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Quarterly Status Report of LASL Rover Program

for Period Ending May 31, 1965

For The Atomic Energy Commission

Bram C. Feldman

(Title Unclassified)

Chief, Reactor, Space and Technology Branch
Division of Classification

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for Period Ending May 31, 1965

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I. KIWI AND PHOEBUS-1

KIWI-B-4E-301

Postmortem Evaluation

Introduction

The full-power test of Kiwi-B-4E-301 was described in the quarterly report for the period ending November 30, 1964 (LA-3212-MS) and most of the results of the test were given in the subsequent quarterly report (LA-3280-MS). Some additional results are summarized below.

Data Analysis

The detailed analysis of the Kiwi-B-4E-301 data is essentially complete. The results have been described in a number of internal laboratory reports and will be compiled in a forthcoming final report.

The complete reactor data for both Experimental Plans V and VI have been recorded on magnetic tape for computer use. The tapes (termed LTAPE) also include averaged quantities, data corrected for offsets and individual calibrations, and pressures converted to absolute units. In addition, several derived quantities have been included on the LTAPES. Typical of these quantities are the following: calculated nozzle chamber temperature, thermal power values derived from the coolant enthalpy increases through the entire reactor system and individual components, and H₂ flow rate corrected for pipe line lags during transients (caused by fluid storage and compressibility effects).

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4. None of the units performed sufficiently well to justify an attempt to make accurate measurements in the direct reactor radiation flux. This is the primary reason LASL is focusing attention on methods of improving line response so that transducers may be mounted under the privy roof shield.

KIWI-B-4 GENERAL

Lag Between Actual and Measured Fuel Temperatures

During reactor operations, a significant lag apparently exists between actual fuel temperatures and those observed at the thermocouple locations in the adjacent unfueled support element graphite. Although the detailed nature and cause of the lag is not understood, it can amount to 4-6 sec on a startup transient. The lag is believed to be due primarily to relatively poor heat transfer across the gap between fuel elements and support elements. The gap size may well vary throughout the core and may be influenced by the temperature difference between the support element and the encircling fuel elements, which may tend at times to bridge around the support element. The time constant should also be influenced by the interstitial H_2 conductivity.

The basic evidence in support of the lag consists of the following three observations:

1. A direct measurement of Kiwi-B-4D-202 in which thermocouples placed in support elements at Station 51 lagged 5-7 sec behind adjacent thermocouples located inside fuel elements.

2. The power-to-core temperature transfer functions which were obtained from the crosscorrelation experiment on the restart of Kiwi-B-4E-301 show more phase lag and less gain than is predicted for the fuel temperatures at the same axial locations.

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3. The measured core temperatures in both Kiwi-B-4D and Kiwi-B-4E lag up to 7 sec behind the calculated fuel temperatures obtained from the HEX transient heat transfer code using flow rate and power as input variables.

Kiwi-B-4 Post-Test Radiation Measurements

Extensive radiation level measurements from the Kiwi-B-4D and Kiwi-B-4E tests have provided detailed information on the radiation environment during ground operation of propulsion reactors. The complexity of test site areas does not permit this information to be obtained by calculation alone, particularly during post-shutdown, the condition of greatest practical interest. The components (direct beam, air scatter, and activation) of the total dose rates after shutdown were separated and identified by comparing measurements taken before and after reactor removal from the test area. With the reactor at a distance from the test location, direct source effects and activation effects were measured separately. The displaced reactors exhibited lower dose fields than expected. No evidence was found of local fission product contamination.

Measurements were taken: (1) at a point 1000 ft from the reactor and in direct view of it; (2) at a point 644 ft from the reactor but shielded from it; (3) at a survey location on the concrete test pad 60 ft from the test point in direct sight of the reactor; and (4) during transit back to the MAD Building in an area free of activation.

The 1000 ft measurements (1) gave evidence of the existence of two activation half-life groupings of ~ 2.5 h and ~ 15 h; this was supported by measurements at other locations. Decay measurements (2) at the 644-ft point were typical of results in shielded locations at distances of 100 to 2000 ft from the reactor. There were differences in the ratio of total dose rate to activation dose rate (since this ratio was a function of both time and distance) but the significant feature was the relatively large fraction of the total dose which was attributable to activation in these shielded locations at decay times of from 5-100 h.

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Results of the (3) measurements indicated that the decay of the activated concrete on the pad differed little from the activated soil off the pad; there was a slightly lower activity on the concrete when points of similar neutron exposure were compared. During the (4) measurements for the Kiwi-B-4E reactor, the radiation fields were less than those expected by a factor of almost 1.5. Moreover, they fell off slightly less rapidly with distance than the experimental values. The differences in slope and in absolute magnitude have not yet been satisfactorily explained.

The information obtained--only a portion of which is summarized above--can be extrapolated to predict the conditions following contemplated reactor runs of higher energy release. This extrapolation shows that facility shields will be needed to control activation and permit the desired early reentry for activities such as dewar filling.

PHOEBUS-1A

Introduction

Phoebus-1A is an experimental reactor of the Kiwi type but incorporating several core modifications intended to reduce external fuel element corrosion. The reactor core is 35 in. in diameter with a design power level of approximately 1100 MW at 70 lb/sec of H₂ flow.

Status

The reactor/test car assembly of Phoebus-1A was moved to Test Cell C on May 11, 1965 for final preparation prior to testing. The following is a summary of the major steps involved in the reactor assembly and the dates on which they occurred.

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Last shipment of reactor parts to NRDS	2/26/65
Arrival of orifice print-out and element loading deck at NRDS	3/19/65
Completion of all element jetting and first core assembly	4/13/65
First cold critical rod position measurements	4/13/65
Completion of reshimming of core with an additional 21 tantalum loaded elements	4/15/65
Second cold critical rod position measurements	4/15/65
Complete assembly of reflector cylinder to core	4/20/65
Completion of brazing of instrumentation leads into access cover plate	4/26/65
Mating of reactor core to pressure vessel dome	4/27/65
Reactor leak checked to 300 psi with simulated nozzle	4/29/65
Reactor installed on test car	4/30/65
Installation of RN-6 nozzle No. 018 on reactor	5/5/65
Leak check of nozzle/pressure vessel seal and the pressure transducer panel (125 psi)	5/10/65

Only minor difficulties were encountered during the reactor assembly. A glue buildup in two clusters (270 H-3 and 270 H-4) prevented proper fitup and alignment of fuel and filler elements. Consequently, the two clusters were removed, disassembled, cleaned, reassembled, and reinstalled.

The two core exit temperature probes (T-167-E and T-168-E) were badly bent during part of the assembly procedure. The probes were straightened but the sheaths had been cracked by the initial bending. Since no replacements were available, the probes were allowed to remain. The channels are still operative (with proper continuity measurements) but will probably fail sometime during reactor operation.

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Design and Analysis

The Phoebus-1A design was modified from Kiwi-B-4E to reduce non-fuel coolant flow rates. Perimeter filler coolant tube impedances were increased by adding inlet orifices. The tie rod coolant flow rate was reduced by decreasing the annular flow area between the tie rod and stainless steel sleeve.

Hydrogen flow tests were performed on the inlet orifices of the perimeter filler tubes. The calculated inlet orifice performances for various orifice sizes and the corresponding test data are shown in Fig. 1. A comparison of the results indicates that the as-built 31-mil orifices behave like sharp-edged orifices with diameters between 31 and 33 mils.

Figure 2 gives results of flow tests made on the support element coolant channels. Analytical predictions (derived by the same methods used in making reactor performance calculations) are also shown. The correlation between predicted values and test data was very satisfactory.

Fig. 1. Phoebus-1A perimeter filler tube inlet orifice flow characteristics.

Fig. 2. Phoebus-1A tie rod channel flow test.

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Core Thermal Performance Analysis

Calculations have been completed for predicting the nominal thermal performance of the Phoebus-1A core using the basic methods described in LA-3170-MS ("Thermal Analysis of the Kiwi-B-4D Reactor," Parts I-V). The predictions were in the form of parametric curves for core pressure drop, fuel channel (mean) exit H_2 temperature, and core power with flow rate as a parameter. In addition, the performance predictions of the tie rod support channel coolant, peripheral coolant, and the (mean) Station 20 core thermocouples were completed. Typical results are given in Figs. 3, 4, and 5.

The results of the predictions for the planned intermediate and full power hold are given in Tables 1 and 2. The hold conditions were defined by flow rates of 60 and 70 lb/sec and (mean) fuel exit temperatures of 3100° and $4500^\circ R$, respectively.

The cluster model used to represent the core (fuel plus support channel) was modified for Phoebus-1A in the following ways:

1. The average fuel channel diameter is 0.096 in.
2. The average fuel channel inlet meter diameter is 0.038 in.
3. There are 29,020 fuel channels and 265 support rod coolant channels in the core resulting in 111 fuel channels per support for the average cluster.
4. Power density in the fuel varies radially from 2% above average next to the unloaded support element to 2% below average farthest from the unloaded element.
5. The axial power distribution was altered slightly to account for the unloaded graphite tips on the elements (see LA-3280-MS, p. 27, Fig. 11).
6. The effect of channel roughness on the coolant pressure drop is included.
7. Hydrogen properties are evaluated at the channel surface temperature in the heat transfer and pressure drop correlations.

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Fig. 3. Predicted Phoebus-1A core pressure drop.

Fig. 4. Prediction of support channel performance.

Fig. 5. Predicted mean Station 20 fuel element temperature.

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Table 1

Summary of Predicted Phoebus-1A Thermal Performance - Intermediate-Power

<u>Component</u>	<u>Inlet Temp. (°R)</u>	<u>Outlet Temp. (°R)</u>	<u>Temp. Diff. (°R)</u>	<u>Inlet Press. (psia)</u>	<u>Outlet Press. (psia)</u>	<u>Press. Diff. (psi)</u>	<u>Flow Rate (lb/sec)</u>	<u>Flow Frac.</u>	<u>Power (MW)</u>
Nozzle Coolant	49	114	65	648	508	140	58	0.97	
Reflector	114	148	34	508	471	37	60.0	1.000	8
Flow Separator	148	148		471	459	12	60.0	1.000	
Core	148			459	392	67	60.0	1.000	633
Fuel		3100					54.9	0.915	(625)
Support		560					3.0	0.050	(5)
Periphery		550					2.1	0.035	(3)
Tubes		680					0.9	0.015	
Cavity		450					1.2	0.020	
Nozzle Chamber	2900			392			60.0	1.000	
Total						256			641
Core Thermocouple Temperature, Station 20						2000°R (av.)			
Maximum Fuel Channel Surface Temperature						3190°R (av.)			

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Table 2Summary of Predicted Phoebus-1A Thermal Performance - Full-Power

<u>Component</u>	<u>Inlet Temp. (°R)</u>	<u>Outlet Temp. (°R)</u>	<u>Temp. Diff. (°R)</u>	<u>Inlet Press. (psia)</u>	<u>Outlet Press. (psia)</u>	<u>Press. Diff. (psi)</u>	<u>Flow Rate (lb/sec)</u>	<u>Flow Frac.</u>	<u>Power (MW)</u>
Nozzle Coolant	54	176	122	975	737	238	68	0.97	
Reflector	176	229	53	737	685	52	70.0	1.000	15
Flow Separator	229	229		685	667	18	70.0	1.000	
Core	229			667	557	110	70.0	1.000	1110
Fuel		4500					63.5	0.907	(1098)
Support		685					4.0	0.057	(7)
Periphery		820					2.5	0.036	(5)
Tubes		940					1.1	0.016	
Cavity		725					1.4	0.020	
Nozzle Chamber	4200			557			70.0	1.000	
Total						418			1125

Core Thermocouple Temperature, Station 20 3060°R (av.)

Maximum Fuel Channel Surface Temperature 4605°R (av.)

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Fig. 6. Predicted radial variations of Station 20 fuel element temperatures, Sector-330. (Arrows indicate fuel elements adjacent to thermocouple locations.)

instrumented with thermocouples are indicated. The predicted (mean) Station 20 thermocouple indication recorded in Tables 1 and 2 were corrected for the difference between support element thermocouple reading and adjacent calculated fuel element temperatures as indicated by the Kiwi-B-4E-301 data (see p. 5).

The irregularity of predicted Station 20 fuel temperatures between the 3 and 6 in. radial locations arose from a mismatch of fuel density and channel diameter between elements and from the asymmetric arrangement of these elements in the core. The radial distribution of core temperatures for the actual run conditions will depend on the core inlet temperature and radial fission distribution.

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Stress and Deflection Analysis of Tab Supported Fuel Elements

Phoebus-1A has several regular clusters of tab-supported fuel elements and some tab-supported elements in irregular clusters at the core periphery. This support scheme eliminates the support block and substitutes a tab on the side of the fuel element tip. The tab transmits the axial core pressure drop load to the pyro washer, pyro cup, support cone, and tie rod. Since the axial load is applied eccentrically to the fuel element center line, a bending moment will act on the fuel element. It is of particular interest to estimate the bending moment (and stresses) transmitted to the glue joint between the unfueled tip and the fuel element.

A parametric stress analysis was performed to study the effects of varying the magnitudes of the eccentricity, load, and length of the exit end of the element free to deflect. In summary, the results of the analysis indicated that relatively small stresses and deflections would be experienced by the element for a wide range of the parameters.

Instrumentation

Eight new channels were added to the Phoebus-1A instrumentation. Three temperature channels (T-445-E through T-447-E) will measure the potential between the W leg and the Ta sheath of existing development core thermocouples. These thermocouples are located at Station 44 and are assigned as follows:

<u>New Channel</u> <u>(Sheath Potential)</u>	<u>Existing Channel</u> <u>(Core Temperature)</u>	<u>Cluster</u> <u>Designation</u>
T-445-E	T-435-E	270/D1/ABG
T-446-E	T-437-E	030/D1/ABG
T-447-E	T-440-E	210/D1/ACD

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Three CEC vibration transducers (channels H-104-E through H-106-E) supplied by Westinghouse will be installed on the nozzle inlet spider and checked for overall performance during the reactor test.

Two pressure measurements were also added to the instrumentation. Channel P-104-E will measure the nozzle shell purge pressure and channel P-105-E will indicate whether the instrumentation shroud coolant is operative by taking a measurement in the purge/coolant gas line. Both channels will be multiplexed and displayed on meters in the control room.

Nineteen W/W-26% Re development thermocouples have been installed in Phoebus-1A. All have ~0.110-in. o.d. Ta sheaths, hard-fired BeO insulators, and 0.020-in. diam wires. One has a vapor-deposited W coating over the sheath, two are bare Ta, and the remaining 16 are coated with Liqui-Moly grease (MoS₂ in an organic grease).

Studies on the protection of Ta against hydriding are being continued. While several coating materials show some promise, a W layer diffusion-bonded to the Ta appears to be best at the moment. Nitride, carbide, and Liqui-Moly grease layers also seem to offer some protection.

Thermocouples with 0.110-in. o.d. Mo sheaths are on order. These should work up to temperatures of ~4200°R and may go to considerably higher temperatures with W coats to protect against carbiding.

The standard Cu-Con thermocouples for cryogenic measurements have MgO insulation and stainless steel sheaths swaged to 0.063-in. o.d. In the past, these thermocouples have shown considerable disparities in readings made near LH₂ temperatures. The Cu-Con thermocouples for Phoebus-1A were calibrated against a very accurate thermocouple. The discrepancies noted during the calibration were similar to those found earlier and are ascribed to strains in the thermocouple wire caused by the swaging operation. Some shifts were observed on thermal cycling from room temperature to 35°R. It is believed that the thermocouples in Phoebus-1A should be accurate to $\pm 5^{\circ}\text{R}$. The next generation of Cu-Con thermocouples will not be swaged.

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Run Profiles

The control systems for the first Phoebus-1A hot run will be similar to those used on the later Kiwi reactors. The major programmed and controlled variables will be flow rate, log power, and average Station 20 material temperature. The flow rate and log power programs are designed to give the desired Station 20 temperature. Thus, the temperature control system applies vernier corrections to log power demand to compensate for errors in the flow rate and log power programs and systems.

A new control scheme will be used during the second hot run. Station 20 temperature, flow rate, and control drum rotation will be programmed. The controlled variables will be temperature and flow rate. The log power control system will be used to get power to the programmer initial conditions. The log power loop will be opened before the programs are started and will stay open for the remainder of the run. Since the drum angle and flow rate programs will be designed to achieve the desired fuel temperature profile, the output of the temperature controller will operate around zero volts. It will be clamped so that it can only change control drum position by $\pm 7.2^\circ$ which is the equivalent of about $\pm 400^\circ\text{R}$ at Station 20.

The programs for the startup and shutdown are shown in Figs. 7, 8, and 9; they are based on achieving the hold levels in Table 3.

Table 3. Planned Hold Levels for Phoebus-1A

<u>Quantity</u>	<u>Power Calibra- tion Hold</u>	<u>Full Power Hold</u>
Reactor flow rate	60 lb/sec	70 lb/sec
Station 20 measured temperature	2000 $^\circ\text{R}$	3010 $^\circ\text{R}$
Fuel exit gas temperature	3100 $^\circ\text{R}$	4500 $^\circ\text{R}$
Chamber temperature	2900 $^\circ\text{R}$	4200 $^\circ\text{R}$
Power	630 MW	1120 MW

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Fig. 7. Flow rate (A) and rod position (B) programs for Phoebe-1A startup.
 Fig. 8. Station 20 temperature programs for startup (A) and shutdown (B).
 Fig. 9. Flow rate (A) and rod position (B) programs for Phoebe-1A shutdown.

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The actual expected power, temperature, flow rate, and rod positions are identical for the first and second runs. The control modes are different, as stated above, and the various programs and control spans are indicated on the figures. Pipe line capacitance and cooling effects account for the difference between venturi flow and reactor flow in Fig. 7A. Attempts have been made in Figs. 8A and 8B to predict the differences between fuel temperature and thermocouple temperature at Station 20. Present plans call for the temperature controller to come out of the clamp at the beginning of the power calibration hold and continue control until 30 sec into the shutdown when its output again clamps.

Temperature Control System

The plan to program control rod angle rather than log power during the second high-power run of Phoebus-1A required the design of a new temperature control system; this is shown in Fig. 10. As developed, it has some advantages over the previous temperature control system.

Fig. 10. Block diagram of Phoebus-1A temperature loop (no log power loop).

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Fig. 11. Open loop transfer function data for reactivity-to-loop power in Phoebus-1A.

advantages of the new temperature control scheme over the old system with its log power inner loop, namely, the new system is much less susceptible to the destabilizing effects of fast periods. The reason for this becomes apparent when the $\delta P / \delta(\delta K_R)$ inner loop transfer function shown below is considered.

$$\frac{\delta P}{\delta(\delta K_R)} = \frac{G_1(S)}{1 + G_1(S) G_5(S)},$$

where

$$G_5(S) = \frac{\delta(\delta K_{FB})}{\delta P} = G_3(S) + G_4(S).$$

If $G_1(S) G_5(S) \gg 1$

$$\frac{\delta P}{\delta(\delta K_R)} = \frac{1}{G_5(S)} \quad .$$

The success of the proposed system is heavily dependent upon the natural stability of the inner loop from δK_R to power. A review of the Kiwi-B-4E-301 data indicates that the static relationships from θ or $\dot{W}$ to T_{20} are as follows:

$$\left. \frac{\Delta T_{20}}{\Delta \theta} \right|_{\dot{W}} = + 65 \frac{^{\circ}\text{R}}{^{\circ}\theta}$$

$$\frac{\Delta T_{20}}{\Delta \dot{W}} \bigg|_{\theta} = +41 \frac{^{\circ}\text{R}}{\text{lb/sec}} \cdot$$

The open loop transfer function data for the reactivity-to-power loop is shown in Fig. 11 for 1120 MW at infinite and 0.5-sec periods. The data point up one of the major

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The $G_1(S)$ term, which cancels out, contains both the gain dependency on power and the gain and phase dependency on period.

A rough idea of the frequency range over which the assumption that $G_1(S) G_5(S) \gg 1$ may be obtained from Fig. 11, which shows $G_1(S) G_5(S)$. At full power, $G_1(S) G_5(S) = 1$ at ~ 0.4 cps with infinite period and at ~ 2 cps with a 0.5-sec period. A typical example of the improvement in stability is that a 0.5-sec period causes an increase of +20 db in gain and 42° in phase lag at 0.4 cps in a log power control system. The same conditions in the new temperature control system cause an increase of 5 db in gain and a decrease of 48° in phase lag.

There is another major advantage with the new control scheme in the region where

$$\frac{\delta P}{\delta(\delta K_R)} = \frac{1}{G_5(S)}.$$

The frequency dependent characteristics of both $G_5(S)$ and $G_2(S)$, the power-to- T_{20} transfer function, are similar because the basic low-frequency poles of both are generated in the reactor heat exchanger. Since $G_5(S)$ is in the denominator of $\delta P / \delta(\delta K_R)$, its poles become zeros in the $\delta P / \delta(\delta K_R)$ transfer function. These zeros cancel the poles in $G_2(S)$ as long as $G_1(S) G_5(S) \gg 1$.

The open and closed loop characteristics of the new temperature control system with a controller transfer function of $\frac{S+1}{1} \frac{\text{Volts}}{\text{Volts}}$ is shown in Fig. 12 for 1120 MW and 70 lb/sec of flow rate. The system is designed for a closed loop bandwidth of 0.4 cps, a phase margin of 75° , and a gain margin of ~ 20 db. The large amount of stability margin is being provided to allow for inaccuracies in the transfer functions

$$\left. \frac{\delta(\delta K_{FB})}{\delta P} \right|_{\dot{W}} \quad \text{and} \quad \left. \frac{\delta T_{20}}{\delta P} \right|_{\dot{W}}. \quad \text{Fortunately, enough data were}$$

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Fig. 12. Transfer functions for Phoebus-1A temperature control system.

desired. A known increase in reactivity of 1.19\$ beyond this level was produced by rotating two control rods to their "out" position. The neutron counting rates before (C_1) and after (C_2) this change were then used to compute shutdown reactivity by means of the formula

$$\Delta k(\text{shutdown}) = 1.19\$ \frac{C_2}{C_2 - C_1} .$$

The resulting value of 2.51\$ was 0.94\$ short of the desired 3.45\$ shutdown.

An additional 21 Ta-loaded support elements (total 69) were added to the core and a recheck of reactivity gave an estimated 3.35\$ shutdown, which was considered to be satisfactory. Allowing 0.2\$ for nozzle, plumbing, etc., the predicted test cell cold critical is at 89° vs 91° specified.

obtained on the Kiwi-B-4E run to alleviate a somewhat uncertain situation, but more experimental data are needed for more precise predictions of control system operation.

MAD Building Checkout of Phoebus-1A

Multiplication measurements (designed to estimate the reactor criticality) were carried out on Phoebus-1A following initial assembly in the MAD building. According to a rough indication obtained by monitoring counting rate from a central source as the Cd safing strips were removed, the reactor was somewhat closer to critical than

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It is felt that the ~ \$1 error in estimating Phoebus-1A reactivity based on core material weights was primarily due to uncertainty in effectiveness of Ta support elements. Kiwi-B-4E experiments in the Kiva also indicated a discrepancy in the curve that has been used. This curve has been revised in Fig. 13 to be more compatible with Phoebus-1A and Kiwi-B-4E data.

In the light of recent information on the nozzle for Phoebus-1A, it appears that H₂ densities used in the original neutronic analysis were somewhat low. Revisions are in progress based on the most up-to-date reactor parameters.

Fig. 13. Effectiveness of Ta support elements in Phoebus-1A.

Fuel Element Photocoverage

Photographs were obtained of the last (hot) half of each face on each fuel element placed in the Phoebus-1A core during assembly. The negatives were of excellent quality but the pictures may be of limited use in postmortem inspection because of changes in the disassembly lighting and some incorrect records made during the assembly photography.

The photographic system for the inspection of fuel elements between Windows H and J in the R-MAD building during Phoebus-1A disassembly (LA-3280-MS, p. 27) has been designed and constructed. All parts are on hand and are being assembled in the clean room of the R-MAD building for checkout before Phoebus-1A disassembly. The simplified photographic system which LASL fabricated for Westinghouse for use in the NRX-A3 disassembly is installed and operating. Experience obtained with the Phoebus-1A assembly photography

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and the installation and checkout of the NRX-A3 system indicates that these systems should operate very well and will yield excellent photographs.

PHOEBUS-1B

Introduction

Phoebus-1B will be a Kiwi type reactor modified to operate at approximately twice the power and flow rate levels of Phoebus-1A. Present design study conditions are 2000 MW power, 4600°R fuel exit temperature, and 140 lb/sec H₂ flow. These levels are subject to revision as may be indicated by the results of analyses.

Status

The periphery design for Phoebus-1B has been selected. The periphery will be circular, utilizing narrow cooled slats, pyrographite strips, and filler elements of matching widths. Each trio of slats, strips, and filler elements will be machined as a subassembly to improve component uniformity and fitup. At present it is planned to use recirculating tie tubes of the Phoebus-2 type in six clusters of the core if the hardware can be obtained in time.

Components unchanged from the Phoebus-1A design or whose design change has been completed are being released for fabrication or procurement as rapidly as possible. The release list to date includes Support Block Regular, Support Rod, and Sleeve Pyro.

Revised values of temperature, pressure, and flow at design point are shown in Table 4.

Design and Analysis

Thermal-hydraulic analysis of the core periphery is under way. ~~OPTION~~ calculations have been made with four, five, and six seals replacing the load rings of the Phoebus-1A reflector cylinder. The temperature and pressure distributions shown in Figs. 14 and 15 (for the four-seal case) are typical of the results obtained. The calculations represent an idealized counter-flow condition for the reflector with no plunger leakage. With the pressure

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Table 4. Phoebus-1B Design Conditions

Thermal Power	2000 MW
Fuel Exit Gas Temperature	4600°R
Total Coolant Flow	123 lb/sec
Periphery By-Pass and Coolant Flow	7.8 lb/sec
Support Rod Coolant Flow	6.4 lb/sec
Support Rod Coolant Exit Temperature	670°R

	Temperature (°R)	Pressure (psia)
Nozzle Coolant Entrance	60	1604
Reflector Inlet	134	1255
Reflector Exit	200	1178
Core Inlet	200	1154
Core Exit Plenum (Mixed)	4244	988

Fig. 14. Phoebus-1B periphery temperature distribution.

Fig. 15. Phoebus-1B periphery pressure distribution.

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profiles so obtained, the radial flow around the plungers is estimated to be 0.8 lb/sec; this is significant compared with the 1.35 lb/sec cavity flow used.

Calculations are being performed to determine the temperatures of the fuel element orifice jets. A quasi three-dimensional model has been set up for the T~~O~~SS code, which solves the general heat conduction equation and determines temperature distributions in three-dimensional irregular bodies. Results obtained to date for Al and Ti orifices indicate that steady-state temperatures at design conditions are below 700°R. Further cases are being prepared to determine the effect of gap size between orifice and fuel element.

The calculated values of RN-6 nozzle operating conditions were received from Rocketdyne. These design case conditions seem quite reasonable. The coolant temperature rise through the tubes is 74°R with a pressure drop of 349 psi. The maximum tube wall temperature is 1450°R. If there were no core perimeter bypass, and therefore no cool film of propellant next to the nozzle tubes, the corresponding values would be 117°R, 433 psi, and 1820°R. This latter condition would require a nozzle inlet pressure of 1695 psia.

The calculations made for the Phoebus-2 tie tubes were repeated for the Phoebus-1B design conditions. With the same design flow rate of 0.047 lb/sec per tube, the temperatures were nearly identical with those in Phoebus-2. The tie tube pressure drop was 109 psi whereas in Phoebus-2 it is 155 psi. The difference is caused by the higher pressure and density levels in Phoebus-1B. These flow conditions result in a mean inner tube temperature approximately 200°R lower than the tie rod temperatures. The difference is one that needs to be considered in using both tie tubes and tie rods in the same core. A tie tube flow rate of 0.03 lb/sec would decrease the above difference to 50°R.

Calculations were also made for reactor conditions of 1000 MW and 4500°R exit temperature. With an orifice sized to control the flow to 0.047 or 0.03 lb/sec at 2000 MW, the maximum tube temperatures at 1000 MW would be 580°R or 720°R, respectively.

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II. PHOEBUS-2

PHOEBUS-2 REACTOR

Introduction

The Phoebus-2 experimental reactor will incorporate several basic design changes to increase the power density and H_2 flow rate from Kiwi and Phoebus-1 levels. The Phoebus-2 fuel region will be approximately 55 in. in diam (as opposed to 35 in. for Kiwi and Phoebus-1 cores) but will retain the same fuel element length. Phoebus-2 will develop 5000 MW of power at 320 lb/sec H_2 flow.

Present plans call for the first Phoebus-2 test in November 1967. Portions of the reactor are presently in the preliminary design stage with other parts in the final design stage.

Phoebus-2 General Design

Additional decisions among alternate design concepts were made during this reporting period as follows:

1. The core geometry will incorporate a circular periphery.
2. Lateral support and core periphery sealing will be by a leaf spring-plunger-load ring (or seal) arrangement.

3. Some major dimensions are:

Core o.d. (measured to o.d. of slats)	= 56.66 in.
Interface cylinder o.d.	= 62.16 in.
Reflector System i.d.	= 62.36 in.
Reflector System o.d.	= 78.36 in.

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4. Some major dimensions established for nozzle and pressure vessel work are:

Nozzle skirt/interface cylinder joint station = 53.375 in.
Nozzle skirt i.d. at forward end = 59.00 in.
Nozzle skirt o.d. at forward end = 61.0 to 62.0 in.
Pressure vessel i.d. (cylindrical section) = 79.36 in.

Phoebus-2 Neutronics

A comparison of four recent design alternatives for Phoebus-2 has been completed. These designs included circular and dodecagonal cores combined with iris and piston ring seals. All reactors contained 75 g of Nb per fuel element, a 7-element cluster structure, and an 8-in. thick Be reflector at 10% void. The primary neutronic differences in these designs were the interface region size and composition, as shown in Table 5. Calculations were performed to determine the U inventory and poison-sheath control worth for each reactor design; the results are given in the table.

Table 5. Comparison of Four Designs for Phoebus-2

Model Designation	A	B	C	D
Core Shape	Circular	Circular	Dodecagonal	Dodecagonal
Seal Type	Iris	Piston Ring	Iris	Piston Ring
Interface Region				
Inner Radius, in.	28.3	28.3	28.2	28.2
Thickness, in.	1.8	2.85	2.45	3.00
Al, kg	400	587	693	642
Inconel, kg	64		65	
Graphite, kg		127		127
Uranium Inventory, kg	286	281	301	285
Control Sheath Worth, \$	12.8	12.4	11.6	12.3

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Fig. 16. Typical Phoebe-2 radial power distribution.

The fuel loadings in each reactor were adjusted to yield comparable power patterns, and a typical fission power profile is depicted in Fig. 16. The core contains six fuel loading regions of which the outer three are approximately one fuel element thick. The max/av and min/av power ratios are approximately 1.2 and 0.8, respectively.

A new, comprehensive neutronic model for Phoebe-2 is being prepared. It will incorporate the most recent tie tube and interface region designs, Ta shim in the core, and a B sheath in the reflector. The model will be based upon the following assumptions, some of which were arbitrarily chosen and others deduced from previous calculations and/or experimental measurements. None should be considered final.

1. Excess cold reactivity with drums at 180°	\$ +2.00
2. Worth of drums at 180° (compared to no drums)	-1.00
3. Worth of W shim	-3.00
4. Worth of axial structure external to core	+4.70
5. Control swing (drums out to drums in)	-7.50

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Fig. 17. Full power hydrogen content and axial distribution for tie tube design of Phoebus-2.

Based on these assumptions, the cold "critical," one-dimensional model will contain \$3 worth of Ta shim, \$3 worth of B at a position equivalent to drums at 118° , and will have a $k_{\text{eff}} = 0.966$ ($-\$4.70$ reactivity defect).

The full power H_2 content and corresponding axial distribution were calculated for the latest tie tube design (0.205 in. and 0.350 in. o.d. for the inner and outer tubes, respectively). The results are shown in Fig. 17. The total core H_2 content is 3.12 lb of which 67% is in the fuel elements and 33% in the tie tubes.

The C density of the loaded fuel elements in the neutronic model was increased to 1.82 g/cm^3 and the unloaded graphite to 1.87 g/cm^3 . Previously, a value of 1.80 g/cm^3 had been used for both types.

In conjunction with planned irradiation experiments, the average B burnup in the control plates and inlet poison plates was calculated to be 0.014% and 0.023%, respectively. It should be noted that the maximum local burnup will be appreciably greater, particularly in the control plates where a max/av factor of ~ 20 might be expected.

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Phoebus-2 Structural

A study layout was made of the preferred interface cylinder and lateral support arrangement for Phoebus-2. It will be updated as more detailed fluid flow and thermal information influences the design. The latest arrangement contains the following general features:

1. Interface cylinder made up of stacked Al rings.
2. A ladder seal arrangement using graphite seals and plungers.
3. Angle-face plungers and seals to provide positive seal seating.
4. Al leaf springs, three layers of 0.080-in. material.
5. A ring reinforcement of the interface cylinder at the nozzle end to resist buckling.

Detailed drawings were made of a new tie tube design. The major changes are those of tube diameter; i.e., an outer tube o.d. of 0.350 in. and an inner tube i.d. of 0.205 in.

The load-deflection analysis of the iris spring system (no longer current) was completed and reported in an internal memorandum in which the derivation is given of the equations for the deflection, stress, and load relationships. Predictions were made of the deflection-load characteristics of the three springs that comprise the model acting in parallel.

Derivation of the equations that describe the dynamic behavior of the reactor structure was initiated. The spring terms have been derived using Lagrange's equations to insure the inclusion of all the coupling terms and work is being continued on the derivation of the friction terms.

Phoebus-2 Fabrication

Tie Tubes

The first developmental assemblies of Phoebus-2 tie tubes at Twigg are 40% through final assembly; these parts should be complete in early June. On the basis of the experience gained during process development,

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revised drawings and specifications are being generated. One major change consists of the elimination of the 3rd braze cycle.

Control Drums

Fabrication development of control drums has started on two different materials. One design (using Al construction) will probably require chem-milling of the inner cylinder. The other design (using stainless steel) probably involves simpler fabrication techniques. The Al design is preferable, however, from the neutronics standpoint.

Beryllium Reflector

Two sets of Be blanks were ordered for the Phoebus-2 reflector and several pieces are already on hand. Drawings specifying the trepanning requirements on these blanks have been released for bid. The trepanning for the control drum will be done as a separate fabrication step since fabricators capable of doing the remaining machining are not generally equipped for trepanning.

Phoebus-2 Fuel Element Thermal Stress Perturbations Due to Coolant Channel Location Displacements

A thermal stress perturbation analysis (see LA-3280-MS, pp 116 and 117) was completed for the Phoebus-2 fuel element configuration (nominal Kiwi-B-4 channel locations with channel diameters increased to 0.110 in.) and the Phoebus-2 operating conditions. The purpose of the study was to determine the variations in the maximum calculated thermal stresses as the locations of the ring of six and the outer twelve channels were moved radially over a range of ± 0.010 in. from the nominal locations.

The input parameters used in the analysis were as follows:

1. Reactor Operating Conditions:

Core inlet temperature	260°R
Core exit temperature	4500°R

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2. Values at Core Axial Station 30:

a) Thermal Inputs

Power density	105.5 Btu/in ³
Hydrogen bulk temperature	2960°R
Heat transfer coefficient	6.73×10^{-3} Btu/sec-in ² -°R
Thermal conductivity	3.99×10^{-4} Btu/sec in.-°R

b) Thermoelastic Inputs

Elastic modulus	1.2×10^6 lb/in.
Thermal expansion coefficient	2.7×10^{-6} °R ⁻¹
Poisson's ratio	0.15

The thermal boundary conditions of heat transfer coefficient and H₂ temperature at each channel surface were held constant for all perturbations. The outer surface was assumed to be adiabatic.

Figure 18 is a plot of typical results for the situation where both sets of holes were moved inward by 0.010 in. The in-plane thermal stress component is plotted around each hole, where it takes on its maximum value, for the nominal location and for the perturbed locations. The complete

Fig. 18. Thermal stress perturbation due to 0.01-in. inward movement of coolant channels in Phoebus-2.

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results of the investigation indicate: (1) the maximum value of the thermal stress is a minimum with the coolant channels in the Kiwi-B-4 nominal locations; (2) the stresses are tensile in the region of the thin (~ 0.023 in.) outer webs for the nominal location; (3) movement of the outer channels inward increases the maximum stresses but tends to put the thin webs (now thicker by the inward relocation) into compression; and (4) the maximum in-plane stress value is not very strongly influenced by movements of the location of the inner ring of six channels.

Phoebus-2 Tie Tube Flow and Pressure Drop Experiments

An experimental program was initiated to determine the flow and pressure drop characteristics of the Phoebus-2 tie tube. Initial data were obtained on a prototype tie tube assembly. The data were taken at ambient temperature H_2 flow with no heat addition. The results indicate that the pressure drop data for the center tube can be reduced to average surface roughness values which agree quite well with an actual profilometer determination. The button pressure losses can be conveniently expressed as a loss coefficient to be used with the velocity head at the exit of the center tube.

It is planned to extend this test program to incorporate the design changes of the component as they occur and to include heat addition, cryogenic H_2 , and heat transfer measurements.

Phoebus-2 Reflector System

Development work on the B_4C/Cu mix for control plate material is being continued (p.110).

Control drum drive shafts for Kiwi reactors accommodated minor misalignment by using two crown-tooth gear couplings, one near each end. Because of the relatively high cost and long fabrication time required for these couplings, it is hoped to develop a simpler flexible coupling for use in Phoebus-2. Steps were taken to procure test samples of 1.5-in. diam

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stainless steel tubing incorporating an integrally formed bellows convolution near each end. Subsequent analysis has indicated too low a natural frequency in torsional vibration with tubing of this size. The diameter of the tubing was therefore increased to 2.5 in. o.d. to keep the natural frequency above 50 cps.

Phoebus-2 Reflector Thermal Analysis

OPTIØN calculations were made of the pressure distribution in the ladder seal system. Plunger leakages were neglected in the first step but were subsequently calculated at each seal station using the previously determined pressure distribution. The results indicate the plunger leakage to be of the same order as the bypass leakage, invalidating the first results. The present OPTIØN code cannot properly handle a problem of this type and so other analytical tools (e.g., Westinghouse codes) are being investigated.

Hand calculations are being made for the most recent configuration of the interface cylinder. It has been decided to flow test this design as soon as possible and specifications for such testing are being prepared.

Phoebus-2 Heat Transfer - General

A parametric study was completed on tie tubes with various tube diameters. This resulted in the selection of an outer tube o.d. of 0.35 in. and an inner tube diameter o.d. of 0.205 in. Studies were made for the full power condition and for a particular point on a possible shut-down profile. The results are shown in Figs. 19 and 20, respectively. The studies of the variation in inner tube diameter showed little sensitivity of pressure drop to changes in diameter. The tie tube analyses will be continued for various other shutdown programs and conditions.

A new calculational model was written for the fuel channels including the unloaded portion of the fuel element and the support block.

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Fig. 19. Characteristics at core center for Phoebus-2 tie tube full power.
Fig. 20. Characteristics at core center for Phoebus-2 tie tube 96 sec after scram.
Fig. 21. Phoebus-2 fuel channel pressure distribution.
Fig. 22. Phoebus-2 fuel channel maximum temperature distribution.

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Calculations have been made with variations in the local/average power ratio. The distributions of pressure and maximum temperature are shown in Figs. 21 and 22, respectively. For a peak-to-average power ratio of 1.2, the core pressure drop is calculated to be 183 psi. These calculations were made with the Reynolds number evaluated at bulk temperature.

The requirements for fuel element orifices were studied. It was found that a range of orifice diameters from 0.080 to 0.030 in. can maintain an exit gas temperature of 4500°R for power densities varying by a ratio of 1.5, max-to-min. This holds true when the maximum-to-average power density ratio is varied from 1.2 to 1.05.

Two-dimensional studies were made of the unloaded graphite region of the periphery. The results indicated that unloaded graphite possessing radial dimensions as presently planned (up to 0.6 in.) would experience undesirably high temperatures and temperature gradients. The calculation was extended to provide a modest amount of cooling for the channels. It appears that single coolant channels for each of the larger filler elements will be adequate and that a channel diameter of 0.085 in. will suffice. Such channels will require metering since it would be desirable to maintain the exit temperature of their coolant at 4300 to 4500°R.

The tie tube button temperatures were reexamined for the new tie tube geometry. Increasing the tie tube flow rate to 0.047 lb/sec decreased the button gas temperature from 465° to 200°R. Previous two-dimensional solutions were used in estimating button temperatures for the new boundary conditions. The results for three geometries are shown in Fig. 23.

Phoebus-2 Exhaust End Mockups

The axial fission profiles in the Phoebus-Zepo core have been observed with various mockups of exhaust end structures. Profiles are reported for the bare core, with 1-3/8 in. of unloaded end tips added to the fuel elements, and with fuel elements to which end tips and various

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Fig. 23. Estimated tie tube button temperatures for three geometries.

profile whereas the lower curve shows detail at the end of the fueled region.

The fission density at the fuel element ends relative to their center is shown by Fig. 25 to depend primarily on the amount of graphite added.

Phoebus-2 Reactivity Contributions of Core Materials

Effectiveness measurements of core materials in Phoebus-2 Zepo gave core-average reactivity coefficients and radial variations for U, graphite, and Nb, and a core-average value for Inconel.

For the U measurements, fuel elements with loadings of 100, 200, 300, and 400 g/cm³ were substituted for unloaded elements. As shown in

support block structures have been added. Table 6 gives a description of each mockup and a summary of the reactivity contribution.

Fission profiles were observed by scanning fuel elements with a well-collimated crystal scintillation detector. The results were fitted by least squares to functions of the type $a_1 e^{-a_2 x} + a_3 \sin(a_4 + a_5 x)$. In each case, $x = 0$ is the end of the loaded portion of the fuel element and the functions are normalized to unity at their highest point. The fission profile data are summarized in Fig. 24. The upper curve illustrates the overall effect of various end mockups on the fission

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Table 6

Phoebus-2 Exhaust End Mockups

No.	Mockup Components	Thickness (in.)	Void (%)	Weight of Materials (kg)					Reactivity (\$)
				C	Nb	Ta	Inc. 718	Mo	
1	Bare core end								
2	Unloaded end tips	1.37	33.7	58.2	9.12		2.71		0.96
3	Support blocks uncoated	2.50	40.0	100.9					
4	Support blocks Ta-coated	2.50	36.6	100.9		24.7	14.0		1.30*
5	Support blocks Nb-coated	2.50	36.1	100.9	16.7		14.0		1.40*
6	Support blocks (Kiwi-B-4 type)	1.50	30.4	66.8	9.53		3.98	1.48	
7	Variation of 2 + 5	1.37	33.0	58.2	12.2		2.71		
		2.50	33.2	100.9	40.1		14.0		

* Additional reactivity when added to Mockup No. 2.

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Fig. 24. Axial fission profile data for Phoebus-2, showing overall effect and detail at end of core.

Fig. 25. Fission density at end of Phoebus-2 core as function of amount of graphite added.

Fig. 26, the sensitivity of reactivity coefficient to the size of fuel-loading change is large near the core surface but disappears as the axis is approached. The radial values for graphite (Fig. 27) represent the effect of inserting 0.25-in. diam graphite rods into channels of the Zepo elements.

Fig. 28 shows overall reactivity contribution of Nb as a function of loading and the corresponding core-average reactivity coefficient. Experimental coefficients in Honeycomb assemblies with various C/U ratios are included to illustrate general consistency with the Zepo results.

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Fig. 26. Uranium reactivity effectiveness vs radius in Phoebus-2 Zepo.
 Fig. 27. Graphite reactivity effectiveness vs radius in Phoebus-2 Zepo.
 Fig. 28. Niobium reactivity effectiveness vs radius in Phoebus-2 Zepo.

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Core-average reactivity coefficients for Zepo and the similar Honeycomb assembly are compared in Table 7. The small value for Inconel in Zepo was the result of adding double-wall support-tube mockups to a 60° sector of the core.

Table 7. Core-Average Reactivity Coefficients

<u>Material</u>	<u>Reactivity (ρ/kg)</u>	
	<u>Zepo</u>	<u>Honeycomb</u>
Uranium	7.7	7.2
Graphite	2.8	3.0
Niobium	-3.2 ⁽¹⁾ -3.6 ⁽²⁾	-2.5 ⁽³⁾
Inconel	-0.65 ⁽⁴⁾	

(1) Zepo type: 75 g, single tube/element

(2) Phoebus-type: 75 g, 19 tubes/element

(3) 0.005 in. foil: 91 g/element

(4) 64% Inconel 600, 36% Inconel 718

Phoebus-2 Boron-Copper Control Vane Measurements

According to measurements in Phoebus-2 Zepo, the proposed B-Cu control plate material effectiveness is identical with the Kiwi boral material at the same concentration of B¹⁰. The matrix material and B¹¹ content appear to have no influence on the neutron absorbing properties.

Test plates of Cu-B₄C (natural B) 2 x 13 x 0.050 in. were fabricated with 20, 40, and 50 v/o B₄C content. Reactivity contributions of samples in the Zepo reflector annulus nearest the core were compared with that of a Kiwi boral plate of the same size. The 0.050-in. thick plates were

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Fig. 29. Effectiveness vs B¹⁰ surface density in Cu-B₄C measured in Phoebus-2 Zepo.

used in single, double, and triple thicknesses in order to provide a wide range of points. As Fig. 29 shows, the effectiveness is a function of the surface density of B¹⁰ alone. The solid curve is the fit obtained with a two-term saturation function

$$Y = \sum_{i=1}^2 a_i (1 - e^{-b_i x})$$

in which $a_1 = 0.612$, $b_1 = 274$, $a_2 = 0.495$, and $b_2 = 25.5$.

Phoebus-2 Radiation Heating in a Dodecagonal Core Phoebus Reactor

The QAD Point Kernel program has been modified to handle explicitly the source regions necessary for estimation of radiation heating from a dodecagonal (12-sided) core. Calculations have been made with this new program version to determine the heating from such a core and to compare them with results from an equivalent cylinder version having the same cross sectional area. Linear thicknesses of the unloaded pyrographite,

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slats, and void region were the same. The outer radii of the interface regions were set equal, with the result that cross sectional areas were equal to within 1% with the regions inside the interface region treated as indicated above. A small region of filler strip was lumped with the pyrographite into a single region and a small void between the interface region and the reflector was neglected. Neither of these approximations should affect the results significantly.

All calculations were for a power level of 5000 MW and a core length of 132.08 cm (52 in.). The axial power density distributions were cosine-shaped with relative values of 0.46 at the inlet end, 1.0 at the peak, and 0.12 at the outlet end. All reported points were calculated at the axial position of peak fission density.

Total heating rate traverses through the interface and reflector regions are given in Fig. 30 for the cylindrical core and at 0° and 15°

for the dodecagonal core. Although the percentage differences between the curves are not large, they seem to persist well into the reflector. The values given here are in good agreement with those reported in LA-3158-MS.

Fig. 30. Total heating rate traverse for cylindrical and dodecagonal Phoebus-2 cores.

Phoebus-2 Tie Tubes

No additional tie tubes were fabricated for Phoebus-2 during the report period and it is expected that no more will be made until the new brazing and heat treating furnace has been installed. The assemblies on hand were pressure checked to 1500 psi, followed by a vacuum leak test; all passed both tests successfully.

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Fifteen ribbed support-Inconel 718 tubing joints brazed with Nicro were tested in tension at various temperatures. All specimens broke in the tubing at **strength** levels similar to those published in the literature. Five specimens were tested at each temperature and the average for each condition is given in Table 8.

Table 8. Ribbed Support - Inconel 718 Tubing Braze Strength

<u>Test Temperature</u>	<u>Ultimate Tensile Strength (psi)</u>	<u>Yield Strength 0.2% Offset (psi)</u>
Room	199,980	176,980
1000°F	149,760	133,300
1325°F	125,980	124,060

Flow tests on the magnetically formed Inconel 718 standoffs, used to align the inner and outer tubes of the tie tube assembly, indicated a pressure drop larger than desired for some unknown reason. A test specimen has been designed for direct comparison of pressure drops when standoffs of different design are tested.

The material for the outer tube has been changed from Inconel 600 to Inconel 718 because of handling problems and for other reasons. It is anticipated that orders for Inconel 718 tubing and rod for Phoebus-2 Cold Flow and Phoebus-2A reactors will be initiated shortly.

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III. NRDS ACTIVITIES

MODIFICATIONS TO TEST CELL C

Introduction

During the past quarter the majority of work needed for Test Cell C modification was placed under contract. The detailed status of major areas is described below.

First Construction Package - LH₂ Dewars and Hot Water Tank

Submittal and approval of vendor drawings for the dewars is essentially complete. Site preparation started during the week of March 15. Temporary and permanent foundations are complete. Erection of the water tank started during the week of April 19. Erection of the first dewar started during the week of May 24.

Second Construction Package - Site Preparation, Piping and Electrical Systems

Work scheduled for completion by May 7 (end of the first 120 days of contract time) is essentially finished. The 3-in. GN₂ underground line to the water tank area originally scheduled during this period has been shifted to the period after the forthcoming reactor run. Scheduled tests have excluded the contractor from the test cell for 9 days during this period. Foundations and associated work for the LN₂ dewars and four high-pressure gas bottles along with the paving under the LH₂ dewars have been added to the work and should be completed by June 18. At the present time,

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the contractor still intends to leave the site approximately June 18 and return October 7. Award of the LN₂ system piping installation with a November completion date, however, will require an earlier return to the job.

Government-Furnished Equipment

Vacuum-Jacketed Pipe

Submittal and approval of shop drawings are essentially complete. Although minor changes have been made and additional work awarded, the scheduled delivery date still holds. The contractor has been asked to make a feasibility study for the revamp of existing spool pieces into a new LN₂ supply line.

Valves

Additional purchase orders were awarded for valves and filters having a total value of \$30,994. Submittal and approval of vendor drawings continues. Purchase orders for approximately 70 N₂ system valves have still to be placed. Approximately 20 valves for the turbine energy exchanger and high pressure LH₂ dewar remain to be purchased.

Flow Systems

Flow Criteria

An extensive list of fluids, flow rates, and pressure requirements which include all phases of reactor operation, both normal and emergency, was published to enable completion of the design of the LASL portion of the modification.

Liquid Hydrogen System

Design drawings of the LH₂ lines in the flow control room, including the high-pressure LH₂ system, are nearly complete. Valve

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specification sheets are being written and surplus valves are being assigned where possible.

Mechanical design for instrumentation of the two 500,000-gal dewars is scheduled for completion by June 15. All hardware is to be on hand by June 30, and fabrication should be finished by August 15. At that time, while the dewar construction is in progress, the instrumentation for studying perlite compaction, temperature gradients, and acoustical motion will be installed in the annular spaces. After the dewars have been delivered to the test cell, instrumentation inside the inner vessel, for measuring temperatures and liquid levels, will be installed.

Recent tests have indicated that adequate reduction in pressure drop in the existing LH₂ pump suction line resulted from replacing part of the 8-in. line with 10-in. line. It will not be necessary, therefore, to replace the existing 8-in. pump suction venturi to accommodate the higher flow rates planned for the Phoebus-1B tests.

Flow Measurements

The 10-in. venturi for the new pump discharge line is being fabricated by the LASL shops and is scheduled for completion by June 15. It will be shipped to Associated Piping to be incorporated in the discharge line and vacuum-jacketed.

In view of the confirmation by test data from Rocketdyne of the previously anticipated difficulties of obtaining good LH₂ flow rate measurements during stall conditions from a meter in the pump discharge, it was decided to include an inlet venturi in the 10-in. ducting of the NFS-3 turbopump. The design-sizing criteria were established as 25 psid at 350 lb/sec. Drawings of this venturi have been completed and sent to Associated Piping. They will fabricate the venturi and incorporate it in the pump suction line.

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It has also become necessary, as a result of gaining knowledge of NFS-3 pump characteristics, to increase the LH₂ bypass line from 4 in. to 6 in. Work has been initiated to accomplish this change.

Emergency High-Pressure Dewar System

The potential for inadvertent turbopump shutdown during reactor operation (such as occurred during the first NRX-A-3 run) was reviewed and the earlier decision to provide for this situation by means of a pressurized LH₂ vessel was reaffirmed. The system will consist of a 3000-gal vessel pressurized to 500 psi connected to the main propellant line through a check valve downstream of the main flow control valve. In the event of a pump shutdown, the adiabatic depressurization of the vessel will provide adequate cooling until such time as the dewar pressure (100 psi) can provide adequate flow.

Liquid Nitrogen System

Foundations for the three 28,000-gal LN₂ dewars have been completed. Because of the transportation problems, LASL was asked to accept two dewars from Warren Air Force Base other than those which had been inspected. An inspection of the substituted dewars has been made to confirm critical dimensions.

Drawings of the LN₂ piping have been completed. Use is planned of 8-in. vacuum jacketed piping removed from the existing LH₂ system and also of valves from valve skids acquired with the dewars.

An order has been placed with Cosmodyne for the bottle-charging N₂ vaporizer. Delivery, originally promised for May 15, has been postponed 30 days.

Gas System

Foundations for the four new gas bottles have been constructed. The bottles have arrived on site but have not been put in place. Drawings

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of piping to integrate the new bottles into the system have been issued. Other gas system modification drawings, including those showing interfaces between new and existing piping, are complete.

Liquid Level Sensing

Liquid level sensing in the two 500,000-gal dewars to be built for Test Cell C has been under investigation. The bids for commercial capacitance-type continuous sensors seemed high, and consideration has been given to the design and fabrication of suitable units at IASL. The desired accuracy of 0.1% places stringent limitations on design. A capacitive sensor must include quite complex compensation measurements to achieve such accuracy.

Recently, very promising results have been obtained by use of time domain reflectometry to locate the gas-liquid interface in a coaxial cylinder geometry. This idea will be pursued since it can be accomplished with only minor modifications of commercial equipment.

The level sensing investigation has also involved construction and evaluation of several other items, including a 65-point sensor, a capacitance sensor, and comparison of capacitance probes made both of conventional tubing and of sintered stainless steel.

Computer and Data Systems

Control Room Automation

The computer portion of the Operations Control System is defined in detail in EG&G Specification No. 1248, "Operation Control System - Computer System Specification." The basic system consists of a 16-bit minimum word length parallel digital computer, with 8K of magnetic core memory, including two high-speed data channels. One channel communicates with a cathode ray tube capable of producing alpha-numeric and profile displays. The second data channel is used with a 200-channel analog

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multiplexer and analog-to-digital unit. The remaining input devices of the basic system include 208 binary input channels, an input/output typewriter, and a paper tape reader/punch. In addition to the CRT display, 12 digital-to-analog output channels drive strip chart recorders. A request for a quotation on this computer system was sent to 22 possible bidders. An evaluation of the technical portion of each bid received is presently in progress. A total cost breakdown of each vendor, including all options, is also being prepared.

Preliminary design on a logic system for automation of key phases of the checklist has been completed. Proposed circuitry and patching techniques are included. Laboratory evaluation of breadboard models using various vendors' equipment is now in progress at EG&G.

Data System

Phoebus-2 reactor tests will require additional narrow-band amplifiers. Since the existing amplifiers have a high failure rate and high maintenance cost, it was decided to purchase enough amplifiers to replace those in the existing system. Requests for bid on 400 units were sent to 18 manufacturers and the responses to these requests are being examined.

Control Room Modification for Phoebus-2 Testing

The upgrading of Test Cell C for Phoebus-2 testing will require control room console layout modifications. These additions will include: (a) LN_2 system for reactor cooldown, (b) high-pressure LH_2 storage system for emergency coolant, (c) thermal water deluge system, (d) LN_2 heat exchanger system for use as a reactor cooldown vaporizer, (e) four gas storage bottles, and (f) the turbine drive system (LH_2 - H_2O heat exchanger). A firm control room layout was completed and will serve as the master drawing for future chassis design as it reflects the new operational philosophy.

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Neutronics

An evaluation of two solid state log amplifier circuits has been completed. The circuits are similar in that they depend on the forward conductance of a transistor. The new log amplifiers offer improved log characteristics over a large current range as well as simplicity of design and maintenance.

Water-LH₂ Heat Exchanger (Turbine Drive System)

The 160-ft long experimental test section of the H₂O-LH₂ counterflow heat exchanger (LA-3280-MS, p. 56) was completed at ACFI in Albuquerque and delivered to NRDS. It consists of two 80-ft lengths, one of which contains a twisted Al swirl generator or turbulator.

The H₂ flow path through the inner pipe has an inside diam of 4 in. and a wall thickness of 3/4 in. The inner pipe is finned with 12 longitudinal fins each 1 1/2 in. high. The Al water jacket has an inside diam of 10 1/4 in. The design flow rates for the test series were 12 1/2 lb/sec of H₂ and 1000 gpm of H₂O.

Tests were conducted under a variety of conditions during five experimental plans including a full design flow rate and actual turbopump bootstrap operation. The dates of the plans were as follows: E.P. I, March 26; E.P. II, April 15; E.P. II-A, April 16; E.P. III, April 20; and E.P. IV, April 29, 1965. Procedures were developed to provide satisfactory startup and steady state operation. Smooth operation appears to depend on exchanger pressurization level and rate of increase of LH₂ flow rate into the exchanger.

Heat transfer measurements confirmed the preliminary model studies. The latter had indicated that the H₂ heat transfer coefficients were about half those predicted by the classical Dittus-Boelter correlation and inclusion of the swirl generator increased the value to an average of 93% of that predicted. This increase appears to be independent of the location

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of the swirl generator, i.e., the liquid region or the gaseous region. Based on these results, it was decided to include the swirl generator throughout the entire heat exchanger length and to reduce the overall length of each section to 320 ft from the previous 480 ft.

Strain measurements indicated high values of axial stresses on the inner pipe which approached the yield stress. This had been anticipated by previous stress code calculations. To alleviate this condition, it was decided to reduce the inner pipe fin height from $1\frac{1}{2}$ to 1 in. and to reduce the wall thickness $\frac{1}{4}$ in. by increasing the inside diameter from 4 to $4\frac{1}{2}$ in.

Design, procurement, and fabrication of the full-scale heat exchanger are proceeding on this basis and design is approximately 50% complete. The full-scale unit consists of four sections, each 320 ft long, and is expected to deliver 50 lb/sec of GH_2 at approximately 500°R . This work will be done by ACFI with an NRDS delivery date at the end of the year.

Reactor Shield

For the anticipated long duration full power runs of Phoebus-1C, it appears desirable to include an external reactor shield similar to that being designed for the Phoebus-2 reactors to reduce the test stand activation (LA-3280-MS, p. 60). Preliminary criteria for such a shield have been developed and various configurations are under study. The first concepts indicate that the H_2O -cooled shield could be fitted on the roof of the car around the reactor.

Outline drawings and specifications for the Phoebus-2 shield have been prepared and the design is firm. Neutron attenuation will be achieved by an arrangement of B-loaded tubes forming the H_2O cooling passages of the shield. Samples of the B-loaded Al tubes have been received from the vendor and are currently undergoing neutron attenuation tests to determine uniformity of mix and shielding effectiveness.

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Turbopumps

Two Mark-9 pumps and two turbines forming the NFS-3 system were delivered to NRDS March 15 to satisfy Rocketdyne's obligation under contract NAS-W-347. Rocketdyne has signed a contract with SNPO to continue development of the E-bladed pump. This NFS-3A program calls for procurement of two new Mark-25 pumps, modification of two existing E-bladed pumps to the Mark 25 configuration, and modification of four Mark-9 Mod. 1 turbines to the five-stage Mark-25 configuration. The test program for the NFS-3A was approved May 20. The delivery of one set of Mark-25 pumps and turbines is scheduled for Dec. 1 and the other for Dec. 15. The NFS-3A single mode can then be made ready in the existing pump room at Test Cell C as the feed system for Phoebus-1B. The initial tests of the first E-bladed pump with the five-stage turbine have been run with the pump attaining speeds up to 32,000 rpm, pump discharge pressure of 1880 psi, LH₂ flow of 125 lb/sec, and turbine flow of 18 lb/sec. Future tests will be made to continue mapping of both pump and turbine performance, speed loop response, specific speed limit loop transfer capabilities, controllability, and endurance.

Additional work has been done on the Mark-9 soft mount bearing non-synchronous whirl problem. Tests have been run with slots cut into the axial flow annulus to spoil the hydrostatic bearing effect, with increased frictional dampening, and with a coned balance piston. Changes were observed in the speeds at which the pump goes into nonsynchronous whirl, but neither the cause of the whirl nor a definite solution to the problem has been found. Three more tests are scheduled in the next quarter with increased dampening and with the balance piston rub rings coned about the pump center of gravity.

The NFS-2 pump and turbine to be used for the Phoebus-1A tests have been refurbished; they were installed at Test Cell C and their performance verified during EP-1.

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R-MAD Building Operations

Phoebus-1A

Assembly of the Phoebus-1A reactor was started during the first reactivity check on April 13. All elements were photographed as stated earlier. The reactor was installed on the test car on April 30, and moved to Test Cell C on May 11. Hook-up, pressure tests, and electrical checks were completed for Experimental Plan I, conducted on May 27.

Disassembly Operations

During the disassembly of Kiwi-B-4E-301, a time study was made in an effort to reduce the time required for future remote disassembly operations. The conclusions were that the examination and photography of fuel elements through the periscopes was the most time-consuming phase. As a result, an automated system was devised to do this (LA-3280-MS, p. 27). ACFI designed and fabricated the system which handles, positions, photographs, and records all faces and the hot end of every element. The electrical control hardware for the system consists of the sequencer console, which contains all the controls, indicators, and solid state circuit boards necessary to interpret, formulate, and convey all logic sequences of the system.

Two film viewers were purchased; one is now at R-MAD, NRDS, and the second will be located at LASL. The viewer is capable of giving 6X, 12X, and 30X powers. In addition, a new film processor and printer have been purchased and are installed at the Technical Services Building at NRDS.

NERVA Support

Support was given to the NERVA Test Organization in the NRX A3 reactor test program. The reactor was moved to Test Cell A on March 5. The final test of several experimental plans was conducted on May 28, and the reactor will be returned to the R-MAD Building for disassembly on June

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2. In the meantime, preparations were completed for the reactor disassembly. These included installation and checkout of the photography system designed and procured as a backup for the LASL system for photography of all elements during disassembly. A Kollmorgen periscope was obtained on a temporary basis from the 401 Area, to replace a periscope procured by the NTO, which proved to be inferior to the Kollmorgen unit. Much effort was expended in improving the weighing system for elements.

Construction

Assembly Bay Addition - In anticipation of the Phoebus-2 Program, criteria have been prepared for an addition to the assembly portion of the R-MAD Building. This includes an additional clean room adjoining the present one, an equipment maintenance area adjoining the present assembly bay, and a core storage vault. This addition comprises approximately 8400 ft².

Security Fence Addition - Criteria have been prepared for a new security fence to enclose the R-MAD compound. This will enclose the present security area, plus the decontamination facility, the hot storage dump, the new storage building, the Beetle Shed, and all of the railroad trackage in the vicinity of the R-MAD Building. The area enclosed will be approximately 41 acres.

Railroad Spur "D" Realignment in R-MAD Dump - The design to reduce the grade and increase the curve radii to enable hot reactors to be stored was approved on March 26. A contract was awarded to Smith Construction Co. Work started on April 5 and was completed on May 21.

Phoebus-1A Preparation

Schedule

A schedule for Phoebus-1A test series was prepared and will consist of six experimental plans as follows: E.P. I, May 27; E.P. II, June 10;

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E.P. III, June 23; E.P. IV, June 25; E.P. V, July 8, and
E.P. VI, July 15, 1965.

Objectives

The primary objective of this test series is to run the reactor for extended duration at full design power. The objectives of each experimental plan are summarized below.

E.P. I - Facility Checkout: The facility system will be checked out and the full-power run flow rate profiles verified. All emergency systems will be operated.

E.P. II - Reactor Calibration Run: This experimental plan will calibrate the neutronic equipment.

E.P. III - Scaled-Down Power Run: The power profile will be verified at low power. All operations to be performed on the full-power run will be coordinated and their noninterference with reactor operation verified.

E.P. IV - First Full-Power Run: The reactor will be operated at full power to the limit of the LH₂ available. The programmed variables for this test will be the same as those on Kiwi-B-4E-301.

E.P. V - Second Full-Power Run: The reactor will again be operated at full power until LH₂ depletion forces shutdown. During the power phase of this test, the reactor power will be programmed by positioning the control rods from a program demand and allowing propellant reactivity to increase reactor power.

E.P. VI: This run is tentative, and will be conducted if further testing is required to meet the primary objective. It will consist of another duration run at full power.

Radiochemistry

The radiochemical program for Phoebus-1A will include, for the first time since the Kiwi-B-1A test of December 7, 1961, analyses of exhaust gas

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samples collected by B-57-C aircraft fitted with wing-tip filter units. It is expected that the results will provide information on the number of atoms released to the gas stream for most of the fission-product chains.

Plans for radiochemical measurements on Phoebus-1A include a new approach, tested on the Kiwi-B-4E core, which involves taking γ -spectrum measurements on 400 pieces of fuel element (40 elements with 10 pieces per element), and on 300 similar size pieces from structural components. These γ data, used in conjunction with results of radiochemical analyses on selected slices, will provide information on radial and axial distribution of fissions, loss of fission products as a function of axial location, and distribution of temperature in the reactor core. A detailed radial distribution of fission density near the core perimeter will be established from the results of radiochemical analyses on fuel samples obtained by taking longitudinal cuts through three of the pieces of fuel.

The equipment for the measuring and recording gross radiation intensities for whole fuel elements has been revised. The entire system, including the equipment for measuring fuel element weights, will be tested the first week in June.

The determination of the number of fissions generated in the calibration run at NRDS will be based on analyses of both Ni and U wires placed in fuel elements near the center of the core. External Ni monitors positioned near the throat of the nozzle will provide a value for fissions produced on all subsequent runs.

Facility Modification

A reactor cover lifting boom has been installed. Because of procurement difficulty the winch motor has not arrived, but a temporary motor has been installed and the boom will be usable for the forthcoming series of tests.

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A 200-gal reservoir has been installed on one of the two original hydraulic pumps, replacing one of 100-gal capacity, and making the two pump packages identical. These two pumps will be operated in parallel to accommodate servo valves. An additional new pump will serve the on-off valve system.

A pressure decay test has been performed on the servo hydraulic system to determine the adequacy of energy stored in accumulators to effect an automatic program of valve actuation in the event of pump failure. Pump failure and subsequent shutdown were simulated, after which a period of nearly 15 min elapsed before system pressure was dissipated by normal leakage through servo valves. It appears therefore that a safe, orderly shutdown would be possible in the event of pump failure.

Failure Analysis

A detailed failure analysis has been performed on all control equipment, such as power supplies, amplifiers, relays, feedback circuits, 110 volt ac power, 28 volt dc power, and protection circuits, facility and reactor controls. The conclusion was reached that from the control standpoint there is no single failure that could be disastrous during a reactor test. Several relatively minor problems were encountered and corrected as a result of this analysis. Examples are the following:

- (a) The two turbine speed channels are both used as inputs to the flow shutdown system. The higher of the two is electronically selected for turbine speed control.
- (b) Separate power supplies were provided for each of the two converters associated with the turbine speed channels.
- (c) A circuit was installed on ACV-16 which may be used to lock the valve open, eliminating all failure modes except the servo valve and hydraulic system.

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Scram and Flow Shutdown Systems

During previous reactor tests the Scram and Shutdown Chains have been tied together, i.e., a Scram would produce a Flow Shutdown, and a Flow Shutdown would produce a Scram. This allows only one action in the event of trouble: stop LH₂ flow and scram all rods. Longer reactor run times and some other test considerations require that different actions be taken depending upon the type of trouble and the time at which it occurs. The following actions have consequently been agreed upon:

1. Scram when in program control of LH₂: have an exponential decay of the LH₂ demand to 20 lb/sec; no flow shutdown.
2. Shutdown: scram the reactor, stop LH₂ flow, close ACV-16, switch PCV-89 to pressure control (initially at 0.6 times the LH₂ pump discharge pressure and increasing automatically to 1.2 times this pressure).

These modes are all options and can be activated or bypassed as the Test Group Leader or Chief Test Operator desires. Some safeguards have been built into the options, e.g., insuring that the scram chain cannot be reset while the LH₂ "program exponential decay" option is active.

Neutronics Logic and Other Modifications

Evaluation of Nozzle Fission Chambers - The use of radiation shields around future Phoebus reactors will require changes in the neutron detection system which currently provides the reactor control signal. Two experimental fission chamber designs will be evaluated during the Phoebus-1A test series. The chambers will be located on the converging section of the nozzle near the neck as shown in Fig. 31. The chambers will be contained in an Al block $1\frac{1}{2}$ in. by $1\frac{3}{4}$ in. by 1-in. thick. Thermon T-1 (a self-hardening thermally conducting paste) will occupy all interstices in the block. The bottom of the block will be shaped to

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conform with the nozzle and a layer of Thermon will be used to insure good thermal contact with the nozzle. Stainless steel straps will secure the block to the nozzle and two thermocouples will monitor the block temperature.

Neutronic Logic - In order to provide a more reliable neutronic feedback signal to the power controller, a logical combination of the four neutronic log power channels was implemented. The circuit is shown below. The circuit adds very few components and eliminates the dependence on a single neutronics channel for control. It also provides a form of averaging since it throws away at least one channel higher than and one channel lower than the controlling channel.

Fig. 31. Proposed location of fission chambers in nozzle.

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Special Projects

Dewar Boil-Off Measurements

Measurements on Dewar A (Test Cell C) indicate boil-off rates of 0.24% per day at 3 weeks after filling and 0.22% per day at 5 weeks after filling. Because of an unfavorable LH₂ delivery schedule, it was not possible to obtain meaningful early data, but both earlier and later measurements will be made. The guaranteed equilibrium boil-off rate is 0.3% per day, and equilibrium was expected some 20 to 30 weeks after filling. The preliminary data indicate a more rapid approach to equilibrium as well as a much better boil-off rate than originally anticipated.

Boil-off tests for the 100,000 gal LH₂ dewar at Test Cell A have been suspended during this report period, but will be resumed after the current NERVA reactor test series.

Cryogenic Thermocouple Calibration Facility

A LH₂ cryostat has been built at the CEL. It will be used to calibrate thermocouples and Rosemount probes for cryogenic applications.

Effluent Gas Sampling Device

A basic scheme for sampling reactor effluent gas has been approved. It consists of a retractable gun barrel and a magazine. The magazine will store capsules and index them into position for insertion into the barrel. The capsules will be driven mechanically through the barrel to a point in the gas stream, and after being filled they will be returned to the magazine. A layout showing the general design of the gun and its relationship to adjacent structures is being prepared.

Hydrogen Flame Radiation Studies

An apparatus for producing hydrogen flames was fabricated and operated in support of radiation studies. A steam generator has been designed, built, and operated to provide a curtain of steam for

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investigation of the effectiveness of steam as a heat shield. The device will be operated near the small tower during Phoebus-1A tests.

Assistance to NTO (ETS-1)

Facilities to charge GH_2 storage bottles have been investigated. The original scheme was to pass dewar boil-off gas, supplemented by LH_2 from the dewar drain line, through a vaporizer to compressors. The system in question was to handle 100 cfm, a capacity which was apparently compatible with anticipated turn-around time when ETS-1 criteria were originally established. Current plans, however, require a faster turn-around time (9 to 10 days), and NTO therefore plans to abandon the existing (incomplete) system in favor of a high capacity pump-vaporizer unit.

Preliminary plans are being reviewed for a test to determine heat loss in the vacuum-jacketed line which connects the LH_2 supply dewar and the run tank.

Equipment Testing Laboratory

Foundation work was started for installation of two 1200 ft³ GN_2 storage vessels. Preliminary piping and instrumentation drawings were made. The bottles were procured as excess property and have been delivered.

Pressure and leak tests were conducted in the static test bay on 110 valves of various types and sizes, seven pressure regulators, three pressure gages, two tubing assemblies, a small pressure vessel, an emergency purge spider, 10 flex hoses, and a small heat exchanger.

Evaluation flow tests using H_2O were completed for a 4-in. Annin pneumatic control valve with a fail-in-place operator, a 4-in. check valve, and a 2-in. Annin control valve with special modified linear plug. Flow tests using hydraulic oil were made on two hydraulic

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filter banks. A GH_2 flow test was performed on an emergency purge spider. Evaluation of a 1/4-in. Hoke ball valve for instrumentation lines was completed using GN_2 .

Improved calibration ($\pm 0.25\%$) for a 2-in. and 4-in. Potter H_2O flow meter were completed and are in progress for an 8-in. model. The liquid-to-gas small flow meter calibration system using N_2 was run and evaluated.

Several rotameters were calibrated. Verification was made of the correction factor for using GH_2 in an air rotameter. Frequency response tests for a 1/2-in. Annin research control valve were completed.

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IV. FUEL ELEMENT FABRICATION, TESTING, AND DEVELOPMENT

TESTING OF GRAPHITE FUEL ELEMENTS

Hydrogen Corrosion Tests of Phoebus-LA Fuel Elements

Visual Examination

A number of hot-gas tested fuel elements have been examined visually as part of the continuing study of their corrosion behavior. This study has had four prime objectives: (a) a rapid quality evaluation of a given production batch; (2) a more long-range investigation of the applicability of nondestructive test techniques; (3) a correlation of element behavior and process parameters for the possible establishment of more rigid production specifications for a given set of reactor conditions; and (4) the ultimate correlation of hot-gas furnace with reactor test results in order to determine a more exact relationship between these two potentially different types of test.

In pursuit of these objectives, a total of 165 Phoebus-LA production batch elements and 13 miscellaneous related elements were examined microscopically. External pinholing and the condition of the glued-tip were tabulated for each element. The element was then sectioned through the center across a given set of corners and the five exposed interior bores were examined using low-power microscopy. The interior corrosion patterns were tabulated and correlated with production batches, coating furnaces, coating profiles, and potential drop data. Other nondestructive techniques (DXT, bore replication, and permeability)

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currently being used and specific coating parameter data were not available for this group of elements with the result that correlations involving these techniques have not been made.

The most frequently encountered unusual phenomenon was a loss of C from behind the bore coatings, termed "punky" graphite as a descriptive nomenclature. This usually took the form of a generalized loss of C--metallography indicates a probable attack of binder materials--to a uniform depth behind the bore coating. In extreme cases, little or no C structure remained and this has been termed "bore corrosion." In many cases the detectable punkiness started at very low axial stations, e.g., 4-5 in. from the cold end. It usually became worse at higher axial stations up to the point at which the normal "corrosion rings" behind transverse liner cracks occurred. The punkiness usually stopped at this point, recurred at Stations 38-40, and continued to the hot end of the element.

The effect is generally not detectable under low-power microscopy unless the element has been tested for approximately 10 min or longer at temperatures above 2050°C. However, one test for 60 min at 1700°C did show punkiness throughout the element length. A more detailed examination--metallography at higher magnifications--would probably reveal the C loss over wider axial stations and under conditions other than those detected in this study.

The results also indicate a probable difference of mechanism between the C loss at low and high axial stations. At lower axial stations, the loss is probably dependent on the H₂ access through coating grain boundaries or through fine coating cracks different from the large transverse cracks which cause corrosion rings. There are some indications that some coating parameter may be responsible for part of the low-range attack. The high-range C loss is probably due to C diffusion through the NbC coating and its removal by the H₂.

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The interior examination revealed the usual occurrence of corrosion rings behind transverse coating cracks on most elements. There were a few elements which had no corrosion rings but only punkiness and bore corrosion. There were also a few cases in which the corrosion rings in a specific bore extended outside the normal range. In most cases there were some differences in the range of occurrence between the outside bores and the inside bores. This probably reflects the different orificing in the furnace test in which the 12 outside bores are orificed differently from the inner seven bores in an attempt to minimize transverse temperature gradients in the element.

The interior examination also revealed the presence of many corrosion holes behind coating chip-outs or other parts where there may have been liner defects. Some of these occurred in the regions where corrosion rings commonly appear but a significant number occurred outside this range.

Exterior examination reveals those corrosion holes which occur in outside bores and which corrode through the wall to the outside surface. Of the 165 production elements, 50% showed exterior pinholes. A total of 229 such holes was found; 42% occurred in the axial range 10-30 (corrosion ring range) and 22% at stations greater than 50 (including glued joints). Since corrosion holes have a directional quality and tend to continue in the direction started, only those holes which start on the outside part of the outer row of holes will tend to penetrate to the surface. Therefore, a precise correspondence between interior and exterior corrosion holes should not be expected.

Glued Joints

A qualitative examination was made of both the exterior and interior condition of the glued joints (see p. 74). A significant number of joints were loose or heavily damaged after hot gas testing. It is believed, however, that some of the damage was associated with

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electrical grip problems and would not have occurred in a reactor test. Because of this testing problem, no percentage figures of good and bad joints could be established.

Potential Drop Examination

Potential drop examinations were made before and after hot-gas tests on several of the elements in the group under study. The difference curves showed some correlation with the onset of C loss at low axial stations for Y-12 elements where the coating was thin or nonexistent. The IASL elements, which had thicker coatings at the cold end, and the remaining portions of the Y-12 elements did not show any particular correlation between PDE curves and any observable effect. There were no DXT or permeability tests on any of these elements. Attempted correlations between observed corrosion phenomena and coating profile, weight loss, loading, etc., showed no significant relationships. The most heavily damaged elements were, of course, among those having the larger weight losses but many elements showed marked degrees of punkiness and relatively low weight losses.

X-Ray Diffraction of Coatings

X-ray diffraction samples were taken from several of the elements after test of the NbC coatings at 5-in. intervals throughout the element length starting at Station 10. Partial results available to date show that the NbC composition fluctuates between $\text{NbC}_{0.98}$ and $\text{NbC}_{0.92}$, the C content tending to decrease towards the hot end. In all elements so far examined, a second NbC phase appears at about Station 30 with compositions of $\text{NbC}_{0.76}$ - $\text{NbC}_{0.78}$. The lattice parameter (and C content) of this second phase increases towards the hot end to that of $\text{NbC}_{0.80}$ - $\text{NbC}_{0.82}$. An estimate of the relative amounts of these two phases indicates that the amount of the second phase (lower C content) increases up to about Station 40 and then decreases again. There were traces of UC-NbC in

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a few cases. Further study is needed on nonequilibrium conditions of C diffusion rates, reaction rates of H_2 with various "NbC" compositions, and the influence of C content of the flowing gas. At present, however, it appears that the x-ray results are consistent with the observed C loss.

Tests of Fuel Elements Under Phoebus-1A Conditions

Eighteen Phoebus-1A production fuel elements were evaluated using three separate 8-min corrosion cycles. This RPT-3 x 8-2250°C test (cf. LA-3280-MS, p. 75) simulates Phoebus-1A run conditions with respect to time and fuel element exit gas temperature of 4500°R. Each element lost 9 to 14 g of C in the cyclic test, and all exhibited considerable pinhole type of corrosion in the last 25 in. If this amount of C were lost in a reactor run, the control rods would have a net change of 13-15°C.

Phoebus-1B Fuel Element Tests

Approximately 10 fuel elements have been corrosion tested under Phoebus-1B running conditions: optically measured surface temperature of 2250°C between Stations 46 and 47; mass flow of H_2 of 280 lb/h; and a nominal electrical power input of 1.3 MW (double flow and double power of Phoebus-1A). The weight loss for a 20-min test was 10-12 g whereas a comparable weight loss in a Phoebus-1A test was 7 g.

Phoebus-1C Development Fuel Element Tests

Over 300 development fuel elements have been corrosion tested using either the RPT-2250°C or RPT-2400°C test. The 2400°C test simulates the Phoebus-1C fuel element exit gas temperature of 2500°C (5000°R).

The performance of current developmental coated elements may be summarized as follows. Loaded elements lost 7-9 g in a RPT-20-2250°C test and 10-12 g in a RPT-30-2250°C test. In the 20- and 30-min

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RPT-2400°C tests, loaded elements lost 10-12 g and 15-20 g, respectively. Unloaded elements lost 4-5 g and 6-8 g in the 20- and 30-min RPT-2250°C tests. A few RPT-60-2250°C tests have been run on unloaded elements with 15-25 g loss. In all the elements tested, the bulk of the C loss is from Station 10 to Station 35.

The onset of pinholing in these elements was found to be substantially delayed to higher temperatures and longer times as compared to Phoebus-1A production elements. The RPT-30-2400°C corrosion test is now being used as a standard for coating evaluation.

Fuel Element Permeability

Fuel element leakage studies have continued using the apparatus described in the preceding progress report (LA-3280-MS). Each end of the specimen is held in a fixture which effects a tight seal by application of hydraulic pressure (about 100 psi air) against a form-fitting flexible boot which in turn contacts and seals the surface of the graphite. The leakage rate is gaged by measuring the flow of 25 psi He through the walls of the sample, and location of leaks is observed by immersing the specimen in a clear mobile fluid (alcohol). It is possible in this apparatus to measure total fuel element leak rate or the leak rate of the body, glue joint, and any 4-in. axial segment separately.

In the search for improved coating adherence, unloaded elements have been made containing different amounts of wood flour. Measurements show the latter increases the permeability of the graphite matrix almost linearly one order of magnitude for additions of between 10 and 60 mg wood flour per g dry C used in the graphite manufacture.

Fast Curing of Fuel Elements

Eighteen pieces of 5½-in. long freshly extruded fuel element material (480 mg U/cm³, bead loaded) were obtained. Starting within 1 h of extrusion, two pieces were placed in an oven at 120°C and heat treated

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for 1 h. One sample had furnace air blowing around and through the holes whereas the other sample was wrapped in Al foil. At the end of this time both samples were hard and apparently completely cured. The sample exposed to circulating air was covered with surface blisters on all outside faces, i.e., points where the surface was raised but unbroken. It had only three bead "bumps" and no "popouts" where the surface was fractured. The sample wrapped in Al foil had only one or two small blisters but had 30 bead bumps about 15 of which had popped out.

Two more elements were cured in the manner described above (1 h at 120°C) at 18 h after extrusion. Neither sample showed any blisters. The air sample had five bead bumps and no popouts. The foil sample had 30 bead bumps with ~ 12 popouts. A sample of this batch (which had been cured by the conventional method used in fuel element manufacture) had 60 bead bumps of which ~ 40 had popped out.

Starting within 2 h after extrusion, a sample was cured overnight. at 100°C (16 h) in an oven which did not have circulating air. The sample was hard and apparently cured and there were 5 to 8 bead bumps none of which popped out.

Several samples were cured in the following flowing atmospheres at 110° and 120°C : O_2 , N_2 , dry NH_3 , liquid H_2O and steam, He, He with HCl steam, and He with neutral steam. The conclusions drawn were as follows: (a) Curing at 120° is completed in 1 h. (b) Blistering of the graphite is governed by the time between extrusion of the sample and its introduction into a 120°C oven. (c) Samples 1 h old blistered badly and 15-h old samples did not blister. (d) Samples sealed during curing at 120°C , compared with those exposed to circulating air, have less blistering, more bead bumps (30-40 vs 5-8), more popouts (15-20 vs 0), and less shrinkage (4 vs 7 mils).

All gases used for flushing in curing at 120°C produced about the same number of popouts except when steam was added. The number of popouts increased with the addition of steam and when liquid H_2O was present the number of popouts increased by orders of magnitude.

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At 100°C, samples cure in 2 or 3 h. Blistering does not occur at this temperature when the sample is placed in the oven within 2 h after extrusion. Bead bumps and popouts are the same as in the 120°C cure. This suggests that curing at 100°C is better than at 120°C.

There is no change in the number of bead bumps or popouts from the cured to the graphitized state.

A fast cured piece (loaded) was coated with NbC. There was poorer NbC adherence on the flats than on the conventionally treated controls. The bores were smooth with no machining.

Thermal Properties of Fuel Elements

The average coefficient of thermal expansion (CTE) of an unloaded graphite rod made with Carbon Products high-CTE graphite flour was measured to be 5.3×10^{-6} in./in.-°C in the temperature range 25-1100°C. This CTE is about 80% higher than that of graphite made with ML flour ($\bar{\alpha} = 2.9 \times 10^{-6}$ in./in.-°C).

Standard loaded and unloaded 19-hole fuel elements were fabricated from this high-CTE graphite flour. The average CTE's, transverse and longitudinal, over the 25-2100°C temperature range of the fuel elements are listed in Table 9. The values of average CTE for similar fuel elements made with LASL graphite flour are given for comparison.

Table 9

Average Coefficient of Thermal Expansion 25-2100°C

Type of Fuel Element	$\bar{\alpha}$ (average CTE) $\times 10^{-6}$ in./in.-°C			
	Transverse (across grain)		Longitudinal (with grain)	
	High-CTE Graphite	LASL Graphite	High CTE Graphite	LASL Graphite
Unloaded	7.2	4.1	6.8	2.9
Loaded (480 mg U/cm ³)	6.1	3.5	6.1	3.1

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It is evident that the use of the high-CTE graphite flour causes a significant increase in the CTE of the loaded and unloaded fuel elements. Since the purpose of increasing the CTE is to make the graphite expansion more compatible with the expansion of the NbC coating, a value for the CTE of NbC over the same temperature range was obtained from Union Carbide Nuclear Corporation (UCNC). This (7.0×10^{-6} in./in.- $^{\circ}\text{C}$) is sufficiently close to the average CTE values for the elements of high-CTE to warrant continuing interest.

The integrated thermal expansion curves of loaded and unloaded high-CTE graphite flour fuel elements and of loaded elements made with regular LASL graphite flour are compared with the expansion data for NbC as determined by UCNC and by LASL in Fig. 32. These curves show that the high-CTE graphite flour does make the expansion of fuel elements fabricated from it more closely approximate the thermal expansion of NbC.

Fig. 32. Comparison of thermal expansion of various fuel elements with that of NbC.

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GRAPHITE FUEL ELEMENT STUDIES

Fuel Element Development

Graphite Gluing Experiments

A number of the experiments outlined in the preceding report (LA-3280-MS, p. 81) have been completed in the program to develop a strong, leak-tight glue joint for fuel elements. The purpose of the glue joint is to attach a tip of unloaded graphite onto the element to achieve a more adherent NbC end coat or to accept a different tip design for support purposes. The initial work involved 19-hole, unloaded, butt-type specimens prepared from graphitized stock for screening experiments. The specimens were glued, cured, graphitized, and evaluated by leak testing after the curing and graphitizing operation and by flexure testing after graphitization.

Basically, five different glues were studied. The P-514 cement was used in the as-received condition and after being milled in a paint mill at four different milling pressures. The higher pressures produced glues which were almost too viscous to use. The purpose of milling was to reduce the size of the filler particle in the glue from a maximum of 0.003-0.005 in. to a smaller value. Three furfuryl base resins (Varcum, 13.75 w/o stripped Varcum, and Duralon) and one phenol formaldehyde base resin (BKS-2600) were used as glues in both the filled and unfilled condition. The fillers used were -400 mesh 1008 graphite flour, Thermax, and a mixture of the two. The amount of filler added to the resin was that required to obtain a viscosity similar to that of P-514 cement.

Three different curing cycles were used for most glues. The surfaces of some of the specimens were wet with a liquid prior to the application of the glue, the purpose being to achieve better penetration of the glue vehicle into the pores of the graphite.

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The standard procedure was used in that the surfaces to be glued were wiped with acetone prior to the application of the glue. The glued joints were held together with a tie wire under 3-lb spring tension during the cure cycle. Six specimens were prepared for each condition to be evaluated.

Some very good glue joints were obtained in these screening experiments. The P-514 cement appeared to improve with the milling operation and the flexure strengths ranged between 1000 and 2000 psi. The number of joints exhibiting leaks in the leak test, however, remained relatively high. The BKS-2600 phenol formaldehyde resin gave similar results, although the number of leaking joints was somewhat less. The Duralon resin yielded improved joint strengths and a decrease in the percentage of leaking joints as compared to the P-514 cement.

The best results thus far have been obtained with Varcum resin, the same material used for the binder in the fabrication of fuel elements. Stripped Varcum, in which some of the monomer content had been removed from the resin, did not produce better joints than the as-received Varcum. With respect to the various fillers used in the glue, -400 mesh 1008 graphite flour, in which the maximum particle size is approximately 0.001 in., has been the most promising material. The preliminary data on butt-joint specimens revealed that a large percentage exhibited no leaks and the strength of the joints averaged close to 2000 psi.

These encouraging results led to a more comprehensive investigation of the Varcum-graphite flour system. The flour content in the glue was standardized at 50 w/o, providing a suitable viscosity for application of the glue. Surface treatments of the joints prior to gluing and curing cycles were investigated. Pre-wetting the surfaces with furfuryl alcohol did not change the strength of the glue joint significantly and was discarded. A glue curing cycle of 20 h to reach 150°C resulted in flexural strength around 2000 psi whereas a 20-h cycle to reach 340°C yielded joint strengths in the neighborhood of 2300 psi. A 21.5-h cure cycle to reach 450°C

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resulted in very poor joint strength, probably due to oxidation of the glue material at this high temperature. The 20-h cycle to reach 340°C was selected for the glue curing process.

Several batches of U-loaded fuel elements being prepared for other purposes had gluing experiments incorporated into their processing wherever possible. All loaded elements were leached at the joint area to remove exposed U prior to gluing. Half of the elements were glued after the 850°C baking operation and half after the 2225°C graphitizing operation in order to determine the optimum place in the overall processing operation for the gluing procedure. The results to date indicate that gluing elements in the graphitized condition with Varcum-graphite flour glue followed by a 20-h cure to 340°C plus a high temperature treatment yields the best results. Elements processed in this manner had no detectable leak at the joints, whereas gluing after the 850°C baking operation gave more leakers, although in general, the leaks were quite small. It is of interest to mention that it is extremely difficult to detect a glue joint during visual examination of a machined fuel element. The test data reported have, for the most part, referred to elements in the machined or leached condition where the worst leaks might be expected and not in the coated condition where leaks might be covered or closed. There are more glued elements now in process which will be evaluated after the NbC coating and the hot gas testing operations to complete the investigation.

Phoebus-2 Zepo Assembly

Nineteen-hole fuel elements, 580 in number, of 16 different U^{235} loadings were extruded to furnish part of a 90° sector in the Phoebus-2 Zepo assembly. Some of the pieces will be used for gamma counting standards and for gluing experiments, as described above. All of the stock was extruded to have an as-graphitized hole diam of 0.105 in. which will then be reamed to the 0.110-in. diam of the Phoebus-2 design. The fuel

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element bores will be coated with NbC after machining. The U loadings range from 67 to 411 mg U/cm³ in the coated elements.

High CTE Graphite

The fuel elements prepared for the Kiwi and Phoebus series of reactors have a graphite structure possessing a much lower coefficient of thermal expansion (CTE) than that of the NbC coat applied by the vapor deposition process. Cracks observed in the NbC coat at room temperature have been attributed, in part, to the large (factor of ~2-3) difference in CTE which exists between the graphite base and the NbC coat (see p. 72).

A special air blown petroleum coke was prepared by the Carbon Products Division of Union Carbide Corporation under an Air Force contract. A 150-lb sample of graphitized flour, ground to a particle size distribution similar to that of the standard 1008 graphite flour, was received for evaluation. This flour was compared with the M-1 and M-2 flours used in fuel element production; the pycnometer density was 2.11 g/cm³ vs 2.18 g/cm³ for the M-1 and M-2 flours. The surface area measurement, however, indicated a value of 4.7 m²/g as compared to 7.3 m²/g for M-1 and 7.2 m²/g for M-2 flour, indicating much less open porosity in the graphite particle.

Tests were made to evaluate the extrudability of the flour. The first extrusions were small scale to conserve material and a tube 0.25 in. o.d. x 0.1 in. i.d. was attempted. The normal binder content of 27-27½% Varcum, used with the M-1 and M-2 flours, resulted in an extremely wet mix and a very limber extrusion. It was possible to drop the binder content to as low as 22½% and still obtain a good extrusion without excessive pressure. On this basis, 19-hole fuel element extrusions, with and without U loading, were produced with a binder content of 22½% and at normal extrusion pressures. The elements were processed through all thermal cycles without difficulty and have been submitted for NbC coating experiments. The graphitized densities with the experimental flour products were similar, possibly slightly higher, than that obtained

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with standard materials. The significant drop in binder level may be due to the difference in available surface areas of the graphite particles; a more porous material, such as the M-1 and M-2 particles, soaks up more resin than the less porous particle of the experimental flour.

Measurements of electrical resistivity indicated that, where the extrusions made with the M-1 and M-2 flours had values around $1.35 \times 10^{-3} \Omega\text{-cm}$, the experimental flour ranged from 1.84 to $1.92 \times 10^{-3} \Omega\text{-cm}$. For extrusions in which the amount of Thermax added to the experimental flour was varied from 11 to 19%, the electrical resistivity ranged from 1.78×10^{-3} to $1.96 \times 10^{-3} \Omega\text{-cm}$. The corresponding densities increased from 1.850 to 1.891 g/cm³. Preliminary measurements indicate that the CTE for the extrusions made with the experimental flour is approximately twice that from the standard fuel elements. Additional material is being supplied by Carbon Products for overall evaluation in the Rover program.

Elements for Coating Research

The adherence of the NbC vapor deposited coating on a graphite base appears to be influenced by the presence or absence of U in the graphite matrix, the U-free graphite providing the more adherent NbC coating. Eight development lots of 16 fuel elements each were extruded to determine, if possible, the level of free U in the matrix which affects the adherence of the coatings. Four lots were loaded with bare (uncoated) UC₂ particles similar to those used in the preparation of the beads and four lots contained high-density UO₂. The loadings in each series were 250, 500, 1000, and 2000 ppm of U, respectively.

In another experiment to obtain an adherent NbC coat on loaded graphite, 16 fuel elements containing 480 mg U/cm³ plus 80 to 100 mg Mo/cm³ were prepared and extruded. Again, the presence of free U in the graphite matrix was considered responsible for the lack of NbC adherence and the Mo was added in an attempt to tie up the free U as UMoC₂ before it could

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influence the coating. A companion lot of U-free elements containing the same amount of Mo was prepared to serve as a control.

A development lot of fuel elements containing 480 mg U/cm³ and 100 g of wood flour per 1000 g of dry C was extruded. An equal number of U-free elements with a similar wood flour additive were extruded as controls for the experiments. The binder level was increased to 29.5% to insure extrudability. The purpose of this experiment is to determine whether a very high level of porosity in loaded fuel elements will increase the adherence of NbC coatings on graphite. Previous data indicate that unloaded extrusions containing 100 g of wood flour per 1000 g of dry C have a density of 1.66 g/cm³ and a permeability value as determined by the leak test of 18 liters/min, indicating a relatively high porosity.

In an attempt to obtain a more adherent NbC coat on the unloaded tip at the hot end of the element, unloaded stock was prepared containing 12.5 g and 25 g of wood flour per 1000 g of dry C. These wood flour levels will yield graphite stock having densities of approximately 1.87 and 1.83 g/cm³, respectively, and leak rates of 2 to 6 liters/min. If the gaseous coating compound penetrates the fine pores of the graphite the mechanical locking action obtained should help to anchor the NbC coat to the graphite.

Brazing of Graphite

The gluing of a tip of unloaded graphite to the hot end of each fuel element has already been described. A parallel development program has been the substitution of a metallic braze joint for the graphite cement employed in the gluing program. A butt-type joint was selected in place of the mortise and tenon used with the glue. Thin metal foils ranging from 0.002-in. to 0.005-in. thick of Nb, Mo, Zr, and Ti, all strong carbide formers, were prepared as the braze metal. Localized heating of the joint by high-frequency current brought the joint and braze metal to a high temperature in less than 1 min. The joints were under 15 psi pressure and in vacuum during the braze cycle.

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The results to date are preliminary but encouraging with respect to the strength and leak rate of the brazed joint. The 0.005-in. thick braze foil was too thick, resulting in too much squeeze-out of molten braze metal. A thickness of 0.002 in., and possibly 0.001 in., may prove satisfactory. Leak-tight joints have been obtained with Mo, Zr, and Nb brazes. Tensile tests of the butt joint indicate breaking loads ranging from 300 to 700 lb with failure in the graphite matrix at the higher loads. This program will be continued and an attempt will be made to investigate the strength of the brazed joint at temperatures approaching reactor operating conditions. It has been demonstrated that the graphite glue is strong at elevated temperature and there will be a similar requirement for carbide joints.

Hydrostatically Pressed Graphite

A number of different graphite mixes have been pressed hydrostatically in an attempt to produce an isotropic graphite possessing a controlled porosity. Most of the mixes contained graphite flour, Thermax, and wood flour, with Varcum as the binder. The wood flour addition amounted to 40 g per 1000 g of dry C and the Varcum ranged from 8 to 26 w/o of the dry ingredients.

Most of the pressings were made at 20,000 psi, processed through the standard fuel element cure and bake cycles, and graphitized at 2500°C. Many of the initial pressings were laminated--a problem related to the size of the pressing and the plastic sack in which the powder was compressed. This was overcome by making pressings 5 in. in diam and by evacuating the plastic sack prior to pressing. Pressings made in 5-in. diam sacks and containing 20% Varcum had graphitized densities of 1.35 g/cm³, too low to be considered useful, but densities above 1.60 g/cm³ have been obtained. It appears that composition of the mix may have a much greater influence on final density than the hydrostatic pressure, since pressures ranging from 9000 to 40,000 psi have shown little, if any, effect on final density.

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The graphitized blocks are machined into cylinders and radiographed for cracks and other flaws. Compression tests and metallographic inspection will be made on selected pieces as part of the preliminary evaluation of the material. Radiographic results thus far have shown many pieces to be free of any defects.

Binder Resin

The evaluation has been completed of the new lot of Varcum 8251 resin, used as the binder in graphite fuel elements. The material should be satisfactory in production use and the drum-to-drum reproducibility is good. DTA traces have been completed as well as a compilation of the viscosity data. The viscosity index values appear to be the most reliable measure of resin performance.

Protective Coating

Fuel Element Coating Development

Coating experiments have been conducted with loaded fuel elements to which Fe and Mo had been added during fabrication. The coatings exhibited the nonadherence characteristic of U-loaded graphite rather than the adherence characteristic of unloaded material. Thus, no relationship has been found between coat adherence and the effects of additives on the crystal ordering of the different components of the matrix graphite.

An experimental coating has been made on an oxide-loaded element from which all the U had been leached. The coat adherence was characteristic of unloaded graphite. The results suggest that the effect of U on matrix graphite is not the basic reason for the nonadherence of coatings on loaded graphite (at least for this type of loading). Similar experiments have been initiated on "bead-loaded" fuel elements.

Experimental coatings have been made in attempts to find methods for reducing "mid-band" corrosion, or corrosion through cracks which exist

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at temperatures below the coating temperature. Up to the present, elements which have been cooled to room temperature and then recoated at $\sim 1600^{\circ}\text{C}$, have performed slightly better in hot H_2 tests than elements which were cooled to 1600°C as a final step after the usual 1900°C coating.

Conditions have been established for the diffusion controlled deposition of TiC , at 1500°C , from gas mixtures containing TiCl_4 . TiC will be evaluated for use as a low-temperature, crack-healing material.

Recovery of U^{235} from Coating Waste

Additional development work has been conducted on the process for the recovery of U^{235} and purified NbCl_5 from the waste residues collected during the vapor coating of fuel elements. Activated charcoal (rather than graphite granules) has been found to be advantageous for use in the reaction tube in which NbOCl_3 is converted to NbCl_5 . With the activated charcoal, a reaction temperature of 350°C was adequate for the conversion, compared with 750°C reported previously.

A pilot-plant recovery run (3.5 kg of residue) has been made with some of the oldest, and most heavily hydrolyzed, residue. A recovery of $> 90\%$ of the Nb (as uncontaminated NbCl_5) was realized. Equipment is being designed which will permit the processing of 25 to 50-kg batches.

Coated Particle Research and Development

Introduction

Laboratory-scale work on the deposition of pyrolytic C (PyC) coatings on fuel particles in 1-in. diam coaters and evaluation of the resulting coated particles have been continued. The studies are being made within the general framework of coated particles of current and potential interest for use in fabrication of Rover fuel elements, the basic requirement being for PyC-coated particles with adequately high U content and good thermal stability at 2300°C and higher temperatures.

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Pyrolytic Carbon Coatings from Methane at Low Temperatures

In the study of PyC coatings deposited on UC₂ particles from CH₄ at low temperatures, the reference substrate was 104- to 147- μ particles with no effective proeutectic C barrier to migration at 2300°C. The reference coatings were nominally 25- μ thick deposited on 25-g charges of the UC₂ particles from CH₄-He mixtures at 2.6 liters (STP)/min in 1-in. diameter coaters.

The dependence of density of the coatings on average deposition rate is shown in Fig. 33, which also includes other comparable

data. The coatings deposited at intermediate rates at 1200° and 1300°C had the best resistance to migration at 2300°C, as shown in Table 10.

The general technique developed at General Atomic was used to infer the preferred orientation of these "best" coatings relative to that of coatings which showed poor resistance to migration at 2300°C. Nominal 40- μ PyC coatings were deposited on graphite discs in fluidized beds of the particles under conditions used to prepare some of the reference coatings. The PyC deposits were removed from the discs and examined by x-ray diffraction methods. The corrected intensity data were fitted to $I(\phi) = \cos^M \phi$, and the value of M was taken as a measure of preferred orientation. Results obtained on particle coatings and disc coatings in the disc runs are summarized in Table 10. The data indicate that for PyC coatings deposited from CH₄ at 1200-1400°C, both high preferred orientation and high density are required for good resistance to migration at 2300°C.

Fig. 33. Variation of PyC coating density with deposition rate.

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Table 10. Pyrocarbon Coatings Deposited from Methane at Low Temperatures

Coating Temp. (°C)	% CH ₄ in Helium	Nominal 25-μ Reference Coatings ⁽¹⁾			Nominal 40-μ Coatings in Disc Runs ⁽²⁾			
		Average Deposition Rate (μ/h)	Pyrocarbon Density (g/cm ³)	Migration in 3 h at 2300°C ⁽³⁾	Particle Coatings ⁽⁴⁾		Coatings on Graphite Discs ⁽⁴⁾	
					Average Deposition Rate (μ/h)	Pyrocarbon Density (g/cm ³)	Pyrocarbon Density (g/cm ³)	Preferred Orientation ⁽⁵⁾ (M)
1400	3.4	7.9	1.61	10	6.9	1.54	1.32	4.0
	20	37	1.62	10	33	1.64	1.55	3.0
	100	172	1.93	9	134	1.94	2.01	1.4
1300	3.4	3.7	1.61	10F	4.2	1.59	1.42	6.6
	20	24	2.02	3	20	1.95	1.97	8.1
	100	87	2.09	7R	85	2.08	2.10	3.3
1400	3.4	1.4	1.68	10	1.3	1.66	1.59	5.9
	20	5.9	2.02	3	7.6	2.08	2.08	14.1
	100	28	2.12	2D	37	2.10	2.12	9.4

- (1) 25 gram charges of 104-147 μ diameter UC₂ particles made in a carbon bed. Initial charge surface area 1100 cm²; final surface area about 2200 cm² for 25 μ coatings and about 3000 cm² for 40 μ coatings. Coated in 1"-diameter coater with 60° cone above 3/32" orifice at 2.63ℓ(STP)/min. total gas flow.
- (2) One 3/16" diam. x 0.040" and one 1/4" diam. x 0.040" graphite disc included with 25 grams of 104-147 μ UC₂ particles.
- (3) Relative migration occurring in most of the particles of the batch. "0" is no migration detectable by microradiography. "10" is penetration of the coating. Rating up to "3" indicates good limitation of migration in these coatings, i.e., "3" is migration limited to inner 1/3 of coating thickness. "D" indicates slight delamination of residual unattacked coating. "R" indicates localized radial migration. "F" indicates faint or diffuse migration.
- (4) Apparent crystallite size, L_c, was <40Å for all of the turbostratic PyC deposited on the graphite discs.
- (5) From fitting $I(\phi) = \cos^M \phi$ to corrected $I(\phi)$ vs. ϕ data.

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A 3/16-in. diam x 0.004-in. thick Pt disc was included with the graphite discs and particles in each of the disc runs. In all cases the deposit on the Pt disc contained a crystalline graphite component, with interplanar spacing (d_{002}) from 3.35 to 3.36 Å and crystallite size (L_c) from 370 to 2100 Å. Pt beads with 62-147 μ diam were prepared by the C bed method, ignited in O_2 to burn off proeutectic C, coated with PyC at 1300°C, and examined by x-ray diffraction. The PyC coating on the Pt beads did not contain a crystalline graphite component.

Pyrolytic Carbon Coatings from Acetylene at Low Temperatures

A number of PyC coatings were deposited on the reference UC_2 substrate from C_2H_2 -He mixtures at 1400°C and 1200°C. The charge size, equipment, and total gas flow were the same as for deposition of coatings from CH_4 -He mixtures.

The metallographic appearance of the coatings deposited from C_2H_2 at 1400°C varied from definite columnar structures for the two lowest rates, 7 and 13 μ/h, to a dark, featureless structure at the highest rate, 94 μ/h. The coating density, however, showed little variation, 1.59 to 1.78 g/cm³, over this wide range of average deposition rates. The resistance of the coatings to migration at 2300°C was consistently poor.

The coatings deposited from C_2H_2 at 1200°C showed progressions of metallographic appearance and resistance to migration at 2300°C which were strikingly similar to those for coatings deposited from CH_4 at 1300°C. The migration resistance passed through a maximum and was good at intermediate average deposition rates of 4 to 8 μ/h. Density varied from 1.55 g/cm³ for a 1.6 μ/h coating to 2.0 g/cm³ for 8-50 μ/h coatings to 1.93 g/cm³ for a 135 μ/h coating. One coating with a density of 1.68 g/cm³ and good resistance to migration at 2300°C is being studied further.

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Pyrolytic Carbon Coatings from Methane at 2000°C

A 1-in. diam coater with a well-defined high-temperature coating zone was built and operated at $2000 \pm 50^\circ\text{C}$. This coater has a hot orifice, rather than a cooled injector as in most experimental high-temperature coaters, to permit deposition of high-temperature PyC coatings on fuel particles under defined conditions.

General Atomic UC_2 particles with a complete proeutectic C shell could be successfully coated at 2000°C in this coater by dropping them directly into the coating zone, provided coating deposition was started immediately and was rapid enough to build up a deposit quickly to protect the proeutectic C shell from mechanical damage. Coating at rates below about $10 \mu/\text{h}$ required precoating at 2000°C with a high-rate coating. Heating these particles at 2300°C for 3 h produced no apparent additional damage but heating at 2500°C for 30 min essentially destroyed them. The results are given in Table 11.

Table 11. PyC Coatings Deposited from CH_4 at 2000°C on GA UC_2 Particles

Batch No. GA-210-ECR	Coating			% U Exposed in 30 min at 2500°C^*	
	Average Thickness (μ)	Average Rate (μ/h)	Density (g/cm^3)	Ind.	Av.
H-5	23	23	2.16	95.6 89.5	93
H-1	21	11	2.16	86.4 91.9	89
H-6	26	6**	2.16	94.8 94.6	95

* Determined by leaching and U analysis; duplicate samples heated separately.

** Outer.

The particles did not survive as well at 2500°C as did particles with nominal 25μ low-temperature coatings on the same GA cores

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(LA-3212-MS). This was probably due to damage to the proeutectic C shells which reduced the number of particles capable of containing the molten carbide.

Coatings were also deposited from CH_4 at 2000°C on the reference UC_2 particles without an effective proeutectic C barrier to migration after they had been precoated at low temperature (required to prevent agglomeration), to compare the migration resistance at 2300°C of the 2000°C coatings with that of the best low-temperature coatings. Results of tests on these particles are recorded in Table 12. It was concluded that the 2000°C coatings were comparable in resistance to migration at 2300°C to the best of the 1300° and 1200°C coatings from CH_4 .

Table 12

PyC Coatings Deposited from CH_4 at 2000°C on Precoated UC_2 Particles

Batch No.	Av Coating Rate (μ/h)	Av Thickness of Intact Coating (μ)	
		As-Coated	3 h at 2300°C
L-5,C-67,H-2 ⁽¹⁾	23	23	23
L-5,C-67,H-4 ⁽¹⁾	9	18	18
L-5,C-67,H-3 ⁽¹⁾	5	18	18
L-5,C-86-87,H-1 ⁽²⁾	24	24	22
L-5,C-86-87,H-4 ⁽²⁾	10	20	18

(1) 10 μ precoat of 1400°C PyC with density of 1.60 g/cm^3

(2) 6 μ precoat of 1300°C PyC with density $\sim 2.06 \text{ g/cm}^3$

GRAPHITE RESEARCH AND DEVELOPMENT

Experimental Graphite Manufacture

A graphite fabrication experiment has been initiated, the purpose of which is to examine the effect of the curing and baking environments on the properties of a molded furfuryl alcohol bonded graphite. Several

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procedures for curing and baking in various environments -- including air, vacuum, and inert atmospheres -- are planned.

The formulation used throughout the experiments described below was as follows: 85 parts graphite flour, 15 parts Thermax, and 24 parts furfuryl alcohol. Moldings of this material were prepared in a 1 $\frac{1}{8}$ -in. diam vacuum molding die at three compacting pressures: 8000, 16,000, and 24,000 psi. Green densities varied from 1.70 to 1.73 g/cm³, depending on compacting pressure.

Curing and baking runs were made under two different environmental conditions. In the first run an air-curing and vacuum-baking procedure was used. The second run was similar to the first in all respects except that curing and baking were performed in a He atmosphere. The compacts were cured over a heating cycle of approximately 50 h to a temperature of 180°C. Baking was done over a 40-h cycle to a temperature of 875°C.

Air curing and vacuum baking produced specimens with bulk densities of ~1.68 g/cm³, whereas the He cured and baked materials had densities of ~1.61 g/cm³. The air-cured and vacuum-baked specimens were subsequently heated in He to graphitizing temperatures of about 2600°C, and the density increased to ~1.72 g/cm³. This high-temperature treatment has not yet been given to the He-cured and baked material.

X-Ray Studies on Graphite

A mathematical expression has been derived for correction of raw intensity data from any pole-figure device using reflection geometry for **absorption** by any thin, low-absorbing material whose preferred orientation is being studied. This and an experimentally determined correction for nonlinear response of the counting system have been applied to Schulz-camera intensity data on a series of pyrocarbons. Intensities of (002) reflections corrected in this manner and plotted as a function of angle to the sample normal were well fitted both by an exponential and by a

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a cosine-power function, except at low intensities. It is believed that the values of the exponents which appear in these functions will be useful in describing orientations and degrees of anisotropy of pyrolytic deposits in general.

Tensile Behavior of Graphite

Additional relaxation tests have been made on H4IM graphite in tension at 2400° and 2500°C in a He atmosphere. The purpose was to demonstrate that, for previously unstrained specimens and for those previously strained only at relatively low unit loads, relaxation rate is independent of initial load. The experiments have been generally successful, although most of the relaxation curves have been stepped and somewhat irregular. The reason is that the loading train used is long, and a very small voltage fluctuation in the power supply changes its temperature and virtual length enough to disturb the load reading seriously. However, enough good data have been collected during electrically quiet periods to demonstrate quite convincingly that, at both 2400° and 2500°C , fractional load remaining on the specimen is a linear function of the logarithm of time. Additional tests of this type will be made to improve the value of the proportionality constant typical of each temperature, and possibly to obtain a few more relaxation curves smooth enough for publication.

By the use of collars which serve as enlarged fiducial shoulders on tensile bars, and by minor modifications to the optical path through which the specimen is viewed, the sensitivity anomaly previously found in the optical strain-measuring system has apparently been eliminated. Reliable room-temperature values of Young's modulus can now be obtained with this equipment, and its use will be extended to elevated temperatures.

Improvement in Structure of Pyrolytic Graphite

Application of torque around the c axis, sufficient to produce about a 10° twist in a specimen of pyrolytic graphite subjected

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simultaneously to high temperature and compression parallel to the c axis, has reduced the proportion of disoriented layers from $\sim 90\%$ to $\sim 56\%$.

Small Particle Theory

In the preparation for analysis of particle-size spectra determined for the solid raw materials used in graphite manufacture, transformations have been developed which permit calculation of mean particle diameter, variance, and skewness directly from Micromerograph, Coulter Counter, and centrifuge data, without use of the log-normal model. A simple method of analyzing Coulter-Counter data, apparently unrecognized by the manufacturer of this instrument, has also been developed.

NONDESTRUCTIVE TESTING

Introduction

Fuel element development has accounted for a good portion of the activities. Instrumentation has also continued to receive attention, both regarding improvement of existing methods of nondestructive testing as well as development of new methods. Probably the most challenging problem at present is that of NbC coating quality evaluation.

Fuel Element Load Measurements

A detailed study of four coated Phoebus-1A fuel elements representing 191, 371, and 480 mg U/cm³ loadings indicates that the γ counting measurements are erroneously high, by 1-3%, when compared with the results of chemical analysis. Experiments have been conducted by varying several of the γ counting parameters such as the base line location and window width. These changes did not affect the load calibration curve and could not cause the discrepancies noted above. Further investigation is in progress.

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Phoebus-2 Zepo fuel elements are being received for load measurements. The necessary standards for a portion of the load curve have been processed and the curve has been constructed.

A program for the LGP-21 computer has been written for the processing of the data from γ counting standards and for the formation of the respective load curves.

Coat Thickness Measurements

A detailed comparison of Phoebus-1A fuel element coating thicknesses as measured by β -backscatter and optical methods is in progress. Routine inspection of the developmental fuel element coat thicknesses is continuing. A series of calibration curves has been obtained for wood flour elements; they are the same as those for unloaded graphite. Several modifications to the existing β -backscatter probes have been completed. These changes increase the versatility of the equipment.

Fuel Element Research and Development

For the fuel element research and development program, the inspections performed and the methods of data retrieval and presentation have been selected to accomplish two primary objectives: (a) to provide data indicating the quality of the coat in the various test batches and (b) to determine correlation between inspections and evaluate the worth of each.

Whole elements are being inspected before and after the hot-gas test. The inspections performed on these elements are: internal eddy current, potential drop examination (PDE), β -backscatter coat thickness measurements, and radiography of the glued tips. Selected elements receive density-times-thickness (DXT) measurements and bore replication. The selection has been arbitrary except that one element has been chosen to represent each type of coating and element combination.

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Longitudinally sectioned elements are also being inspected. These are representative samples of the coating batches before the hot-gas test. They are subjected to β -backscatter scan, eddy-current scan, and replication. Unloaded, longitudinally sectioned elements are radiographed. In addition, selected samples are cut from these elements and are inspected by metallography. They will be inspected by electron microscopy when a proper sample preparation technique has been developed.

A new method of reporting the data on hot-gas-tested elements has been established. The DXT and PDE data are reported on graphs, examples of which are shown in Fig. 34. Beta-backscatter (BBS) data, when available, are shown on these graphs for convenience. DXT difference (after hot-gas test minus before hot-gas test) is proportional to mass loss. Potential drop difference (after hot-gas test minus before hot-gas test) is directly proportional to electrical conductivity change.

Fig. 34. Examples of PDE and DXT and some BBS data.

The bottom illustration in Fig. 34 shows a deviation which is as yet unexplained. This indicates one aspect of the PDE which is different from the DXT examination. There are many parameters which may affect the PDE as opposed to only two in the DXT method. Conductivity changes may be caused by cross-sectional changes in the element, indicating mass loss. However, they may also be caused by material changes, e.g., U migration, degree of graphitization, contact resistivity, and others. These factors are not separable and may not be apparent in visual inspection.

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Internal eddy current traces are not being reported due to the complexity of evaluation; however, they are being used to aid evaluation of bore replicas. The eddy current traces agree with the bore replicas in indicating that the eddy current inspection is affected largely by the condition of the coat, and only to a small degree by that of the material behind the coat.

Metallographic inspection has been completed on selected portions of the longitudinally sectioned elements and grain size measurements have been made. Typical micrographs are shown in the upper part of Fig. 35 and an interface is seen in the lower part.

Instrumentation Development

Gamma-Count Instrumentation

Routine quality control of the enriched uranium used in the beaded fuel has indicated varying U^{234} content, ranging from about 0.2% to 1.0%. About 30% of the total γ radiation emitted from the beaded fuel is from the U^{234} . The presence of this radiation can lead to serious errors in the determination of the U content of fuel elements. In the present system the U^{234} contribution has been minimized but not excluded, and so experiments are being made to evaluate this contribution to the measured radiation.

Fig. 35. Typical micrographs (above) and interface (below) of coated fuel element.

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It has been determined that breakup (or migration) of the beaded fuel will apparently increase the count rate as measured by the γ scanners. An experiment designed to evaluate the magnitude of this effect is in progress. It appears that the temperature threshold for the effect is in the vicinity of 2300°C for Phoebus-1A elements.

A γ scanner for the inspection of unpackaged fuel elements is under construction in order to eliminate the need for the special packaging procedures presently employed.

Electron Microscope Examination of Fuel Components

Major difficulties have been encountered in the preparation of graphite samples for electron microscopic examination. The sample preparation techniques, as developed at Battelle-Northwest (Hanford), have not been successfully reproduced at LASL. The Battelle samples have produced electron micrographs of very good relief, showing structure of both the graphite flakes and the binder. The attempts to reproduce the Battelle techniques have involved investigations of the following variables: (a) mounting alloy material, (b) rotary and vibratory polishing materials, (c) oxidation either before or after etch, (d) voltage and gas flow rates, (e) etch-gas material (Ar, Kr, Xe), (f) shaped accelerating fields, and (g) radio-frequency and particulate ionization systems. These experiments have resulted in a much improved sample; however, results are not of the quality required for satisfactory electron micrograph interpretation. These experiments are being continued with the goal of developing a sample preparation technique comparable to that of Battelle.

Neutron Transmission Measurements

The polyethylene moderator, the polyethylene shielding, and boral shields have been completed for the neutron transmission system. The electronic counting system has been installed. The checkouts to date have shown that count rates are not well within statistics and that there is possibly some system instability.

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The neutron transmission system will be utilized for the following experiments:

1. The detection of B^{10} distribution in B-loaded poison rods for Rover fuel element containers.
2. The detection of B^{10} distribution in boral shielding material for the Phoebus-2 reactor.

Bore Replication

Pressure Forming Processes

The possibility of U^{235} contamination arising from flaking, post-test elements, and sectioned experimental elements has resulted in handling procedure modifications, e.g., lined trays in which the elements are prepared for replication and a furnace-end baffle to prevent the spread of contamination in the event of replica blowout during the heat forming cycle.

Post-gas-tested elements have been replicated at reduced pressure to prevent replica blowout. This decreases the amount of microscopic bore coating detail obtained, producing a replica which shows only gross defects. In an attempt to replicate post-gas-tested elements at higher pressures, polyvinyl chloride tubing with a wall thickness of 0.027 in., from the Borden Chemical Co., will be utilized instead of the present 0.016-in. thick material.

Construction of a thermocouple scanner to be used for the determination of furnace temperature profiles has been completed. It will be utilized for the determination of optimum heater winding profiles for pressure and vacuum replica furnaces.

Vacuum Forming Processes

A vacuum forming replica experiment was conducted in an environmental test chamber; nine bores of an unloaded, NbC-coated fuel

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element were loaded with size 16 polyvinyl chloride tubing. Nine of the remaining bores were loaded with size 18 tubing. The replica blanks were unpressurized and were given an initial stretch which ranged from 0 to 3 in. in a total length of 30 in. The main results of the experiment were: (a) the integrity of the size 16 tubing was maintained during the experiment; however, the replica blanks of size 18 tubing collapsed, and (b) the replicas of size 16 tubing were of poor quality, exhibiting no great detail. A conclusion from (b) is that the replica blanks should be prepressurized before being subjected to the thermal-expansion pressure; preliminary calculations show this to be true.

Ultrasonic Study of NbC Coating Adherence

The Fokker Bond Tester (FBT) has been received and an evaluation program started. On the limited number of support blocks inspected to date the correlation between the FBT results, thermal conductivity test results, and destructive test data, has been encouraging.

The principle of operation of the FBT is that of sensing the resonance point shift and the amount of damping of a ceramic ultrasonic transducer. The unit has a sweep frequency oscillator which is applied to the transducer. At some frequency, determined by the bond, the transducer has a resonant point and the power supplied decreases. When any material is coupled to the transducer, the resonant point will be shifted and the power supplied will be changed. This shift is proportional to the amount of coupling between the transducer and the material. In operation, the unit is coupled to a sample with a thickness equal to that of the coating under examination. The unit is then calibrated in this zero-bond condition so that it will indicate 100% unbond. The samples are then compared with this condition. Any adherence to the base material will cause a shift in the resonant point and a change in the power supplied. These changes are indicated on a cathode-ray tube and a meter on the unit.

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Thermal Conductivity Study of NbC Coating Adherence

Work to improve the present self-heating thermistor probe for thermal conductivity measurement is being continued. Matched thermistor pairs have been ordered; these should reduce the drift that has been observed in the present arrangement.

A somewhat different approach to the measurement of relative thermal conductivities of the NbC coatings is being studied. The specimen will be scan-heated in order to introduce transient heat point-by-point in a known sequence over the surface. An infrared-sensitive radiometer will then scan the specimen, in a similar sequence, at a constant time after heating. The surface temperature should be somewhat higher in the region of a poorly-bonded coating and lower in the region of a well-bonded coating. Since the radiation flux emitted from the specimen varies with the surface temperature, it should be possible to detect areas of poor adherence.

Coordination with other Installations

NDT Conference at LASL

The First Conference on Nondestructive Testing of NERVA and Rover Fuel Elements was held in Los Alamos on April 7, 8, and 9, 1965. It was attended by representatives of SNPO-Cleveland and Washington, Aerojet-General, Westinghouse, Oak Ridge, and LASL. Nondestructive testing subjects presented during this meeting included: γ -counting, β -backscatter, eddy current, DXT, PDE, bore replication, electron microscopy, and R&D planning. All participating organizations are cooperating in the issuance of transactions of the meeting.

Coordination with Westinghouse

A β -backscatter bore probe, designed by Oak Ridge, Y-12 Plant and constructed at WAFF, has been evaluated at LASL. Although the basic NbC thickness calibration is very similar to that of the LASL probes,

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differences in the geometry of the WAFF probe appear to produce measurable differences in the performance.

A group of 14 coated fuel elements, fabricated by WAFF, has been examined by γ -counting and β -backscatter methods at WAFF, LASL, and Y-12. The agreement between the β -backscatter results is good but there is poor agreement between the γ -count results. The latter discrepancies are attributable to the fact that no load standards have been exchanged for this type element. (Beta-backscatter standards have been circulated previously among the three organizations.)

Coordination with Oak Ridge

The γ -count discrepancy on load measurements of coated elements continues to exist. Chemical analysis indicates the Y-12 load values may be slightly greater than the actual value (about 1%) for Phoebus-1A elements. The LASL γ -count load values may be as much as 3% above the correct (analytical) value. A comparison of the γ energy sensitivity between the Y-12 and LASL counters reveals a definite difference. This difference in detector response, coupled with certain U migration conditions in the fuel element, could cause the observed discrepancy in load measurements. Experiments are in progress to attempt to determine the exact causes of this discrepancy. A standards-exchange program for Phoebus-1B elements is presently being generated.

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V. MATERIALS DEVELOPMENT

CERMET DEVELOPMENT PROGRAM

W-Mo-Re Alloy Development

Alloys Including ThO₂

The determination of an acceptable ductility value, based on the present comparative test procedures, must be resolved for design and other purposes. Because of the recent advances in the cermet technology it now becomes necessary to compare the true ductility as measured by standard tensile tests with that of the Brinell indentation ductility test. A program has been initiated to investigate the mechanical properties of wrought sheet of the alloys under study. Since this material would not be representative of a powder cermet, the program has been expanded to include tensile specimens prepared in the same manner as ductility disc type specimens, i.e., powder metallurgy techniques. The W-Mo alloys to be investigated will include 80/20, 70/30, 60/40, and 50/50 a/o ratios plus 0, 7.0, 12.0, and 17.0 a/o Re.

Earlier work showed that ThO₂ additions to W-Mo base alloys containing 12 a/o and 17 a/o Re improved the ductility of the alloy but produced little or no improvement when UO₂ was added to form the cermet. Recent work was aimed at determining the effect of ThO₂ content on sintered density, grain size, and ductility of this matrix alloy after four successively higher thermal treatments. The base alloy used contained

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70 a/o W - 20 a/o Mo + 12 a/o Re, to which was added 0, 0.5, 1.5, and 3 w/o ThO₂.

The initial sintering treatment of 1700°C for 3 h in H₂ produced a very fine grain size and relatively high ductility, although each incremental increase in ThO₂ decreased the sintered density about 1%. At the higher sintering temperature the density was virtually constant for all compositions. After the 2350°C for 1.5 h in H₂ treatment, the grain size decreased as the ThO₂ content increased.

The d⁴ ductility values, for samples sintered at 1700°C for 3 h in H₂, ranged from a high of 285 to a low of 209. Specimens given an additional treatment at 2340°C for 1.5 h in H₂ showed the usual decrease in ductility. However, for this series, the ductility increased proportionately with decreasing grain size and increasing ThO₂ content.

A third series of specimens sintered at 1700°C for 3 h in H₂ plus 2340°C for 0.5 h in vacuum were wrapped in Mo foil and given an additional treatment at 2550°C for 10 min in H₂. The sample containing no ThO₂ increased in ductility, whereas an additional loss in ductility was noted for the ThO₂-bearing samples. These results were contrary to those obtained previously. A final series was sintered at 1700°C for 3 h in H₂ plus 2340°C for 0.5 h in vacuum, after which they were coated with W from WF₆ prior to a final thermal treatment of 2700°C for 10 min in H₂. The ductility values were slightly higher than those following the 2550°C treatment. This is perhaps due to somewhat thicker specimens and/or better protection against H₂ during cooling and/or improved homogeneity.

Inconsistencies in the ductility values, which may have originated in minor changes in the thermal treatment, indicated the necessity for repeating a portion of the program with stricter control over all variables. The base alloy was the same as before with 0, 1.5, and 3.0 w/o ThO₂ added. Ductility test discs for the three alloys were pressed at 60 tsi. The pressed discs were sintered at 2350°C for 1.5 h in H₂ and furnace cooled

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in H_2 . With one exception, the ductility values showed a consistent upward trend with increasing ThO_2 content for each temperature, regardless of cooling atmosphere. Based upon the data obtained on these two independent experiments as well as earlier preliminary data, it may be concluded that ThO_2 additions to W-Mo-Re alloys do indeed restrict grain growth at the higher processing temperatures resulting in improved ductilities as compared with the alloys containing no ThO_2 .

Alloys Without ThO_2

The factors that might contribute to inferior density (involving the Kirkendall effect) in sintered alloys have been discussed many times. A series of alloys was prepared in an attempt to learn more about this phenomenon, and to see if the Sylvania W-Mo alloy powders would give better sintering results. The alloys investigated consisted of blends of the Sylvania 70/30 and 80/20 w/o W-Mo alloy powders and powder blends of 70/30 and 80/20 w/o W-Mo proportions to which was added 0, 10 w/o (8.5 a/o) and 20 w/o (17 a/o) Re.

After obtaining the green density on over 200 discs, representative discs of each composition were sintered as follows: (a) 17 h in H_2 at $1400^\circ C$; (b) 19 h in H_2 at $1550^\circ C$; (c) 3 h in H_2 at $1700^\circ C$; (d) 1.5 h in H_2 at $2350^\circ C$. In addition to these separate cycles, discs were also sintered in the following cycle combinations: (a) and (c); (b) and (c); (a) and (d); (b) and (d); (c) and (d); (a), (c), and (d); (b), (c), and (d). Densities were obtained on all the discs for the 11 different combinations of sintering cycles. The significant data are presented in Fig. 36.

It is evident that the alloy densities have a small spread in the green condition and when sintered at $2350^\circ C$, but have a wide spread for the sintering cycles with $1700^\circ C$ as maximum. The lower density values are associated with the presence of Re, increased Mo content, coarser Sylvania alloy powder or coarser Mo powder in the blends. These observations not only confirm earlier work, but show that low-temperature sintering accentuates the effects.

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Fig. 36. Density vs sintering temperature of W-Mo mixtures without Re (dashed lines) and with 17 a/o Re added (solid lines).

If pretreated at 1400°C, the 2350°C densities (not shown in the figure) were usually somewhat lower. The best results obtained at the 2350°C temperature (96-97% dense) are somewhat better than those measured in earlier work for W-Mo-Re alloys at this temperature. The improvement could be due to somewhat longer time at temperature, H₂ atmosphere instead of vacuum, and somewhat finer particle size.

The Brinell indentation ductility test was run only on selective samples for this series. The following conclusions were reached after careful study of the density and ductility data for the various thermal treatments:

(1) In general, the alloy powder showed no significant improvement in the sintered densities over the blended powder. In fact, the blended powder, at nearly comparable densities, had slightly higher ductility in nearly all cases than the alloy powder.

(2) The introduction of a low-temperature sintering treatment prior to the high-temperature treatment resulted in significantly lower densities for the Re-bearing material.

(3) The highest densities were obtained on specimens heated directly to the highest sintering temperature. The 70/30 w/o W-Mo alloys with and without Re showed an increase in sintered density with decreasing average particle size of the powders.

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(4) In all but one case, the 70/30 w/o W-Mo samples, with and without Re, had higher ductilities than the comparable sample in the 80/20 w/o W-Mo series. Similarly, the density of the 70/30 w/o W-Mo samples with and without Re were lower than for comparable samples of the 80/20 W-Mo series.

High-Temperature Testing of UO₂ Cermets

The cermets studied were based on a W-Mo alloy matrix of 70/30 a/o ratio containing either 12 or 17 a/o Re; the UO₂ content was constant at 40 v/o. A standard proportion of 4 v/o Y₂O₃ was added to most of the cermets as a possible stabilizer of the UO₂ at 2600°C. The Y₂O₃ was dispersed in the metal matrix or added to the UO₂. Specimens containing no Y₂O₃ were included for comparison purposes. Sintering and ductility test results for these alloys were presented in the previous quarterly report (LA-3280-MS, p. 127). All the cermets were pressed into disc form, and key compositions were also pressed into the form of thin rectangles. The rectangular specimens were tested first because they would correlate better with past work. Measurement of the rectangles, W-coated 20 w/o, showed a coating thickness of approximately 4 mils.

The fuel loss results are shown graphically in Fig. 37. The specimens without Y₂O₃ had a test life comparable to previous results with the 30-min cycle and 20 w/o W-coat. The specimens containing 4 v/o Y₂O₃ cycled every 30 min survived for time periods 3-5 times longer than those containing no Y₂O₃. The 5-min cycle reduced specimen life about 50% compared with the 30-min cycle but the relative superiority of the Y₂O₃-bearing samples was maintained. The four compositions containing Y₂O₃ followed the same sequence and pattern in both the 30-min and 5-min cycles.

From these preliminary data, it appears that the 12 a/o Re samples are more favorable to low fuel loss than the 17 a/o Re bearing samples. Moreover, for the 12 a/o Re samples, the Y₂O₃ was more effective in these specimens when added to the fuel instead of dispersed through the metal matrix.

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Fig. 37. Cumulative fuel loss upon heating at 2600°C of various 70/30 a/o W-Mo cermets containing 40 v/o UO_2 ; dashed lines are for 4 v/o Y_2O_3 addition.

Previous work on specimens treated at 2600°C under cyclic testing conditions revealed no free U on metallographic examination, although it was suspected to be present from reports by other investigators. It was believed that the free U in these specimens was very diffuse or that the cermets were not in a condition suitable for U detection. However, specimens from this series revealed a yellowish metallic phase (apparently U) within or adjoining the UO_2 agglomerates of those cermets not containing Y_2O_3 , especially the specimens with UO_2 loss as high as 2%. The cermets containing Y_2O_3 did not show this yellowish metallic phase even in the three specimens with fuel losses near 1.5%.

The W coatings were quite uniform for the 14 specimens studied, yet there were considerably more open grain boundaries associated with the coatings of the Y_2O_3 -free specimens, even though these had far less exposure time at 2600°C.

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Subject to confirmation by additional testing, the combination of fuel-loss data and metallography tends to support previous speculation regarding the deterioration of W coats. It had been suspected that the products of UO_2 instability (free U and substoichiometric UO_2) diffused more rapidly through the core and into the coat-grain boundaries than did stoichiometric UO_2 .

The presence of foreign matter in the W coat-grain boundaries would weaken the bond, and so reduce resistance to thermal and mechanical stresses from cycling, with added strains and embrittlement from the hydriding and expansion of free U. With repeated cycling, involving alternate hydriding and dehydriding as well as re-solution and precipitation of the U, considerable activity can be envisaged within the invaded grain boundaries. It seems plausible that W grains would eventually be separated by such actions in the form of "wedging" or "ratchetting" mechanisms. Probably the free U plays the greater role in such deterioration.

It is apparent from the test results previously discussed that Y_2O_3 additions to the cermet do, indeed, stabilize the UO_2 , thereby minimizing the formation of free U. This, in turn, is reflected in the improvement in the results of the life cycle test of the Y_2O_3 -bearing specimens.

A summary of test observations appears to support the above reasoning:

(1) W-coated cermets containing no Y_2O_3 perform well for cycle-free heating at 2600°C , but deteriorate rapidly with increasing cycle frequency. Such specimens also appear to be sensitive to hydriding at low temperatures when cooled in a H_2 atmosphere.

(2) W-coated cermets containing Y_2O_3 are not only superior on all observed types of treatments, but also resist deterioration by cycling and seem relatively insensitive to hydriding.

It appeared that the cermets containing Y_2O_3 had demonstrated adequate cycling capability (15 to 30 cycles) as far as number of

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cycles was concerned. Additional tests were run to determine the effect of running time without cycling. Tests lasting at least 4 h without cycling were compared with the same two materials cycled every 30 min. For the cermet containing no Y_2O_3 , the elimination of cycling more than doubled the test life with 0.54% loss at 5 h. For the Y_2O_3 -bearing cermet the test life increased from 5 to 13.5 h with 1.46% fuel loss.

Vapor Phase Coating of Tungsten

Cermet specimens were coated with tungsten from H_2-WF_6 mixtures at various pressures and flow rates and at temperatures ranging from 300° to $1000^\circ C$. It was predicted previously that if the deposition reaction is controlled by gas diffusion at the surface, then the deposition rate should decrease at high temperatures and pressures after reaching a maximum. Indeed, this was the case for the deposition pressures and flow rates investigated. The data for all specimens coated under the conditions indicated are given in Fig. 38. The highest coating rate was obtained at the lowest pressure, indicating that the deposition rate is controlled by gas diffusion at the sample surface for the higher temperatures.

Slightly below $500^\circ C$ at the point of inflection on the curve for 1 atm (H_2-WF_6 of 300/30), the gas diffusion rate becomes controlling and the deposition rate increased more slowly up to $800^\circ C$. Above $800^\circ C$ the turbulence effect due to increasing temperature caused a drop in coating rate even though the reaction kinetics at this temperature must be such that the reaction $WF_6 + 3H_2 \rightarrow W + 6HF$ is essentially instantaneous. The point of inflection for the curve of deposition at 5-in. Hg is probably at $500^\circ C$ or slightly above; and the point where gas diffusion becomes controlling for deposition at 10 torr is probably near $600^\circ C$. The curves for W deposition at lower gas flow all show a leveling above $600^\circ C$, due to depletion of the reactants in the gas stream.

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Earlier work demonstrated that crack-free tubes could be extruded from coarse W powders using methocel-H₂O with and without glycerin as a lubricant. However, the green density of the tube extrusion was very low and, in some cases, the extruded shape had insufficient strength in handling for subsequent processing. It was decided, therefore, to investigate the combination of coarse and fine W powder mixtures of various proportions to increase the green density and also to dry the mixes to varying extents to raise the pressure needed to increase density.

Attempts were made to extrude tubes from various mixtures of 6.5 μ , 2.5 μ , 1.5 μ , and 0.7 μ W powders with methocel-H₂O as a lubricant. In all cases, cracking occurred on the external surface on standing in air shortly after extrusion. When the work was repeated with glycerin added to the methocel-H₂O mix to suppress the drying rate of the as-extruded tubes, an immediate improvement was realized and for the various combinations investigated, crack-free tubes were produced. The density remained low in the as-extruded as well as in the sintered conditions. The highest density in the sintered conditions was for a batch of W powder of three different particle sizes, 0.7 μ , 1.5 μ , 2.5 μ , blended in the ratio 50/25/25, respectively, using a methocel-H₂O glycerin lubricant. This batch was split into two equal portions and dried to different degrees of dryness.

Fig. 38. Rate of deposition of tungsten vs temperature on cermet pins.

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The wetter mix extruded at 4 tons gage pressure to a density of less than 40% of theoretical, whereas the dry mix extruded at 10 tons to an as-extruded density of 50% of theoretical. The sintered densities, following 1700°C for 3 plus 3 h in H₂ plus 2200°C for 1 h in vacuum, were 85 and 89% of theoretical, respectively. These are the highest densities obtained to date on powder extruded tubes.

Metallographic results on these sintered tubes showed considerable carbide phase attributed to the lubricants, and so other lubricants were investigated. Tungsten disulfide, WS₂, appears useful as a lubricant in the extrusion of W-UO₂ tubes, provided the S can be removed to leave only W. Several runs were made in vacuum or H₂ up to 1100°C to determine decomposition or reduction temperatures for pure WS₂ powder. Essentially no decomposition occurred at 10⁻⁴ torr up to 1100°C. However, in 1 atm of flowing H₂, reduction of WS₂ by the formation of H₂S reached a detectable rate at 650°C, and the reaction rate increased with temperature. WS₂ held at 800°C in flowing H₂ for 40 h lost 50 w/o of the S present; whereas, WS₂ in flowing H₂ at 1000°C was reduced to W (gravimetric measurements) after 6 h. Extrusions of W plus 1.6 w/o WS₂ were heated at 1000°C for 7.5 h and then chemically analyzed for S; 90 ppm were found, compared with 0.28% S in the as-extruded piece.

Magnetic Compaction of Tubes

Four tubes of W-20 w/o Mo were magnetically compacted from new powders and with an addition of cetyl alcohol as a binder. The W powder was a 1:1 mixture of 2.5 μ and 5.2 μ particle sizes and the Mo powder had an average particle size of 3.4 μ. The cetyl alcohol ranged from 0.3 to 0.5 w/o. The loose powder was loaded into the long tube using a combination of vibration and air evacuation. The tubes were compacted at the 17.5 kV power setting using an annealed Cu pusher.

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The compacted tubes were processed through nondestructive inspection in the green state and after the sintering operation. At each inspection there was no evidence of cracks or internal defects. Wafers from the green tube were evaluated for radial density variations and the results indicated little or no variation within the error in measurement. The tubes were presintered at 1100°C for 2 h in H₂, followed by a 1700°C sinter in H₂. The pertinent data are reported in Table 13. Metallographic

Table 13. Density of W-20 w/o Mo Magnetically Compacted Tubes

Sample No.	w/o Cetyl Alcohol	Green Density (% theor.)	Sintered		
			Open Pores (%)	Closed Pores (%)	Density (% theor.)
MS-054A	0.3	72.95	13.78	3.07	83.15
MS-054B	0.3	72.95	13.53	2.77	83.70
MS-055A	0.5	72.65	12.92	1.90	85.18
MS-055B	0.5	72.65	15.35	1.87	82.78

evaluation indicated that the sintered material was quite sound and circumferential cracks were completely eliminated in the samples containing 0.5 w/o cetyl alcohol.

Another experiment with W powder was made to evaluate graphite as a mandrel material, but the mandrels could not be removed from the compacted tubes. The interesting effect, however, was that a section through the W tube indicated a unique columnar structure not observed in any compacted material in the past. The significance of this structure is not understood but it will be investigated further.

MISCELLANEOUS REACTOR COMPONENTS

Phoebus-1A Thermocouples

Two thermocouple assemblies, each 25 ft long, were prepared by welding and brazing techniques. The thermocouples will be inserted into

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the exit gas area to record exit gas temperatures. The assemblies were complex and a W-coated Ta tube was brazed to a connector which had been tack welded to the shielded couple. Magnetically formed standoffs also were brazed in two locations and a threaded fitting was brazed to the shield.

Phoebus-1A Wrapper

The change in a sector of the Phoebus-1A periphery, replacing the standard jagged section with a section of circular geometry, necessitated that special wrapper panels be fabricated to match the new graphite panels. A total of 43 wrapper sections were fabricated per specifications and delivered for the reactor assembly.

Phoebus-2 Control Plates

As noted in LA-3280-MS, Cu-50 v/o B₄C is being considered as a replacement for the Al-20 w/o B¹⁰ control plates used in the Kiwi and Phoebus-1 series of reactors. The use of normal B₄C avoids the expensive and critical B¹⁰ and the Cu-B₄C material has much better thermal conductivity than the Al-B¹⁰ plates. A number of 0.050-in. thick strips of Cu-B₄C material in which the B₄C loading ranged from 20 to 50 v/o were prepared by hot pressing the blended powders and rolling the canned pressing to the desired thickness. The results of evaluation of the strips by neutron absorption measurements are plotted in Fig. 38A. The data show that Cu-50 v/o B₄C strip 0.100 in. thick is essentially the equivalent of the Al-20 w/o B¹⁰ control plates used previously.

The preliminary fabrication results indicated that the commercial, angular B₄C particle would be very difficult to incorporate into Cu to the extent of 50 v/o to yield a strip which could be formed to the required contours without cracking. It is possible to roll flat strip of Cu-50 v/o B₄C using angular particles but the strength of the rolled material would be low and fabricability poor. Consequently, a spherical B₄C particle was considered of prime importance for this particular application.

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Fig. 38A. Neutron Absorption effectiveness of Cu-B₄C strips.

Several small batches of commercial B₄C were run in the high-enthalpy, low-velocity, Thermal Dynamics plasma gun. Batches of 100 g of powder were run through the gun three times, investigating different nozzle configurations. All of the runs were made in He containing 1% H₂. The degree of spheroidization and soundness of the particle after each pass through the gun was determined by microscopic examination. Approximately 1 kg of -150 mesh, rounded B₄C powder was produced for pressing and rolling studies of Cu-B₄C plates. It was concluded that particles larger than 150 mesh could not be spheroidized successfully in the plasma gun. Larger particles were believed necessary for the control plate application requiring 50 v/o B₄C.

In experiments with a drop tower, it was found that commercial, angular B₄C particles could be spheroidized and it appears that almost any size of B₄C sphere can be produced in quantity. Temperature control still is under investigation since metallographic results indicate some over-heating and second phase formation within the particles. Selected ranges of B₄C particle sizes have been blended with Cu and are being pressed into plates which will be rolled into strip for fabrication evaluation.

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A new device utilizing an Ar plasma in a high-frequency field has been built and the results of the preliminary spheroidization experiments appear very promising.

PHASE AND THERMODYNAMIC STUDIES

Vaporization Studies of the U-C System

The pressure of $U(g)$ and $UC_2(g)$ and the resulting partial heats of vaporization of $U(g)$ and $C(g)$ have been measured as a function of composition from $UC + U_{(1)}$ to $UC_2 + C$. An abrupt pressure change with composition was found near $UC_{1.0}$ and a much smaller change within the UC_2 region. The partial heat of vaporization of $U(g)$ at $\sim 2200^\circ K$ shows a similar rapid change in value through $UC_{1.0}$ with a maximum near $UC_{1.1}$ at 166 ± 2 kcal/mole. Another maximum occurs in the UC_2 region at 148 ± 2 kcal/mole and $UC_{1.85}$. The heat of vaporization based on the $UC_2 + C$ region is too low. This results from the fact that the UC_2 phase changes composition with temperature.

The measured pressures are consistent with congruent vaporization near $UC_{1.1}$. Evidence has been obtained which supports a value near 126 kcal/mole for the heat of vaporization of U. Using this value, the measured pressures in the U-C system show excellent agreement with the other thermodynamic measurements.

Vaporization Studies of the Ti-C System

Mass spectrometer Knudsen cell studies have been made of the variation of Ti pressures within the Ti-C system. A value of 4.3×10^{-8} atm was found for the pressure of Ti over the two-phase region TiC saturated with graphite at $2220^\circ K$. From this, a value of -36.2 kcal/mole was computed for $\Delta f_f^\circ (2220^\circ K)$, which compares favorably with -35.5 ± 0.5 kcal/mole reported in the literature. At $1925^\circ K$, a pressure drop of ~ 4 orders of

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magnitude was observed for the Ti pressure measured over the two-phase region Ti-TiC ($\sim 2.9 \times 10^{-6}$ atm) compared with that measured over TiC saturated with graphite ($\sim 1.8 \times 10^{-10}$ atm).

Vaporization Studies of the U-Ti-C System

A preliminary investigation has been initiated of metal pressure variation within the ternary system U-Ti-C. Six compositions have been monitored thus far; in each, the Ti^+ , U^+ , and UC_2^+ intensities have been recorded over a temperature range of $\sim 200^\circ C$. Three nominal compositions, $U_{0.5}Ti_{0.5}C_{2.5}$, $U_{0.5}Ti_{0.5}C_{1.5}$, and $U_{0.5}Ti_{0.5}C_{2.5}$, appear to fall within the same three-phase area of an isothermal cut at $2000^\circ C$.

Rare Earth Metal-Carbon Systems

The solid state, tetragonal to cubic transition temperatures and MC_2 -C eutectic melting temperatures have been determined for the TbC_2 , HoC_2 , and TmC_2 phases. The results are listed in Table 14.

Table 14. Transition and Eutectic Temperatures

<u>Dicarbide</u>	<u>Tet. $\rightarrow$ Cubic Trans. Temp., $^\circ C$ $\pm 20^\circ C$</u>	<u>MC_2-C Eutectic Temperature $\pm 25^\circ C$</u>
TbC_2	1285	2275
HoC_2	1305	2370
TmC_2	1355	2245

Lattice constants, from x-ray diffraction, have been determined at room temperature and at temperatures just above and just below the transition temperature. The results are given in Table 15.

The room temperature form of LuC_2 , prepared by arc melting 99.9% pure Lu and C, was not found to be body-centered tetragonal as reported by previous workers. The x-ray powder pattern is quite complex and has not

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Table 15. Lattice Constants of Dicarbides

Compound	Temp. (°C)	Structure	a_o (Å)	c_o (Å)
LaC ₂	> 1060	fcc	5.995 ± 0.005	-
LaC ₂	< 1060	bct	4.032 ± 0.005	6.54 ± 0.01
LaC ₂	Room	bct	3.935 ± 0.005	6.578 ± 0.005
TbC ₂	> 1285	fcc	5.684 ± 0.006	
TbC ₂	< 1285	bct	3.793 ± 0.005	6.228 ± 0.010
TbC ₂	Room	bct	3.692 ± 0.005	6.228 ± 0.005
LuC ₂	> 1500	fcc	5.500 ± 0.005	

yet been solved. This structure appears to exist up to the transformation temperature, but more work will be done near the transition temperature to see if the body-centered tetragonal structure can be found. Work will also be done to determine the basis for the difference between the observed powder patterns and those reported by previous workers.

Microhardness measurements have been obtained on some of the lanthanide dicarbides and on YC₂. Prior to the measurements, the samples were annealed for 4 h at 1500°C. It was necessary to keep a layer of kerosene over the polished specimens during the determinations to keep the oxidation of the samples at a minimum. The results obtained are shown in Table 16.

Table 16. Knoop Microhardness (KN) 400-g Load of Dicarbides

Dicarbide	Microhardness (kg/mm ²)	Dicarbide	Microhardness (kg/mm ²)
LaC ₂	215 ± 20	DyC ₂	392 ± 16
CeC ₂	219 ± 7	HoC ₂	611 ± 40
PrC ₂	166 ± 12	ErC ₂	660 ± 65
NbC ₂	213 ± 13	TmC ₂	648 ± 72
SmC ₂	224 ± 23	YbC ₂	168 ± 3
GdC ₂	281 ± 31	LuC ₂	621 ± 29
TbC ₂	370 ± 9	YC ₂	400 ± 25

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Neutron Diffraction Studies

A sample of the YC_2 phase has been examined by high-temperature neutron-diffraction. At $1100^\circ C$ a complete pattern appears to be isomorphous with tetragonal UC_2 , and at $1400^\circ C$ it appears to be similar to that of cubic UC_2 . Neither pattern has been fully analyzed. The transition was observed at $1340^\circ C$ by neutron diffraction.

Refractory U and Th Systems

Precipitation of U from U-rich phases of UC, UN, UP, and UAs and precipitation of Th from Th-rich ThP and ThN has been observed metallographically. The data confirm published reports which indicate a homogeneity range in UC and ThP.

ANALYTICAL CHEMISTRY RESEARCH

Determination of O_2 in PyC-Coated UC_2 Beads

A successful sample-handling device was developed to facilitate the determination of O_2 in UC_2 beads having highly impervious PyC coatings. Weighed samples of the beads are crushed and loaded into the crucible within the inert atmosphere of a furnace that is an integral part of an inert-carrier-gas apparatus for determining O_2 . Release of O_2 from uncrushed beads of this type requires high temperatures (2900° to $3000^\circ C$) that are impractical because of excessively high apparatus blanks and rapid failure of available crucible materials, whereas prior crushing results in release of O_2 at temperatures of $2600^\circ C$. Crushing must be done in the inert atmosphere of the analytical apparatus because the exposed cores are extremely reactive and are readily contaminated with O_2 . Some duplex-coated beads and all triplex-coated beads investigated require crushing to insure release of O_2 during analysis, and the crushing operation is not detrimental to the determination of O_2 in any coated bead sample. Therefore, all bead samples are crushed prior to analysis.

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Determination of U in NbCl₃ by X-Ray Fluorescence

An x-ray fluorescence spectrographic method was developed for determining 0.1 to 6% of U in NbCl₃. Samples weighing approximately 500 mg were dissolved in HNO₃-HF, and Bi added to serve as an internal standard. The ratio of the intensities of the L α ₁ lines for U and Bi was measured and compared with intensity ratios of solutions of known U content. The relative standard deviation of a single measurement varied from 12 to 1.5%, respectively, for U concentrations in the range of 0.1 to 6%.

MISCELLANEOUS

Stress Analysis

A theoretical study has been made of thermal stresses in an 8-in. stainless steel flange when cooled by an internal LH₂ flow of 1 lb/sec. The temperature profiles were generated with the aid of the HEX heat transfer program, and the stresses calculated with the aid of the SCIRG thermal stress program. Preliminary results indicate that the maximum thermal stress is reached within 1000 sec and that the magnitude of this stress is below 40,000 psi. The behavior of 10-in. flanges and the effects of different flow rates are being studied.

Two-Phase Flow

A large amount of pressure drop punched-card data on two component, two-phase flow of noncryogenic fluids in pipes is being analyzed by machine. Before attempting any new correlations, the important parameters are being found by looking at them one at a time. To date, Reynolds number has been found to be important, pipe diameter is important only in some regions, pressure level is important, and flow rate is not important. The last two findings are of particular interest because of disagreement in the literature on these points.

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VI. ROVER FLIGHT SAFETY

THE KIWI-TNT TEST

Introduction

The Kiwi-TNT test conducted on Jan. 12, 1965 was described in the preceding quarterly report (LA-3280-MS, p. 135). As shown in Fig. 39 the pressure shell from the reactor has been reconstructed on a plane surface. Although two or three pieces are still missing, the recovered portions fit together very well.

Fig. 39. Reconstruction on flat surface of pieces recovered from Kiwi-TNT pressure shell.

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Analysis of the Kiwi-TNT Experiment

The objectives of the analysis were (a) to understand the Kiwi-TNT experiment in the sense of normalizing existing calculational methods to the results and (b) to extend the method of different reactivities and reactivity insertion rates. Pertinent results of such parametric studies are presented in a manner useful for estimating the hazards of conceptual accidents to Kiwi-B-size reactors. The techniques developed will be applied to the Phoebus-size reactor and, in particular, an attempt will be made to study in some detail the conceptual water insertion accident for this reactor.

The major pertinent experimental results are the prompt alpha, i.e., reciprocal prompt period (1600 sec^{-1} to 1680 sec^{-1}), the total number of fissions $[(3.1 \pm 0.3) \times 10^{20}]$, and the estimate of the chemical energy ($220 \pm 100 \text{ MW-sec}$) which would have caused comparable damage to the reactor, test car, and Test Cell C. The value for the chemical energy equivalent is somewhat larger than the kinetic energy of reactor fragments required for comparison with computation. The correction is very highly geometry-dependent and in the absence of precise information, 150 MW-sec (with some undefined uncertainty) was chosen as the kinetic energy to which the code was normalized (see p. 119).

The prompt alpha apparently was constant for over 10 msec before the time of peak power; the assumption of a step insertion of reactivity is consequently reasonable and proper. This constant alpha was verified by the control vane records which indicated that all vanes were fully out before the time of peak power. The reactor was only about 60¢ sub-delayed critical just before the experiment and the full insertion of reactivity resulted in a state 7.3¢ above prompt critical. Thus, from the excess reactivity, the prompt alpha, and the effective delayed neutron

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fraction (0.0078), the neutron lifetime can be estimated as follows:

$$l = \frac{\beta^{\$} p}{\alpha} = \frac{7.8 \times 10^{-3} \times 7.3}{1600} = 35.6 \times 10^{-6} \text{ sec.}$$

This value agrees well with multigroup neutron transport code estimates and it is used in the RAC code, described later.

Kiwi-TNT Kinetic Energy Estimate

The Fastax Movies

The Fastax movies (4000 frames/sec) of the Kiwi-TNT experiment show early motion both axially and radially at about the time of peak power. The hot material ejected axially, toward the mirror structure, accelerated rapidly and reached a peak velocity of $7-8 \times 10^4$ cm/sec at about 1 m above the core. This is close to the peak core surface velocity predicted by the RAC code (p.123). The agreement may be fortuitous, however, as the "early vapor" or "hot dust" has little "body" behind it. The mirror, flashbulb, and poison plate structures clearly are not carried away with this earliest material; dense material may be close behind, but this is hypothetical. The agreement nevertheless is reassuring and better than qualitative.

Pressure Transducer Measurements

Pressure transducers were placed at several positions from the Kiwi-TNT location. Of these, only two placed at the maximum distance (100 ft) produced a signal at the proper time, the others apparently recorded an electromagnetic signal at the time of peak power. The transducers that worked indicated an overpressure of 3-5 psi (250-600 MW-sec chemical energy) at their distance as compared to 2-3 psi estimated from the threshold and damage indications. The estimate of 150 MW-sec for normalization was obtained with a modest weighting of these transducer data.

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Comparison with H.E. Effects

An extensive study has been made of high-explosive "destruct" concepts. In particular, a series of experiments was completed in which four cylinders of H.E. were placed within a full diameter, half height of an inert Kiwi core with mocked reflector and pressure shell. Two such experiments were performed with six, rather than four, cylinders of H.E. These cylinders were exploded simultaneously and cloud velocity, cloud size, particle size distributions, and similar studies were initiated. The Fastax movies (1000-7000 frames/sec) of these explosions (for H.E. masses between 60-170 lb) bear a qualitative, but remarkably similar appearance to the Kiwi-TNT Fastax movies, if black (cold graphite dust) expanding cloud is substituted for the bright (hot graphite dust or vapor) Kiwi-TNT cloud.

The surface velocity of the expanding "black" cloud was measured in most of the H.E. experiments. These velocities seem to depend critically upon the number of charges and their arrangement within the core as much as on the total mass. The best that can be said is that the Kiwi-TNT cloud surface velocity was less than that caused by 170 lb H.E. arranged in four charges and less than that caused by 60 lb H.E. arranged in six charges, each set placed in the half-height core. A 300-lb, four-charge arrangement in a single full-scale reactor mockup definitely appears more violent and surface velocities are higher than those in the Kiwi-TNT.

Photographic Results

A number of experiments were conducted with the various films from Kiwi-TNT. For the most part, these efforts were made necessary (and results were of doubtful value in some cases) by the loss of the time code on both north bunker Fastax cameras and by the overabundance of event markers on both of the core end Dynafax cameras. Measured and derived times of events on the various films were determined and reported to the extent that the values of the pertinent constants and parameters of the photo system were known.

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Densities of the high-speed core Dynafax film from both the hot run and dry run films were read using a densitometer aperture the size of the core image on the film. The results were used to check the time tie derivations mentioned above and to check the flash bulb operation at early times. All evidence indicates that the flash bulbs fired properly.

Two or three frames in the 16-mm movie prints from the Fastax cameras show a blue glow, presumably from ionized air surrounding the reactor. Some two or three frames (0.5 to 0.75 msec) prior to these all the rabbits appear to be obscured by rising vapors. Identification of the glow on the Fastax films was uncertain and so an attempt was made to produce color enlargements of selected frames while adjusting the color balance and print densities to emphasize any such blue glow. This effort has been partially successful and further prints are being made. It is hoped that from such prints it will be possible to identify the frame where the glow has maximum intensity and to identify that frame with peak power. Then by working backwards through the photo timing this can be checked against the coded time of peak power as indicated by the neutronics and data system.

Pin Experiment

The LASL interpretation and analysis of the pin measurements (LA-3280-MS, p. 144) have been compared with that of EG&G. Confidence was good in eight of the 12 measurements, poor in four, and one channel did not produce any identifiable signals. Two measurements with poor confidence and the channel which failed were from an experiment to determine collapse of the holes in the core. A summary of the core and reflector cylinder displacement time histories is shown in Fig. 40.

Brightness Temperature Measurement

Three radiometrically calibrated cameras were operated by EG&G to obtain the visible luminous mass brightness as a function of time. The maximum value for the visible brightness (2750-6750 Å) lies between 35 and

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Fig. 40. Summary of core and reflector cylinder displacement histories in Kiwi-TNT.

46 W/cm²-ster, and occurred between 10 and 30 msec. The corresponding maximum brightness temperature lies between 3250° and 3640°K.

A radiometrically calibrated spectrograph was operated by LASL to record the spectral brightness of the luminous mass as a function of time. The spectrum was generally a continuum. Absorption, believed to be due to C₂, occurred at times from 0 to 10 msec and absorption and emission due to CO was observed later (approximately 100 msec). The maximum brightness temperature recorded by the spectrograph is 3200°K at about 25 msec.

The difference between the two brightness temperatures arises from the camera analysis which picked the brightest spot of the rather inhomogeneous luminous mass whereas the spectrograph slit integrated the light emitted from a larger area. It should be pointed out that these measurements provide information on surface temperature only and that the observed brightness temperature (by definition) is a lower limit to the true surface temperature.

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The RAC Code: Modification and Normalization

The RAC code has been described in LAMS-2930 and only the significant modifications and normalization scheme will be discussed here. This normalization is conducted in the (original) RAC plane geometry code. Extensions to cylindrical and two-dimensional (cylindrical-plane) geometries exist and pertinent studies will be reported subsequently.

Normalization to the two energies (3.1×10^{20} fissions and K.E. = 150 MW-sec) was accomplished through two adjustable constants buried within the code. These are a lumped temperature-expansion coefficient of reactivity (denoted here by A) and a multiplier (B) associated with the graphite vapor equation of state. The quantity A, in units of reactivity change/energy unit (i.e., dollars/ $^{\circ}\text{C}$, $\Delta k/\text{fission}$, etc.), determines primarily the fission yield. In fact, for a symmetrical burst (no vapor pressures, kinetic energy or damage to the core), the energy release is given by

$$E = \frac{2\Delta k_p}{A}$$

and the problem is solved.

To compute the kinetic energy, however, the vapor pressures (if such exist) must be known throughout the core and the mechanical work, $\int P(t)dV(t)$, performed must be computed as a function of time and space in the problem. These vapor pressures are calculated by application of three functions, the first two of which are obtained by integrations of the Clausius-Clapeyron equation. At and below the carbon triple point (3960°K and 100 atm), the function $P_B = P_0 e^{-L'/RT}$ is forced to pass through the 1-atm point at 3600°K as well as the triple point. By requiring the function P_B to predict these two points correctly, the quantity L' is no longer equal to the latent heat of sublimation but the functional form is correct. Above the triple point, data are nonexistent. A second function, P_C (of the same form as the above equation), is required to pass through the triple point, with an "effective" latent heat (L') less than that used below the triple point.

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These two functions are employed only as limiting values. The pressure function used for the mechanical work calculation is a modified "perfect" gas; it has the form $P_D = BRNT$, in which T is the absolute temperature, N is the molar density, R is the gas constant, and B is the "kinetic energy" adjustable constant, suitable for normalization purposes. As long as temperatures are rising, the magnitude of P_D is not allowed to exceed the limiting functions P_B or P_C for the existing temperatures. If this should be the case, the code requires the energy produced in the next time cycle to raise the temperature of the region. If P_D should be less than P_B or P_C , the code requires the energy produced to create vapor (change N) at constant temperature. By this technique a function has been introduced which is dependent upon both the temperature and the vapor density. Additional details and complications could be incorporated but it is not believed that much more detail could be justified on the basis of existing knowledge of graphite vapor.

To normalize the code to the TNT results, a series of calculations were completed in which the parameters A and B were varied with the requirement that the total fissions always be 3.1×10^{20} . The kinetic energy in this set was uncontrolled. Initial conditions required were a prompt alpha of 1600 sec^{-1} (7.3\$ over prompt critical and a neutron lifetime of $35.5 \text{ } \mu\text{sec}$) and a very low initial power. The kinetic energy is nearly linearly proportional to B , except for the higher values.

The magnitude of B is such that for a given temperature and vapor density, the vapor pressure (P_D) is less than that for a perfect gas by a factor of ~ 5 . A qualitative justification for this approach is obtained by noting that in the two-phase region of the thermodynamic PV diagram, the vapor pressure is always less than that for a perfect gas. At the critical point for example, vapor pressures are less than perfect gas pressures by factors of ~ 3 or 4 . Below this temperature (near 9000°K for carbon) vapor pressures drop exponentially. The functional form is given by the equation $P_B = P_0 e^{-L'/RT}$.

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The first parametric study using this normalization was a series of step insertions of reactivity, ranging from 50¢ to 15\$ above prompt critical. Fission yields, maximum pressures, and kinetic energies are shown in Figs. 41, 42, and 43 as functions of the excess reactivity above prompt critical. Figure 44 gives the kinetic energy as a function of the total fissions. For heat energy conversions to kinetic energy, an efficiency may be defined for either the total energy release or for the energy above that required to raise some part of the core to the sublimation temperature. This last value is close to 1.2×10^{20} fissions.

The fraction of the core converted to vapor is given as a function of position within the core for the several reactivities in Fig. 45. The odd shape of the curves for 3.5\$ and 7.3\$ reflects the behavior at the triple point where full liquefaction is required before additional vapor can be created. The vaporization calculations are dependent upon the assumed molecular weight of the vapor and since this is essentially unknown, the vapor was taken to be monatomic. The latent heat of vaporization (or sublimation) is reputed to be a slowly varying function of the molecular weight (i.e., for the C_1 molecules, $L = 172$ kcal/mole; for C_2 , $L \approx 195$; for C_3 , $L \approx 188$; etc.). Thus, for example, if C_3 is the most probable molecule, the fraction vaporized should be increased over that given in Fig. 45 by a factor of $3 \times 172/188 = 2.74$.

The second parametric study for the Kiwi-B type reactor is a series of problems in which reactivity is inserted at a uniform, never-ending rate. Total kinetic energies, fission yields, and maximum reactivities attained during the problems (all for several initial powers) are illustrated in Figs. 46, 47, and 48 vs the reactivity insertion rate. Within the uncertainties of the calculation, these kinetic energies also fall on the curve of Fig. 44.

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Fig. 41. Calculated fission yield vs reactivity above prompt critical in Kiwi-B-type reactor.

Fig. 42. Calculated maximum pressure vs reactivity above prompt critical.

Fig. 43. Calculated kinetic energy vs reactivity above prompt critical.

Fig. 44. Calculated kinetic energy vs total fissions.

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Fig. 45. Fuel converted to vapor calculated as function of core position in Kiwi-B type reactor for several reactivities.

Fig. 46. Calculated kinetic energy vs reactivity insertion rate in Kiwi-B type reactor.

Fig. 47. Calculated fission yields vs reactivity insertion rate in Kiwi-B type reactor.

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Fig. 48. Calculated maximum reactivity vs reactivity insertion rate.

In summary this normalization now allows a precise calculation of the fission energy release and an estimate, that is accurate within a factor of 2 or less, of the kinetic energy for an excursion involving a Kiwi reactor, assuming the reactivity history can be described accurately. The reactivity, reactivity insertion rate, or energy density at which the kinetic energy first appears (the threshold) is accurately fixed.

Kiwi-TNT External Experiments

The graphite extrusion samples containing coated particles, located outside the Kiwi-TNT reactor (LA-3280-MS, p. 157), have been examined both metallographically and visually. The metallographic examination has provided a picture of the damage to the matrix and to the coated particles in the samples. The visual observations of matrix damage are related to damage to the coated particles. Where the graphite matrix was completely powdered or broken with many major cracks, the coated particles were completely destroyed and the U was distributed throughout the matrix. This occurred where the fissions/g U^{235} were highest. The

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minor matrix cracks were generally associated with cracked and broken coated particles; U was located in the matrix cracks. Even where no macroscopically visible matrix damage was observed, metallographic examination showed coated particle cracking and very small matrix cracks. Where the fissions per gram of U^{235} were lowest, the UC_2 cores of the coated particles had melted but the particles remained intact. Photomicrographs covering the range of particle damage observed are shown in Figs. 49 and 50.

A number of conclusions can be drawn concerning the relationship between sample damage and the conditions of the experiment. The variation of both matrix damage and coated particle damage with fissions/g U^{235} overshadowed any other trend. More matrix damage was observed with the

Fig. 49. Upper: Coated particles (x50) completely destroyed at 5×10^{15} fissions/g U^{235} ; lower: coated particles (x50) broken at 2×10^{15} fissions/g U^{235} .

Fig. 50. Upper: Coated particles (x100) cracked at 6×10^{14} fissions/g U^{235} ; lower: coated particles (x250) melted but coating intact at 3×10^{14} fissions/g U^{235} .

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Fig. 51. Relationship between UC_2 particle core diameter, fissions per gram U^{235} , and damage.

samples having high particle volume loading. Coated particle damage was affected by the UC_2 core size and to a smaller extent by the coating thickness. Figure 51 indicates the relationship between the particle damage, the UC_2 core diameter, and fissions/g U^{235} . Particles with larger cores showed more damage at a given flux than those with small cores. For particles with the same core size range, those with thicker coatings showed slightly less damage. No significant variation in matrix damage or coated particle damage was found due to sample temperature at the time of the test.

The four samples containing coated particles with UO_2 cores showed more damage to both matrix and particle at a given flux than coated particles of comparable size with UC_2 cores. The damage appears to have been initiated by cracking and rupture of the individual coated particles. The damage to coated UO_2 particles is influenced mainly by the fissions/g U^{235} , with minor variations due to particle sizes and composition.

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OTHER ROVER FLIGHT SAFETY ACTIVITIES

Destruct Experiments with Chemical Explosives

Several runs have been made to obtain good data on the fragmentation by chemical explosives of a beaded fuel sample at room temperature. A balloon technique was successfully employed to collect fuel fragments without any secondary **breakup**. Runs made in the furnace geometry indicated that bad secondary breakup occurred using bare walls. Good results were obtained when the cavity was filled with lightly packed glass wool.

Initial tests using fuel heated to 2500°C prior to fragmentation uncovered several difficulties in the explosive trigger design and overheating of the explosive from the flowing He atmosphere. These problems have been solved and a successful high temperature fragmentation achieved. Results are now being analyzed.

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VII. ROVER FACILITIES ENGINEERING

Introduction

Work has been completed at LASL on all Rover line item construction projects with funding prior to FY 1964. All FY 1964 and FY 1965 Rover projects are still open.

FY 1964 Rover Construction

Furnace Facility, Building DP-207: Final acceptance of the building was taken on May 21 and installation of the four furnaces has begun. Installation drawings are 80% complete for a fifth furnace which will be installed in Building DP-206.

Subassembly Building Addition, TA-18: Construction work has started and is scheduled to be completed in September.

Ultrasonic Cleaning Facility, TA-18: Criteria drawings are finished and A-E services for this facility are presently being negotiated by the AEC. The ultrasonic cleaner itself has been ordered.

FY 1965 Rover Construction

Phoebus Development Facilities

Nondestructive Test Additions, TA-8: Construction of the Building 70 addition is finished and the Building 22 addition is 60% complete. Completion of this last phase of construction is scheduled for July 15.

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Mechanical Test Facility Addition, TA-46: Design is complete and drawings are being issued to The Zia Company for construction.

Laboratory Modifications for Graphite Research, Building SM-29: Design is 50% complete and some equipment has been ordered.

Standards Laboratory, TA-46: Design is 20% complete.

Cooling Water System Replacement, TA-46: Some changes in design, which is approximately 35% complete, will be necessary as a result of requirements of the using group.

Core Support Test Facility, TA-46: Design of the building is finished and bids will be opened on June 15.

Gaseous Hydrogen Supply System, TA-3 to TA-46: The surplus storage tanks obtained from Warren Air Force Base have been delivered to TA-46. There is some question concerning the adequacy of the TA-3 hydrogen dewar and a new dewar may be installed at TA-46.

Remote Liquid Hydrogen Facility, TA-46: This facility has the lowest priority of the eight Phoebus subprojects and design has not yet been started.

Miscellaneous Rover Construction

Pulsed Accelerator Building, TA-18: Design of the building is complete and bids will be opened on June 17.

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VIII. PUBLICATIONS

"Radiation Environmental Analysis," Notes for the 1962 National Science Foundation Advanced Subject Matter Institute on Nuclear Rocket Propulsion. Univ. of Florida, 1964. pp. 475-613.

"Nondestructive Inspection of Fuel Elements for Nuclear Rockets," Proc. Intern. Conf. on Nondestructive Testing, London, England, 1963. p. 43. Butterworth, 1964.

"Liquid Hydrogen as a Coolant/Propellant for Nuclear Rockets," in "Technology and Uses of Liquid Hydrogen," R.B. Scott, et al., eds. p. 181. Macmillan, 1964.

"Longitudinal Tensile Behavior of H⁴LM Graphite in Nitrogen to 2750°C," J. Nucl. Mater., 13, 57 (1964).

"High-Current Coaxial-Arc Thrustors," Bull. Am. Phys. Soc., Ser. 2, 10, 23 (1965) (Abstract).

"Temperature Dependent Kinetics of Coupled Core Reactors," LA-3148-MS.

"Radiation Heating Rate Estimates for a Phoebus Reactor," LA-3158-MS.

"Cryogenic Tensile Testing of Project Rover Materials," LA-3161.

"Thermal Analysis of the Kiwi-B-4D Reactor. Parts I-V," LA-3170-MS.

"A New Instrument for Uranium Determination in Rover Fuel Elements," LA-3196-MS.

"Treatment of Niobium Pentachloride - Rover Project Waste," LA-3142-MS.

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