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April 1969

Westinghouse Astronuclear Laboratory

WESTINGHOUSE ASTRONUCLEAR LABORATORY
CONTRIBUTION TO THE
TWENTY - EIGHTH HIGH TEMPERATURE FUELS
COMMITTEE MEETING
13,14,15 MAY 1969
(TITLE UNCLASSIFIED)

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SECTION 1

(U) INTRODUCTION

(U) A new series of reactor tests has been inaugurated since the last report to the High Temperature Fuels Committee. The small reactors, designated PEWEE-1 and PEWEE-2, are a joint venture with LASL. The reactors are intended primarily as tests of fuel development; although the core size and number of fuel elements is relatively small, each element is tested in an environment equivalent to that of full scale rocket reactors.

(U) As with the first of the series, PEWEE-1, the fuels tested in PEWEE-2 will represent experimental materials and variants of present production parameters. The results will be fed back for use in further PEWEE tests and ultimately in further rocket reactor tests.

(U) Electrically powered tests are still being carried on with individual fuel elements and fuel element clusters. These tests are intended to serve a variety of functions from exploration of degradation mechanisms to quality control. Not the least among these functions is the use of the tests for screening the large number of controllable parameters available in fuel element production and structure.

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SECTION 2

(CRD) RESULTS OF THE PEWEE-1 REACTOR TEST (U)

(U) The first test of the PEWEE series was run late in 1968. The reactor was disassembled at the reactor test site after the test and components sent to the hot cells at Los Alamos for detailed examination and evaluation. A large amount of information in the form of film strips and metallography is now becoming available. However, this report is based on a preliminary evaluation of less detailed data.

2.1 (CRD) PEWEE-1 Test Conditions (U)

(CRD) The test was intended to be one of multiple restart capabilities, not simple endurance. The first startup was a calibration run to about 90 percent of power with the exit gas temperature reaching about 2200°C for about two minutes. Subsequent behavior of the fuel may have been influenced by this short heat.

(CRD) Two 20-minute cycles were successfully run at full power. Another cycle was aborted because of the failure of peripheral structural components. During the full power runs, the exit gas temperatures were about 2300°C. No metallographic evidence of fuel migration indicating materials temperatures above 2475°C has been found.

2.2 (CRD) Westinghouse Fuel Elements in PEWEE-1 (U)

(CRD) A total of 130 Westinghouse produced fuel elements were tested in PEWEE-1. The parameters considered in this group were: additives to the graphite fuel matrix; fuel loading; and the process used for depositing the NbC protective coating. The numbers of elements of each type tested are shown in Table I*. The standard matrix designation refers to a composite of graphite flour, lampblack, and condensed Varcum similar to those used in previous reactors. Modifications of the standard matrix were made by impregnating with niobium resinate, or by blending in powdered niobium carbide (2 1/2 v/o) before extrusion.

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* Coating process designations are explained in the Appendix.

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TABLE 1

(CRD) BREAKDOWN OF WESTINGHOUSE FUEL ELEMENTS IN PEWEE-1 (U)

Fuel Loading, mg/cm ³		525	525	477	269	269
Matrix Type		Std.	Res.	Add.	Add.	Std.
Coated By	Coating Type					
Westinghouse	GED + Mo	35	17	24	17	
Westinghouse	GEM	6	6	6	13	
Y-12	GEM					6

Matrix Code:

- Std. - NRX-A6 type matrix; flour, lampblack, and Varcum
- Res. - Std. matrix impregnated with niobium resinate
- Add. - Std. ingredients with 2.5 v/o NbC powder added

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(CRD) The coatings were either of the type previously used with carbon supplied to the carbide by diffusion from the matrix (GED) or utilized carbon supplied in the gas phase as methane to form the carbide (GEM). The GED coatings were over-coated with molybdenum as in NRX-A6. A small number of Westinghouse elements were prepared with GEM coatings at the Y-12 plant. The coating profile for each of the processes used is shown in Figure 1.

(CRD) In addition to the usual internal coatings on the coolant channel surfaces, the PEWEE-1 elements were coated with NbC over their external surfaces. The axial load bearing surfaces at the hot ends of the elements were graphite-NbC composite bodies joined to the fuel element with an unfueled graphite transition piece by brazing.

2.3 (CRD) Fuel Element Performance in PEWEE-1 (U)

(U) Corrosion of the external surfaces of the fuel elements accounted for a much higher proportion of the overall weight loss than in previous reactors. This is attributed to reactor structure related patterns of hydrogen flow between the fuel elements rather than to fuel properties. A summary of preliminary weight loss data is presented in Table 2; these data represent an examination of about 75 percent of the fuel elements tested.

(CRD) One of the regions of high local weight loss due to external surface corrosion was about 10 to 15 inches from the cold, inlet end of the core. The attribution of this weight loss to external corrosion is based on bore appearance and other detailed visual examinations. An example of the appearance of a badly corroded element is shown in Figure 2. The mechanism of attack was apparently similar to the phenomenon labeled "midband" corrosion in the early NRX-A reactors, resulting from inadequate protection by the NbC coating. With GEM coats, the thickest external coatings (those on the Y-12 coated elements) gave the best external behavior and the thinnest coatings, the worst.

(CRD) Another point of surface damage in varying degree was on the non-fueled graphite transition piece of the hot end tips as shown in Figure 3. The degree of damage varied from no corrosion to very severe coolant channel exposure. Most

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TABLE 2

(CRD) PEWEE-1 GROSS WEIGHT LOSSES BY COATING/MATRIX COMBINATION

		Avg. Wt. Loss	Wt. Loss Range	No. in Core	No. Evaluated
		g	g		
Coating/Matrix (fuel Loading, mg/cm ³)					
GED+Mo/					
Std.	(525)	18.6	15.8-25.0	35	26
Res.	(525)	15.9	13.8-18.2	17	15
Add.	(477)	18.6	15.2-22.6	24	20
Add.	(269)	16.9	15.8-18.5	17	16
Westinghouse GEM/					
Std.	(525)	32.4	22.8-44.5	6	3
Res.	(525)	18.0	13.8-21.7	6	5
Add.	(477)	31.8	31.8	6	1
Add.	(269)	27.4	16.3-45.7	13	9
Y-12 GEM/					
Std.	(269)	10.6	9.8-12.0	6	6

* Includes both surface and bore corrosion. Correction for surface losses will indicate fuel element performance in terms comparable to the results of previous reactor tests and electrically powered corrosion tests.

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of the damage appeared to originate on the external surfaces near the joint between the graphite transition piece and the composite tip. Detailed studies of the extent of tip corrosion on each face of 114 elements have shown that one of the major factors influencing tip corrosion was, again, the thickness of the external coating. Of the Westinghouse supplied elements, the GEM coated elements, which had the thinnest (about 0.3 mils) external coatings, exhibited the worst tip corrosion; the thicker (≥ 0.8 mils) GED coatings exhibited good performance; and the Y-12 GEM coated elements, which had the thickest (1.4 to 1.9 mils) coatings gave the best performance. When GEM and GED coatings were of approximately the same thickness, similar severity of tip corrosion was observed.

(CRD) A limited number of the PEWEE-1 fuel elements have been sliced open to observe bore corrosion characteristics. Conclusions concerning bore behavior are considered preliminary pending a more complete examination. Behavior of the GED plus Mo coated elements appeared to be similar to that observed with this type of element in the NRX-A6 core; midband effects were mostly absent, and very small internal corrosion pockets were found about 40 to 50 inches from the inlet end.

(CRD) The GEM coated elements gave evidence that fuel bead hydrolysis had occurred causing points of damage to the bore coatings. Internal corrosion pockets were found which were similar in size, appearance, and location to those found in deliberately hydrolysed and corrosion tested (with electric power) PEWEE-1 type elements. These pockets were the major cause of pinholing of GEM coated elements. The GEM coated elements with 2.5 v/o NbC additive showed the worst hydrolysis effects. Aside from the hydrolysis effects, the bores of the Westinghouse elements behaved well.

(CRD) Another, but less severe, component of tip corrosion was of internal origin and occurred in the transition piece at or near either of the braze joints to produce pinholes or holes. The graphite-NbC composite tips appeared unaffected by either internal or external corrosion.

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SECTION 3

(CRD) OUT OF REACTOR FUEL DEVELOPMENT (U)

(CRD) The number of materials modifications and variations of production parameters available to the NERVA fuel system is too great to permit evaluation of any but a few of the possibilities in reactor tests. Therefore, electrically powered corrosion tests are used to correlate physical properties with corrosion behavior, to test experimental materials, to investigate specific problem areas and phenomena, and to screen potential reactor quality fuel elements. Thus, only the most promising experiments are carried to reactor testing. Among the materials, processes, and phenomena now under consideration in this phase of the program are: graphites with thermal expansion characteristics similar to those of the carbide protection coatings; additives to promote graphitization of the fuel matrix binder phase; various processes for depositing NbC coatings; ZrC coatings deposited over a NbC basecoat by various processes as a hot end barrier to carbon diffusion; and the origin of fuel bead hydrolysis damage. The goal of the program is the development of fuel elements having a multiple restart capability with low corrosion losses. Therefore, most corrosion testing is done as series of ten minute cycles, typically with four cycles per test, under simulated reactor thermal and flow conditions.

3.1 (CRD) Summary of Recent Corrosion Data (U)

(CRD) A variety of fuel element types, ranging from the NRX-A6 type to high expansion ZrC coated types, have been corrosion tested. It is of interest to examine comparative corrosion performance of the various types subjected to four 10 minute cycles in order to realize the fuel improvements since the NRX-A6 element. The results will be discussed in terms of hot end (station 35-50) and midband (station 0-35) behavior.

(CRD) A summary of hot end corrosion data after four 10 minute test cycles for the major types of elements tested is shown in Table 3. The average is shown, along with the lowest value achieved. The weight loss results clearly show that both ZrC coated elements on low or high expansion matrices, and high expansion matrices, and high expansion elements with or without ZrC are superior to the NRX-A6 type of fuel element or to the GEM NbC - low expansion element.

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TABLE 3
(CRD) HOT END CYCLIC HYDROGEN CORROSION
TEST PERFORMANCE OF SOME RECENT FUEL ELEMENTS (U)

Coating	Matrix ^(a)	Four 10 Minute Cycles Weight Loss, 35 - 50 Inches (grams)	
		Lowest	Typical
HED + Mo Overcoat (NRX - A6 Type)	LE	8.6	10
GEM	LE	4.8	7
GEM + GED NbC Overcoat (LASL PW-1 Type)	LE	4.2	6
GEM + GEM ZrC Overcoat	LE	4.6	5.5
GEM + GEM ZrC Overcoat	LE	2.1	4.8
GEM + HED ZrC Overcoat	LE	4.1	5.1
GEM + GED + GEM ZrC + GEM ZrC	LE	2.4	--
GEM	HE 75% POCO	2.3	3.7
GEM	HE 100% POCO	3.8	4.2
GEM + GEM ZrC Overcoat	HE AXM/Gil.	5.7	6.0
GEM + GEM ZrC Overcoat	HE 75% CP-3	3.3	3.7
GEM + GED ZrC Overcoat	HE 75% POCO	--	3.9
GEM + GED ZrC Overcoat	HE 100% POCO	2.4	3.4
GEM + GED ZrC Overcoat	HE 75% CP-3	--	4.0

(a) LE = Low Expansion; HE = High Expansion

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(CRD) Qualitative hot end ratings of the various types, based upon visual appearance after test is shown in Table 4. These results show that high expansion matrices were those most free of gross corrosion; however certain ZrC coatings on low expansion matrices also gave very good results.

(CRD) Observations of some axially sliced corrosion tested fuel elements have shown a tendency for the coatings to blister. The blisters have occurred with both low and high expansion matrices. Limited metallographic studies of the blisters have shown that they occur in one of two basic forms, coating-coating or coating-matrix. The coating-coating type usually starts as a separation of the ZrC coating into two layers of roughly 1/3 and 2/3 of the original thickness, with subsequent blistering when the outermost ZrC layer bows into the coolant channel. This type of blister usually extended into the channel for less than five mils and were generally found in this region between stations 30 and 45. A few coating-coating blisters have been observed as starting with delamination between the ZrC and NbC coatings.

(CRD) The most detrimental of the blisters as far as effects on corrosion behavior is the coating-matrix type. Some of these blisters have been very large, being up to one inch long and blistered to the extent of almost closing a bore. In this case, the combined NbC-ZrC duplex coating bends away from the matrix leaving a large void between the coating and graphite substrate. This type of blister occurs only at positions where fuel bead migration is also observed. A layer of pyrocarbon is usually observed on the underside of the coating and it is believed this layer formed when the corrosion products decompose at the coating interface. Deposits up to 15 mils have been observed and the shape of the deposits indicate that the blister occurred very early in the test, probably during the startup ramp.

(CRD) The midband performances of all of the types shown in Table 3 were all good, typical weight losses being 1 to 3 grams, and the results primarily depending upon coating batch. The NRX-A6 coating type, HED + Mo, was found to be less effective in preventing "center bore" corrosion than the other types.

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TABLE 4

(CRD) RATING OF HOT END PERFORMANCE CHARACTERISTICS
OF VARIOUS TYPES OF DEVELOPMENTAL FUEL ELEMENTS (U)

Coating Type	Hot End Performance	
	Low Expansion	High Expansion
HED + Mo, NRX-A6 Type	Fair	--
GEM, PW-1	Good	Excellent
GEM + GED NbC LASL PW-1	Good	--
GEM + HED ZrC	Very Good	--
GEM + GED ZrC	--	Excellent
GEM + HEM ZrC	Very Good	--
GEM + GEM ZrC*	Poor	Poor
GEM + GEM ZrC	--	Excellent

* Results "Poor" because of coatings blistered in the hydrogen corrosion test to cause pinholes and hot end corrosion failures. Further work is required in order to achieve the desired ZrC coat microstructure.

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3.2 (CRD) Hydrolysis Studies (U)

(CRD) Detailed studies of the effects of hydrolysis upon GEM coated fuel elements have shown that beads at the coating-matrix interface are affected. Metallography studies suggest that the core of a fuel bead became exposed to atmospheric moisture because a coating crack penetrated into the UC_2 . When hydrolysis occurred the resulting expansion of the core caused the coating directly above the bead to flake; local corrosion pockets and pinholing resulted when the element was corrosion tested.

(CRD) Detailed studies of flaked bore coating in both Y-12 and WANL GEM coated PW-1 type fuel elements which were subjected to uncontrolled hydrolyzing storage have shown that flaking was related to coating thickness, i.e., the number of coating cracks, and not coating manufacturer. For each manufacturer flaked bore occurred at a frequency of 1 flake per 5 lineal bore inches at a coating thickness below 1.3 mils.

(CRD) The Y-12 elements which had relatively thick midband coatings experienced flaking only at low stations where corrosion test temperatures were low and therefore these elements had only a small number of corrosion effects caused by flaking. The Westinghouse elements, which had thin midband coatings, showed flaking up to approximately station 25 where test temperatures were high and therefore corrosion testing produced internal corrosion pockets and pinholes in these elements. The pockets dropped to a minimum of 0 - 1 pocket per 5 lineal bore inches at coating thicknesses exceeding 1.7 mils.

(CRD) Fueled elements from four Y-12 NRX-A6 coating batches were tested to determine if the pinholes observed in the reactor were due to fuel bead hydrolysis. The tests were conducted at either typical hydrogen-helium differential pressures (hydrogen overpressure of 30 psi at station 20), or high hydrogen overpressures (80 psi at station 20). In addition, an element purposely hydrolyzed at $95^{\circ}C$ and approximately 100 percent relative humidity was tested. The results are given in Table 5.

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TABLE 5
(CRD) RESULTS OF NRX-A6 PINHOLING EXPERIMENT (U)

Element	Batch	Time	ΔP at Station 20 (H_2 - He)	ΔW	Avg. Pinholes in Reactor	Pinholes in Test
16-64404	8713/1613	30+30	30	12.7	8.2	0
20-66498	8723/1623	30+30	80	14.5	9.0	19
20-66486	0734/1634	30+30	80	11.5	15.0	0
21-67757	8742/1642	30+30	30	12.7	9.0	0
16-64402*	8713/1613	17.6	71	37.4	8.2	83

* Hydrolyzed at 95°C, 100% R.H.

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(CRD) The conclusions based on the above test results and sliced element evaluations are:

1) Hydrolysis of fuel beads may have been a contributing factor to pinholding in the Y-12 NRX-A6 reactor elements. (Although these elements were molybdenum overcoated and hydrolysis would not normally be expected, the coating crack structure and adherence was such that the molybdenum may not have effectively sealed off access areas for hydrolyzing atmospheres).

2) Based on the limited test data above, it appears that the magnitude of the hydrogen overpressure (30 psi versus 80 psi) did not significantly affect the corrosion behavior of these elements. Batch 8723 which pinholed in the above tests, also showed marginal corrosion behavior by visual appearance in the qualification test indicating that elements from this batch may have experienced early degradation rather than an effect of overpressure.

(CRD) The hydrolysis test performed on elements from three of the above batches resulted in gross hydrolysis damage at the joint area. The hydrolysis in this area occurred in fuel beads that were migrated during the tip brazing operation and resulted in severer cracking of the matrix similar to that observed in many of the Y-12 elements produced for the PW-1 core.

3.3 (CRD) High Expansion Graphite Matrices

(CRD) The purpose of this experiment was to evaluate the performance characteristics of LASL ZrC coatings upon WANL and LASL high expansion matrices. The LASL elements were coated at LASL using a GEM NbC plus GEM ZrC process. The Westinghouse elements received GEM NbC plus GED ZrC coatings and a Mo overcoat.

(CRD) The WANL matrices were manufactured of POCO AXZ flour (both 100% and 75%). The LASL matrices were of Robinson coke (75%); a low expansion control group of elements (M-3 flour) were also made by LASL.

(CRD) The corrosion tests were performed by WANL. Most of the elements exhibited at least capability for three 10 minute cycles, and some four cycles. Failure of the elements to reach the test target of four cycles was usually caused by

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end face regression of the WANL elements (graphite non-fuel tips), and breaking at the composite-fuel joint of the LASL elements. The results showed:

- 1) Midband performance, in general, was good.
- 2) Hot end performance of all elements including the low expansion elements was good, typical weight losses being less than about one gram per cycle.
- 3) The best hot end performance was of the high expansion elements, WANL or LASL, coated by the GEM plus GED process.

3.4 (CRD) Catalyzed Binder Study (U)

(CRD) Some preliminary results have been obtained to increase the degree of graphitization of binder carbon for the varcum system. Relying on a time-temperature treatment of the carbon is prohibitive since temperatures of greater than 3000°C are necessary to initiate this transformation in a hard carbon. This process may be effected, however, through the addition of additives to catalyze the graphitization at a lower temperature. This catalyzing agent may be any of a number of metallic elements or components, or even a soft carbon in some cases.

(CRD) Preliminary investigations indicated the prime metallic candidates to be elemental aluminum and/or copper. X-ray diffraction studies revealed that these two did institute graphitization at temperatures below 2100°C. Peak position, shape, height, and width showed a decrease in interlayer spacing, increased crystallographic ordering, an increase in percent graphitization, and an increase in crystallite size.

(CRD) Aluminum was the most successful agent used. Additions of >3% AL increased the percent graphitization from an original value of about 26 percent to values of ≥90 percent. A mixture of AL and Cu also yielded good results. Judging from the curves and evaluations, percentages of AL and/or Cu of 3 - 7 percent will be sufficient to catalyze the process.

(CRD) In order to more fully evaluate the effects of additions on strength, CTE, etc., batches of 0.375 inch x 12 inch diameter rods have been extruded to provide samples for analysis. These have been cured, baked, and graphitized

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and are in the process of being sectioned. In addition to metallic catalyzing agents, batches were also extruded to study pitch-varcum combinations, graphitized thermax, variations in thermax content, and effects of sized flour.

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APPENDIX 1

(CRD) EXPLANATION OF COATING PROCESS DEFINITIONS (U)

- GED - A carbide deposition process with gas flow from the end of the fuel element corresponding to the reactor inlet (GE). Carbon provided by diffusion from the graphite substrate (D).
- HED - Similar to GED, but with the coating gases flowing from the reactor exit end of the fuel element (HE). This flow pattern tends to give a thicker hot end coating.
- GEM - A carbide coating process with gas flow from the reactor inlet end (GE), but with carbon provided in the gas phase by methane (M) addition to the coating gases.
- Q - A molybdenum coating deposited by thermal decomposition of molybdenum carbonyl. This coating is deposited over a carbide coating to seal cracks.
- X - ZrC. Standard coating designations refer to NbC. However, ZrC coatings are of interest for hot end protection as a barrier to carbon diffusion. Therefore, ZrC coatings are being tested both alone and in conjunction with NbC coatings. The ZrC deposition processes are analagous to those for NbC.
- GEM+GEM - GEM NbC coat with a GEM ZrC coat deposited over it.
- GEM+GED - GEM NbC coat with a GED ZrC coat deposited over it.

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Figure 1. (CRD) Average Bore Coat Profiles of Westinghouse Supplied Fuel (U)

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Figure 2. (CRD) Example of Severe Corrosion of an External Fuel Element Surface in the Midband Region (U)

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Figure 3. (CRD) Example of External Corrosion Local
to the Tip Transition Piece (CRD)

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