1100	1	T, °C 1100	20.35	Creep rate - 27	13	Elongation 4	Warm-worked.			
						ORM-8	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1400	1	1100	20.35	16	- 15	-				
						ORM-8	70	9.5 × 10 ¹⁸	2.5 × 10 ¹⁹	1400	1	1100	20.35	- 73	190	-				
						ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1400	1	1100	18.28	- 64	210	- 6				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1650	1	900	22.64	- 74	520	-10				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1200	1	900	22.64	- 91	900	-17				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1300	1	900	22.64	- 61	190	- 7				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1400	1	900	22.64	- 56	160	-				
		Tensile	W	Rod	MKI42(PM)-B	ORM-9	70	2.5 × 10 ¹⁹	5.1 × 10 ¹⁸					Yield Strength	Tensile Strength	Elongation	To be tested. To be tested.			
						ORM-10	70	9.3 × 10 ¹⁹	1.7 × 10 ¹⁹											
						Resistivity	W	Rod	MKI42(PM)-D	ORM-7	70	2.5 × 10 ¹⁹	5.3 × 10 ¹⁸	650	1	-196			Resistivity 8	
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	650	1	-196		12		
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	650	1	-196		72		
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	1900	1	-196		1		
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	550	1	-196		1		
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	450	1	-196		14		
Hardness	W	Sheet	411-1	ORM-15	70	1.3 × 10 ²⁰	3.5 × 10 ¹⁹	650	1	Load 650 500 grams	DPH Hardness 80	Single crystal.								
6	To determine the in-reactor creep properties of refractory metals and alloys.	Creep-rupture	W	Rod	MKI37(PM)-Y	ORM-6	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1100	1	T, °C 1100	20.35	Creep rate - 27	13	Elongation 4	Warm-worked.			
						ORM-8	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1400	1	1100	20.35	16	- 15	-				
						ORM-8	70	9.5 × 10 ¹⁸	2.5 × 10 ¹⁹	1400	1	1100	20.35	- 73	190	-				
						ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1400	1	1100	18.28	- 64	210	- 6				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1650	1	900	22.64	- 74	520	-10				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1200	1	900	22.64	- 91	900	-17				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1300	1	900	22.64	- 61	190	- 7				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1400	1	900	22.64	- 56	160	-				
		Tensile	W	Rod	MKI42(PM)-B	ORM-9	70	2.5 × 10 ¹⁹	5.1 × 10 ¹⁸					Yield Strength	Tensile Strength	Elongation	To be tested. To be tested.			
						ORM-10	70	9.3 × 10 ¹⁹	1.7 × 10 ¹⁹											
						Resistivity	W	Rod	MKI42(PM)-D	ORM-7	70	2.5 × 10 ¹⁹	5.3 × 10 ¹⁸	650	1	-196			Resistivity 8	
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	650	1	-196		12		
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	650	1	-196		72		
										ORM-11	70	1.2 × 10 ²⁰	3.3 × 10 ¹⁹	1900	1	-196		1		
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	550	1	-196		1		
										ORM-14	70	6.2 × 10 ¹⁹	1.2 × 10 ¹⁹	450	1	-196		14		
Hardness	W	Sheet	411-1	ORM-15	70	1.3 × 10 ²⁰	3.5 × 10 ¹⁹	650	1	Load 650 500 grams	DPH Hardness 80	Single crystal.								
7	To determine the in-reactor creep properties of refractory metals and alloys.	Creep-rupture	W	Rod	MKI37(PM)-Y	ORM-6	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1100	1	T, °C 1100	20.35	Creep rate - 27	13	Elongation 4	Warm-worked.			
						ORM-8	70	2.0 × 10 ¹⁸	3.7 × 10 ¹⁸	1400	1	1100	20.35	16	- 15	-				
						ORM-8	70	9.5 × 10 ¹⁸	2.5 × 10 ¹⁹	1400	1	1100	20.35	- 73	190	-				
						ORM-19	70	3.9 × 10 ²⁰	8.2 × 10 ¹⁹	1400	1	1100	18.28	- 64	210	- 6				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1650	1	900	22.64	- 74	520	-10				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1200	1	900	22.64	- 91	900	-17				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1300	1	900	22.64	- 61	190	- 7				
						ORM-17	70	7.5 × 10 ¹⁹	1.6 × 10 ¹⁹	1400	1	900	22.64	- 56	160	-				
		Tensile	W	Rod	MKI42(PM)-B	ORM-9	70	2.5 × 10 ¹⁹	5.1 × 10 ¹⁸					Yield Strength	Tensile Strength	Elongation	To be tested. To be tested.			
						ORM-10	70	9.3 × 10 ¹⁹	1.7 × 10 ¹⁹											
						Resistivity	W	Rod	MKI42(PM)-D	ORM-7										

^aAll materials are in the recrystallized condition unless otherwise noted.

^bMaterial lot numbers are followed by parenthetical expressions denoting powder metallurgy (PM) or arc-cast (AC) processing. The last letter or number identifies the sheet or rod stock fabricated from the specified lot. Seven or more test specimens are generally machined from each sheet or rod stock.

^cThe listed irradiation conditions represent approximate values for specimens which were irradiated and represent target values for specimens to be irradiated. A summary of capsule irradiations, designations, and the number and type of specimens is given in Table 2.2.

^dProperty changes represent increases due to the indicated irradiations and test conditions when compared to a corresponding unirradiated test specimen. Negative values represent a decrease in the specified property.

TABLE 4.2
IRRADIATION DATA FOR REFRACTORY-METAL CAPSULES

Capsule	Test Specimens		Quantity	Facility	Irradiation Conditions ^a		
	Material	Type ^b			Exposure, hr	Dosage, n/cm ² Thermal	(E _n ≥ 1 Mev)
ORM-31	W	CR(R)	4	ORR-A2	400	c	c
	W	H-R	2				
	W	T	2				
ORM-32	W	CR(R)	4	ORR-A2	20	5.8 x 10 ^{18d}	2.9 x 10 ^{18d}
	W	H-R	2				
	W	T	2				
ORM-33	W	CR(R)	4	ORR-A2	80	2.3 x 10 ^{19d}	1.2 x 10 ^{19d}
	W	H-R	2				
	W	T	2				
ORM-34	W	CR(R)	4	ORR-A2	480	1.4 x 10 ^{20d}	6.9 x 10 ^{19d}
	W	H-R	2				
	W	T	2				
ORM-35	W	CR(R)	4	ORR-A2 (Being irradiated)		3.0 x 10 ^{20e}	1.5 x 10 ^{20e}
	W	H-R	2				
	W	T	0				
ORM-36	W	CR(R)	4	ORR-A2	813	2.5 x 10 ^{20d}	1.2 x 10 ^{20d}
	W	H-R	2				
	W	T	2				
ORM-37 ^f	W	CR(R)	4	ORR-A2		4 x 10 ^{19e}	6.9 x 10 ^{19e}
	W	H-R	2				
	W	T	2				
ORM-38	Mo	CR(F)	16	ORR (Being assembled)		3.0 x 10 ^{20e}	5.4 x 10 ^{19e}
	Mo	H-R	5				
	W	H-R	9				
	W	T	15				

^aAll irradiations at reactor ambient temperature.

^bCoded as follows: CR = creep-rupture specimen

(R) = round specimen

(F) = flat type

H-R = hardness and resistivity

T = tensile specimen

^cCapsule inadvertently positioned slightly above reactor midplane. Estimation of dosage cannot be made until dosimeters are processed and data evaluated.

^dEstimated dosage.

^eRequested dosage.

^fSpecimens are shielded by a cadmium jacket.

The design of the ORM-38 capsule consists of modifications of the single-stage ORM-31 capsule features. Details of this new capsule can be seen in Figures 4.1 and 4.2. The two lower stages designed to contain round specimens are similar to the slotted double concentric shell design⁴ of ORM-31 except for the number and location of the slots and holes. Figure 4.1 shows the details of the center stage loaded with resistivity specimens. Five of the specimen positions have radial slots for the resistivity specimens with integrally welded wire emf measuring and current lead tabs. The partially assembled ORM-38 capsule is shown in Figure 4.2. The newly designed segmented sheet specimen holder is shown mounted on the central cooling water tube. Not shown in this photograph are the wedge blocks and draw bolts that force the segments against the inside wall of the capsule and against the specimens for optimum heat transfer.

⁴"High-Temperature Materials Program Progress Report No. 43, Part A," GE-NMPO, GEMP-43A, January 29, 1965, p. 41, and Figures 4.1 and 4.2, p. 45.

Fig. 4.1—Photograph of capsule ORM-38 showing stage containing resistivity specimens (Neg. P65-6-10C)

Fig. 4.2—Photograph of capsule ORM-38 showing stage containing flat creep-rupture specimens (Neg. P65-6-10B)

A new series of ambient-temperature capsules is planned for ETR irradiation. These irradiations will be used primarily for the high-dose conditions of the dose-dependency studies discussed above. The first two capsules, designated GEFP2-232 and -233, will contain tungsten tensile, creep-rupture, and resistivity specimens. They will be irradiated for a full reactor cycle and a half reactor cycle, respectively. A third capsule (GEFP2-234) is scheduled to be irradiated for a period of two reactor cycles. It will contain molybdenum resistivity and creep-rupture specimens in addition to the tungsten specimens that will be used in the dose-dependency studies. Each capsule will contain a total of 21 specimens loaded into seven tubes of a new capsule design.

W - 25Re Creep-Rupture Tests

The results of 800°C and 900°C creep-rupture tests on W - 25Re sheet specimens are presented in Table 4.3. A stress versus creep rate plot is shown in Figure 4.3. The tests at 900°C show very little difference in creep rates between control specimens and those specimens irradiated to a fast neutron dose of about 2×10^{19} nvt (ORM-21). The rupture lives of the two irradiated specimens were greater than the control specimens by factors of 1.4 and 2.2 (Figure 4.4).

TABLE 4.3
SUMMARY OF CREEP-RUPTURE TESTS ON W - 25Re SPECIMENS^a

Specimen No.	Specimen Sheet No.	Condition	Test Temperature, °C	Stress, kg/mm ²	Linear Creep Rate, sec ⁻¹	Rupture Life, hr	Elongation, percent in 2.54 cm gage length
736	3	Control	800	70.31	1.83×10^{-7}	(24 - 309) ^b	(36.73) ^c
737	3	Control	800	77.34	1.95×10^{-6}	24.2	—
731	3	ORM-21 ^d	800	77.34	6.31×10^{-8}	382.2	30.4
735	3	ORM-21 + anneal ^e	800	77.34	2.19×10^{-7}	139	30.54
740	4	Control	800	75.70	1.18×10^{-6}	30.3	28.8
751	4	ORM-21	800	75.70	1.53×10^{-8}	(28 - 353)	—
			842	75.70	2.55×10^{-7}	(353 - 388)	(27.57) ^c
744	4	ORM-20 ^f	800	75.70	1.35×10^{-8}	(0 - 235)	—
			840	75.70	2.13×10^{-8}	(235 - 598)	Test discontinued
721	2 ^g	ORM-16 ^h	800	70.39	1.31×10^{-7}	283.1	32.6
722 ⁱ	2 ^g	ORM-16 + anneal ^j	800	70.39	5.83×10^{-7}	56.77	—
727	2 ^g	Control	800	70.39	2.35×10^{-7}	182.6	33.5
739	3	Control	900	66.79	3.58×10^{-6}	14.56	42.1
738	3	Control	900	70.31	1.34×10^{-5}	3.87	33.1
730	3	Control	900	73.83	3.00×10^{-5}	1.30	41.0
733	3	ORM-21	900	73.83	2.50×10^{-5}	1.75	35.7
734	3	ORM-21	900	70.31	6.79×10^{-6}	8.63	38.2
728	3	ORM-20 + anneal ^k	900	70.31	7.69×10^{-8}	(481 - 647)	—
			900	73.83	$\sim 2.63 \times 10^{-7}$	(647 - 698)	(24.0) ^c
747	4	Control	900	63.28	2.56×10^{-6}	21.44	37.26

^aSpecimen from 0.051-cm sheet recrystallized by vendor at 1600°C for 0.5 hour in hydrogen and annealed at 1750°C for 1 hour in hydrogen following specimen fabrication, unless otherwise noted.

^bNumbers in parentheses indicate time interval in which linear creep rates were determined for those specimens which were tested under different conditions.

^cFinal elongation for those specimens which were tested under different conditions.

^dCapsule ORM-21 irradiated in ORR-F2 at reactor ambient temperature to an approximate dosage of 2×10^{19} nvt ($E_n \geq 1$ Mev).

^eAnnealed at 1494°C for 30 hours in hydrogen.

^fCapsule ORM-20 irradiated in ORR-F2 at reactor ambient temperature to an approximate dosage of 8×10^{19} nvt ($E_n \geq 1$ Mev).

^gAnnealed at 1900°C for 1 hour in hydrogen following specimen fabrication.

^hCapsule ORM-16 irradiated in ORR-F2 at reactor ambient temperature to an approximate dosage of 3.7×10^{19} nvt ($E_n \geq 1$ Mev).

ⁱA power failure occurred when on test for 42 hours and specimen cooled under load; test was restarted and continued on to failure.

^jAnnealed at 1700°C for 30 hours in hydrogen.

^kAnnealed at 1700°C for 1 hour in hydrogen.

Fig. 4.3 – Linear creep rate of W – 25Re specimens tested at 800°C and 900°C in hydrogen

Fig. 4.4 – Stress-rupture strength of W – 25Re specimens tested at 900°C in hydrogen

Specimen No. 728, which was irradiated in ORM-20 to a fast neutron dose ($\sim 8 \times 10^{19}$ nvt) that was a factor of about four greater than those irradiated in capsule ORM-21, was post-irradiation annealed at 1700°C for 1 hour in a hydrogen atmosphere and then creep-rupture tested at 900°C. As a result of irradiation to this higher dosage, the specimen exhibited a creep-rate which was approximately two orders of magnitude lower than that observed for the control specimen (No. 730) or for the specimen (No. 733) irradiated at the lower dosage when tested at the same stress level. It is evident that annealing at 1700°C did not remove the irradiation-induced strengthening of W - 25Re. Similar irradiation hardening has been observed⁵ in tungsten specimens irradiated to the higher dose level.

At a test temperature of 800°C, the specimens irradiated to fast neutron dosages of 2×10^{19} (Nos. 731 and 751) and 8×10^{19} nvt (No. 744) showed approximately the same decrease in creep rate when compared to control specimens. This is in contrast to the results at 900°C where a pronounced decrease in the creep rates was observed for the specimens irradiated to 8×10^{19} nvt but not for those irradiated to 2×10^{19} nvt. Further testing of W - 25Re will be required before any mechanism for the apparent saturation irradiation hardening effect observed at 800°C may be postulated.

Additional annealing studies were conducted in the 800°C tests. One specimen (No. 735) post-irradiation annealed at 1494°C ($0.54 T_m$) for 30 hours in a hydrogen atmosphere showed an increase in the creep rate and a significant decrease in the rupture life. Elongation-versus-time curves for the as-irradiated, irradiated plus annealed, and control specimens are shown in Figure 4.5. The marked difference in the creep properties between the as-irradiated specimen and the control specimen is apparent. Specimen total elongations do not appear to be significantly affected by the irradiations. The ductility of W - 25Re, similar to that of the Mo and W specimens also irradiated at ambient reactor temperatures, was not significantly affected as a result of neutron irradiation to doses up to about 8×10^{19} nvt. For the annealed specimen, although the 1494°C annealing temperature is greater than one-half the absolute melting temperature (based on an estimated 3000°C solidus temperature for W - 25Re), annealing times longer than 30 hours apparently are required to reach complete recovery. It has been shown⁶ that annealing irradiated tung-

Fig. 4.5 - Elongation versus time for W - 25Re specimens tested at 800°C and 73.34 kg/mm²

⁵"Fourth Annual Report - High-Temperature Materials and Reactor Component Development Programs, Volume I - Materials," GE-NMPO, GEMP-334, February 26, 1965, Figure 2.1, p. 59.

⁶Ibid., Figure 2.16, p. 69.

sten specimens at $0.54 T_m$ for 30 hours results in complete recovery, even for specimens irradiated to the higher neutron dose.

Resistivity Tests

Equipment Modifications - Additional modifications were made to the specimen holding fixture for use in measuring the resistivity at liquid nitrogen temperatures (-196°C). As shown in Figure 4.6, one of the copper current contacts was replaced with a removable Teflon cup containing mercury. This method allows more flexibility for testing various specimens and particularly for testing those specimens which have small pieces broken from the ends as a result of the remote handling treatments. Also, the knife edges formerly used for measuring emf were replaced with a small metal block containing a V-groove machined to position a 0.05 cm diameter tungsten wire so that it would contact the specimen normal to its axis. Measuring the emf values with the knife edge technique was satisfactory except that the edges would become blunted and consequently had to be replaced rather frequently. With the new wire-type emf contacts, short lengths (1.2 cm) of tungsten wire can be used for several measurements and then replaced before the bearing surface area is appreciably changed. This new emf measuring technique requires less contact pressure (applied torque)⁷ and also results in a more consistent separation distance which is not as sensitive to contact pressure as that observed with the knife edge technique.

Fig. 4.6 - Sketch of resistivity specimen holding fixture

Isothermal Resistivity Recovery Measurements at -196°C - Annealing studies were initiated on molybdenum resistivity specimens that were irradiated to a fast neutron dose of about 3×10^{19} nvt. These specimens will be annealed at homologous temperatures similar to those considered for the tungsten specimens⁷ discussed in the previous progress report. The resistivity recovery due to the migration of irradiation-induced defects in the temperature region of $0.31 T_m$ ($\sim 620^\circ\text{C}$) will be investigated. Since annealing was initiated on only one of the four irradiated specimens and the two control specimens at one temperature and only for a time period of 180 minutes, the preliminary data will not be presented in this progress report. It is planned to anneal the molybdenum specimens for much longer times at each temperature than were the tungsten specimens.⁸

Elevated-Temperature Resistivity Measurements - Resistivity measurements on unirradiated tungsten specimens as a function of temperature up to 1130°C showed good reproducibility when measurements were made on the cooling leg of the test.⁹

⁷"High-Temperature Materials Program Progress Report No. 41, Part A," GE-NMPO, GEMP-41A, November 30, 1964, p. 52.

⁸"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, pp. 90-92.

⁹Ibid., p. 93.

Isothermal studies were initiated on an irradiated tungsten specimen. Since the emf leads are welded to the test specimen and are not disturbed throughout the test, the uncertainty in the effective specimen length has been essentially eliminated when compared to that encountered with the technique using knife edge contacts. The elevated-temperature resistivity fixture showed good performance throughout the test. It is estimated that resistivity changes as small as 0.05 percent can be detected. Because of the high sensitivity and also because tungsten metal has a relatively large thermal coefficient of resistivity, the furnace temperature control became the limiting factor.

With the present furnace temperature control system, the best temperature variation that could be maintained was $\pm 0.1^\circ\text{C}$ at a value of 292.5°C for times less than 1 hour. Over a longer period of test time, the temperature variations occasionally ranged up to $\pm 0.5^\circ\text{C}$. However, the data obtained over a period of about 3 hours appears to be fairly good. By constantly monitoring the specimen temperature the large fluctuations may be corrected. A plot of the resistivity of the irradiated specimen and the corresponding specimen temperature as a function of time are plotted in Figure 4.7. The resistivity of an unirradiated specimen at this temperature is shown for reference. It is apparent that a significant portion of the irradiation-induced defects still remain even after 340 minutes of testing. The furnace temperature increase reflected in a significant change in resistivity as clearly seen for test times between 70 and 100 minutes.

Annealing studies of irradiated tungsten specimens at higher temperatures (860°C) resulted in such variations in resistivity values that it was evident that the furnace temperature control must be considerably improved. Therefore, the tests were temporarily discontinued until the furnace and associated control system can be modified to control the furnace temperature within $\pm 0.05^\circ\text{C}$ at temperature levels up to 1000°C .

Hardness Tests

The hot hardness testing of worked (unirradiated) molybdenum discussed in the previous progress report¹⁰ was completed. Analysis of the data, however, is continuing and the final results will be discussed in a later progress report. The preliminary data are shown in Figure 4.8. The upper curve was obtained from indentations made after annealing for 1 hour at the indicated temperature starting at room temperature and continuing up to 1200°C . Following this high-temperature heat treatment (specimen is now recrystallized), another pass made across the same sample resulted in the data points shown on the lower curve. A sharp drop in hardness of the worked material can be noted at 900° to 1000°C . Of more significance, however, is the slight rise in the hardness value at 150°C ($0.15 T_m$) and the nonlinear (on semi-log plot) portion of the hardness-versus-temperature curve at temperatures above 300°C as compared to the linearity of the hardness values following the recrystallization treatment.

The similarity of the hardness values of the recrystallized unirradiated molybdenum specimen with that of the recrystallized molybdenum control specimen, previously reported,¹¹ is clearly shown in Figure 4.9. These curves were obtained by drawing smooth lines through the points of Figure 4.8 and from the data on the irradiated and control specimens presented in the previous progress report.¹¹ Based on the curves shown in Figure 4.9, the following observations may be made:

1. Both the irradiated and worked specimens show discontinuities in the hardness values at temperatures near 150°C ($0.15 T_m$).

¹⁰Ibid.

¹¹Ibid., p. 94.

Fig. 4.7 – Resistivity and temperature of irradiated tungsten as a function of time

Fig. 4.8 - Hardness of molybdenum as a function of temperature

- Both the worked and the irradiated specimen hardness values deviate from linearity (on semi-log plot) at a temperature of above approximately 300°C indicating a possible similar defect structure and, therefore, a similar hardening mechanism in this temperature range.
- The hardness values for the irradiated specimen show a change in slope near 700°C; the hardness values of the worked specimen indicate a similar but not as obvious change in the same temperature region.
- Both the worked and the irradiated specimens show full recovery at a temperature slightly below 1100°C (conventional recrystallization temperature).
- It appears that the increase in hardness values of worked and of neutron-irradiated molybdenum may be attributed to three separate but additive components, each being prominent at different test temperatures.

The slight difference in hardness values of the two molybdenum materials at the elevated temperatures is probably due to minor variations in fabrication history and chemistry since the materials were obtained from different vendors. Both materials, however, were prepared by the arc-cast process; the respective chemical analyses are shown in Table 4.4.

Fig. 4.9 – Comparison of the hot hardness values of worked and irradiated molybdenum specimens

TABLE 4.4
CHEMICAL ANALYSIS OF MOLYBDENUM
SPECIMENS USED IN
HARDNESS MEASUREMENTS

Element ^a	Analysis	
	Content, wt %	
	Worked ^b	Irradiated ^c
Carbon	0.014	0.018
Oxygen	0.0017	0.0020
Nitrogen	0.0019	0.0006

^aSubstitutional impurities are reported to be less than 100 ppm by weight.

^bArc-cast Ingot 935D, sheet number M(6); 0.051-cm-thick specimen in worked condition.

^cArc-cast Ingot KDM 515A, sheet number M(10); 0.051-cm-thick specimen in the recrystallized condition.

HEAT RESISTANT METALS AND ALLOYS PROGRAM

Status of Irradiations

Elevated-temperature capsule GEFP2-139¹² was disassembled and the six Hastelloy N and 12 A-286 specimens examined. All appeared to be in good condition. Two of the Hastelloy N specimens have an abnormally high induced radioactivity compared to the other four Hastelloy N specimens. The half-life and/or gamma energies of smears from these two specimens will be determined with a gamma counter to identify the radioactive isotope. Gamma scanning of the specimens is also planned.

Another elevated-temperature capsule (GEFP2-148) containing Hastelloy N and A-286 specimens was fabricated and is being irradiated at 650°C in the ETR-14N-SE facility.

A fourth elevated-temperature capsule (GEFP 151) for irradiation of 18 Hastelloy N (INOR-8) specimens is being fabricated. It is scheduled for irradiation at 650°C during ETR Cycle 74. Six of the specimens are from a welded plate assembly furnished by the MSRE project, ORNL. The joint assembly consists of a 2.54-cm-thick plate welded to a 2.86-cm-thick plate using a U-weld joint preparation. The deposited weld metal is in the center of the gage length of the creep-rupture specimen.

Hastelloy N Creep-Rupture Tests

A series of Hastelloy N specimens irradiated in capsule GEFP2-128 at ambient temperature ($\sim 1 \times 10^{20}$ nvt, $E_n \geq 1$ Mev) was creep-rupture tested at 816°C in air. Plots of stress versus time to rupture are presented in Figure 4.10. Two control data points are also shown. The ORNL control data represents tests of the same heat as reported by ORNL. The GE-NMPO control curve was drawn parallel to the ORNL data line based on the assumption that the slopes should be the same. Additional GE-NMPO control tests are planned to verify this assumption. The irradiated stress-rupture data appear to show

Fig. 4.10 – Stress-rupture properties of Hastelloy N (INOR-8; Heat 5081) at 816°C

¹²Ibid., p. 96.

convergence with the control data at stresses below 8 kg/mm². This tendency for recovery at the lower stress levels has been previously observed¹³ in Hastelloy X irradiated specimens when tested at 870°C.

These rupture tests (816°C) show a smaller reduction in the time to rupture due to irradiation than the previously reported 650°C test results.¹⁴ The 650°C test results exhibited an approximate factor of 10 reduction in time to rupture for the same irradiation conditions; the 816°C tests show a reduction factor of 3.5 in the rupture life of irradiated specimens compared to the control specimens at the higher stress levels. A factor of 1.6 reduction was observed at the lower stress levels.

Creep tests are presently being conducted on specimens irradiated in capsule GEFP2-128 which were post-irradiation annealed at 871°C for 4 hours in air. These tests will be performed at test temperatures of 650°C and 816°C.

WORK PLANNED FOR NEXT PERIOD

Creep-rupture testing of (1) molybdenum specimens at the lower temperatures ($\sim 0.31 T_m$) will be continued and (2) W - 25Re specimens made from a special heat (GE-NMPO produced) with the intent of defining the change in flow mechanism in the 800° to 900°C temperature range will be initiated.

Hot-hardness tests on single-crystal tungsten specimens irradiated to three different dose levels will be performed. The influence of dose on hardness, resistivity, and creep rupture will be compared and evaluated.

The resistivity annealing furnace and power supply will be modified to obtain better ($\pm 0.05^\circ\text{C}$) control. Following equipment check-out and calibration, elevated-temperature isothermal annealing recovery of irradiated tungsten will be resumed. Isothermal annealing studies of irradiated molybdenum will be continued in the temperature region of $0.31 T_m$.

Post-irradiation creep-rupture tests of irradiated Hastelloy N will continue at test temperatures of 650°C and 815°C. Emphasis will be placed on studies of the creep-rupture properties of post-irradiation annealed specimens.

Theoretical studies on neutron-induced defects in the BCC metals will continue with emphasis on the absolute number of defect clusters produced in neutron-irradiated α -iron per unit exposure.

Computer results of the fractional abundances of original and product elements (Re and Os) formed from tungsten in a thermal neutron environment will be compared to radiochemical analysis of irradiated tungsten specimens.

¹³"Second Annual Report - High-Temperature Materials and Reactor Component Development Programs, Volume 1 - Materials," GE-NMPO, GEMP-177A, February 28, 1963, Figure 2.1-20, p. 80.

¹⁴"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, p. 97.

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5. HIGH-TEMPERATURE THERMOCOUPLE AND ELECTRIC MATERIALS RESEARCH

(57014)

The objective of this program is to identify and establish properties of reliable high-temperature thermocouples and electrical and electronic materials to provide instrumentation and electrical components for long-life, high-temperature reactors and reactor experiments.

HIGH-TEMPERATURE THERMOCOUPLES

Thermoelement Development

Work was continued to develop metallic thermocouple materials that would operate in a nuclear environment to 1800°C with adequate output and sensitivity and with minor change in thermoelectric signal over at least 2 years' time in a 10^{14} nv thermal neutron flux. The thermoelectric characteristics of several metals and alloys¹ having low thermal neutron cross sections were compared in the previous progress report.² The specific elements evaluated were Mo, Nb, Ta, Ru, Os, Cr, Zr, Ni, and several alloys. This evaluation showed Mo, Mo - 1Ru, and Mo - 1Os to be good positive thermoelements. A completely satisfactory negative leg has not yet been developed. The Nb - 10Mo alloy, previously reported to be inhomogeneous,³ and additional Nb - 20Mo and Nb - 40Mo alloys, made by methods previously reported,⁴ were heat-treated at 2250°C for 4 hours to obtain complete solid solution. The thermoelectric characteristics of these alloys are shown in Figure 5.1 which includes the previously determined Mo and Nb curves for comparison purposes. The Nb - 10Mo alloy is positive thermoelectrically with respect to pure Nb whereas the Nb - 20Mo and Nb - 40Mo alloys are negative with respect to Nb. Work during this report period showed the homogeneous Nb - 20Mo alloy to be considerably more negative than previously reported.⁵ Since the Nb - 20Mo and Nb - 40Mo alloys have good potential for use as negative thermoelements, the fabricability of these alloys to wire will be ascertained.

For reference purposes, the thermoelectric characteristic of chromium was compared to W - 25Re. A curve showing the thermoelectric output of chromium as a function of temperature is included in Figure 5.1.

In the previous progress report⁶ a difference was noted in the thermoelectric output of commercial wire and the powder metallurgy rod specimens for both molybdenum and niobium. Spectrographic analyses revealed a higher silicon and iron content in the com-

¹All alloy compositions are in atomic percent unless otherwise noted.

²"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, Fig. 5.1, p. 102.

³Ibid., p. 101.

⁴"High-Temperature Materials Program Progress Report No. 27, Part B," GE-NMPO, GEMP-27B, September 30, 1963, pp. 69-70.

⁵"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, p. 101.

⁶Ibid.

Fig. 5.1 – Thermoelectric output of Cr and the Nb-Mo alloy systems versus W – 25Re commercial wire as function of temperature

mercial molybdenum wire and a higher zirconium content in the niobium rod than in the corresponding wire specimens. These compositional differences probably account for the differences in thermoelectric output.

SHEATH MATERIAL EVALUATION

Work was initiated to determine the best possible sheath and electrical insulating materials to be used with a small diameter (2.5 mm) W/W – 25Re thermocouple probe being developed for measuring temperatures up to 2450°C for at least 1 hour in a graphite – hydrogen environment.

Molybdenum is commonly used as a sheath material for high-temperature probes. However, because it forms a eutectic at 1.8 weight percent of carbon, which melts at 2200°C, molybdenum must be isolated from the graphite if it is to maintain its integrity. To evaluate the use of coatings to protect molybdenum sheathing, a 0.05 mm coating of Ta and 0.075 mm coatings of flame-sprayed CaO-stabilized ZrO₂, and 0.075 mm HfO₂ were applied to Mo tubing; the samples were then encapsulated in graphite and tested at 2450°C in H₂ for 1.5 hours. None of the coatings gave complete protection to the molybdenum tubing. The tantalum, converted to the carbide, apparently was too thin to prevent diffusion of carbon into the molybdenum. Cracks occurred in the ceramic coatings, probably as a result of differential expansion between the coatings and the molybdenum, which permitted transport of carbon into the molybdenum.

Osmium was also investigated for use as a carbon barrier on thermocouple sheaths. A 3-mm-OD rod of osmium did not increase in weight when heated in contact with graphite at 2450°C for 1.5 hours in hydrogen. Metallographically, no evidence of osmium carbides was found and X-ray diffraction analysis indicated only pure osmium to be present. These results indicate that osmium may be a good sheath coating material to prevent carbon diffusion into a thermocouple system.

INSULATOR MATERIAL EVALUATION

For separating and electrically insulating the W/W - 25Re thermocouple legs, BeO, MgO, and HfO₂ were evaluated. Insulators were encapsulated in a graphite cylinder and heated in inert gas at 2450°C for 1.5 hours to evaluate the stability of each oxide in a graphite environment. These conditions were more severe than in normal usage where the oxide specimens would be separated from graphite by a sheath in an actual thermocouple probe. Only the HfO₂ remained intact. The HfO₂ specimen developed on its outer surface a 0.05-mm-thick layer of what is believed to be HfC. Electrical resistance measurements showed this outer layer to be a good electrical conductor. Although the center portion of the HfO₂ specimen changed in color from a light gray to black, its low-temperature electrical resistivity remained very high. Of the oxides tested, HfO₂ was the most resistant to carbon attack. Carbon penetrated the HfO₂-coated specimen to a depth of only 0.05 mm, indicating that HfO₂ may be a successful carbon barrier.

The ability of a refractory oxide electrical insulator to remain thermally stable and to partially retain its electrical insulating properties depends on the ability of the thermocouple sheath to contain the dissociation products formed by the oxides at high temperatures. Small capsules of either molybdenum or Mo - 50Re were machined to approximately 4 mm OD by 3.25 mm ID by 25 mm long. Small rod-like samples of MgO, BeO, and HfO₂ having an OD of approximately 2.5 mm were sealed in the metal container by electron-beam welding plugs into the ends. All capsules were heated to 2450°C for 1.5 hours in a hydrogen environment.

Two molybdenum capsules and six Mo - 50Re capsules all containing MgO ruptured and the OD expanded approximately 4 percent. This was probably caused by the high vapor pressure of MgO, and/or by water vapor pressure which formed as hydrogen diffused through the wall of the capsule and combined with oxygen of dissociated MgO. The general appearance of the MgO was not altered radically by the test. Little or no dimensional change was noted. Reheating the MgO specimens in the ruptured capsules to 2450°C for 1.5 hours in hydrogen caused a loss in OD of 5 percent, but again the appearance of the specimen remained essentially unchanged. These tests indicate that MgO may be a satisfactory insulator for limited time periods.

Two Mo capsules and two Mo - 50Re capsules containing HfO₂ were intact after test and showed an increase in OD of less than 1 percent, indicating that a lower vapor pressure was developed inside these capsules than inside the capsules containing MgO. The HfO₂ was black after testing, indicating some physical or chemical change.

Rhenium-encapsulated HfO₂ was also tested. The HfO₂ remained essentially unchanged except for a slight darkening, indicating that rhenium may be a better sheath material for HfO₂ than Mo or Mo - 50Re.

Two Mo - 50Re capsules containing BeO were leak-tight after testing but the contents had melted.

Based on the sheath and insulator evaluation, HfO₂, because of its higher melting point, greater resistance to graphite, and greater stability in hydrogen, is probably the best of the three oxides studied for high-temperature insulation.

HIGH-TEMPERATURE METAL-TO-CERAMIC SEALS

High-temperature gas-pressure bonding techniques were used to fabricate niobium-to-Lucalox⁷ seals. These seals were tested for more than 1200 hours at 1600°C without failure. Similar seals withstood 100 thermal cycles at 300°C per minute to a maximum temperature of 1450°C.

HIGH-TEMPERATURE GRADED SEALS

The addition of graded zones of mixed niobium and alumina powders at the interface of niobium and Lucalox resulted in hermetic seals capable of maintaining complete integrity for long-time periods at high temperature and remaining resistant to rapid thermal cycling. Present work is concerned with the continued evaluation and testing of these graded niobium-to-Lucalox seals.

Life Testing and Thermal Cycle Testing

A graded niobium-to-Lucalox seal fabricated by gas-pressure bonding at 700 kg/cm² for 1 hour at 1650°C was held at 1600°C for 1234 hours and remained vacuum tight. This seal was fabricated with three transitional zones between the niobium and Lucalox.

A second niobium-to-Lucalox seal with five transitional zones between the metal and ceramic remained vacuum tight after 1076 hours at 1600°C.

Other seals of this type, with three or five transitional zones between the niobium and Lucalox, were thermal cycled between 650°C and 1450°C at heating and cooling rates of 300°C per minute for 100 cycles and remained vacuum tight. A photomicrograph of the seal with five transitional zones after thermal cycling is shown in Figure 5.2.

No failures occurred in any of the seals life-tested and thermal-cycle-tested.

Fig. 5.2 – A photomicrograph showing the structure at the interface of a niobium-to-Lucalox graded seal after 100 thermal cycles from 650° to 1450°C at heating and cooling rates of 300°C per minute (100X)

⁷Trademark of the General Electric Company for commercial polycrystalline alumina.

Diffusion Studies of Niobium-to-Lucalox Bonds

An electron microprobe was used to determine the interdiffusion of niobium and alumina in the bonded regions of niobium-to-Lucalox seals. Studies were performed on metal-to-ceramic bonds that had been held at 1600°C for 115 hours and 466 hours and at 1950°C for 6.5 hours.

Interdiffusion was found to be quite limited. The first specimen with a history of 115 hours at 1600°C showed only an interdiffusion zone of 5.3 microns. Another specimen with 466 hours at 1600°C had diffusion zone widths of approximately 6.7 microns, while the specimen with 6.5 hours at 1950°C had diffusion zone widths of approximately 6.1 microns.

These data indicate that diffusion effects in niobium-to-alumina seals should not be a problem in long-life operation and also indicate that the niobium - alumina system is chemically compatible.

WORK PLANNED FOR NEXT PERIOD

The development of a thermocouple system for a graphite reactor will be continued. Alloys of Cr-Os, Mo-Ru, and Mo-Ru-Os will be evaluated thermoelectrically to obtain better negative thermoelements with low neutron cross sections. The resistivity study of refractory oxides will be continued. In-pile testing of W/W - 25Re thermocouple systems to measure transmutation effects will be initiated.

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6. APPENDIX

The "High-Temperature Materials Program Progress Reports" previously issued in this series are listed below. The first two reports were each issued as one document, containing both the classified and unclassified portions. The subsequent reports were issued as two documents; part A, the unclassified portion and part B, the classified portion.

Report No.	Report Period	Publication Date
GEMP-1	May 1961 - June 30, 1961	July 15, 1961
GEMP-2	July 1, 1961 - July 31, 1961	August 15, 1961
GEMP-3, A and B	July 1, 1961 - August 31, 1961	September 15, 1961
GEMP-4, A and B	August 1, 1961 - September 30, 1961	October 15, 1961
GEMP-5, A and B	August 15, 1961 - October 15, 1961	November 15, 1961
GEMP-6, A and B	September 15, 1961 - November 15, 1961	December 15, 1961
GEMP-7, A and B	October 15, 1961 - December 15, 1961	January 15, 1962
GEMP-106, A and B		
(First Annual Report) Calendar Year 1961		February 28, 1962
GEMP-9, A and B	January 1, 1962 - February 15, 1962	March 30, 1962
GEMP-10, A and B	January 1, 1962 - March 15, 1962	April 16, 1962
GEMP-11, A and B	February 15, 1962 - April 15, 1962	May 15, 1962
GEMP-12, A and B	March 15, 1962 - May 15, 1962	June 15, 1962
GEMP-13, A and B	April 15, 1962 - June 15, 1962	July 31, 1962
GEMP-14, A and B	May 15, 1962 - July 15, 1962	August 15, 1962
GEMP-15, A and B	June 15, 1962 - August 15, 1962	September 14, 1962
GEMP-16, A and B	July 15, 1962 - September 15, 1962	October 15, 1962
GEMP-17, A and B	August 15, 1962 - October 15, 1962	November 15, 1962
GEMP-18, A and B	September 15, 1962 - November 15, 1962	December 14, 1962
GEMP-19, A and B	October 15, 1962 - December 15, 1962	January 25, 1963
GEMP-177, A and B		
(Second Annual Report) Calendar Year 1962		February 28, 1963
GEMP-21, A and B	January 1, 1963 - February 15, 1963	April 23, 1963
GEMP-22, A and B	January 1, 1963 - March 15, 1963	April 30, 1963
GEMP-23, A and B	February 15, 1963 - April 15, 1963	May 31, 1963
GEMP-24, A and B	March 15, 1963 - May 15, 1963	June 28, 1963
GEMP-25, A and B	April 15, 1963 - June 15, 1963	July 31, 1963
GEMP-26, A and B	May 15, 1963 - July 15, 1963	August 16, 1963
GEMP-27, A and B	June 15, 1963 - August 15, 1963	September 30, 1963
GEMP-28, A and B	July 15, 1963 - September 15, 1963	November 11, 1963
GEMP-29, A and B	August 15, 1963 - October 15, 1963	November 30, 1963
GEMP-30, A and B	September 15, 1963 - November 15, 1963	December 31, 1963

<u>Report No.</u>	<u>Report Period</u>	<u>Publication Date</u>
GEMP-31, A and B	October 15, 1963 - December 15, 1963	January 24, 1964
GEMP-270, A and B		
(Third Annual Report)	January 1, 1963 - January 31, 1964	February 28, 1964
GEMP-34, A and B	February 1, 1964 - March 15, 1964	April 15, 1964
GEMP-35, A and B	February 1, 1964 - April 15, 1964	May 28, 1964
GEMP-36, A and B	March 15, 1964 - May 15, 1964	June 19, 1964
GEMP-37, A and B	April 15, 1964 - June 15, 1964	July 31, 1964
GEMP-38, A and B	May 15, 1964 - July 15, 1964	August 19, 1964
GEMP-39, A and B	June 15, 1964 - August 15, 1964	September 30, 1964
GEMP-40, A and B	July 15, 1964 - September 15, 1964	October 15, 1964
GEMP-41, A and B	August 15, 1964 - October 15, 1964	November 30, 1964
GEMP-42, A and B	September 15, 1964 - November 15, 1964	December 18, 1964
GEMP-43, A and B	October 15, 1964 - December 15, 1964	January 29, 1965
GEMP-334, A and B		
(Fourth Annual Report)	January 1, 1964 - January 31, 1965	February 26, 1965
GEMP-46, A and B	February 1, 1965 - March 15, 1965	April 15, 1965
GEMP-47, A and B	February 1, 1965 - April 15, 1965	May 28, 1965
GEMP-48, A and B	March 15, 1965 - May 15, 1965	June 25, 1965

ATOMIC PRODUCTS DIVISION

GENERAL ELECTRIC