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Westinghouse
Astronuclear
Laboratory

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DATA ITEM T-119

OUTGASSING AND
VAPORIZATION EFFECT OF
PYROFOIL LAYERS BONDED
WITH PETROLATUM

FINAL REPORT

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DATA ITEM T-119

OUTGASSING AND VAPORIZATION EFFECT OF PYROFOIL LAYERS BONDED WITH PETROLATUM FINAL REPORT

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ABSTRACT

The outgassing and vaporization effects under rapid heat up transients are experimentally investigated for multiple layers of pyrofoil bonded with petrolatum. Both thermalized rerolled pyrofoil (PDS 30141-1) and unthermalized pyrofoil (PDS 30182-2) are included.

The pyrofoil layers are shaped to simulate their application in the peripheral region of the R-1 reactor. Maximum heat up temperature ramps at 75° per second are applied to the layers until a hot side temperature of 2500° is attained. An overtest at a heat up ramp of 100°R per second and a hot side temperature of 5600°R are also evaluated. These two sets of temperature conditions bracket the maximum conditions anticipated for pyrofoil usage in the R-1, 10 hour 60 cycle reactor.

Visual observations show that the vaporization and outgassing produce some blistering of the pyrofoil surfaces and some positive net dimensional expansion. Analysis of these effects show that petrolatum bonded pyrofoil layers are satisfactory for use in the R-1 reactor.

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1.0 INTRODUCTION

1.1 TEST IDENTIFICATION

1.1.1 Type of Report

This report is a complete Final Report.

1.1.2 Applicable Scope of Test Document

The applicable Scope of Test Document Number is 64DE-312-2, Scope of Test for Outgassing and Vaporization Effect of Pyrofoil Layers Bonded with Petrolatum.

1.1.3 Functional Category of Test

This is a Development test.

1.1.4 Contractor

The Westinghouse Astronuclear Laboratory (WANL/14683), under Subcontract NP-1, CY-70, is the contractor.

1.1.5 Test Facility, Location and Dates of Test

These tests were performed at the Westinghouse Astronuclear Engineering Mechanics Laboratory, Furnace Room, Building No. 8; May 28, 1969 through July 22, 1970.

1.2 PURPOSE

The objective of these tests was to determine whether outgassing and vaporization of pyrofoil layers bonded together with petrolatum on a simulated wrapper geometry would cause catastrophic bubbling or cracking of the pyrofoil when exposed to reactor heat-up rates.

The data obtained were used to support analysis needed to verify the acceptability of outgassing and vaporization effects of the petrolatum bonded pyrofoil assembly in the R-1 reactor.

2.0 REFERENCES AND ASSUMPTIONS

2.1 REFERENCES

2.1.1 Specification

EC-677564 - Part 1. Performance/Design and Qualification Requirements for Core Periphery Forming a Part of CP-677555 Nuclear Subsystem

WANL-PDS-30189-1 - Purchasing Department Specification for Bonding Compound (Petrolatum)

WANL-PDS-30182-2 - Purchasing Department Specification for Graphite Tape Unthermalized

WANL-PDS-30141-1 - Purchasing Department Specification for Graphite Tape Thermalized and Rerolled

2.1.2 Data Item Description

Form 9 - Data Item Description, T-119, dated April 2, 1969.

2.1.3 Scope of Test Document

64DE-312-2 - Outgassing and Vaporization Effect of Pyrofoil Layers Bonded with Petrolatum

64DE-143 - Outgassing and Vaporization Effect of Pyrofoil bonded with Petrolatum

2.1.4 Data Release Memorandum

52559 - Outgassing and Vaporization Effect of Pyrofoil Layers Bonded with Petrolatum - 64DE-312-2, dated July 23, 1970.

52985 - Outgassing and Vaporization Effect of Pyrofoil Layers Bonded with Petrolatum - 64DE-312-2, T-119 Summary Report dated August 19, 1970.

51254 - Zr-C-UC-Graphite-Material Properties Data

52533 - R-1 Core Radial Expansion of a High Expansion Graphite Core

2.1.5 TME

WANL-TME-2722 - Thermal Expansion Characteristics of Unthermalized and Thermalized Rerolled Pyrofoil

2.1.6 Drawing

939J806, Sheet 4 - Core Periphery with Pyrofoil Wrapper for R-1

940J422 - Corrosion Resistant Core Periphery

711J744 - Furnace, Pyrofoil Outgassing Test Fixture

ATEMCO Part No. 5392 - Bend Press Assembly

2.2 ASSUMPTIONS

That the performance of a sample of 26 layers of pyrofoil shaped on a bed with longitudinal vees simulating the corners of fuel elements would simulate the behavior of a similar area of reactor periphery.

That the use of argon instead of hydrogen as the gas environment would not significantly change the test results.

3.0 DESCRIPTION OF TEST ARTICLE

The test article consists of 26 layers of thermalized or unthermalized pyrofoil folded into a V shaped configuration over an area of approximately 18 x 6 inches (See Figures 3-1 and 3-2). The Vees run longitudinally over the 18 inch dimension. The pyrofoil layers are cut longitudinally into Z shaped strips and assembled according to Reactor Drawing Layout 939J806, sheet 4. The pyrofoil material is nominally 0.0055-inch thick and bonded together with petrolatum purchased from Gulf Oil Corporation according to PDS 30189-1. The folding into Z shaped strips is accomplished on a special bend press (ATEMCO Part No. 5392). The testing was done on equipment built according to Drawing 711J744.

Photographic displays are shown of the test bed assembled in the furnace rig (see Figure 3-3) and the assembled rig and instrumentation (see Figure 3-4).

Figure 3-1 Results from Checkout Run Showing Hot Side
of Unthermalized Pyrofoil without Gaps

Figure 3-2 Results of Checkout Run Showing Cold Side of Unthermalized Pyrofoil without Gaps

Figure 3-3 Test Bed Assembled in the Furnace Rig

Figure 3-4 Assembled Rig and Instrumentation

4.0 SUMMARY OF TESTS

4.1 NARRATIVE

DRM 52559 summarizes most of the various tests which were run and any deviations from the scope of test document as issued. Due to late delivery of additional Q.C. approved pyrofoil material, the final test was run following the issue of DRM 52559 and the results are documented in DRM 52985.

4.2 TEST CONDITIONS

The initial test conditions called for a $150^{\circ}\text{R}/\text{sec.}$ ramp rate of temperature and 4900°R maximum temperature with 8 psi and 30 psi bundling pressures. These conditions were to satisfy the requirements of the R-1, 75 minute reactor incorporating a full length pyrofoil wrapper. Later test conditions were modified to attain 2500°R on the fuel interface layer of pyrofoil. This condition simulated the fuel interface temperature at Station 17.0, the end of shortened pyrofoil wrapper in the R-1, 10 hour, 60 cycle reactor.

Two builds each of thermalized and unthermalized pyrofoil were to be tested according to the above conditions.

4.3 TEST RESULTS

4.3.1 Recorded Data

As the objective of the test was to obtain visual data such as bubbling and cracking in the pyrofoil strips, there is a minimum of numerical data to report. All attempts to attain 4500°R (before the test requirements were changed to 2500°R) on unthermalized pyrofoil, ended in arcing of the equipment with some destructive results as the pyrofoil began to conduct current better than the heating bed. However, the petrolatum was completely eliminated from the test configuration before 2500°R was attained, so the lack of accomplishing the full 4500°R did not prevent getting the desired information on petrolatum effects.

Boron nitride insulating paint was used to successfully overcome the electrical insulation problem without seriously reducing the heat transfer capability, and a successful run was made to 2537°R on a thermalized rerolled pyrofoil specimen assembly. A thermocouple trace of the heatup ramp to this temperature is shown in Figure 4-1.

The final test was run on thermalized rerolled pyrofoil at as high a compaction pressure and as high a maximum temperature ramp rate as could be obtained. During the course of this test, the evolution of gas was so violent that the cover of the box containing the equipment was lifted and temporarily pulled the thermocouples from their moorings. The violence of the disruption negated the readings from that point in time but the tungsten tip of the thermocouple contacting the hottest pyrofoil layer melted. It requires approximately 5600°R to melt tungsten thus, the highest temperature can be pegged at that point. The total thickness expansion of the pyrofoil ranged from .025-inch to .046-inch over the heater surface. The expansion was caused by blistering of the pyrofoil layers with more being observed on the hottest layers than on the coldest. No tearing or lack of functional capability was observed.

Similarly, the observations from other tests showed that considerable blistering of the pyrofoil occurred with the layers next to the heated surface showing more of this effect than those farther away. In no case was there any tearing or destructive effect. There was less evolution of gas observed when the test sample was allowed to sit around for two or three weeks before testing due to evaporation of the benzene solvent of the petrolatum.

The initial thickness of the pyrofoil assembly included 0.014 inch of petrolatum. When the thermalized reactor grade pyrofoil was tested to 2500°R on the hot side, the loss of petrolatum due to vaporization should cause a net compaction of the layers of 0.014 inch.

Figure 4-1 Temperature Ramp on Hot Side Pyrofoil Layer

However, there was a net expansion of 0.010 inch measured after the heating cycle. This indicates an actual permanent expansion of 0.024 inch ($0.010 + 0.014$) in the pyrofoil pieces due to the heating cycle.

The permanent growth, which is due to blister formation, is very soft and spongy and cannot be measured accurately in its free state. There has to be a compression of the layers before any firmness is reached, and this may amount to as much as 0.003 inch to 0.004 inch. Figures 4-2 and 4-3 show typical results of petrolatum evolution from the coldest and hottest sides of thermalized rerolled pyrofoil assembly specimens.

4.3.2 Discussion of Data

Attainment of the requested parameters of this test was limited by equipment capability. This was particularly at the requested rate of rise in temperature of $150^{\circ}\text{R}/\text{sec}$. The heat transfer necessary to accomplish this rate required electric heating which in turn required that the heating bed be at a higher temperature than the pyrofoil specimen. When the insulation barrier between the heating bed and pyrofoil broke down, the pyrofoil would become part of the electric circuit with a consequent reduction in rate of temperature rise. However, it was evident that there was considerable heat lag in the assembled pyrofoil layers and the rate of heat rise of the hottest layers next to the heater bed was greater than the coldest layers away from the heater bed. The best thermal gradient which could be determined from chart records was approximately $75^{\circ}\text{R}/\text{sec}$. If a top temperature of 5600°R is assumed from evidence of melting in the final run, then the rate of rise in that test would have been well over $100^{\circ}\text{R}/\text{sec}$. The absorption of carbon by the tungsten wires of the thermocouple undoubtedly changed the accuracy of the thermocouple's response so that lower than actual values were recorded. The evolution of dense smoke precluded the use of optical equipment for a temperature check during the test runs.

Figure 4-2 Results of Petrolatum Evolution from Thermalized Pyrofoil
for Coldest Layers with Gaps Away from the Heater Bed

Figure 4-3 Results of Petrolatum Evolution from Thermalized Pyrofoil for Hottest Layers with Gaps Adjacent to the Heater Bed

What is meaningful about the test is that the petrolatum was completely evaporated before any equipment failure was encountered and no soot or ashes were ever found between the pyrofoil layers. No differences were found in pyrofoil appearances from the range of pressure applied to the test bed. The main effect of pressure variation was to make the pyrofoil carry more electric current as the pressure was increased. No significant differences were found in the appearance of the thermalized rerolled (PDS 30141-1) vs. unthermalized (PDS 30182-2) pyrofoil materials tested.

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5.0 ANALYSES

(U) The intent of this test was to expand the results of Scope of Test 64DE-143, Out-gassing and Vaporization of Pyrofoil Bonded with Petrolatum, to conditions more typical of the reactor. Simulation of the reactor periphery required that a significant area of periphery be tested to insure that any gas generated would have to travel a reasonable distance between layers of the simulated periphery before escaping. The basic data requirements of the test were qualitative in nature and analysis was limited to interpretation of the results of visual examination of the specimen including microscopic determinations of incipient or actual damage.

(U) When it was observed that thermalized rerolled pyrofoil would form blisters under rapid heat up conditions, despite previous high temperature processing, measurements were taken to determine how much growth was involved. In the range where the 2500°R temperature was measured, next to the heater for the thermalized-rerolled pyrofoil material, the outside layer of the 0.155-thick specimen assembly was measured by thermocouple to be 1300°R. The average material temperature was, therefore:

$$\frac{2500^{\circ}\text{R} + 1300^{\circ}\text{R}}{2} = 1900^{\circ}\text{R}$$

(U) From Figure 4-5 of TME-2722, Thermal Expansion Characteristics and Thermalized Rerolled Pyrofoil, the pyrofoil thickness is shown to have an average percent expansion of 1.2 percent at 1900°R for subsequent heat up cycles after volatilization of petrolatum is complete. The growth after the release of the compaction pressure (recorded in the test log sheets to be 15 to 30 psi) was measured in the free state to be 0.010 inch. The percent expansion from the original test condition was determined to be:

Change in specimen thickness at
Room Temperature, 530°R, in the
free state is, $\Delta t = .010$ inch

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Initial specimen
thickness prior to
startup of test
assembly, $t = .155$ inch

Percent expansion,
$$\frac{\Delta t}{t} \times 100 = \frac{.010}{.155} \times 100 = 6.5\%$$

(U) The multiple cycle thermal expansion of the thickness as the average pyrofoil material temperature heats up to 1900°R is thus: Initial growth + subsequent cycle growth;

$$6.5\% + 1.2\% = 7.7\%$$

(CRD) From DRM 51254 (ZrC-UC-Graphite-Materials Properties Data), it was determined that the expected maximum core expansion of Station 17.0 where the fuel periphery interface is 2500°R is .758 percent. The increase in core radius of 19.4755 inches is:

$$(.00758) (19.4755) \text{ inches} = .148 \text{ inch}$$

(CRD) From DRM 52533 (R-1 Core Radial Expansion of a High Expansion Graphite Core and a Composite Core), the 2.3 σ maximum radial thermal expansion based on 30 v/o UC-ZrC-C high expansion fuel is determined to be .352 inch. The lateral support system is designed to accommodate this maximum expansion. Because the core expands only .148 inch at Station 17.0 the remaining expansion capability of the lateral support system is the difference, or

$$\Delta t = .352 \text{ inch} - .148 \text{ inch} = .204 \text{ inch}$$

(CRD) In terms of allowable percent expansion of initial .155-inch of wrapper material thickness, this amounts to:

$$\frac{\Delta t}{t} \times 100 = \frac{.204}{.155} \times 100 = 132\%$$

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(U) The expansion of 7.7% for the thermalized rerolled pyrofoil is thus a small fraction of the total expansion available at Station 17.0 in the lateral support.

(CRD) The $150^{\circ}\text{R}/\text{sec.}$ ramp is characteristic of the aft end of the reactor where the maximum fuel material temperature is nominally 4450°R. At Station 17.0, where the significant thickness of pyrofoil ends, the temperature at the fuel interface is 2500°R. The ramp rate is proportionately reduced from $150^{\circ}\text{R}/\text{sec.}$ to $83^{\circ}\text{R}/\text{sec.}$ This is within range of the ramp rates ($75^{\circ}\text{R}/\text{sec.}$ to $100^{\circ}\text{R}/\text{sec.}$) achieved in the test.

(CRD) The test was originally designed for the R-1, 75 minute reactor, and was modified as design requirements were changed to support the current R-1, 10 hour, 60 cycle reactor. The current periphery design (Drawing 940J422) includes four thermalized layers of laminated pyrofoil bonded with petrolatum. As the previous design required 26 to 30 individual pyrofoil layers, the problem has been reduced an order of magnitude with relationship to the total weight of petrolatum involved.

(U) While the reactor configuration changed from the R-1, 75 minute to the R-1, 10 hour, 60 cycle reactor, the test assembly configuration was not changed because the laminated pyrofoil was not available and it was felt that the already assembled specimen would provide a more conservative test due to the larger weight of petrolatum involved.

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6.0 CONCLUSIONS

In drawing conclusions from the test results, it should be pointed out that a number of the test parameters applicable to the hot end of the wrapper in a 75 minute reactor could not be attained due to equipment limitations. The test conditions attained were compatible with the requirements at Station 17.0 of the current R-1, 10 hour reactor. The problem has been reduced in the current R-1 reactor an order of magnitude with relationship to the larger weight of petrolatum involved as the previous design required 25 to 29 layers of petrolatum between the individual pyrofoil layers.

Based on the results of tests on the present test specimen size and previous tests with small specimen size, there is no evidence of catastrophic damage due to thermal outgassing and vaporization of petrolatum bonded pyrofoil layers.

All specimens were examined microscopically and found to be free of tears or any products of decomposition such as ash. Blistering appears to be common in all test results. *This appears to provide a soft cushion that does not interact adversely with the lateral support system.*

	TITLE Outgassing and Vaporization Effect of Pyrofoil Layers Bonded with Petrolatum	
	DOCUMENT NO. WANL TME-2728	DATE 9/1/70
ABSTRACT Outgassing and Vaporization of decomposition products from a simulated Petrolatum bonded wrapper area of approximately 108 sq. in. of thermalized rerolled or unthermalized pyrofoil layers did not cause catastrophic bubbling, cracking, or significant thickness growth changes to pyrofoil assemblies for heat ramps up to 100° R/sec.		
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