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Nuclear Materials & Propulsion Operation

**HIGH-TEMPERATURE MATERIALS PROGRAM
PROGRESS REPORT No. 59, Part A**

May 31, 1966

GENERAL **ELECTRIC**
ATOMIC PRODUCTS DIVISION

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**HIGH-TEMPERATURE MATERIALS PROGRAM
PROGRESS REPORT No. 59, Part A**

May 31, 1966

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

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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	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

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1. INTRODUCTION AND SUMMARY

Introduction

This report is the unclassified portion of the fifty-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 February 1, 1966 to April 15, 1966, on four of the specific development programs in process. Three of the remaining programs are reported in the classified portion of this report, GEMP-59, Part B.

Ten other research and development efforts covered by the contract are reported in alternate months.

Summary

OXIDATION-RESISTANT FUEL ELEMENT MATERIALS RESEARCH (57001)

Laboratory testing of Fe-Cr-Al-Y-clad Fe-UO₂ fueled specimens was continued at 750°, 950°, and 1100°C in air. Three specimens clad with 2541 alloy were in good condition and had limited uranium concentration on the cladding surface after completing 10,000 hours of testing at 1100°C. Similar specimens tested for less time at 950°C have shown no uranium on the surface. Specimens clad with the lower chromium content 1541 alloy failed due to oxidation at 1100°C but showed good stability at 950°C.

Two in-pile tests of 1541-clad Fe - 40UO₂ specimens were completed. Experiment LTFC-3 was conducted in the LITR at 950°C for 2406 hours in flowing air. No fission gas release was detected and, although the burnup was 3.5 percent, the specimen was in excellent condition. Experiment ORFC-1 was operated in the ORR for 731 hours at indicated temperature of 730°C and to a burnup of 3.5 percent. An increase in fission product release after 400 hours is attributed to a localized temperature excursion which caused incipient melting in the cladding alloy.

Ductility evaluations of a series of Fe-Cr-Al-Y alloys which have been in oxidation tests for as long as 10,000 hours show that the alloys containing 25 percent chromium age-harden at 450°C and 550°C and are brittle at room temperature. None of the alloys increased in hardness at 730°C, and softening occurred at higher temperatures with corresponding good ductility below room temperature.

HIGH-TEMPERATURE REACTOR MATERIALS RESEARCH (57003)

The stress-rupture and creep characteristics of arc-cast W - 25Re sheet were determined in hydrogen from 1600° to 2600°C. Below 2000°C, the rupture life of W - 25Re is greater

than that for arc-cast unalloyed tungsten. Above 2000°C the rupture life of tungsten is greater. The ductility of the W - 25Re, based on the strain at rupture, was found to be a maximum at 1850°C.

Additional creep-rupture tests of powder metallurgy W-Re-Mo sheet for compositions ranging from W - 18Re - 10Mo to W - 27Re - 30Mo were performed at 1600°C and 2200°C. The results indicate a significant decrease in the ductility of the ternary alloys in the region of the phase boundary. Of the alloys tested, W - 18Re - 20Mo is the most ductile and creep resistant at 1600°C and 4.22 kg/mm² whereas W - 18Re - 10Mo is the most ductile and creep resistant at 2200°C and 0.56 kg/mm².

Studies dealing with the analysis of combined first-, second-, and third-stage creep data for arc-cast tungsten at 2400°C were made using four different expressions. The effectiveness of the modified deLacombe equation does not appear to be much better than the t^3 or deLacombe equations for stresses below 1.05 kg/mm², but at this stress the superiority of the modified deLacombe equation in expressing the creep curve is obvious.

Metallographic and Laue back-reflection diffraction studies of arc-cast tungsten, creep tested at 1800°C and 2400°C, were made in an attempt to identify a difference in the creep mechanisms. At 1800°C, coarse and fine slip bands are very evident with some grains showing a network of cross-slip lines. At 2400°C, "cell" formation becomes predominant with the large subgrains or "cells" outlined by rows of dislocations.

The thermal conductivity of W - 20UO₂ (vol %) was determined by the radial heat flow techniques for the temperature range of 620° to 2300°C. Data were also obtained for W - 40UO₂ (vol %) from 400° to 900°C.

HIGH-TEMPERATURE THERMOCOUPLE AND ELECTRICAL MATERIALS RESEARCH (57014)

Studies were performed on the compatibility of calcium oxide with W, W - 5Re, and Ta at 2450°C for 1 hour in hydrogen and in a carbonaceous hydrogen environment. Tungsten and W - 5Re were found to be compatible with CaO in hydrogen but not in carbonaceous hydrogen. Ta and CaO appear to be compatible in both environments at 2450°C.

The thermoelectric signal from a W - 3Re / W - 25Re thermocouple using synthesized alloys to represent 6 months in a 10^{14} nvt thermal neutron flux has larger induced errors at low temperature and smaller errors at high temperature than the W / W - 25Re thermocouple.

REFRACTORY-METAL ALLOY RESEARCH AND DEVELOPMENT (57015)

The ductility of welded Mo sheet processed with purification techniques developed in this program was confirmed. Good ductility and weldability were also observed in Mo sheet containing minor rhenium and tungsten additions if purification techniques to remove carbon and oxygen were used.

Studies of the influence of stress-relief treatments from 1425° to 1700°C on the ductility of W - 30Re - 30Mo sheet showed that the preferred structure and ductility were achieved at temperatures from 1650° to 1700°C.

Tooling was procured for producing small W - 30Re - 30Mo seamless tubing; four primary extrusion techniques are being studied.

2. OXIDATION-RESISTANT FUEL ELEMENT MATERIALS RESEARCH

(57001)

The objective of this research is to develop and evaluate oxidation-resistant fuel element materials markedly superior to presently available fuel materials in terms of high-temperature capabilities, long-time service (high burnup), and containment of fission products in environments of steam, air, carbon dioxide, or other oxidizing coolants.

FUEL ELEMENT MATERIALS EVALUATIONS

Fuel element cladding alloys of the Fe-Cr-Al-Y composition have been developed under this job which have demonstrated excellent stability in air to 1100°C and in steam to 730°C for periods in excess of 5000 hours. The current efforts in this program are aimed at the completion of 10,000-hour laboratory evaluations of the compatibility of the Fe-Cr-Al-Y alloy with various forms of UO₂ at temperatures of 750° to 1100°C. Four in-pile experiments have been performed and are being evaluated to determine burnup capability and fission product retentivity of the fuel element system as a function of time and temperature.

10,000-Hour Laboratory Test Program

The objective of the current laboratory test program is to demonstrate the time - temperature capability of both 2541- and 1541-clad¹ Fe - 40UO₂ fuel element specimens at temperatures of 750°, 950°, and 1100°C in air for times in excess of 10,000 hours. Cylindrical specimens 1.05 cm in diameter by 2.54 cm in length with cladding of 0.5 mm thickness are being tested. Dispersion-type fueled cores of Fe - 40UO₂ were sintered at 1200°C in hydrogen, machined to fit in the cladding tubes, assembled with end plates, sealed by electron-beam welding, and hot-gas pressure bonded at 1100°C or 1250°C under 700 kg/cm² helium pressure. A sufficient number of specimens were tested at each temperature to permit destructive evaluations at interim time periods. During the testing period, non-destructive analyses were made periodically by X-ray fluorescence analysis or alpha-counting the cladding surface for uranium diffusion.

The status of the long-time test program is given in Table 2.1. A total of 27 specimens representing six different material systems or fuel forms continued on test at the end of the report period. Four specimens clad with 1541 alloy were terminated because of failure due to oxidation after 2300 hours at 1100°C. The failure of the 1541 alloy to provide long-time oxidation resistance at 1100°C was not unexpected since the lower chromium alloy was developed for lower temperature application in steam and air and was not originally intended for service above 950°C. Eight specimens clad with 2541 alloy completed their scheduled test time of 2,500, 7,000, or 10,000 hours in good condition and were retired for metallographic evaluation and other analyses. Of these eight, only the three tested at

¹Designation for Fe-Cr-Al-Y alloys. The first two digits designate Cr content; the third, Al content, and the fourth, Y content; i.e., 2541 is Fe - 25Cr - 4Al - 1Y, 0541 is Fe - 5Cr - 4Al - 1Y, etc.

TABLE 2.1

STATUS OF CONTINUING 10,000-HOUR TEST PROGRAM OF Fe-Cr-Al-Y-CLAD FUELED SPECIMENS

Cladding	Core Composition, vol %	Type Fuel ^a	Specimen No.	Test Temperature, °C	Current Test Time, hr	Status
2541	Fe - 50UO ₂	KM-A	FAU-38	950	13,044	
2541	Fe - 40UO ₂	AR	FU-30	1100	10,030	Completed
2541	Fe - 40UO ₂	GE-A	FAU-53	750	5,648	
			FAU-54	750	5,648	
			FAU-55	950	5,043	Completed
			FAU-56	950	5,734	
			FAU-57	950	5,734	
			FAU-60	1100	5,683	
1541	Fe - 40UO ₂	GE-A	FAU-61	750	2,693	
			FAU-62	750	2,693	
			FAU-63	750	2,693	
			FAU-64	950	2,874	
			FAU-65	950	2,874	
			FAU-66	950	2,874	
			FAU-67	950	2,874	
			FAU-68	1100	2,300	Failed due to oxidation
			FAU-70	1100	2,300	Failed due to oxidation
			FAU-71	1100	2,300	Failed due to oxidation
			FAU-72	1100	2,300	Failed due to oxidation
2541	Fe - 40UO ₂	KM-A	FAU-22	1100	10,035	Completed
2541	Fe - 40UO ₂	S-KM	FSU-22	950	5,734	
			FSU-23	950	5,734	
			FSU-24	950	5,734	
			FSU-26	750	5,000	Completed
			FSU-27	750	5,648	
			FSU-28	750	5,648	
			FSU-29	750	5,648	
2541	Cr - 50UO ₂	KM-A	CAU-28	1100	10,005	Completed
2541 (Fe - 25Cr) - 40UO ₂		GE-A	FCAU-6	750	5,008	Completed
			FCAU-7	750	5,648	
			FCAU-8	750	5,648	
			FCAU-9	950	5,043	Completed
			FCAU-10	950	5,734	
			FCAU-12	950	5,734	
2541 (Fe - 25Cr) - 40UO ₂		S-KM	FCSU-1	750	5,648	
			FCSU-4	950	5,043	Completed
			FCSU-5	950	5,734	
			FCSU-6	950	5,734	
			FCSU-14	1100	5,683	

^aAR - As-received ceramic grade UO₂.GE-A - UO₂ agglomerated at GE-NMPO, -100/+200 mesh size.KM-A - Commercial agglomerated UO₂, -100/+200 mesh size.S-KM - UO₂ plasma-arc spheroidized at GE-NMPO, -120/+200 mesh size.

1100°C for 10,000 hours (specimens No. FU-30, FAU-22, and CAU-28) showed any significant uranium concentration on the surface of the cladding. The specimens tested at 750°C and 950°C for 5000 hours showed no uranium on the surface.

Metallographic examination was completed for five specimens previously tested at 950° to 1100°C for test periods of 2,000 to 10,000 hours. The compositions, test times, and results are shown in Table 2.2. None of the specimens had measurable concentrations of uranium on the surface. The microstructures of the two specimens tested at 950°C are

TABLE 2.2
SUMMARY OF METALLOGRAPHIC ANALYSIS OF THERMAL STABILITY SPECIMENS

Specimen No.	Cladding	Core Composition, vol %	Fuel Form	Test Conditions		Surface U Concentration, %	Oxide Thickness, microns	
				Temperature, °C	Time, hr		Cladding Surface	Interface
FAU-17	2541	Fe-50UO ₂	Agglomerated	950	10,015	<0.02	5.0	3.5
FAU-27	2541	Fe-40UO ₂	Agglomerated	950	7,025	<0.02	5.0	5.0
FAU-58	2541	Fe-40UO ₂	Agglomerated	1100	2,000	<0.02	10.1	5.0
FCSU-7	2541	(Fe-25Cr)-40UO ₂	Spheroidized	1100	2,595	<0.02	9.4	Nil
FCAU-13	2541	(Fe-25Cr)-40UO ₂	Agglomerated	1100	2,595	<0.02	10.6	Nil

shown in Figure 2.1. One specimen (FAU-27) contained 40 volume percent UO₂ fuel and the other (FAU-17) contained 50 volume percent fuel in an iron matrix. An oxide layer 3 to 5 microns in thickness formed at the core - cladding interface of these specimens. A partial oxide barrier also formed around some of the fuel particles near the interface. Some intergranular oxidation is evident in the cladding near the interface and some internal oxidation is apparent in the 2541 cladding.

The microstructures of two specimens tested at 1100 °C for 2595 hours are shown in Figure 2.2. Both specimens contained 40 volume percent UO₂ fuel in a matrix of Fe - 25Cr (wt %). Specimen FCSU-7 contained plasma-arc spheroidized fuel and specimen FCAU-13 contained agglomerated fuel. Neither specimen showed an interfacial barrier but some oxide

Fe-50UO₂, specimen FAU-17,
after 10,015 hours (Neg. 7026)

Fe-40UO₂, specimen FAU-27,
after 7025 hours (Neg. 7027)

Fig. 2.1 - Photomicrographs of 2541-clad Fe-UO₂ specimens showing oxide barrier formed at interface after testing at 950°C (200X)

Fe-25Cr-40UO₂, specimen FSCU-7;
spheroidized fuel (Neg. 7029)

Fe-25Cr-40UO₂, specimen FCAU-13;
agglomerated fuel (Neg. 7030)

Fig. 2.2 - Photomicrographs of 2541-clad Fe-Cr-UO₂ specimens tested at 1100°C for 2595 hours (200X)

was observed on the fuel particles near the core - cladding interface. The agglomerated fuel particles had considerable void formation around them as a result of shrinkage. In both specimens, internal oxidation of the cladding occurred near the interface but to a greater extent in the specimen containing agglomerated fuel. The cladding of these two specimens appeared similar to that of UN fueled specimens reported previously² although the degree of penetration and concentration of the oxide and/or nitride is less when UO₂ fuel is used.

In-Pile Test Program

In-pile testing of the Fe-Cr-Al-Y-clad Fe-UO₂ system was discontinued during this report period. This program was planned to define the fission product retention capabilities in terms of temperature and burnup. The C-48 facility in the LITR was used for fission product retention determinations and the F-2 facility in the ORR was planned for high burnup tests. During the course of this test program, three tests were completed in the LITR and one in the ORR.

1541-Clad Fe-UO₂ at 950°C (LTFC-3) - Dynamic in-pile experiment LTFC-3 was completed after 2406.6 hours of irradiation in the LITR. An airflow over the fuel element specimen of about 1700 liters per minute was required to maintain the maximum temperature at about 950°C. The test specimen experienced 19 thermal cycles due to reactor

²"Fifth Annual Report - High-Temperature Materials Programs, Part A," GE-NMPO, GEMP-400A, February 28, 1966, p. 141.

shutdowns. Upon reactor startup, the temperature would peak at 1020 °C for periods of approximately 5 minutes.

Fractional release data obtained from the analysis of samples of the discharge air stream for escaped fission products indicated that steady-state release remained fairly constant (maximum $R/B = 4 \times 10^{-8}$ for Kr^{87} to 2×10^{-7} for Xe^{133}) throughout the test. The release rate is as low or lower than any previous tests which indicate excellent specimen integrity.

The fuel specimen was removed from the test capsule and examined at ORNL. The specimen had a light gray coating over most of the fueled portion similar to the specimen from LTFC-2.³ The excellent condition of the specimen after removal from the cartridge is shown in Figure 2.3.

Fig. 2.3—Post-test condition of 1541-clad Fe-40UO₂ specimen LTFC-3 after irradiation in LITR for 2406 hours at 950°C in flowing air coolant. Burnup was about 2.4×10^{20} fissions per cm³ (3.5%) and no release of fission products was noted.
(Neg. ORNL, R-27820)

Dimensional measurements on the diameter of the specimen after irradiation showed no detectable changes. Hardness tests along the length of the specimen showed a Rockwell 15N hardness of 66 to 67 which is about a 5 point increase from the annealed hardness of the cladding alloy. No change in weight of the specimen was found. A gamma scan along the fueled length of the specimen indicated that it had been exposed to a uniform flux from end to end. The specimen was sectioned at ORNL and will be shipped to GE-NMPO for metallographic examination.

1541-Clad Fe-UO₂ Test in ORR (ORFC-1)—The first of a series of high-burnup specimens was a 1541-clad Fe-25UO₂ (70% enriched) specimen. This cartridge was irradiated in the F-2 facility of the ORR for 731 hours at an indicated temperature of 750 °C. A coolant airflow of about 4800 liters per minute was used over the specimen.

The fission product release data shown in Table 2.3 indicate that the first significant release occurred after the mid-cycle shutdown of the ORR after 479 hours of operation. Another increase in fission gas release was detected after the third shutdown with 727 hours of operation. This indicated that a major rupture of the specimen cladding probably occurred and it was decided to terminate the test a short time later.

The total burnup at the time of termination was calculated to be 1.9×10^{20} fissions/cm³, or about 3.5 percent of the total uranium atoms in the core.

The specimen was removed from the cartridge and examined in the hot cell at ORNL. The appearance of the specimen as-tested is shown in Figure 2.4. In one area near the

³“High-Temperature Materials Program Progress Report No. 55, Part A,” GE-NMPO, GEMP-55A, January 21, 1966, p. 20.

TABLE 2.3
SUMMARY OF ANALYSES OF CHARCOAL TRAPS FOR RELEASE OF
FISSION GASES FROM IN-PILE TEST ORFC-1

Trap No.	Time at Temperature, hr	Fractional Release (R/B ^a)				
		Kr ^{85m}	Kr ⁸⁷	Kr ⁸⁸	Xe ¹³³	Xe ¹³⁵
1	75.0	3×10^{-7}	2×10^{-8}	7×10^{-8}	5×10^{-7}	1×10^{-7}
2	454.8	2×10^{-7}	3×10^{-8}	3×10^{-8}	5×10^{-7}	5×10^{-8}
Shutdown	473.3					
3	479.7	1×10^{-7}	1×10^{-7}	—	7×10^{-7}	2×10^{-6}
Shutdown	590.9					
4	592.6	1×10^{-7}	1×10^{-7}	1×10^{-7}	2×10^{-6}	1×10^{-6}
5	665.1	5×10^{-7}	2×10^{-7}	3×10^{-7}	3×10^{-6}	9×10^{-7}
Shutdown	727.2					
6	727.5	7×10^{-4}	3×10^{-4}	4×10^{-4}	9×10^{-2}	2×10^{-3}
7	729.3	6×10^{-4}	3×10^{-4}	3×10^{-4}	2×10^{-2}	7×10^{-4}

^aR/B is ratio of fission product released to those born.

Fig. 2.4—Post-test condition of 1541-clad Fe-25UO₂ specimen ORFC-1 after irradiation in ORR for 731 hours at 750°C in flowing air coolant. Burnup was 1.9×10^{20} fissions per cm³ (3.5%). Release of fission products was noted after about 480 hours. (Neg. ORNL, R-27823)

downstream end, the specimen appeared to have suffered overheating and possibly incipient melting. Another indication of over temperature was the sticking of the alumina insert in the bore of the specimen which indicated melting had occurred. The alumina insert was used to hold the three thermocouples in place against the ID of the specimen and in one area, the ceramic insert was joined sufficiently that it had to be driven out. Whatever over-temperature may have occurred was not detected by the thermocouples.

The specimen had a thin black, glossy coating over the fueled area when viewed under diffuse light, but under direct light the cladding appeared clean and metallic. A gamma scan was made over the length of the specimen and indicated that the neutron flux peaked at the cooler end of the specimen and dropped off sharply to the hotter downstream end. The diameter of the specimen was unchanged from pre-test measurements at the cooler end, showed growth ranging from 1 to 3.4 percent at the center of the fueled section, and as much as 4 percent growth in the area of the incipient melting.

Superficial hardness readings were made along the fueled portion of the specimen and averaged 68 (Rockwell 15N) at the cooler end and 62 to 64 from mid-section to the hotter end. However, the reading at the hot end may be low because of the irregular surface of the cladding.

The specimen was sectioned at ORNL and is being transferred to GE-NMPO for metallographic analysis.

ALLOY DEVELOPMENT

The objectives of the alloy development program are to develop Fe-Cr-Al-Y alloys having improved structural properties without sacrificing oxidation or corrosion resistance. A major portion of this work has been the measurement of the oxidation resistance of a series of Fe-Cr-Al-Y alloys in air at 450°, 550°, 730°, 900°, and 1100°C for times up to 10,000 hours. The progressive weight gain data for these alloys were presented previously.⁴ The 10,000 hours of testing have been completed for all alloys and data on hardness and ductility have been obtained. The hardnesses of specimens tested for 10,000 hours are given in Table 2.4. These data show that the 25 percent Cr alloys age harden at 450°C and 550°C. According to the basic Fe-Cr equilibrium diagram, precipitation of alpha prime, a Cr-rich ferrite phase, occurs at temperatures below 520°C. At 550°C, sigma phase (FeCr) occurs. The 15-percent Cr alloys lie outside the alpha plus sigma region, and thus only exhibit alpha prime precipitation below about 500°C.

TABLE 2.4
MICROHARDNESS OF Fe-Cr-Al-Y ALLOYS
OXIDIZED FOR 10,000 HOURS

Alloy	Heat No.	Test Temperature, °C	Hardness (DPH), 100-g load	
			Center	Edge
2541	MS-36	As-annealed	200	200
		450	335	331
		550	362	359
		730	199	204
		900	190	190
		1100	173	168
2541	MS-42	730	206	221
		900	221	213
		1100	181	193
1541	MS-38	As-annealed	175	175
		450	226	215
		550	179	169
		730	171	172
		900	166	168
		1100	146	141
1541	MS-41	730	181	181
		900	206	200
		1100	170	165
1041	M-239	730	193	175
		900	181	193
		1100	151	152
0561	M-236	730	206	206
		900	193	206
		1100	181	161
1540	M-230	As-annealed	165	165
		450	208	205
		550	160	159
		730	156	154
2540	M-232	730	181	181

⁴Ibid., p. 25.

None of the alloys exhibited significant change in hardness after exposure at 730°C. At 900°C and 1100°C, softening occurred probably as a result of the depletion of aluminum from the alloys because of oxidation.

The hardness data are shown graphically as functions of test time at 450°C and 550°C in Figures 2.5 and 2.6. At 450°C the 25 percent Cr alloys exhibited the greatest hardness increase followed by the 15 percent Cr alloys. The alloys made with ferrochromium (heats No. MS-41 and MS-42) hardened more than the alloys made with high-purity Cr. The 0561 alloy exhibited slight hardening, possibly associated with precipitation of phases in the Fe-Al system such as Fe₃Al. The 1041 alloy did not exhibit hardening at 450°C. At 550°C, only the 25 percent Cr alloys exhibited significant hardening, as expected from the Fe-Cr-Al equilibrium diagram. The sharp drops in hardness shown in Figures 2.5 and 2.6 for four of the alloys were caused by re-solutioning of the precipitate during temperature excursions caused in turn by furnace malfunctions. The specimens were over-heated by 50° to 75°C for short periods of time.

Fig. 2.5—Hardness versus time at 450°C for various Fe-Cr-Al-Y alloys

Fig. 2.6—Hardness versus time at 550°C for various Fe-Cr-Al-Y alloys

Specimens of various alloys were selected for bend testing, and the results are shown in Table 2.5. As expected from the hardness data, the 25 percent Cr alloy exhibited the most embrittlement after aging at 450°C and 550°C. The good ductility of heat No. MS-41 (1541 alloy) after aging at 450°C was unexpected, particularly since heat No. MS-38 (1541 alloy) was brittle at room temperature. An examination of the microstructures indicates that the precipitate in heat No. MS-41 is resolvable as shown in Figure 2.7. No precipitate is visible in MS-38 after 1000 hours as shown in Figure 2.8, and the structure remained the same up to 10,000 hours. These data suggest that over-aging occurred in heat No. MS-41 even though no hardness decrease occurred. In fact, the hardness was greater in heat No. MS-41 than in heat No. MS-38. Examination of the structure of heat No. MS-42, a 2541 alloy made with ferrochromium (as was MS-41), also indicates extensive visible precipitation at 450°C as shown in Figure 2.9. For some reason, the alloys made with ferrochromium exhibit faster precipitate growth (possibly over-aging) than alloys made with high-purity Cr. The reasons for this are not clear but it may be possible to pre-age the 1541 alloys for service below about 500°C. Above 500°C, the precipitate will be taken into solution and re-precipitate when the temperature is reduced.

Referring to Table 2.5, none of the alloys exhibited embrittlement after exposure at 730°C, all exhibiting 90-degree bends at -78°C. At 900°C and 1100°C some embrittlement is noted. The embrittlement is probably caused by the intergranular oxides which are formed at the higher test temperatures. The oxide stringers act as notches as indicated in Figure 2.10.

TABLE 2.5

BEND TEST DATA FOR Fe-Cr-Al-Y ALLOYS AFTER OXIDATION TESTING

Alloy	Oxidation Test		Hardness (R-15N)	Bend Temperature, ^a °C					
	Temperature, °C	Time, hr		250	170	100	R. T.	0°C	-78°C
2541	450	6,000	82.3	○	●				
(MS-42)	550	5,000	78.9		○	●			
	730	10,000	66.4						○
	900	10,000	66.1					○	●
	1100	10,000	62.7					○	●
	As-annealed								○
1541	450	6,000	78.2					○	●
(MS-41)	550	6,000	66.4						○
	730	10,000	62.2						○
1541	450	6,000	72.5			○	●		
(MS-38)									
2540	450	6,000	81.8		○	●			
(M-232)	550	10,000	76.8		○	●			
1540	450	8,000	73.8				○	●	
(M-230)	550	6,000	59.9						○
1041	450	6,000	62.5						○
(M-239)	550	10,000	61.2						○
	730	10,000	60.6						○
0561	450	2,000	68.6						○
(M-236)	550	2,000	63.9						○
	730	10,000	63.4						○
	900	10,000	61.8					○	●
	1100	9,000	57.0				○	●	
	As-annealed								○

^aCode: ○ Bend of 90° without fracture.

● Fracture of bend angle of less than 20°. All duplicate specimen tests.

Fig. 2.7 – Photomicrograph of 1541 alloy (MS-41) after aging for 4000 hours at 450°C (Neg. 7064; etchant, 10% oxalic acid; 250X)

Fig. 2.8 – Photomicrograph of 1541 alloy (MS-38) after aging for 1000 hours at 450°C (Neg. 7063; etchant, 10% oxalic acid; 250X)

Fig. 2.9 – Photomicrograph of 2541 alloy (MS-42) after aging for 4000 hours at 450°C (Neg. 7062; etchant, 10% oxalic acid; 250X)

Fig. 2.10 - Photomicrograph of 0561 alloy after oxidizing for 9000 hours at 1100°C then bending to fracture at 0°C, showing fracture grain-boundary oxide (Neg. 7061; as-polished; 250X)

WORK PLANNED FOR NEXT PERIOD

Laboratory testing of the Fe-Cr-Al-Y-clad specimens under the 10,000-hour test program will be completed. Metallographic and X-ray fluorescence analyses of all specimens will be in progress.

In-pile test specimens LTFC-2, LTFC-3, and ORFC-1 will be metallographically evaluated. The alloy development phase of this job will be completed with evaluations of the oxidation test specimens.

3. HIGH-TEMPERATURE REACTOR MATERIALS RESEARCH

(57003)

The purpose of this program is to measure and evaluate high-temperature physical and mechanical properties of commercially available and newly developed refractory materials being considered for use in fueled and non-fueled high-temperature (1000° to 3000°C) reactor applications.

MECHANICAL PROPERTY EVALUATIONS

Creep-Rupture Studies

W - 25Re - The previously reported^{1,2} evaluation of the creep-rupture characteristics of W - 25Re sheet (0.05 cm thick) obtained from seven different commercial vendors showed the results to vary considerably in terms of creep resistance, rupture life, and ductility at rupture when tested at 1600°C and 4.22 kg/mm² and at 2000°C and 2.11 kg/mm² in hydrogen. Two vendors provided arc-cast material and the others provided wrought powder metallurgy material. In view of these variations, the results obtained for the arc-cast material were considered representative and reproducible properties for W - 25Re since it was previously demonstrated³ that arc-cast unalloyed molybdenum sheet provided the same results independent of vendor, whereas the wrought powder metallurgy material did not.

Additional creep-rupture tests of arc-cast W - 25Re sheet (Ev-1) were performed at temperatures from 1600° to 2600°C in a hydrogen atmosphere. The results, presented in Table 3.1, give the time to various percentages of strain and rupture along with the total strain at rupture. The data, presented in terms of the Larson-Miller parameter, are shown in Figure 3.1. A computer program was used to give the line of best fit and the optimum C value for each percentage of strain and rupture time.

Typical creep curves obtained with the W - 25Re sheet specimens are shown in Figures 3.2 and 3.3 for 1600°C and 2400°C. At 1600°C, the curves exhibit a significant amount of primary creep as well as a detectable amount of secondary creep, but the major portion of the creep process is seen to be confined to third-stage creep. At 2400°C there is no detectable amount of primary or secondary creep and third-stage-type creep occurs from time zero. The indication is that strain hardening is non-existent or is not sufficient to overcome the effect of annealing during the initial portion of creep for W - 25Re at 2400°C. This was not the case for arc-cast, unalloyed tungsten because when tested from 1600° to 3000°C, primary and secondary stages of creep were observed at all temperatures.⁴

Although the primary reason for the rhenium addition to tungsten is to obtain increased ductility during fabrication, the creep resistance and rupture life of W - 25Re exceeds that

¹"High-Temperature Materials Program Progress Report No. 53, Part A," GE-NMPO, GEMP-53A, November 26, 1965, p. 40.

²"High-Temperature Materials Program Progress Report No. 55, Part A," GE-NMPO, GEMP-55A, January 21, 1966, p. 29.

³P. N. Flagella, "High Temperature Stress-Rupture Characteristics of Mo and Some Mo Alloys," GE-NMPO, GE-TM 65-4-39, June 1965.

⁴"High-Temperature Materials Program Progress Report No. 47, Part A," GE-NMPO, GEMP-47A, May 28, 1965, p. 30.

TABLE 3.1
CREEP-RUPTURE RESULTS FOR ARC-CAST
W - 25Re SHEET^a IN HYDROGEN^b

Temperature, °C	Stress, kg/mm ²	Time to Indicated Strain, hr							Rupture	
		0.2%	0.5%	1%	2%	3%	5%	10%	Time, hr	Strain, %
1600	7.03	0.20	0.64	1.36	2.60	3.75	5.73	9.55	24.0	86
1600	5.62	0.29	1.13	2.60	5.70	8.70	14.0	24.6	62.1	84
1600	4.92	0.72	2.08	5.05	11.5	17.9	29.0	50.7	119	73
1600	4.22	1.00	2.95	7.10	16.2	25.5	42.0	75.9	195	87
1600	3.37	0.94	4.40	15.3	42.7	70.0	117	213	513	71
1600	2.11	3.70	20.1	96.2	375	513	894	1445	c	c
2000	3.52	0.06	0.12	0.20	0.35	0.47	0.68	1.06	2.40	81
2000	2.11	0.50	1.10	1.88	3.23	4.43	6.30	9.85	21.9	86
2000	1.41	2.00	5.00	8.50	15.1	20.1	28.6	46.9	107	103
2000	1.05	3.60	10.8	22.0	39.5	53.8	77.2	121	266	96
2200	0.56	3.96	11.6	21.3	38.1	53.0	80.1	129	252	54
2400	0.70	0.12	0.33	0.62	1.10	1.48	2.15	3.54	7.30	44
2400	0.56	0.49	1.16	2.15	3.95	5.60	8.25	12.7	27.7	40
2400	0.46	1.00	2.45	4.55	8.40	11.8	17.3	25.4	33.1	28
2400	0.35	2.64	6.47	12.6	24.2	33.7	48.8	65.6	82.0	26
2600	0.35	0.32	0.85	1.68	3.01	3.91	4.91	6.38	7.04	20

^a0.05-cm-thick sheet specimen with 0.64-cm by 2.54-cm gage section.

^bAnnealed 2 hrs at test temperature prior to test.

^cTerminated at 2,685.5 hours with 34.2% strain.

Fig. 3.1 - Larson-Miller parameter for arc-cast W - 25Re sheet

Fig. 3.2—Creep curves for arc-cast W-25Re sheet at 1600°C in hydrogen

Fig. 3.3—Creep curves for arc-cast W-25Re sheet at 2400°C in hydrogen

of unalloyed tungsten at 1600°C for the same stress (see Figure 3.4). The difference appears to decrease as the temperature is increased. Above 2000°C, the rupture life for tungsten exceeds that of W-25Re for the same conditions. This same relationship between the two materials is also observed when comparing the time required to give 10 percent strain. This decrease in creep resistance and rupture life above 2000°C for W-25Re when compared to tungsten is apparently related to the different modes of creep discussed above.

Fig. 3.4—Stress-rupture curves for arc-cast W-25Re compared to arc-cast W, both tested in hydrogen

An interesting observation for the arc-cast W-25Re sheet is shown in Figure 3.5. The ductility of the material, based on the total strain at rupture, is apparently related to temperature. Above and below 1850°C, the ductility decreases and appears to approach zero near the melting point of the material. The same basic shape and trend was observed for the unalloyed arc-cast tungsten⁵ from 1600° to 3000°C except that the maximum ductility occurred at approximately 2250°C. In addition, the tungsten data also exhibited a minimum at 1800°C with the ductility increasing below this temperature.

Included in Table 3.1 are the results of a creep test of arc-cast W-25Re sheet in hydrogen at 1600°C and 2.11 kg/mm². The specimen was operated continuously at the test conditions and terminated without rupture after 2685.5 hours. The total strain at this point was 34.2 percent. Examination of the specimen after testing indicated that significantly more strain would have occurred before rupture with the implication that the increased time at temperature has no deleterious effect on the ductility of the material. (Compare to data in Figure 3.5.) The creep curve for this test is given in Figure 3.6. Although the major portion of the curve consists of third-stage creep, primary and secondary stages of creep are apparent. This is consistent with the observations discussed earlier with regard to Figures 3.2 and 3.3.

⁵Ibid., p. 33.

Fig. 3.5 – Ductility versus temperature for arc-cast W-25Re sheet from stress-rupture tests in hydrogen

Fig. 3.6 – Creep curve for arc-cast W-25Re at 1600°C and 2.11 kg/mm² in hydrogen

W-Re-Mo Alloys – Previous reports⁶⁻⁸ presented the creep-rupture results of various W-Re and W-Re-Mo alloys in sheet form fabricated by the powder metallurgy process. The tests were part of the evaluation of materials fabricated by the "Refractory-Metal Alloy Research and Development" program at GE-NMPO (prior to FY-66 part of this task; now job No. 57015). Additional creep-rupture tests were performed at 1600°C and 2200°C using W-Re-Mo sheets of still different compositions in an attempt to determine the most creep-resistant material with reasonable ductility before and after exposure to high temperatures.

A summary of the creep-rupture results for all the compositions investigated to date at 1600°C and 4.22 kg/mm² is given in Table 3.2. A similar summary for the same compositions at 2200°C and 0.56 kg/mm² is presented in Table 3.3. Typical creep curves are given in Figures 3.7 and 3.8 for the W – Re – 10Mo alloys creep-rupture tested at 1600°C and 2200°C, respectively. In both figures, the curves show the increased creep resistance due to the decreasing amount of rhenium (increasing amount of tungsten), as might be expected. While this effect is quite pronounced in the data for the 10-percent Mo alloys, it was not consistently observed in the data for the various other compositions.⁹

TABLE 3.2
CREEP-RUPTURE RESULTS^a FOR W-RE-MO ALLOYS^b AT 1600°C AND 4.22 kg/mm² IN HYDROGEN

Composition ^c	Time to Indicated Strain, hr						Rupture		Linear Creep Rate, min ⁻¹	Hardness (DPH) ^d	
	0.5%	1%	2%	3%	5%	10%	Time, hr	Strain, %		Before	After
W – 26Re	1.7	4.2	9.2	13.5	21.5	35.8	45.4	15	3.9×10^{-5}	524	475
W – 27Re	0.8	2.0	4.9	7.7	13.3	24.5	39.6	21	5.9×10^{-5}	550	475
W – 28Re	0.2	0.5	1.6	2.7	5.5	13.3	36.8	40	1.0×10^{-4}	572	550
W – 29Re	0.1	0.2	0.6	1.2	2.5	6.4	26.3	54	2.1×10^{-4}	627	575
W – 18Re – 10Mo	2.6	6.9	16.9	26.1	39.3	—	48.5	8.5	1.7×10^{-5}	454	381
W – 20Re – 10Mo	3.5	7.9	17.2	25.8	38.4	—	50.3	9.0	1.8×10^{-5}	487	369
W – 22Re – 10Mo	3.2	6.5	13.1	19.3	28.4	—	36.6	7.9	2.5×10^{-5}	466	412
W – 24Re – 10Mo	1.9	4.3	9.0	13.8	20.7	—	25.8	7.7	3.5×10^{-5}	441	423
W – 26Re – 10Mo	1.9	4.0	7.7	10.6	—	—	16.4	6.0	4.0×10^{-5}	485	450
W – 26Re – 10Mo	1.6	3.3	6.5	9.3	13.3	—	17.2	9.0	4.7×10^{-5}	502	460
W – 27Re – 10Mo	1.7	3.2	6.4	9.6	15.2	24.7	28.7	14	5.2×10^{-5}	530	480
W – 27Re – 10Mo	1.2	2.4	4.5	6.4	9.5	13.6	13.6	10	—	528	453
W – 28Re – 10Mo	0.4	1.0	2.5	4.1	7.5	14.8	25.1	28	1.0×10^{-4}	540	405
W – 29Re – 10Mo	0.05	0.2	0.6	1.0	2.2	5.8	16.4	29	2.3×10^{-4}	600	530
W – 18Re – 20Mo	2.0	5.2	11.6	18.1	30.1	55.7	95.7	23	2.6×10^{-5}	456	384
W – 20Re – 20Mo	1.5	3.7	8.7	13.0	19.8	—	25.5	9	3.3×10^{-5}	466	396
W – 24Re – 20Mo	1.4	3.0	5.6	7.3	—	—	9.2	4.4	4.7×10^{-5}	447	412
W – 26Re – 20Mo	1.9	3.5	6.1	7.8	10.1	—	12.2	9	4.7×10^{-5}	495	415
W – 26Re – 20Mo	0.9	1.9	3.8	5.2	7.2	9.1	9.2	12	—	463	412
W – 27Re – 20Mo	1.4	2.7	5.0	7.1	10.0	13.5	14.5	16	5.8×10^{-5}	495	425
W – 27Re – 20Mo	1.2	2.1	3.8	5.0	6.7	8.4	8.4	10	—	432	402
W – 28Re – 20Mo	1.1	2.2	4.3	6.4	9.7	14.7	17.0	13	7.4×10^{-5}	525	465
W – 29Re – 20Mo	0.5	1.3	2.8	4.3	7.0	11.9	17.2	21	1.1×10^{-4}	525	500
W – 17Re – 30Mo	1.1	2.8	8.2	15.4	33.0	72.3	99.4	16	1.9×10^{-5}	376	374
W – 21Re – 30Mo	1.1	2.5	5.1	7.2	10.1	—	11.4	7.0	5.9×10^{-5}	482	384
W – 25Re – 30Mo	1.5	2.8	5.3	7.6	10.6	13.9	14.8	14	6.1×10^{-5}	405	398
W – 27Re – 30Mo	0.9	1.6	2.7	3.7	5.1	—	6.6	9.5	9.0×10^{-5}	438	396

^aAfter annealing at 1600°C for 2 hours in H₂.

^b0.05-cm-thick sheet specimens with 0.63-cm by 2.54-cm gage section.

^cAtomic percent.

^d5-kg load.

⁶Ibid., p. 79.

⁷"High-Temperature Materials Program Progress Report No. 49, Part A," GE-NMPO, GEMP-49A, July 28, 1965, p. 48.

⁸"High-Temperature Materials Program Progress Report No. 51, Part A," GE-NMPO, GEMP-51A, September 30, 1965, p. 31.

⁹"High-Temperature Materials Program Progress Report No. 49, Part A," GE-NMPO, GEMP-49A, July 28, 1965, p. 53.

Fig. 3.7 – Creep curves for W – Re – 10Mo tested at 1600°C and 4.22 kg/mm² in hydrogen

Fig. 3.8 – Creep curves for W – Re – 10Mo tested at 2200°C and 0.56 kg/mm² in hydrogen

TABLE 3.3
 CREEP-RUPTURE RESULTS^a FOR W-Re-Mo ALLOYS^b AT 2200°C AND 0.56 kg/mm² IN HYDROGEN

Composition ^c	Time to Indicated Strain, hr						Rupture		Linear Creep Rate, min ⁻¹	Hardness (DPH) ^d	
	0.5%	1%	2%	3%	5%	10%	Time, hr	Strain, %		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-18Re-10Mo	1.7	3.6	7.8	11.8	20.6	42.2	128.2	46	3.8×10^{-5}	454	364
W-20Re-10Mo	1.1	2.4	5.3	8.5	15.0	31.5	96.1	42	5.1×10^{-5}	494	381
W-22Re-10Mo	1.0	2.3	4.8	7.6	13.1	26.6	73.0	40	6.1×10^{-5}	473	402
W-24Re-10Mo	1.0	2.0	4.0	6.3	10.7	21.6	55.4	36	7.6×10^{-5}	473	406
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-26Re-10Mo	0.5	1.0	2.0	3.0	4.8	9.2	15.6	25	1.7×10^{-4}	516	376
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-27Re-10Mo	0.4	0.8	1.6	2.5	4.2	8.5	23.9	38	1.9×10^{-4}	516	374
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-18Re-20Mo	0.8	1.7	3.5	5.5	9.7	20.2	54.7	43	7.9×10^{-5}	454	427
W-20Re-20Mo	1.2	2.7	5.8	8.8	15.0	28.1	56.7	31	5.4×10^{-5}	447	376
W-24Re-20Mo	1.3	2.8	5.9	9.3	16.2	33.4	73.6	30	4.8×10^{-5}	441	424
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-26Re-20Mo	1.1	2.2	4.6	7.2	12.4	25.4	51.5	26	6.4×10^{-5}	438	402
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-27Re-20Mo	0.7	1.4	3.0	4.5	7.4	14.0	24.2	26	1.1×10^{-4}	460	366
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
W-17Re-30Mo	21.2	43.0	86.5	121	166	—	173.7	6	3.8×10^{-6}	384	386
W-21Re-30Mo	1.5	3.2	6.8	10.6	18.2	35.7	70.5	30	4.4×10^{-5}	418	369
W-23Re-30Mo	1.7	3.4	6.8	10.3	17.1	33.1	68.6	31	4.9×10^{-5}	426	367
W-25Re-30Mo	0.9	1.9	3.9	6.0	10.1	19.2	28.8	18	8.1×10^{-5}	412	400
W-27Re-30Mo	0.8	1.8	3.9	6.0	10.1	20.5	43.2	31	7.8×10^{-5}	426	391

^aAfter annealing at 2200°C for 2 hours in H₂.

^b0.05-cm-thick sheet specimens with 0.63-cm by 2.54-cm gage section.

^cAtomic percent.

^d5-kg load.

Some of the alloy compositions employed in this study existed in the region of complete solid solution (α) while others were located in the alpha plus sigma region.¹⁰ It was possible, therefore, to observe the effect of the additional phase (σ) on the ductility as measured by the elongation at rupture. This effect was found to be fairly pronounced as shown in Figure 3.9 for tests at 2200°C and 0.56 kg/mm². As the composition approaches the phase boundary from either side (~26% Re) the ductility at temperature decreases significantly.

Figure 3.10 presents similar data for the tests at 1600°C and 4.22 kg/mm². The curves at 1600°C do not show the sharp transition at the phase boundary but are more diffused; however, the effect is still apparent. The additional observation noted at 1600°C is the significant increase in ductility obtained in the two-phase region as opposed to the single-phase region. This rate of increase of ductility with increasing sigma phase is apparently reduced as the Mo content is increased. Results of the ductility evaluation indicate that this technique may be used to identify or confirm the location of phase boundaries within an equilibrium diagram.

¹⁰"High-Temperature Materials Program Progress Report No. 55, Part A," GE-NMPO, GEMP-55A, January 21, 1966, p. 68.

Fig. 3.9 – Composition effect on rupture strain at 2200°C and 0.56 kg/mm²

Fig. 3.10 – Composition effect on rupture strain at 1600°C and 4.22 kg/mm²

The effect on the creep resistance of the appearance of the sigma phase in the various alloys is not apparent. Figures 3.11 and 3.12 show the time required to give 5 percent strain for the various compositions at the same temperature and stress. The time required to yield 5 percent strain decreases, in most cases, as the Re content is increased (W content decreased) at constant Mo content.

Fig. 3.11 – Composition effect on time to give 5 percent strain at 2200°C and 0.56 kg/mm²

Photomicrographs of the various W-Re-Mo specimens both before and after test are shown in Figure 3.13. No significant differences in the corresponding structures are observed except for some of the materials containing the higher amounts of Re. These high Re specimens exhibit pronounced sigma phase, but, as noted, the amount of sigma phase decreases as the amount of Mo is increased. The grain boundary separation due to the strain is also similar for each alloy at both temperatures.

From an overall appraisal of the test data, it appears that at 1600°C the most ductile alloy with the best creep resistance is W – 18Re – 20Mo. At 2200°C the alloy that appears to contain both desirable characteristics is W – 18Re – 10Mo. Further evaluations such as fabricability and room-temperature ductility both before and after testing are necessary before the best composition can be determined.

Creep Analysis Techniques

Studies dealing with the analysis of combined first-, second-, and third-stage creep data were continued. While previous analyses¹¹ were concerned with the form:

$$\epsilon = a + bt^{1/2} + ct + dt^{3/2} \quad (3.1)$$

¹¹Ibid., p. 41.

Fig. 3.12 – Composition effect on time to give 5 percent strain at 1600°C and 4.22 kg/mm²

the studies during this present report period were involved with the evaluations of the following equation forms:

$$\epsilon = a + bt^{1/3} + ct + dt^{4/3} \quad (3.2)$$

$$\epsilon = a + bt^{1/3} + ct + dt^3 \quad (3.3)$$

$$\epsilon = a + bt^m + ct^n \quad (3.4)$$

$$\epsilon = a + bt^m + ct^n + dt^p \quad (3.5)$$

where a , b , c , d , m , n , and p are constants. Some study of the Andrade equation was undertaken but early results indicated that this expression was not very effective in analyses of this type. For this reason no additional studies of the Andrade equation are planned.

It will be noted that the expression involving the $t^{4/3}$ term is a modified Cottrell-Aytenkin¹² equation with the final term serving to provide the accelerating creep rates characteristic of this portion of the creep process. The expression involving t^3 represents the form proposed by Graham and Wallis¹³ for use in describing the entire creep curve. It may also be noted that the expression involving two variable exponents is the deLacombe¹⁴ equation while the form involving three variable exponents is a modification of the deLacombe equation and was proposed for the first time in the current study. Least squares procedures were employed in each case to yield values for the equation constants and, whereas this

¹²A. H. Cottrell and V. Aytekin, "Andrade's Creep Law and the Flow of Zinc Crystals," *Nature*, Vol. 160, 1947, p. 328.

¹³A. Graham and K. F. A. Wallis, "Relationships Between Long and Short Time Creep and Tensile Properties of a Commercial Alloy," *Journal of Iron and Steel Institute*, Vol. 179, 1955, p. 105.

¹⁴J. deLacombe, "A Method of Representing Creep Curves," *Rev. Metal.*, Vol. 36, 1939, p. 178.

Fig. 3.13 – Photomicrographs of W-Re-Mo (at. %) sheet before and after creep-rupture testing (250X)

Fig. 3.13 (Cont.) – Photomicrographs of W-Re-Mo (at. %) sheet before and after creep-rupture testing (250X)

Fig. 3.13 (Cont.) - Photomicrographs of W-Re-Mo (at. %) sheet before and after creep-rupture testing (250X)

approach is fairly straightforward when the $t^{4/3}$ and t^3 equations are considered, special non-linear regression analyses were necessary in the solution of the expression involving variable exponents.

In evaluating the effectiveness of the above equation forms the creep data for arc-cast tungsten at 2400°C were employed. A summary of the results obtained is presented in Table 3.4 where the creep equation constants are listed along with the value for the standard deviation, μ (corrected for the number of equation constants). Of course, the smaller the value for μ , the closer the creep equation comes to passing through all the experimental points and the more effective the representation.

TABLE 3.4
COMBINED FIRST-, SECOND-, AND THIRD-STAGE CREEP DATA FOR ARC-CAST TUNGSTEN
AT 2400°C ANALYZED IN TERMS OF VARIOUS EQUATIONS

I. $\epsilon = a + bt^{1/3} + ct + dt^{4/3}$								
Stress, kg/mm ²	a x 10 ³	b x 10 ³	c	d	$\mu \times 10^4$			
0.46	-1.70	2.21	8.99×10^{-7}	1.72×10^{-6}	10.9			
0.56	-2.49	3.83	-3.09×10^{-5}	7.26×10^{-6}	11.5			
0.70	-2.01	5.92	-7.77×10^{-5}	2.84×10^{-5}	12.6			
0.84	-9.25	14.10	-5.96×10^{-4}	1.22×10^{-4}	28.3			
1.05	10.05	21.90	-1.90×10^{-3}	5.04×10^{-4}	33.2			
II. $\epsilon = a + bt^{1/3} + ct + dt^3$								
Stress, kg/mm ²	a x 10 ³	b x 10 ³	c	d	$\mu \times 10^4$			
0.46	0.45	1.195	2.67×10^{-5}	3.17×10^{-13}	7.08			
0.56	0.48	1.928	5.36×10^{-5}	4.58×10^{-12}	3.31			
0.70	1.081	3.427	1.46×10^{-4}	1.26×10^{-10}	3.45			
0.84	0.313	6.022	2.69×10^{-4}	9.66×10^{-10}	8.34			
1.05	-1.24	11.91	4.14×10^{-4}	3.82×10^{-8}	24.0			
III. $\epsilon = a + bt^m + ct^n$								
Stress, kg/mm ²	a x 10 ³	b	m	c	n	$\mu \times 10^4$		
0.46	2.01	2.20×10^{-4}	0.734	1.13×10^{-9}	2.12	7.42		
0.56	1.96	6.56×10^{-4}	0.65	9.19×10^{-9}	2.10	2.98		
0.70	4.15	1.06×10^{-3}	0.708	2.81×10^{-9}	2.58	3.83		
0.84	5.71	1.86×10^{-3}	0.713	2.06×10^{-9}	2.90	8.09		
1.05	10.60	2.58×10^{-3}	0.808	9.87×10^{-21}	8.98	8.70		
IV. $\epsilon = a + bt^m + ct^n + dt^p$								
Stress, kg/mm ²	a x 10 ³	b	m	c	n	d	p	$\mu \times 10^4$
0.46	1.20	4.92×10^{-17}	3.88	1.87×10^{-6}	1.29	5.54×10^{-4}	0.545	7.21
0.56	1.27	3.43×10^{-11}	2.74	9.74×10^{-6}	1.18	1.15×10^{-3}	0.499	2.80
0.70	3.54	8.79×10^{-34}	10.84	7.51×10^{-8}	2.10	1.38×10^{-3}	0.647	3.59
0.84	2.87	6.24×10^{-11}	3.43	1.93×10^{-4}	1.06	4.02×10^{-3}	0.423	7.45
1.05	(-32.59)	2.74×10^{-25}	11.1	8.21×10^{-4}	1.03	32.60	1.048×10^{-4}	2.87

In general it was found that, for the data employed in this analysis, the least effective equation form was that involving the $t^{4/3}$ term. This expression is even inferior to the third-degree polynomial in $t^{1/2}$, results for which were presented in the last report.¹⁵ The expression involving t^3 was definitely superior to the equation involving $t^{4/3}$ and the third-degree polynomial in $t^{1/2}$. It was found to be just about as effective as the deLacombe

¹⁵"High-Temperature Materials Program Progress Report No. 55, Part A," GE-NMPO, GEMP-55A, January 21, 1966, p. 42.

equation. While the effectiveness of the modified deLacombe equation (involving three variable exponents) does not appear to be much better than the t^3 or deLacombe equations at stresses below 1.05 kg/mm^2 , the better results obtained at 1.05 kg/mm^2 are particularly striking. None of the other equations studied was capable of providing as low a value of μ as that obtained with this more complex expression. Some disadvantage might be assigned to the large negative value of ϵ_0 but positive strain values are obtained with this expression for time values beyond 1 minute.

Several examples of the effective representations afforded by the various equation types studied are presented in Figure 3.14. It is clear that the t^3 equation type is definitely superior to the $t^{4/3}$ expression. Also the deLacombe equation is seen to provide excellent representations particularly at 1.05 kg/mm^2 . Finally, the effectiveness of the modified deLacombe equation is demonstrated by the excellent representation of the data at 1.05 kg/mm^2 .

When the linear portion of the creep curve is extrapolated into the third-stage region, differences, δ , between this plot and the experimentally measured strain values in third stage can be calculated. Plots of the logarithm of these differences versus the logarithm of time were found to be linear by Graham and Walles. Slope measurements led to a value of 3.0 for the exponent on time for use in equation (3.3). Similar studies using the creep data for arc-cast tungsten at various temperatures from 2000° to 2600°C led to identical results except that larger slopes were obtained. Two such examples are shown in Figure 3.15 for the data at 2200°C and 2400°C . Distinct linearity is observed although in these instances the slopes are approximately 5.6 at 2200°C and 4.7 at 2400°C . These results suggest a slight revision in the largest exponent in equation (3.3), at least for the data employed in this analysis. Some additional studies are planned to investigate the merits of this suggestion.

Studies of Creep Mechanisms

The creep behavior of tungsten has been observed to undergo a definite change between 1800° and 2400°C . For example, plots of stress-rupture and linear creep rate results obtained at 2000°C and 2200°C on metal pre-annealed at 2400°C for 1 hour show a marked change in slope.¹⁶ To explain this behavior, more thorough studies have been made on two specially prepared tungsten specimens.

Arc-cast tungsten of high purity was fabricated into 0.15-cm-thick sheet. Two creep test specimens were cut from this sheet and pre-annealed in hydrogen at 2800°C for 2 hours. This was done so that the grain structure of both specimens would be identical before initiation of the test. One specimen was tested in hydrogen at 1800°C at 2.46 kg/mm^2 for 8 hours and the other was tested in hydrogen at 2400°C at 0.56 kg/mm^2 for 8 hours. Although the former underwent an elongation of 13.6 percent and the latter only 7.8 percent, both tests were stopped approximately halfway through the linear creep stage and rapidly cooled in hydrogen to room temperature to prevent any major changes in the structure. Test specimens were then examined by metallography and X-ray diffraction to characterize differences which might explain their phenomenological behavior. Another sample of the same arc-cast tungsten sheet which had received only the pre-annealing treatment was used as a standard reference material in this study.

The grain size after annealing ranged from 2 to 10 mm in diameter and remained constant during test; hence, individual grains were large enough to be examined by the Laue back-reflection technique. With the standard photographic method, exposures of 1 hour using unfiltered Cu radiation at 40 kv and 20 ma gave good results for a specimen-to-

¹⁶"High-Temperature Materials Program Progress Report No. 49, Part A," GE-NMPO, GEMP-49A, July 28, 1965, p. 31.

Fig. 3.14—Combined first-, second-, and third-stage creep data for arc-cast tungsten at 2400°C represented by various equation forms

Fig. 3.14 (Cont.) – Combined first-, second-, and third-stage creep data for arc-cast tungsten at 2400°C. represented by various equation forms

Fig. 3.15—Strain differences, δ , versus time for arc-cast tungsten data at 2200°C and 2400°C

film distance of 3 cm. When the Polaroid attachment was used under the same conditions, exposures of 1.5 minutes were found to be satisfactory.

The metallographic analysis was made on sections cut from the tungsten sheet. These sections were: longitudinal – looking at the sheet surface perpendicular to the stress axis; transverse – looking at the cross section of the sheet down the stress axis; and edge – looking along the edge of the sheet perpendicular to the stress axis. Specimens were mounted, polished, and etched electrolytically in a 2 percent NaOH solution to develop dislocation etch pits.

Surfaces of the tested specimens are shown in Figure 3.16. Both samples are highly distorted with a wrinkled surface appearance. There are no major external differences, although the specimen tested at 1800°C contains many more fine crystallographic slip bands than the one tested at 2400°C which has some grains containing practically no slip lines.

Photomicrographs of the specimen tested at 1800°C ($0.56 T_m$ where T_m is the absolute melting temperature) are presented in Figure 3.17 as longitudinal (a), transverse (b), and edge (c). All three views show the formation of numerous small subgrains between rows of slip lines. In Figure 3.17a and c, the parallel slip traces make an angle of about 45 degrees to the stress direction. Slip lines in the grains of Figure 3.17b, having different orientations, meet at about 90 degrees along the grain boundary. It is of interest to point out that the long axis of the dislocation etch pits is roughly parallel to the polygonization walls which outline the slip bands.

The presence of severe strain in this material is also revealed in the Laue back-reflection photographs shown in Figure 3.18. These were taken of the grain shown in Figure 3.16a and of the right-hand grain in Figure 3.16b. Note, in particular, the extreme elongation in the stress-axis direction. The multiplicity of small spots for each Laue reflection is characteristic of a structure containing many subgrains misoriented by tilt boundaries containing many piled-up dislocations.

The appearance of various grains after test at 2400°C ($0.73 T_m$) is illustrated in Figure 3.19, longitudinal (a), transverse (b), and edge (c). These photomicrographs show a rather random distribution of dislocation etch pits throughout the grains, with well-identified "cells" (major subgrains) which contain smaller subgrain boundaries in the process of formation. In the views perpendicular to the stress direction, there is a tendency for alignment of dislocations into boundaries at 45 degrees to this direction. However, these boundaries are much less pronounced than in the specimen tested at 1800°C. Grains having an orientation near [011] or [113] have been chosen to illustrate the substructure because it has previously been found that electrolytic etching more readily develops dislocation etch pits on these surfaces than on [001] or [112].

Laue back-reflection photographs of the grains shown in Figure 3.19a and b are shown in Figure 3.20a and b, respectively. Note that the individual Laue reflections are fairly coherent because the angular spread of a given spot is relatively small. This is a further indication that the substructure is composed of rather large perfect cells separated from each other by rows of dislocations producing a small angular tilt boundary. The new (smaller) subgrains have not as yet become sufficiently pronounced to produce additional Laue splitting.

The test temperatures of 1800° and 2400°C are homologous temperatures of $0.56 T_m$ and $0.73 T_m$, respectively. At temperatures above about $0.5 T_m$, the creep rate of metals is diffusion controlled, as Dorn¹⁷ has conclusively demonstrated. Below one-half

¹⁷J. F. Dorn, *Creep and Recovery*, ASTM, Cleveland, 1956, p. 225.

a. Tested for 8 hours in hydrogen at 1800°C and 2.46 kg/mm²
(Neg. 6661)

b. Tested for 8 hours in hydrogen at 2400°C and 0.56 kg/mm²
(Neg. 6659)

Fig. 3.16 – Photomicrographs of arc-cast tungsten after creep testing (7X)

a. Longitudinal section (Neg. 6672)

b. Transverse section (Neg. 6669)

Fig. 3.17 – Photomicrographs of arc-cast tungsten after creep testing for 8 hours in hydrogen at 1800°C and 2.46 kg/mm² (250X)

c. Edge section (Neg. 6671)

Fig. 3.17 (Cont.) – Photomicrographs of arc-cast tungsten after creep testing for 8 hours in hydrogen at 1800°C and 2.46 kg/mm² (250X)

the absolute melting temperature, creep occurs by the movement of dislocations along slip planes in the metal. The specimens studied were tested in a temperature region where both of these processes are competing; the predominant mechanism undoubtedly depends not only upon the temperature but also the creep rate itself.

The most dramatic demonstration that different mechanisms of creep are operating in these two samples is seen in Figure 3.21. At 1800°C, coarse and fine slip bands are very evident, with some grains showing a network of cross-slip lines. The specimen tested at 2400°C shows that cell formation has become well developed. The large subgrains or cells, as outlined by rows of dislocation etch pits, are generally equiaxed in appearance.

Differences between creep behavior at these two temperatures can also be seen by a more detailed examination of previous Figures taken at high magnification (250X). Figure 3.17 (1800°C test specimen) reveals bands of the major slip system running at 45 degrees to the stress axis, intersected by wavy lines of a secondary slip system. The presence of easy cross-slip in bcc metals is believed to be due to their high stacking-fault energy. From Figure 3.18, the crystallographic slip direction is [111], and the Laue reflections are roughly six times as long in this direction as they are wide. Hence, the primary mode of deformation under these conditions of intermediate temperature and higher strain rate is a general disordering of the structure by grain boundary migration and intragranular slip. Glide of dislocations in the [111] direction along planes favorably oriented to the stress direction would produce the slip lines and bands which have been observed. At this temperature self-diffusion is relatively slow so that the dislocations progress through the metal by glide only on close-packed atomic planes.

For the 2400°C test specimen, the degree and mechanism of deformation is markedly different. At the higher temperature and lower strain rate, the creep process appears to be mainly governed by self-diffusion. Dislocations now move not only by glide but by climb and therefore can bypass obstacles to reach the surface. This leads to the formation of

a. Longitudinal section (Neg. M-9160A)

b. Transverse section (stress axis perpendicular to page)
(Neg. M-9159(6))

Fig. 3.18 – Laue back-reflection photographs of arc-cast tungsten after creep testing for 8 hours in hydrogen at 1800°C and 2.46 kg/mm²

a. Longitudinal section (Neg. 6667)

b. Transverse section (Neg. 6665)

Fig. 3.19 – Photomicrographs of arc-cast tungsten after creep testing for 8 hours in hydrogen at 2400°C and 0.56 kg/mm² (250X)

c. Edge section (Neg. 6668)

Fig. 3.19 (Cont.) – Photomicrographs of arc-cast tungsten after creep testing for 8 hours in hydrogen at 2400°C and 0.56 kg/mm² (250X)

structurally perfect subgrains separated from each other by small tilt boundaries containing entrapped dislocations. This polygonization is shown quite well in Figures 3.21b and 3.19. The random distribution of dislocation etch pits in Figure 3.19 implies a greater freedom of movement through the metal. The substructure is less strained as the back-reflection photograph in Figure 3.20 illustrates. The reflections are composed of large individual spots very near each other which indicates much less distortion of the grain structure. These observations are in accord with the cell formation described by Wood and his collaborators.¹⁸

A change in the mechanism of deformation during creep has also been observed in tantalum between $0.6 T_m$ and $0.9 T_m$ by Green.¹⁹ He found that the coarse slip-band deformation was very important at high stresses, while the subgrain size increased with temperature at lower stresses. It is of interest that such a change occurs at about $0.6 T_m$ for tungsten, also.

One other bcc metal recently studied was a niobium alloy by Harris.²⁰ It was concluded that crystallographic slip was predominant at 980°C ($0.55 T_m$) but becomes less important as the temperature is increased. Cell-formation begins to occur at 1205°C ($0.65 T_m$) and becomes the controlling mechanism at 1315°C ($0.70 T_m$). These results are noteworthy because they agree remarkably well with those for tantalum and tungsten. At a homologous temperature of $0.6 T_m$ for these three bcc metals, a transition in the deformation mechanism has been observed. Below this temperature, creep is not diffusion-controlled but dislocation glide produces crystallographic slip and cross-slip. Above $0.6 T_m$, however, self-diffusion begins to predominate by the mechanism of dislocation climb.

¹⁸W. A. Wood, et al., *Journal of Institute of Metals*, Vol. 75, 1948–1949, p. 693; *ibid.*, Vol. 76, 1949–1950, p. 237; *ibid.*, Vol. 77, 1950, p. 423; *ibid.*, Vol. 79, 1951, p. 1959.

¹⁹W. V. Green, *Trans. AIME*, Vol. 233, 1965, p. 1818.

²⁰B. Harris, *Journal of Less Common Metals*, Vol. 9, 1965, p. 244.

a. Longitudinal section (Neg. M-9158A)

b. Transverse section (stress axis perpendicular to page) (Neg. M-9157)

Fig. 3.20 – Laue back-reflection photographs of arc-cast tungsten after creep testing for 8 hours in hydrogen at 2400°C and 0.56 kg/mm²

a. Tested for 8 hours in hydrogen at 1800°C and 2.46 kg/mm²
(Neg. 6664)

b. Tested for 8 hours in hydrogen at 2400°C and 0.56 kg/mm²
(Neg. 6663)

Fig. 3.21 – Photomicrographs of arc-cast tungsten after creep testing (18X)

PHYSICAL PROPERTY EVALUATION

Thermal Conductivity

The thermal conductivity of W – 20UO₂ (vol %) was measured from 620° to 2300°C and that of W – 40UO₂ was extended from the previously reported²¹ 900° to 2300°C range down to 400°C. The new data for the W – 40UO₂ have higher values than would be obtained by extrapolating the linear equation²¹ from 900° to 400°C. The density of the specimens was 97.4 percent of theoretical for the W – 20UO₂ and 95 percent for the W – 40UO₂.

Figure 3.22 shows the data for these compositions compared to curves for tungsten,²² uranium dioxide, and W – 60UO₂ which have been previously reported.²³ The influence of composition on the thermal conductivity of W-UO₂ compacts can be readily seen from this comparison. Studies conducted to date have not been sufficient to determine the mechanisms of heat transport in this material nor the effect of shape and orientation of the oxide particles on the thermal conductivity.

All specimens used in this study were prepared by a Dynapak process and measurements were made in a plane perpendicular to the direction of compaction. Examination of these materials under a microscope shows some preferred orientation; i.e., the oxide particles are somewhat lenticular in shape with the major axis lying perpendicular to the direction of compaction or in the direction of heat flow in the measurements. This situation probably yields a higher conductivity than would be obtained in the direction of pressing or perpendicular to this major axis of the particle since the heat path through the matrix material (tungsten) would be lengthened and possibly more heat would flow through the low-conductivity phase of the system (UO₂).

Figures 3.23 and 3.24 are photomicrographs showing the comparison in structure of the two materials. These photographs were made from plugs which were machined from the center of the specimen and represent the as-received condition.

Specimens for measuring the electrical resistivities of these materials and pure tungsten have been partially fabricated. It is anticipated that these measurements will aid in understanding the mechanisms of thermal conductivity operative in these materials.

WORK PLANNED FOR NEXT PERIOD

Stress-rupture and creep characteristics of various compositions of W-Re-Mo alloys with the Mo content between 40 and 65 percent will be investigated at 1600°C and 2200°C.

The effect of varying the loading rate on the initiation of first-stage creep for molybdenum at 1600°C will be investigated further.

Investigations of equation forms for describing the strain – time behavior for the entire creep curve (first, second, and third stages) will be continued.

Enthalpy and heat capacity measurements for tantalum and some of its alloys will be made to 2000°C.

²¹"High-Temperature Materials Program Progress Report No. 55, Part A," GE-NMPO, GEMP-55A, January 21, 1966, p. 52.

²²V. S. Gumenyuk and V. V. Lebedev, "Investigating the Heat and Electrical Conductivities of Tungsten and Graphite at High Temperatures," *Fizika Metallov and Metallovedeniye*, 2, January 1961, NR/pp 29–31, Translation USAEC-NP-TR-733.

²³A. D. Feith, "Thermal Conductivity of Several Ceramic Materials to 2500°C," *Advances in Thermophysical Properties at Extreme Temperatures and Pressures*, ASME, 1965, p. 328.

Fig. 3.22 – Thermal conductivity versus temperature for W-UO₂ and composites

Fig. 3.23 – Photomicrograph showing as-received condition of W – 40UO₂
(vol %) thermal conductivity specimen (Neg. 6595, 10X)

Fig. 3.24 – Photomicrograph showing as-received condition of W – 20UO₂
(vol %) thermal conductivity specimen (Neg. 6597, 10X)

4. HIGH-TEMPERATURE THERMOCOUPLE AND ELECTRIC MATERIALS RESEARCH

(57014)

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

DEVELOPMENT OF THERMOCOUPLES FOR A GRAPHITE - HYDROGEN ENVIRONMENT

Evaluation of CaO

Work was continued on the evaluation of CaO as a usable thermocouple insulator at 2450°C in a carbonaceous hydrogen environment. The test specimens consist of small sealed refractory-metal capsules containing CaO completely encapsulated in graphite. A previous 1-hour test of W - 5Re-sheathed¹ CaO in hydrogen and encapsulated in graphite resulted in rupturing of the W - 5Re sheaths and loss of the CaO.²

Two electron-beam-welded W - 5Re capsules (2.4 mm OD, 0.25 mm wall thickness, and 2 cm long) containing 0.038 and 0.0475 of CaO, respectively, were tested for 1 hour at 2450°C in hydrogen only. Both capsules developed longitudinal cracks, but only one leaked. There was practically no increase in the OD of the capsules which suggests that no excessive internal pressure developed.

In another experiment two empty W - 5Re capsules were tested in a carbonaceous hydrogen atmosphere at 2450°C. Both capsules showed an increase in OD dimensions due to carburization but the swelling was 25 percent less than when CaO was present in the capsule.² Similar results were obtained using an extruded tungsten capsule containing CaO; after 1 hour at 2450°C in a hydrogen atmosphere (no carbon present), a tungsten capsule remained leak tight with a negligible change in weight and dimensions. After an additional 1 hour at 2450°C in carbonaceous hydrogen, the capsule cracked and lost all the CaO. These results demonstrate a carbonaceous atmosphere at 2450°C increases the internal pressures in W-Re and W capsules containing CaO.

Previous work³ using Ta as a sheath material over HfO₂ electrical insulators indicated that Ta reduced the propensity of the HfO₂ becoming carburized. Two Ta capsules with approximately the same dimensions as the W - 5Re capsules and containing CaO were prepared for testing in a carbonaceous hydrogen environment at 2450°C for 1 hour. The capsules remained leak tight after testing (as determined by helium mass spectrograph techniques) although the OD and length dimensions increased 4 percent due to carburization of the tantalum. Visual examination after opening the capsules showed the CaO to be essentially unchanged although some did adhere to the Ta wall. This test further serves to

¹All compositions are in weight percent unless otherwise specified.

²"High-Temperature Materials Program Progress Report No. 55, Part A," GE-NMPO, GEMP-55A, January 21, 1966, pp. 55-56.

³"High-Temperature Materials Program Progress Report No. 53, Part A," GE-NMPO, GEMP-53A, November 26, 1965, p. 54.

indicate that by reducing the carbon activity over the CaO within the capsule reduces the probability of rupture.

CARBON PENETRATION RATE STUDIES

It has been demonstrated³ that 0.25 mm thick Ta sheath is adequate to protect HfO₂ from carburization at 2450°C in a hydrogen and graphite environment for a period of 1 hour. On the otherhand, HfO₂ encapsulated in W became carburized after 1 hour. To determine the criticality of time for damaging carburization to occur at 2450°C, capsules were prepared from both extruded W and Ta tubing with a 0.24 mm wall thickness. A rod of hard-fired HfO₂ was encapsulated inside each 2-cm-long capsule. The W capsule was heated for 0.25 hour and the Ta capsule for 3 hours at 2450°C in a carbonaceous hydrogen environment. Post-test inspection showed the HfO₂ inside the W capsule was completely carburized over its entire surface and was highly conductive. The HfO₂ encapsulated in the Ta capsule and was not carburized as confirmed by its practically zero electrical conductivity on the outer surface.

Thermoelectric Changes in W - 3Re From Nuclear Transmutation

Studies on the effects of nuclear transmutation, using the synthesized compositions technique employed on the W / W - 25Re thermocouple system,⁴ has been extended to include the W - 3Re / W - 25Re thermocouple system.

A W - 3Re rod, prepared from high-purity powders, was compared thermoelectrically with a rod of W - 3.81Re - 0.38OS which represents W - 3Re after being irradiated with 10^{14} nv thermal flux for 6 months. Figure 4.1 shows the thermoelectric output of the two rods versus tungsten commercial wire as a function of temperature. The difference in thermoelectric potential between the 2 curves represents the amount of emf change due to 6 months at 10^{14} nv thermal flux transmutation in a W - 3Re versus W - 25Re thermocouple caused by the W - 3Re leg only. The thermoelectric emf change of the W - 25Re leg only for the same flux was previously determined.⁵

The total thermoelectric transmutation effect change in W - 3Re / W - 25Re thermocouple is the sum of these two changes. Figure 4.2 shows this emf change converted to a temperature error that would exist if a standard chart for W - 3Re / W - 25Re thermocouple were used to determine temperature. The temperature error increases rather rapidly to approximately 65°C at 600°C and then increases rather slowly to about 75°C at 2000°C. Comparing these data with the data obtained for the W / W - 25Re thermocouple, it is observed that the W - 3Re versus W - 25Re thermocouple has a greater error below approximately 1300°C and a lesser error above 1300°C.

Reactor Testing of Thermocouples

The post-irradiation testing of reactor irradiated W and W - 20Re thermocouple wires has been temporarily delayed because of their high radioactivity and difficulty of handling in this condition. When the radioactivity decays to a lower level thermoelectric measurement will be made.

A capsule for in-pile testing of W / W - 25Re thermocouple is nearly ready for shipment to Oak Ridge.

WORK PLANNED FOR NEXT PERIOD

Determination of the thermoelectric changes due to nuclear transmutation in the W - 3Re versus W - 25Re and the W - 5Re versus W - 25Re thermocouples will be continued.

⁴"Fourth Annual Report - High-Temperature Materials and Reactor Component Development Programs, Volume I - Materials," GE-NMPO, GEMP-334A, February 26, 1965, pp. 222-224.

⁵"High-Temperature Materials Program Progress Report No. 43, Part A," GE-NMPO, GEMP-43A, January 29, 1965, p. 72.

Fig. 4.1—Relative thermoelectric output changes as a function of temperature for W-3Re and W-3Re (wt %) doped with Re and Os to represent transmutations corresponding to 6 months exposure at 10^{14} nv flux versus W commercial wire

Fig. 4.2—Expected deviation from initial calibration of a W-3Re/W-25Re thermocouple alloyed to represent a transmuted composition after 6 months in a 10^{14} neutron thermal flux

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5. REFRACTORY-METAL ALLOY RESEARCH AND DEVELOPMENT

(57015)

The overall objective of this program is to advance the technology of refractory metals to permit the early utilization of these materials in high-temperature reactors. Specifically, the program encompasses development of new refractory-metal alloys to improve the temperature and life capabilities of nuclear power plants.

PURIFICATION OF Mo AND Mo ALLOYS

Investigations were continued on techniques for obtaining high-purity molybdenum by long-time heat treatment in hydrogen. Studies were continued on a range of molybdenum alloy compositions having both useful room-temperature ductility and good strength at high temperatures. Recently, activities have been extended to the preparation of dispersion-strengthened-type refractory-metal alloys.

Purification of Mo

An objective of this program is to obtain Mo stock that will retain its ductility after heating to 1600°C or above, and after welding. Commercial arc-cast Mo loses room-temperature ductility when heated at 1600°C and above, and powder metallurgy Mo is not considered weldable. Creep-rupture data on commercial Mo and Mo alloys showed that the Mo-TZM alloy is not as strong as Mo - 50Re;^{1,2} therefore, another objective of this program is to develop Mo alloys with greater creep resistance than Mo-TZM but of lower alloy content than Mo - 50Re.

Techniques were previously developed that reduce the carbon and oxygen contaminants in pressed powder metal compacts to less than 10 ppm each and as low as 5 ppm in many cases. This is accomplished by exposing the compacted powders for prolonged periods at 1000°C in wet hydrogen followed by another long-time treatment in dry hydrogen, first at 1000°C and then at 1450°C, to remove carbon and dissolved oxygen.

After receiving these treatments, laboratory-produced high-purity Mo sheet was compared for weldability with commercial powder-metallurgy and arc-cast sheets. Results of these tests, shown in Table 5.1, indicate that purification of the Mo powder improves the weld structure without the loss of ductility as shown in the arc-cast material. A possible cause of the porosity noted is that carbon and oxygen form carbon monoxide gas during the welding operation.

In addition to the problem of porosity, two other problem areas developed during work with the purified Mo sheet stock: cracks of unknown origin appeared during cold-rolling

¹All compositions are in atomic percent unless otherwise specified.

²P. N. Flagella, "High-Temperature Stress-Rupture Characteristics of Mo and Some Mo Alloys," GE-NMPO, GE-TM 65-4-39, presented at AIAA Propulsion Joint Specialists Conference, Colorado Springs, Colorado, June 1965.

TABLE 5.1
COMPARISON OF WELDABILITY AND BEND TESTS ON MOLYBDENUM SHEET
FROM THREE SOURCES

Material	Room-Temperature Bend Tests			Condition of Welds
	As-Received	After 2 Hour Heat Treatment at 2400°C	After Welding	
Commercial powder-metallurgy sheet	90° bends	90° bends (fine-grained)	Not tested	Large blow holes (unweldable)
Commercial arc-cast sheet	90° bends	Brittle ^a	Brittle	Excellent
GE-NMPO powder-metallurgy sheet	90° bends	90° bends (fine-grained)	45°-90° bends	Microporosity

^aCoarsened to very large grains.

sheet and material believed to be defect-free cracked during a high-speed sheet forming operation. The cracking problem was eliminated by increasing the care to prevent contamination while handling the green powder compacts and by taking heavier passes in initial hot-rolling reduction stages.

Since the cause of the second problem appeared to be strain-rate sensitivity of Mo sheet, an experiment was designed to determine if the stress relieving temperature used influences its forming properties. Specimens were prepared from high-purity 0.050-cm-thick Mo sheet stock. All specimens were stress-relieved for 20 minutes in hydrogen at the following temperatures: 1450°, 1600°, 1800°, 2000°, and 2400°C. The range of temperatures covered gave stress-relieved specimens having progressively larger grain sizes.

Room-temperature bends of 90 degrees were then successfully performed both parallel and transverse to the direction of the final cold-rolling operation. The ability of a sheet to take a 90-degree or greater bend is considered to be a good criterion of the ductility of Mo sheet. As all sheets tested were given 90-degree bends without breaking, it appears that impurities have a greater effect on strain-rate sensitivity than does the stress relieving temperature.

Ductility of EB-Welded Purified Mo – To establish the effects of EB welds on purified Mo, sheets of purified Mo were EB-welded and then subjected to room-temperature and -50°C bend tests. Results are shown in Table 5.2. Figure 5.1 shows the welded specimens after bend testing.

TABLE 5.2
DUCTILITY OF EB-WELDED PURIFIED Mo SHEET^a

Sheet Designation	As-Welded	As-Welded and Stress-Relieved ^b	
	Room-Temperature Bend, degrees	Room-Temperature Bend, degrees	-50°C Bend, degrees
3	10	50	30
4	45	45	45
5	10	>90	70
6	>90	>90	>90
7	30	>90	—
8	30	>90	>90
9	45	>90	>90

^aBends conducted with 2T radius punch in accordance with recommendations of the Materials Advisory Board for refractory sheet.

^bStress-relieved for 2 hours at 1450°C in vacuum after welding.

Fig. 5.1 – Bends completed at room temperature and at -50°C on 0.050-cm-thick electron beam welded Mo sheet. Specimens were stress-relieved for 2 hours at 1450°C in hydrogen before bending. (Neg. P66-4-2)

These tests show that EB-welded, purified Mo has good ductility at temperatures as low as -50°C instead of the commonly accepted 100°C ductile-to-brittle transition temperature (DBTT) found in commercial material.

As is characteristic of most body-centered cubic metals, molybdenum is strain-rate sensitive; i. e., high deformation rates cause a loss of ductility and apparent embrittlement. Table 5.3 shows the initial results of three different bend rates recently used in evaluating EB-welded Mo sheet at various temperatures. No conclusions are drawn at this time but it now appears that, at low punch speeds, good bends can be made at temperatures as low as -50°C .

TABLE 5.3
EFFECTS OF TEMPERATURE AND DEFORMATION RATE ON
DUCTILITY OF PURIFIED EB-WELDED Mo SHEET^a

Test Temperature, $^{\circ}\text{C}$	Bend, degrees		
	Punch Speed 0.25 cm/min	Punch Speed 0.025 cm/min	Punch Speed 0.009 cm/min
200	>90	—	—
150	>90	—	—
100	>90	—	—
20	>90 ^b	—	—
-50	—	5, 5 ^c	—
-50	—	—	35, 60 ^c

^aWelds were not stress-relieved before testing.

^bShowed edge crack after bending to 45-60 degrees; weld area remained sound.

^cResults shown for two tests.

Purification of Mo Alloys

The purification program also included studies performed on three molybdenum alloys: Mo - 5Re, Mo - 3Re - 5W, and Mo - 5W. All alloys were purified by treating the compacts for 600 hours in wet hydrogen (dewpoint 5°C) plus 48 hours at 1400°C in dry hydrogen (dewpoint -55°C) and then processing to sheet. As shown in Figure 5.2, there was a marked difference in coloration and shrinkage among the compacts in the presintered stage. Appearance of the compacts after sintering for 3 hours at 2540°C in hydrogen is also shown in Figure 5.2.

Weld porosity of the alloys was evaluated by making an EB weld fusion across each sheet. A contact X-ray photographic print of the completed welds is shown in Figure 5.3. The Mo - 5Re alloy weld area shows many voids, whereas the Mo - 3Re - 5W and Mo - 5W welds contain very few voids. Apparently, the addition of 5 percent tungsten significantly reduces the porosity in welds areas. Creep-rupture tests and a chemical analysis for trace impurities will be performed on the alloys.

Because carbon, oxygen, and volatile trace elements appeared to be forming voids in welds of Mo and Mo-base alloys, a purification program was initiated to reduce these contaminants. A 500-gram batch of high-purity Mo powder, made by the fluorine dissociation process, was obtained from ORNL. The spectrochemical analysis of this batch, in ppm, was:

Ag - <1	Co - <10	Na - <100
Al - <1	Cr - <10	Ni - 30
B - <20	Cu - 2	Si - 4
Be - <1	F - 113	Sn - <10
Bi - <1	Fe - 10	Ti - 15
C - 42	Mg - <1	V - <20
Cd - <10	Mn - <1	

Oxygen analysis was 1.36%.

Fig. 5.2 - Bottom row: unalloyed Mo, Mo - 5Re, and Mo - 3Re before sintering. Top row: purified and fully sintered compacts of the same compositions. (Neg. P66-2-18)

Fig. 5.3 - X-rays of welds in alloyed Mo sheet. White dots in the Mo - 5Re specimen are internal porous areas not visually observed on the welded sheet. Pores are not noted in Mo - 3Re - 5W alloy. Possible small pores in the Mo - 5W alloy appear only at the starting end of the weld (right side). (Neg. 8079)

The powder was compacted to bars for sheet rolling, and the sheets are currently undergoing a 600-hour water-vapor - hydrogen treatment to reduce carbon content. These treatments are also expected to remove essentially all the oxygen and fluorine.

After purification, the material will be processed to sheet stock and subjected to a complete property evaluation for comparison with commercial and laboratory-produced material.

W - 30Re - 30Mo ALLOY DEVELOPMENT

A total of 29 W-Re-Mo alloys have been evaluated for use in this program. Of these, W - 25Re - 30Mo and W - 30Re - 30Mo alloys were selected for advanced development and evaluation. Sheets from trial production runs and tubing made from these two alloys are being used in fuel element development programs. Work during this reporting period was largely confined to the W - 30Re - 30Mo alloy.

Influence of Stress-Relief Temperature on Ductility

High-quality W - 30Re - 30Mo alloy sheet can be produced to a maximum width of about 11 cm with available in-house rolling equipment. In fabricating prototype reactor core components, the sheets must be bent parallel to the rolling direction to achieve the desired length rather than bending transverse to the rolling direction which is normally desired. A series of 0.025-cm-thick W - 30Re - 30Mo sheet specimens was treated, at 25°C temperature intervals from 1425° to 1700°C in hydrogen, to evaluate the effect of stress relief on bend ductility parallel to the rolling direction. A punch speed of 5 cm/min over a 2T radius was used, starting with the specimens stress-relieved at the highest temperature, until a failure was detected. Thereafter, the specimens stress-relieved at the lower temperatures were tested at a punch speed of 0.125 cm/min.

The bend test data given in Table 5.4 show that good bend ductility parallel to the rolling direction is achieved at stress-relief temperatures of 1575° to 1700°C. As shown in Figure 5.4, multiple cracks tend to occur in grain boundaries on the tensile stress side of specimens stress-relieved below 1575°C. Also, specimens stress-relieved in the temperature range from 1550° to 1625°C showed numerous cracks in the sigma phase particles in the bend-deformed metal structure. The conclusion, therefore, is that stress-relief temperatures of 1650°C should be used for working and forming operations.

TABLE 5.4

ROOM-TEMPERATURE DUCTILITY OF W - 30Re - 30Mo ALLOY^a

Lab No.	Stress-Relief Temperature, °C ^b	Hardness ^c (Rockwell-A)	Bend Punch Speed, cm/min	Bend Test Results, degrees
1	1400	71.6	0.125	Broke, >45 <90
2	1425	70.6	0.125	Broke, >45 <90
3	1450	70.6	0.125	Broke, >45 <90
4	1475	70.2	0.125	Broke, >45 <90
5	1500	70.2	0.125	Broke, >45 <90
6	1525	70.0	0.125	Cracked, >90
7	1550	69.0	5.0	Cracked, >90
8	1575	69.8	5.0	>90
9	1600	69.6	5.0	>90
10	1625	69.6	5.0	>90
11	1650	69.2	5.0	>90
12	1675	68.8	5.0	>90
13	1700	67.6	5.0	>90

^aBend crease was parallel to rolling direction; bend radius was approximately 2T.

^bStress-relieved for 20 minutes in hydrogen at indicated temperatures.

^cAverage of four readings.

Development of Tube Drawing Procedure

The objectives of the tube drawing program are to select the best of several possible techniques for a trial production run of W - 30Re - 30Mo tubing and to prepare starting material for the trial production run. Basic starting material is 1.40-cm-OD extruded tube having an ID-to-OD ratio of 0.723 to 0.745. The starting size permits clean-up to a 0.818 finished ID-to-OD ratio.

The tube drawing techniques under consideration are shown in Table 5.5. Results to date indicate that W - 30Re - 30Mo tubing down to 0.474-cm ID can be obtained through sinking, moving-mandrel drawing, or leachable-core-filler drawing. Stress relieving at 1650°C between drawings produces better results than stress relieving at 1450°C.

The remaining problem is to demonstrate that at least one of the techniques will be suited to draw tubing down to an ID of 0.343 cm.

WORK PLANNED FOR NEXT PERIOD

Trial production runs of small seamless refractory-metal tubing will be made using procedures described in this report. Nuclear standards of quality are required in the tubing.

Work will be initiated on investigation of the composition and processing techniques for Ta- and Cr-base alloys.

Fig. 5.4 – Cross section through room-temperature bends of W – 30Re – 30Mo alloy. Bends were made after stress-relief treatments at temperatures ranging from 1425°C to 1700°C at 25°C intervals as indicated. Fold or crease at bend is parallel to rolling direction of the sheet. (As-polished, 200X.)

Fig. 5.4(Cont.)—Cross section through room-temperature bends of W-30Re-30Mo alloy. Bends were made after stress-relief treatments at temperatures ranging from 1425° to 1700°C at 25°C intervals as indicated. Fold or crease at bend is parallel to rolling direction of the sheet. (As-polished 200X.)

TABLE 5.5
W - 30Re - 30Mo TUBE DRAWING TECHNIQUES BEING EVALUATED

Extrusion Piece No.	Stress-Relief Temperature, °C	Basic Drawing Process	Lubricant on OD Drawing Surface	Current Status
1	1650	Draw on Mo-Hf removable	WS ₂ ^b	OK at 0.90 cm OD
2	1450	filler core ^a		
3	1650	Draw on hardened tool-	GE ^d	Continued
4	1450	steel mandrel ^c		
5	1650	Draw on hardened tool-	WS ₂ ^b	OK at 0.90 cm OD
6	1450	steel mandrel ^c		
7	1650	Sink - no mandrel nor core	WS ₂ ^b	OK at 0.90 cm OD
8	1450	filler during drawing ^e		
9	1650	Originally reserves, now	WS ₂ ^b	-
10	1450	processing as extrusion piece No. 5		

^aMolybdenum with Hf leachable filler placed in tube after honing the ID and grinding the OD to 0.818 ID/OD ratio.

^bA commercial lubricant.

^cNo lubricant on mandrel.

^dA tungsten drawing lubricant obtained from GE-LMCD which gives higher drawing loads than WS₂ lube. Both specimens 3 and 4 continued after 2 draws with WS₂ lube.

^eTube sized to 0.818 ID/OD ratio prior to drawing.

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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

Report No.	Report Period	Publication Date
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
GEMP-49, A and B	April 15, 1965 – June 15, 1965	July 28, 1965
GEMP-50, A and B	May 15, 1965 – July 15, 1965	August 20, 1965
GEMP-51, A and B	June 15, 1965 – August 15, 1965	September 30, 1965
GEMP-52, A and B	July 15, 1965 – September 15, 1965	October 20, 1965
GEMP-53, A and B	August 15, 1965 – October 15, 1965	November 26, 1965
GEMP-54, A and B	September 15, 1965 – November 15, 1965	December 20, 1965
GEMP-55, A and B	October 15, 1965 – December 15, 1965	January 21, 1966
GEMP-400, A and B (Fifth Annual Report)	January 1, 1965 – January 31, 1966	February 28, 1966
GEMP-58, A and B	February 1, 1966 – March 15, 1966	April 29, 1966

ATOMIC PRODUCTS DIVISION

GENERAL ELECTRIC