INTERIM REPORT ON HTLTR MOCKUP
RUN NUMBER 3

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INTERIM REPORT ON HTLTR
MOCKUP RUN NUMBER 4

INTRODUCTION

Herein are presented results obtained during mockup Run Number 4. The mockup was designed and built by Equipment Development Unit for research and development in support of the High Temperature Lattice Test Reactor (HTLTR). Data obtained during Run Number 3 were presented in BNWL-CC-425.

This was the final scheduled run in the test series and was performed to determine the integrity of the control rod, safety blade, heater elements and thermocouples when exposed to a graphite core temperature of 1000°C for a period of 42 days. This is estimated to be the equivalent of approximately four years of accumulated operating time at 1000°C in the HTLTR. It was believed that if the components on test survived a test of this duration they would be satisfactory for use in the reactor.

SUMMARY AND CONCLUSIONS

Run Number 4 was terminated after 1010 hours of operation at a graphite core temperature of 1000°C. Subsequent to heated operation the mockup was cooled and components on test, viz: control rod, safety blade, heating elements and thermocouples were removed and inspected. The interior of the mockup at the end of Run Number 4 is shown in Figure 1.

Corrosion of that portion of the heating elements exposed to the circulating mockup atmosphere was minimal and can be seen in Figure 2.

Graphite samples located within the mockup exhibited an average weight loss rate of 1.2×10^{-6} gm-hr⁻¹-cm⁻². This rate is approximately 65% of the average rate observed during Run Number 3.

Maximum gas contaminant concentrations observed at the blower discharge during the run were 2350 vppm H₂O, 300 vppm CO₂, and 2450 vppm CO. These

concentrations had decreased to 70 vppm H_2O , 30 vppm CO_2 , and 600 vppm CO by the end of the run. Maximum hydrogen concentration observed in the mockup outlet (heat exchanger inlet) gas was 3200 vppm. The only evacuation in which water was observed at the vacuum pump discharge was the final, heated (290°C) evacuation which resulted in collecting approximately 0.5 liters of water. A total of 26.5 liters of water were removed in the molecular sieve towers during the run.

The chromatograph to be used at the reactor for analyzing O_2 , CO_2 and CO was included in the mockup sampling system to permit shakedown of the chromatograph prior to installation at the reactor site. Satisfactory operation was achieved after minor modifications were made to the chromatograph circuitry; e.g., replacing the range attenuators and lengthening the sampling time cycle.

Control rod and safety blade scrams totalled 67 and 68, respectively, forty-three of which were performed at a core temperature of 1000°C . Safety blade clutch slippage, noted in the previous two runs, was not encountered due to the larger magnetic clutch employed during this run. The long duration test revealed a boron transport problem not previously encountered, as evidenced by the deposits seen in Figure 3. Tests of the deposited material indicated a high boron concentration and it is postulated that water vapor present in the mockup oxidized boron carbide present in the safety blade forming volatile metaboric acid (HBO_2). The metaboric acid was thermally decomposed when exposed to the high temperature within the mockup and was deposited on the safety blade sheath in the form of boron oxide glass (B_2O_3), which is liquid at 1000°C . The glass then coalesced on the safety blade sheath in a manner not unsimilar to that noted in dropwise condensation. Boron transport in this or any other manner is intolerable and therefore renders

B₄C an unsatisfactory candidate for use as the safety blade poison. An alternate poison material, consisting of co-precipitated Gadolinia (GD₂O₃)-Europie (Eu₂O₃), and nickel, is now under consideration.

No evidence of TD nickel support rod corrosion of the magnitude noted in Run Number 3 was apparent upon disassembly of the control rod. This major reduction in corrosion is attributed to the use of a UO₂-graphite material containing fewer contaminant(s) than that used in Run Number 3. Control rod and safety blade scram times averaged 0.65 seconds and 0.93 seconds, respectively, throughout the run.

Calibration of the 1/8" OD inconel sheathed, chromel-alumel thermocouples at the conclusion of the run indicated a maximum error at 2000°F of -3.9% and an average error of -1.89% for eleven thermocouples. An error of less than 0.1% was noted for an unexposed thermocouple. The drift in calibration after exposure is attributed to carbon diffusion into the thermocouple junction thus changing the junction characteristics. Except for the grounded junction, no carbon transport into the wires was evident. Consequently, the ungrounded thermocouples to be used in the HTLTR should experience less carbon contamination of the junction than the grounded thermocouples used in the mockup.

One stainless steel heat exchanger tube was removed and sectioned to permit inspection of the tube for carbon deposition. The maximum tube deposition noted was 1.5 mg/cm². This tube had been in service during the four runs completed thus far and can be seen in Figure 4.

DISCUSSION

Gas System

The run was initiated by alternately evacuating the system and filling with fresh nitrogen nine times. Three of the evacuations were performed at elevated temperatures from 175°C to 300°C. Four hundred and

sixty ml of water were collected in the vacuum pump discharge during the final 290°C evacuation. This was the only water collected during the nine evacuations. Evacuation periods ranged from 2 hours to 20 hours and pressures of 19 torr to 32 torr were attained. Pressure data obtained during one of the evacuations are presented in Figure 5.

Results of an inleakage check performed at the end of the third evacuation are presented in Table 1.

TABLE 1

<u>Time after start, Min.</u>	<u>Gage Press., in. H₂O</u>	<u>Barometric Press., in H₂O</u>	<u>Absolute Press., in H₂O</u>
0	-384.2	399.7	15.5
5	-382.5	↓	17.2
10	-380.0		19.7
15	-376.3		23.4
20	-374.0		25.7
25	-371.0		28.7
30	-368.5		31.2
35	-365.0		34.7
45	-359.0		40.7
50	-356.0		43.7
60	-351.0		48.7
70	-344.0		55.7
80	-339.0		60.7
100	-326.0		73.7
120	-315.0		84.7

An inleakage rate, based on the above data, was calculated to be 1.1 scfm.

Periodic sampling and analysis of the gas streams at the blower discharge (mockup inlet), heat exchanger inlet (mockup outlet) and moisture removal system outlet was performed throughout the run. Water concentrations in these three streams are shown in Figure 6. Figures 7 and 8 describe carbon dioxide concentrations obtained throughout the run using infrared and chromatographic gas analysis techniques. The same type of data for carbon monoxide are presented in Figures 9 and 10. Samples of the mockup outlet gas were obtained and analyzed for hydrogen using a mass spectrograph. Figure 11 exhibits hydrogen concentration in the mockup outlet gas stream during the run.

These figures indicate a general decrease in contaminant concentration during the run. This is due to depletion of water within the system in conjunction with a continuous purge of the recirculating gas. In addition to loss by purging, water and some carbon dioxide was removed in the moisture removal towers.

Electrical System

The four graphite heating elements were in excellent condition at the end of the run with the exception of one location on a single element which was opposite a viewing port. A photograph of the elements upon completion of the run is shown in Figure 2.

The grounding wedges and sliding collets shown in Figures 12 and 13 have been used in each of the four runs completed thus far.

Electrical resistances of the individual heater elements showed no change when measured before and after the run with a Wheatstone Bridge.

Prior to starting Run Number 4, the heating circuit was energized for a short period while measuring current passing through the individual, parallel heating elements. Voltage drop values between the bus bars were obtained simultaneous with the current measurements

permitting calculation of the installed resistances reported in Table II.

TABLE II

<u>Location</u>	<u>Current, amps</u>	<u>Volts</u>	<u>Resistance, ohm</u>
TR	152	9.00	0.0592
BR	156	9.20	0.0590
BL	160	9.10	0.0570
TL	154	9.10	0.0591

The above procedure was repeated at the conclusion of the run with the results listed in Table III.

TABLE III

<u>Location</u>	<u>Current, amps</u>	<u>Volts</u>	<u>Resistance, ohm</u>
TR	159	9.50	0.0598
BR	160	9.50	0.0594
BL	170	9.55	0.0561
TL	154	9.45	0.0613
TL (recheck)	156	9.45	0.060

The above values are not true heating element resistances due to the fact the voltage drop was measured across the bus bars rather than the elements themselves. They do, however, give an indication as to how closely the installed resistances, including contact resistance, compare before and after the run.

Figure 14 depicts the total heater circuit resistance and total circuit current throughout the run.

Temperature Measurement

Figures 15, 16, 17 and 18 depict the temperature history during the run at various locations within the graphite core and thermal insulation. Graphite core temperatures were obtained using 1/8" OD inconel sheathed, grounded junction, chromel-alumel thermocouples. Wire diameter was 0.020 of an inch and was insulated from the sheath by crushed MgO beads. Thermal insulation temperatures were obtained using the 1/16" OD inconel sheathed, chromel-alumel thermocouples installed prior to Run Number 1.

Subsequent to Run Number 4, the graphite core thermocouples were removed and calibrated. The calibration results are listed in Table IV.

TABLE IV

TC No.	Calibration Temp., °F	Error %	Calibration Temp., °F	Error %	Calibration Temp., °F	Error %	Calibration Temp., °F	Error %
2	510	±1.05	1010	±0.14	1530	-0.51	2000	-1.01
3	↓	±2.12	↓	±0.27	↓	-0.62	↓	-0.34
4	↓	±1.43	↓	-0.02	↓	-0.84	↓	-0.85
6	↓	±0.9	↓	-9.27	↓	-9.88	↓	-1.33
7	↓	±2.03	↓	±0.11	↓	-9.91	↓	-1.4
8	↓	±1.19	↓	-0.49	↓	-1.38	↓	-1.96
10	↓	±1.39	↓	-0.57	↓	-1.8	↓	-2.7
11	↓	±0.93	↓	-0.37	↓	-1.03	↓	-1.41
12	↓	±0.86	↓	-1.3	↓	-2.19	↓	-2.86
14	↓	±0.9	↓	-1.12	↓	-2.49	↓	-3.91
16	↓	±1.03	↓	-1.28	↓	-2.33	↓	-2.95
Reference	↓	±0.54	↓	±0.44	↓	±0.32	↓	±0.07

Thermocouple number 14 was submitted for metallurgic examination in that calibration changes of the magnitude shown in Table IV were not expected. The thermocouple was sectioned at the junction and analyzed to determine if the calibration shift was caused by diffusion of a contaminant(s) into the junction. Using microprobe techniques, it was qualitatively determined that carbon had diffused into the sheath and junction. Previous work indicates that bare wire, chromel-alumel thermocouples are not satisfactory for measurement of graphite temperatures above 900°C because of a resultant drift in calibration due to carbonization of the wires.⁽¹⁾ It is therefore postulated that

(1) F. A. Reichardt. Early Work on Thermocouples to Measure Graphite Temperature of 800°C and Higher, DPR-25, p. 5. Atomic Energy Establishment Winfrith, Dorchester, Dorset, Great Britain, May, 1961.

the calibration change noted at the completion of Run Number 4 is the result of carbon transport into the thermocouple junction.

In addition to junction examination, thermocouple Number 14 was sectioned and examined approximately $1/4$ " above the junction. No carbon was detected in the wire at this location and, consequently, diffusion of carbon into the ungrounded (not in direct contact with the sheathing material) HTLTR thermocouple junctions should not be as extensive as that encountered in the mockup.

Photomicrographs of portions of one of the thermocouples used to monitor heater element temperatures are shown in Figure 19.

Samples

Small samples of graphite were placed within the mockup and remained there throughout the run to permit evaluation of corrosion occurring during the run. These samples were $1/2$ " x $1/2$ " x 3" and were slotted, forming fins approximately $1/32$ " thick, to provide a large reaction surface area. Weight changes associated with these samples are reported in Table V.

TABLE V

Sample ⁽¹⁾ Number	Initial Weight, gm	Final Weight, gm	Weight Change, gm	Weight Loss ⁽²⁾
				Rate x 10 ⁶ , gm-hr ⁻¹ -Cm ⁻²
1E	11.6992	11.3847	-0.3145	-1.335
2E	11.3176	11.0702	-0.2474	-1.05
3E	11.6788	11.5614	-0.1174	-0.494
4E	12.2462	11.9579	-0.2883	-0.122
5E ⁽³⁾	12.7565	12.5622	-0.1943	-0.825
6E ⁽³⁾	12.2703	12.0042	-0.2661	-1.13
7W	11.5230	11.5124	-0.0106	-0.045
8W	12.7103	12.2356	-0.4747	-2.01
9W	11.3352	11.2941	-0.0411	-0.174
10W	11.4670	10.8159	-0.6511	-2.76
11W	12.6691	12.1896	-0.4795	-2.04

(1) W refers to west end of mockup
E refers to east end of mockup

(2) Based on 1250 hours of exposure above 700°C and an exposed surface area of 189 cm².

(3) These samples were placed on the access opening brick floor.

One sample of B₄C and one of B-Zr-graphite left in the mockup throughout the run exhibited respective weight change rates of -0.266×10^{-6} gm-hr⁻¹-Cm⁻² and 5.80×10^{-6} gm-hr⁻¹-Cm⁻².

In addition to those samples which remained in the mockup during the run, there were graphite samples which were periodically inserted into and removed from the graphite core. The data obtained from these samples are shown in Table VI.

TABLE VI

Sample Number	8	2	2	2	2
Exposure time, hr	22.92	45.50	23.50	23.75	44.58
Core Temp., °F	1720	1800	1800	1800	1800
Initial wt., gm	13.4367	13.3716	13.3594	13.3514	13.3424
Final wt., gm	13.4265	13.3594	13.3514	13.3424	13.3199
Wt. change, gm	-0.0102	-0.0122	-0.008	-0.009	-0.0225
Wt. change rate ⁽¹⁾ x 10 ⁶ , gm-hr ⁻¹ -Cm ⁻²	-2.36	-1.42	-1.8	-2.0	-2.67

Sample Number	3	3	3
Exposure time, hr	17.08	23.83	23.5
Core Temp., °F	1805	1805	1805
Initial wt., gm	13.8735	13.8650	13.8614
Final wt., gm	13.8650	13.8614	13.8564
Wt. change, gm	-0.0085	-0.0036	-0.005
Wt. Change rate ⁽¹⁾ x 10 ⁶ , gm-hr ⁻¹ -Cm ⁻²	-2.63	-0.80	-1.13

(1) Based on an exposed surface area of 189 Cm².

Safety Blade

A total of sixty-eight safety blade scrams were completed, forty-three of which were performed at a core temperature of 1000°C. Measured safety blade scram times are shown in Figure 20.

Examination of the safety blade at the completion of the run revealed an opaque, solid material deposited on the hot end of the TD nickel sheath as seen in Figure 3. Tests performed on the material indicated a high boron content and it is thought the material was transported from B₄C present in the safety blade. The postulated mechanism by which transport occurred is shown by the following reactions:

The fact boron transport did occur renders B₄C an unsatisfactory candidate for safety blade poison material. Consequently, new materials, e.g., co-precipitated Gd₂O₃-Eu₂O₃-Ni, Gd₂O₃-Eu₂O₃-graphite and Gd₂O₃-Eu₂O₃-ceramic, will be investigated for inclusion in the safety blade design.

Slippage of the safety blade drive clutch was not encountered during this run due to the higher power clutch installed prior to this run.

As in previous runs a low flow (600 cfh) of cool gas was directed into the safety blade housing. A maximum safety blade housing flange temperature of 110°F was noted.

Control Rods

No control rod malfunctions were noted during sixty-seven scrams. Forty-three of the scrams were performed at a core temperature of 1000°C.

Measured control rod scram times are shown in Figure 20.

The extensive corrosion of the UO_2 -graphite portion of the TD nickel support rod encountered during Run Number 3 was not in evidence at the completion of Run Number 4. This is due, principally, to a change in the method of preparing the UO_2 -graphite mixture used in the cylinders.

Operating History

To permit comparison of certain data and curves, the following operating highlights are presented:

<u>Date</u>	<u>Comment</u>
9/1/65	Recirculation blower turned on.
9/2/65	Moisture removal towers valved into gas system.
9/3/65	Evacuated mockup and refilled with fresh nitrogen.
9/7/65	Evacuated mockup and refilled with fresh nitrogen.
9/9/65	Evacuated mockup and refilled with fresh nitrogen. Changed moisture removal towers.
9/10/65	Evacuated mockup and refilled with fresh nitrogen.
9/12/65	Blower off for approximately five hours.
9/13/65	Increased flow through mockup from 1,170 cfh to 10,600 cfh.
9/14/65	Changed moisture removal towers.
9/16/65	Blower off for approximately nine hours.
9/28/65	Reduced flow through mockup from 10,600 cfh to 6,600 cfh.
10/8/65	Reduced flow through mockup from 6,600 cfh to 5,600 cfh.
10/11/65	Reduced flow through mockup from 5,600 cfh to 4,600 cfh.
10/18/65	Reduced flow through mockup from 4,600 cfh to 1,200 cfh.
10/22/65	Changed moisture removal towers.

<u>Date</u>	<u>Comment</u>
10/27/65	Changed moisture removal towers.
12/2/65	Changed moisture removal towers.
12/3/65	Start of cooldown.

FIGURE 1
Mockup Interior After Run Number 4

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FIGURE 2
Heating Elements Exposed During Run Number 4

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FIGURE 3
Boron Oxide Deposits on Safety Blade

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FIGURE 4
Carbon Deposition on Inside of Heat Exchanger Tube

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FIGURE 5
Absolute Pressure vs. Time
Run Number 4
Evacuation Number 1

FIGURE 6
H₂O Concentration vs. Time

FIGURE 7
CO₂ Concentration (Infrared) vs. Time

FIGURE 8
CO₂ Concentration (Chromatograph) vs. Time

FIGURE 9
CO Concentration (Infrared) vs. Time

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FIGURE 10
CO Concentration (Chromatograph) vs. Time

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FIGURE 11
H₂ Concentration vs. Time

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FIGURE 12
Grounding Wedges and Positioning Bolts

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FIGURE 13
Sliding Collets

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FIGURE 14
Total Circuit Current and Resistance vs. Time

FIGURE 15
Heater Element Temperature vs. Time

FIGURE 16
Horizontal Blade Temperature vs. Time

FIGURE 17
Control Rod Temperature vs. Time

FIGURE 18
Thermal Insulation Temperature vs. Time

Position 1

Position 2

Position 3

Position 4

Position 5

FIGURE 19
1/8" OD Inconel Sheath, 1000 Hours at 1000 °C 135X

FIGURE 20
Scram Time vs. Scram Number