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INFORMAL AEC RESEARCH
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MASTER

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EXPOSURE OF HTGR CANDIDATE CORE PLATE AND
THERMAL INSULATION MATERIALS TO IMPURE HELIUM
AT 1650°F TO 1850°F FOR 3000 HOURS

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INTRODUCTION

Several materials that are being considered for use in HTGR structures (i.e., thermal insulation, plenum shroud, core plate, and pressure vessels) have been subjected to an 18,000-hr exposure at 1100°F and 1375°F under the following conditions: (1) purified helium, (2) helium containing 300 ppm CO and H₂, respectively, and (3) helium containing 3000 ppm CO and H₂, respectively. In March 1965, it was decided to extend these experiments to include temperatures of 1650°F and 1850°F on some of the previously tested materials and on new materials. Accordingly, four materials were exposed for 1500 hr and two materials were exposed for 3000 hr at these new temperatures in helium containing 3000 ppm CO and H₂. Mechanical tests and metallographic examinations were performed periodically to evaluate changes in mechanical properties.

The test was originally planned for an 18,000-hr exposure; however, results of mechanical property tests and metallographic examination of samples taken after the 500-hr and the 1500-hr exposures indicated that sufficient data had been obtained to allow termination of the tests on four of the ~~six~~ materials. Tests on specimens of 430 stainless steel and Hastelloy X were continued for an additional 1500 hr, for a total of 3000 hr, before experimentation was discontinued.

ENVIRONMENTAL CONDITIONS

The environmental conditions of the experiments were as follows. The specimens were exposed in helium containing 3000 ppm CO and H₂ at 1650°F and 1850°F. The CO and H₂ were generated by feeding water into hot graphite under controlled conditions. The gas was bled into helium and then passed over the specimens in muffle furnaces at the above temperatures. The furnaces were shut down after the 500-hr exposure, the 1500-hr exposure, and the 3000-hr exposure, and mechanical properties were evaluated after each shutdown. Major components of the control system are described below.

Furnace Controls

A four-section control panel was fabricated with a separate section for each of the four furnaces. Each panel consisted of a control thermocouple, a Variac control, a timer, an ammeter, and a voltmeter; a safety

device was included in case a furnace should go out of control because of a short circuit or a burnout of the control thermocouple. The controls were operated at 110 V, and each furnace was operated at 208 V.

Thermocouples

Three thermocouples were wired to the test specimens, one at each end and one in the center. Thermocouple readings were continuously recorded on two Minneapolis-Honeywell 15-point strip chart recorders. In addition, two thermocouples were located near the furnace winding. One was used on the controller, the other on the Sim-Ply-Trol safety device.

Environmental Gas System

A four-cylinder helium gas manifold with a regulator was installed with valving that allowed operation of one cylinder at a time. One cylinder was kept in reserve and was used while changing the cylinders in the manifold. From the manifold, the gas flow entered two hot traps, one containing uranium chips and the other containing titanium chips. The furnace with uranium chips was at 1000°F, and the furnace with titanium chips was at 1400°F. From the hot traps, a portion of the gas was diverted through flow regulators, through water bubblers to pick up moisture, and then into two high-temperature (2150°F to 2200°F) Kanthal furnaces containing mullite tubes packed with graphite. The ends of each mullite tube were packed with quartz wool, allowing the tubes to be set so that the graphite on the exit side was in the center of the furnace hot zone. The high-temperature furnaces converted the water in the gas into oxygen and hydrogen, and, as the gas passed through the graphite, the CO impurity was formed. The diverted gas was then returned to the main gas flow line. This method controlled the quantity of impurities in each system. From the junction of the diverted gas line and the main gas line, the gas was fed into a manifold that was designed to allow passage of the gas through both furnaces, through any one furnace, or through the gas chromatograph. It was also possible to bypass all furnaces and analyze the gas prior to entering the furnaces.

If the temperature in the high-temperature Kanthal furnaces dropped below 2100°F, there was a possibility that water would enter the test specimen area. It was therefore important to check for moisture. There were two methods of detecting moisture in this system: (1) a dew-point indicator with a sensitivity of 0.0003% vol and (2) a container of silicagel with indicator. As a safety feature, two solenoids, controlled by a thermocouple in each of the high-temperature furnaces, were inserted in the input line to the furnaces.

MATERIALS

At the start of the experiment, the following six materials were exposed:

1. 0.065-in. Rene 41 strip (precipitation hardened)
2. 0.065-in. Inconel X strip (precipitation hardened)
3. 0.060-in. Hastelloy X (solution annealed)
4. 0.060-in. 304 stainless steel strip (solution annealed)
5. 0.076-in. 430 stainless steel strip (cold rolled and annealed at 1450°F)
6. 0.061-in. Incoloy 800 strip (solution annealed)

The chemical analysis of the materials is shown in Table 1.

HEAT TREATMENT

The heat treatments given to the materials prior to startup were as follows:

1. Rene 41--solution annealed at 2150°F, hardened for 4 hr at 1650°F, air cooled
2. 430 stainless steel--annealed 1/2 hr at 1450°F
3. 304 stainless steel--cold rolled and annealed at 1950°F, water quenched
4. Hastelloy X--solution annealed at 2150°F, water quenched
5. Incoloy 800--solution annealed at 2150°F, air cooled
6. Inconel X--solution annealed at 2100°F for 2 hr and air cooled, aged 24 hr at 1550°F and air cooled, reaged 20 hr at 1300°F and air cooled

The samples were 4-in. long strips, cut to standard ASTM specifications for tensile bars with a 1-in. gauge length. The width of the tensile bars was 1/4 in. at gauge location.

EVALUATIONS

Mechanical properties of the various materials before exposure and after the three exposures (500 hr, 1500 hr, 3000 hr) are shown in Figs. 1 through 3 and in Table 2. A series of photomicrographs (Figs. 4 through 42) shows various changes in structure during the exposures. Rene 41 and Inconel X were overaged because of exposure to temperature above their aging temperature. Both materials showed severe intergranular attack

Table 1
CHEMICAL ANALYSIS*
(%-wt)

Elements	Rene 41	430 SS	304 SS	Hastelloy X	Incoloy 800	Inconel X
Cr	19.03	16.30	17.3	20.77	19.99	14.87
Fe	2.16	Bal.	Bal.	18.63	45.24	6.55
C	0.09	0.059	0.041	0.11	0.04	0.03
Si	0.15	0.37	0.64	0.71	0.40	0.31
Co	11.02	----	----	2.32	----	---
Ni	Bal.	0.25	9.5	Bal.	33.04	73.36
Mn	0.05	0.41	1.52	0.71	0.85	0.56
Mo	9.69	----	0.2	9.03	----	---
S	0.010	0.010	----	0.003	0.007	0.007
Al	1.51	----	----	----	----	0.71
Ti	3.19	----	0.03	----	----	2.70
Other	B = 0.005	P = 0.017 Cu = 0.20		P = 0.012 W = 0.66	Cu = 0.41	Cu = 0.04, Cb and Ta = 0.84

* Manufacturer's analysis.

Table 2
MECHANICAL PROPERTIES

Material	Before Exposure			500 Hours					
	Tensile Strength (psi $\times 10^3$)	Yield Strength		Tensile Strength		Yield Strength		Elongation	
		0.2% Offset (psi $\times 10^3$)	Elongation (%/1 in.)	(psi $\times 10^3$)		0.2% Offset (psi $\times 10^3$)		(%/1 in.)	
				1650°F	1850°F	1650°F	1850°F	1650°F	1850°F
Rene 41	191.0	132.3	21	145.3	165.6	107.7	101.0	4	19
430 SS	79.2	51.6	32.5	67.4	67.1	38.5	34.6	35.5	21
304 SS	92.1	45.0	75	89.0	80.8	32.7	27.2	59	40.5
Hastelloy X	116.8	64.1	42	115.5	105.5	49.1	46.0	25	23.5
Incoloy 800	79.1	40.5	44	79.1	75.2	28.1	26.4	35.5	23
Inconel X	173.8	113.2	26	160.0	144.2	91.6	76.0	26	29.5

Table 2 (Continued)

Material	1500 Hours						3000 Hours					
	Tensile Strength (psi × 10 ³)		Yield Strength 0.2% Offset (psi × 10 ³)		Elongation (%/1 in.)		Tensile Strength (psi × 10 ³)		Yield Strength 0.2% Offset (psi × 10 ³)		Elongation (%/1 in.)	
	1650°F	1850°F	1650°F	1850°F	1650°F	1850°F	1650°F	1850°F	1650°F	1850°F	1650°F	1850°F
Rene 41	129.3	148.0	102.4	87.4	4	17	-----	-----	-----	-----	--	--
430 SS	80.0	60.4	42.1	33.6	27.5	25.5	78.2	93.6	42.6	61.5	8	(a)
304 SS	89.6	76.4	32.4	25.2	27.5	33	-----	-----	-----	-----	--	--
Hastelloy X	101.8	91.8	46.0	44.4	28	25	103.2	93.2	46.4	44.4	28	28
Incoloy 800	63.9	68.6	26.9	24.8	13	22.5	-----	-----	-----	-----	--	--
Inconel X	153.1	124.9	90.7	59.3	29.5	27	-----	-----	-----	-----	--	--

^a Broke out of gauge.

and carburization followed by oxidation (see Figs. 6 through 9, 18, and 19, 29 through 32, and 33 through 36). Type 304 stainless steel showed grain growth and sensitivity at 1650°F and 1850°F and intergranular attack at 1850°F (see Figs. 13, 27, and 28). Incoloy 800 showed grain growth and intergranular attack at both test temperatures (see Figs. 16, 17, and 23 through 26).

Mechanical property measurements and metallographic examination showed that Hastelloy X and 430 stainless steel were the materials least affected by exposures at 1650°F and 1850°F. Of these two materials, the least affected at 1850°F was Hastelloy X. It is even more apparent from the metallographic examination that Hastelloy X is the better material for use at both temperatures; however, 430 stainless steel, the less expensive material, was not significantly changed at 1650°F and therefore should be considered for a thermal insulation material up to that temperature. A comparison of the photomicrographs in Figs. 11, 22, and 40 (430 stainless steel) and in Figs. 15, 38, and 42 (Hastelloy X) shows that grain growth and the formation of martensite with a possible intermetallic compound had occurred in the 430 stainless steel after the 1850°F exposure. Diamond penetration hardness measurements were also made on the two materials (see Table 3). The results of these measurements verified the formation of martensite in the 430 stainless steel that had been exposed at 1850°F.

Table 3
HARDNESS TESTS AFTER 3000 HOURS

Material	Temperature (°F)	Diamond Penetration Hardness	Rockwell
Hastelloy X	1650	293	R _C 28
Hastelloy X	1850	278	R _C 27
430 SS Ferrite	1650	181	R _b 89
430 SS Martensite	1650	237	---
430 SS Ferrite	1850	221	R _b 97
430 SS Martensite	1850	420	R _C 42

The results of the gas analysis at the input and the exit of the furnaces are plotted in Fig. 43. It is clear from the figure that most of the carburization had occurred by 1500 hr of exposure, and then substantial equilibrium was achieved. Figure 43 clearly shows that during the first 100 hr of exposure, virtually all of the CO introduced into the specimen area was diffused

into the specimens. Metallographic examinations showed that Rene 41 and Inconel X were the primary absorbers of CO and that some time after 500 hr of exposure they had developed an oxide layer that prevented the diffusion of carbon.

Figure 5 is a photograph of the specimens, as installed in the furnaces. The photograph was made after the first 500 hr of exposure. The gauge length of the specimens is 1 in., and the width is approximately 1/4 in.

Creep testing was attempted, with limited success. Specimens of Hastelloy X, exposed for 1500 hr at 1650°F and 1850°F, were placed in duplicate in the arc weld creep testing machines in a purified helium atmosphere. The duplicate specimens exposed at 1650°F were tested at 1650°F, and those exposed at 1850°F were tested at 1850°F. Standard ASTM procedures for creep testing were observed. Specimens were in tension at stresses of 2800 psi at 1650°F and 850 psi at 1850°F. Based on published data, these stresses were selected to give 1/2% total elongation in 1000 hr. Platinum strips were spot welded to the specimens and the strains measured with optical cathetometers.

One of the specimens exposed at 1650°F ruptured in 550 hr and a second stage creep could not be measured. The other specimen at 1650°F was exposed for 1700 hr, and then the exposure was discontinued. First stage creep was completed in 500 hr, with a strain of 0.0163 in./in. From 500 hr to 1700 hr, there was no measurable plastic strain.

At the 1850°F exposure, one of the furnaces burned out after 17 hr, and no data could be obtained. The other furnace burned out after 365 hr. A plastic strain of 0.002 in./in. was measured on the second specimen for the first stage creep after 125 hr. There was no measurable plastic strain between the 125-hr exposure and the time the furnace burned out.

CONCLUSIONS

The following conclusions can be drawn from the experiments:

1. The results obtained in this experiment show type 430 stainless steel to be the logical choice for metallic thermal insulation in an HTGR.
2. Of the alloys tested at 1850°F, Hastelloy X was least affected by the HTGR environment.
3. Because Hastelloy X is prohibitively expensive in foil thickness materials, 430 stainless steel should be considered for thermal insulation, providing the temperature does not exceed 1650°F.

4. It appears that carburization from small partial pressures of carbon monoxide in helium quickly reaches a saturation point; therefore, carburization of material of structural thickness would not pose a threat to reactor safety.

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Fig. 2--Mechanical property changes of 430 stainless steel and Incoloy 800

Fig. 3--Mechanical property changes of Hastelloy X and 304 stainless steel

M-11490-1

(100×)

Fig. 4--430 SS annealed at 1450°F--etched (2% Nital)

M-12063-2

(1/2×)

Fig. 5--Specimens after 500-hr exposure--unetched

M-12092-1

(100×)

Fig. 6--Rene 41, 500-hr exposure, 1650°F--etched (10% Oxalic)

M-12092-2

(100×)

Fig. 7--Rene 41, 500-hr exposure, 1650°F--unetched

M12098-1

(100×)

Fig. 8--Rene 41, 500-hr exposure, 1850^oF--etched (10% Oxalic)

M12098-2

(100×)

Fig. 9--Rene 41, 500-hr exposure, 1850^oF--etched (Kallings)

M-12097-1

(100×)

Fig. 10--430 SS, 500-hr exposure, 1650°F--etched (2% Nital)

M-12103-1

(100×)

Fig. 11--430 SS, 500-hr exposure, 1850°F--etched (2% Nital)

M-12096-1

(100×)

Fig. 12--304 SS, 500-hr exposure, 1650°F--etched (10% Oxalic)

M-12102-1

(100×)

Fig. 13--304 SS, 500-hr exposure, 1850°F--etched (10% Oxalic)

M-12095-1

(100×)

Fig. 14--Hastelloy X, 500-hr exposure, 1650°F--etched (10% Oxalic)

M-12101-1

(100×)

Fig. 15--Hastelloy X, 500-hr exposure, 1850°F--etched (10% Oxalic)

M-12094-1

(100×)

Fig. 16--Incoloy 800, 500-hr exposure, 1650^oF--etched (10% Oxalic)

M-12100-1

(100×)

Fig. 17--Incoloy 800, 500-hr exposure, 1850^oF--etched (10% Oxalic)

M-12087-1

(100×)

Fig. 18--Inconel X, as treated and installed--etched (10% Oxalic)

M-12093-1

(100×)

Fig. 19--Inconel X, 500-hr exposure, 1650°F--etched (10% Oxalic)

M-12099-1

(100×)

Fig. 20--Inconel X, 500-hr exposure, 1850° F--etched (10% Oxalic)

M-13184-1

(100×)

Fig. 21--430 SS, 1500-hr exposure, 1650° F--etched (Villemas)

M-13185-1

(100×)

Fig. 22--430 SS, 1500-hr exposure, 1850^oF--etched (Villelias)

M-13175-1

(100×)

Fig. 23--Incoloy 800, 1500-hr exposure, 1650^oF--etched (10% Oxalic)

M-13180-1

(100×)

Fig. 24--Incoloy 800, 1500-hr exposure, 1850°F--unetched

M-13174-2

(100×)

Fig. 25--Incoloy 800, 1500-hr exposure, 1650°F--unetched

M-13180-2

(100×)

Fig. 26--Incoloy 800, 1500-hr exposure, 1850° F--etched (10% Oxalic)

M-13174-1

(100×)

Fig. 27--304 SS, 1500-hr exposure, 1650° F--etched (10% Oxalic)

M-13179-1

(100×)

Fig. 28--304 SS, 1500-hr exposure, 1850°F--etched (10% Oxalic)

M-13182-2

(100×)

Fig. 29--Inconel X, 1500-hr exposure, 1850°F--unetched

M-13182-1

(100×)

Fig. 30--Inconel X, 1500-hr exposure, 1850°F--etched (10% Oxalic)

M-13177-2

(100×)

Fig. 31--Inconel X, 1500-hr exposure, 1650°F--unetched

M-13177-1

(100×)

Fig. 32--Inconel X, 1500-hr exposure, 1650°F--etched (10% Oxalic)

M-13178-2

(100×)

Fig. 33--Rene 41, 1500-hr exposure, 1650°F--unetched

M-13178-1

(100×)

Fig. 34--Rene 41, 1500-hr exposure, 1650°F--etched (10% Oxalic)

M-13183-2

(100×)

Fig. 35--Rene 41, 1500-hr exposure, 1850°F--unetched

M-13183-1

(100×)

Fig. 36--Rene 41, 1500-hr exposure, 1850^oF--etched (10% Oxalic)

M-13176-1

(100×)

Fig. 37--Hastelloy X, 1500-hr exposure, 1650^oF--etched (10% Oxalic)

M-13181-1

(100×)

Fig. 38--Hastelloy X, 1500-hr exposure, 1850^oF--etched (10% Oxalic)

M-14791-1

(100×)

Fig. 39--430 SS, 3000-hr exposure, 1650^oF--etched (2% Nital)

M-14790-1

(100×)

Fig. 40--430 SS, 3000-hr exposure, 1850°F--etched (2% Nital)

M-14788-1

(100×)

Fig. 41--Hastelloy X, 3000-hr exposure, 1650°F--etched (10% Oxalic)

M-14789-1

(100×)

Fig. 42--Hastelloy X, 3000-hr exposure,
1850°F--etched (10% Oxalic)

Fig. 43--Gas analysis