**Westinghouse
Astronuclear
Laboratory**

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WANL-TME-2723
SEPTEMBER 1970

DATA ITEM T-119

SHIELD ELEMENT CORROSION

FINAL REPORT

MASTER

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Westinghouse Astronuclear Laboratory

Subcontract NP-1
CODE IDENT NO. 14683

WANL-TME-2723
SEPTEMBER 1970

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ABSTRACT

Specimens of BATH shield material were tested to determine their resistance to hydriding, dehydriding, nitriding, and contact corrosion with structural material when exposed to flowing hydrogen or nitrogen gas at a pressure of 600 psia and a temperature of 900°R for a period of two hours.

Five specimens were tested in hydrogen and nitrogen gas. Two specimens were used for control. The specimens tested were in contact with 6061-T6 aluminum at one end and with type 304 stainless steel at the other end.

Test results indicate no corrosion or decomposition of the specimens or holders. There was no significant change in hydrogen or nitrogen content after a 2-hour exposure at the above conditions. Further, no surface spalling or debris generation of particles occurred greater than .007-inch. Based on these results, BATH shield development material in combination with 6061-T6 aluminum and Type 304 stainless steel should meet the requirements of Paragraph 3.3.6 when exposed to a more severe thermal environment than specified in Paragraph 3.1.2.4.2.3 of ECC-677562, dated July 16, 1970.

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1.0 INTRODUCTION

1.1 TEST IDENTIFICATION

1.1.1 Type of Report

This is a final report.

1.1.2 Applicable Scope of Test Document

The Applicable Scope of Test Document is 62DT-190, and Rev. 1, " Shield Element Corrosion Test. "

1.1.3 Functional Category of Test

This is a development type test to demonstrate corrosion resistance of the shield materials in contact with reactor structural material when exposed to a reactor environment.

1.1.4 Contractor

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

1.1.5 Test Facility and Location

WANL Test Cell 5, Large, Pa., during the period October 29 to November 25, 1969.

1.2 PURPOSE OF TEST

This test is intended to demonstrate satisfactory corrosion resistance of the shield materials in contact with reactor structural material when exposed to reactor environment. The results of this test will indicate that the shield material is corrosion resistant in reactor environments in either of two modes of corrosion:

Surface corrosion including hydriding, dehydriding or nitriding,
Contact corrosion or electrolytic corrosion.

2.0 REFERENCES AND DOCUMENTATION

2.1 REFERENCES

2.1.1 Specification

ECC-677562, Paragraph 3.1.2.4.2.3

2.1.2 Data Item Description

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

2.1.3 Scope of Test Document.

62DT-190- Shield Element Corrosion Test, and Revision 1, dated February 23, 1969.

2.1.4 Drawing List

Drawing No. 100E807, Sheet 1, Shield Corrosion Test Fixture Assembly.

2.2 DOCUMENTATION

	<u>Document No.</u>	<u>Date</u>	<u>Dept.</u>	<u>Subject</u>	<u>Purpose</u>
2.2.1	<u>Procedures</u>				
	TFLP-109	August 18, 1969	Thermal & Fluid Flow Laboratory	Flow Tests in Cell 5	To present the approved steps for the proper operation of Test Cell 5 flow experiments.

2.2.2 Test Plan

DRM 51303, issued 4/10/69.

2.2.3 Test Scope

The original scope of test document was transmitted under letter RD-1332, dated January 23, 1969, and Revision 1 was transmitted under letter RD-1382, dated February 13, 1969.

2.2.4 Data Release Memorandums

1. DRM 51419, Preliminary Results for End of Coodown
2. DRM 52943, Temperature Distributions Within a Non-Rotating Vehicle.

3.0 DESCRIPTION OF TEST ARTICLE

The test article consisted of 12 BATH cylindrical specimens 2-inches in length by 1/2-inch diameter, as per Item 3 of Drawing 800C507. See Figure 3-1 (2 sheets) for pre-test photographs of the specimens. Material for the specimens was ordered under Purchase Requisition No. 94412, 96064, and 94409. Pressing of the material was performed by Sylvania Corp., under Purchase Requisition 97264, and the extruding into bars was done by Alcoa Corp., under Purchase Requisition 97265. This was the second lot of development material procured.

Five specimens were tested in a hydrogen atmosphere; five were tested in a nitrogen atmosphere; and two specimens were reserved for control purposes.

The test fixture for this test series is represented schematically in Figure 3-2. The BATH specimens were held in place by two holders, one of which was fabricated from Type 304 stainless steel and the other from 6061-T6 aluminum. The holders were fabricated with four "fingers" machined to provide a slip fit with the specimen outside diameter. Each of the fingers was drilled to provide passage for the gas flowing over the specimen outer surface. At temperature, the specimens were to expand and provide face-to-face contact with the holders.

A screen assembly consisting of 0.007 inch mesh mounted at the exit of the fixture completes the design. This mesh corresponds to that used at the fuel element inlet.

Figure 3-1. Pretest Specimens (Sheet 1 of 2)

Figure 3-1. Pretest Specimens (Sheet 2 of 2)

Figure 3-2. Shield Element Corrosion Test Fixture

4.0 SUMMARY OF TESTS

4.1 NARRATIVE

Prior to the start of testing, an aluminum specimen was fabricated to the same dimensions as the BATH specimens. The aluminum specimen was used in checking out the assembly of the fixture, and for the facility flow loop checkout test.

Prior to the start of testing each of the BATH specimens was weighed on an analytical balance. Pre- and post-test weights are presented in Section 4.3, and are the average of three separate weighings.

Pre- and post-test photographs of the specimens ends are presented for the ends in contact with the aluminum and stainless steel holders.

For the actual testing steady-state run time began when T_{out} reached 415°F. Startup and cooldown times are included in the Test Results, Section 4.3.

It must be noted that the start-up times for the nitrogen tests were much longer than for the hydrogen tests. This is due to the difference in heat capacities between the two gases. The first five tests, Specimens A85906 through A85910, were run with hydrogen with the pressure vessel un-insulated. Approximately 20 - 25°F temperature difference existed between T_{in} and T_{out} for the hydrogen series.

However, the first nitrogen test, Specimen A85913, produced a very slow start-up and a large difference in $T_{in} - T_{out}$ of approximately 130°F at a time of 2 hours and 40 minutes. At this point the decision was made to begin steady-state and control on the average of T_{in} and T_{out} . Subsequently the fixture was insulated to reduce heat loss and the remainder of the tests reached steady-state conditions in approximately 1 hour and 20 minutes. The control temperature remained the average of T_{in} and T_{out} .

No debris was trapped in the 7 mil screen in any of the tests.

4.2 TEST CONDITIONS

The following conditions were used for the hydrogen and nitrogen corrosion tests:

Gas temperature:	$900^{\circ}\text{R} \pm 25^{\circ}\text{R}$
Pressure:	$600 \text{ PSIA} \pm 10 \text{ PSIA}$
Flow Rate:	0.01 lb/sec.
*Material temperature:	$900^{\circ}\text{R} \pm 25^{\circ}\text{R}$
Time:	2 hours at $900^{\circ} \pm 25^{\circ}\text{R}$

- * NOTE: Material temperature was assumed to equal exit gas temperature or the average of T_{in} and T_{out} .

4.2.1 Discussion of Test Conditions

In the following paragraphs the Laboratory Test conditions of the BATH Specimen will be compared with various shield rated operating conditions expected during the life of the reactor. The shield reactor conditions shown in Table 4-1 were extracted from the reactor State Point for the graphite core, Drawing 957A979.

TABLE 4-1
COMPARISON OF TEST CONDITIONS WITH REACTOR CONDITIONS

	Test Condition Shield Speciman	Rated Power	Reactor Condition Cooldown* Storage*	
Gas Temperature	$900^{\circ}\text{R} \pm 25$	275°R		
Pressure	$600 \pm 10 \text{ psia}$	647 psia		
Flow Rate	$.01 \text{ lbs/sec}$	91.2 lbs/sec		
Mass Velocity	$.2 \text{ lbs/sec in}^2$	$.8 \text{ lbs/sec in}^2$		
Maximum Temperature	900°R	400°R	850°R	540°R
Time	2 hrs	(1)	30 hrs	1 year

(1) Normal mode endurance 600 minutes at rated temperature (accumulated in up to 60 cycles of varying duration up to 60 minutes maximum per cycle).

* Estimated range of temperatures from perliminary analysis.

Comparison of the test and reactor temperature environment of the BATH material in Table 4-1 shows that the 900°R test temperature is much higher than the 575°R rated operation gas temperature environment within the reactor shield region. Thus, from a chemical corrosion standpoint (reaction of BATH material with H₂ environment) the component test represents an over test. The chemical reaction rates, such as one would expect in this case, generally increase rapidly with increasing temperature if reacting specie availability is assumed on the reactivity surface.

To assure that active H₂ is available to the reacting surface, hydrogen was continually fed to the laboratory specimen. The mass reactivity in the test configuration flow channel was 0.2 lb H₂/sec. in.². This compares with a mass velocity of 0.8 lb H₂/sec. in.² through the reactor shield coolant channels. The corresponding linear velocity through the test and reactor flow channels was 234 ft/sec and 266 ft/sec respectively. The linear velocity corresponds on almost a one-to-one basis, test to reactor conditions, as shown in Table 4-1. Thus, in comparison of mass velocities and linear velocities, it is seen that the availability of hydrogen to react with, at a much elevated temperature, the BATH material surfaces was assumed in the test. The difference in mass velocity between 0.2 lb/sec. in.² in test and the 0.8 lb/sec. in.² in the reactor flow channels will have negligible affect upon availability of hydrogen to the BATH surface, since in both cases, the flow channels are operating in the turbulent flow regime.

Because the shield material is also subjected to reactor operating environment other than rated conditions, specifically cooldown and storage environment, the test conditions are also compared in Table 4-1 with estimated reactor temperature conditions, during these modes of operation.

The chemical corrosion of the BATH material during cooldown and space soak is judged to be minimal because in the space environmental pressure of 10⁻⁹ torr, the availability of hydrogen reactants is only that residual amount remaining in the reactor shield region.

The comparison of the cooldown and space soak temperatures of BATH and the test conditions are also of interest in assessing the compatibility of the BATH material with its supporting Al structures. This was also an objective of the corrosion test.

As shown on Table 4-1, the test temperature of 900°R exceeds both the estimated maximum temperatures of the reactor shield during cooldown (850°R) and space soak (545°R).

The maximum temperature during cooldown, will occur between 17 and 25 hours after the last cooldown pulse.

In summary, comparisons of test environment vs reactor environment show that for chemical reaction of H_2 with BATH material:

- a. Temperature levels of Test (900°R) are very extreme relative to reactor conditions, thus extreme corrosion rates would result if BATH were shown to be reactive with H_2 .
- b. H_2 availability for reaction to the BATH surface was assumed by maintaining sufficient H_2 flow.
- c. The test time (2-hours) does not meet the present NERVA 10 hour requirement. However, the test results are valid from a corrosion rate indication and applicable to the 10-hour reactor.
- d. The 900°R test temperature is compatible with the maximum temperature range (850°R) exacted during cooldown and above the 545°R expected space soak temperature. Thus the test results will be indicative of the BATH material compatibility with its supporting structure for two hours of testing. Although the two hours of testing is not adequate to fully judge the time effects of materials compatibility, it is judged sufficient to identify any major materials incompatibility problem.

4.3 TEST RESULTS

4.3.1 Recorded Data

Table 4-2 presents test data summary of conditions imposed on specimens for two hours. The photographs (2x magnification) on Figures 4-1 through 4-4 show pre- and post-test conditions of the ends in contact with the structural materials. Table 4-3 lists pre- and post-test specimen weights. Table 4-4 lists the pre- and post-test specimen chemistry.

4.3.2 Discussion of Data

Extracted from the Scope of Test document, page 2, is a tabulation of required data for all tests:

- a.) Gas temperature
- b.) Gas pressure
- c.) Gas flow rate
- d.) Material temperature
- e.) Duration of test
- f.) Surface comparison of sample before and after test. (Both surface exposed to gas and surface in contact with structural material.) The comparison may be made by photographs, profilometer or other.
- g.) Analyze debris, if any, caught on flow screen. Spectrographic analysis is acceptable. Screen opening .007-inch.
- h.) Chemistry of sample before and after test to determine gain or loss of hydrogen or nitrogen. (Chemistry or crystallography or other techniques acceptable)
Check weight change or sample.

Comparison with data of Section 4.3.1 show full compliance with Scope of Test requirements.

PRE-TEST

POST-TEST

Figure 4-1. Ends in Contact With AISI Type 304 Stainless - GH_2 Gas
(Specimens A85906-A85910)

PRE-TEST

POST-TEST

Figure 4-2. Ends in Contact with 6061-T6 Aluminum - GH_2 Gas
(Specimens A85906-A85910)

PRE-TEST

POST-TEST

Figure 4-3. Ends in Contact With AISI Type 304 Stainless - GN_2 Gas
(Specimens A85913-A85917)

PRE-TEST

POST-TEST

Figure 4-4. Ends in Contact With 6061-T6 Aluminum - GN_2 Gas
(Specimens A85913-A85917)

TABLE 4-2
TEST DATA SUMMARY

Specimen A85906 - GH₂

<u>Time (min.)</u>	<u>P_{in} (PSIA)</u>	<u>T_{out} (°F)</u>	<u>Ẇ (lb/sec.)</u>
0	567.65	415	.0094
15	569.65	442	.0096
30	567.65	440	.0098
45	565.65	440	.0099
60	567.65	442	.0099
75	565.65	442	.0100
90	565.65	442	.0101
105	568.65	443	.0101
120	569.65	443	.0102

NOTE: Start-up Time: 23 min.
 Cooldown Time: 40 min.

Specimen A85907 - GH₂

0	604.48	425	.0102
10	605.48	460	.0102
20	608.48	455	.0103
30	607.48	457	.0103
40	605.48	457	.0104
50	603.48	458	.0105
60	603.48	450	.0105
70	601.48	465	.0106
80	602.48	442	.0107
90	600.48	435	.0107
100	596.48	430	.0107
110	593.48	430	.0107
120	593.48	430	.0107

NOTE: Start-up Time: 19 min.
 Cooldown Time: 40 min.

TABLE 4-2 (CONTINUED)
Specimen A85908 - GH₂

<u>Time (min.)</u>	<u>P_{in} (PSIA)</u>	<u>T_{out} (°F)</u>	<u>Ẇ lb/sec.)</u>
0	602.36	418	.0107
10	600.36	435	.0102
20	601.36	442	.0103
30	600.36	450	.0104
40	602.36	450	.0104
50	602.36	450	.0105
60	604.36	455	.0106
70	604.36	455	.0106
80	604.36	455	.0107
90	604.36	455	.0107
100	603.36	455	.0107
110	600.36	455	.0107
120	599.36	455	.0107

NOTE: Start-up Time: 20 min.
 Cooldown Time: 40 min.

Specimen A85909 - GH₂

0	605.34	417	.0099
10	605.34	435	.0100
20	604.34	440	.0101
30	603.34	445	.0101
40	603.34	450	.0101
50	600.34	450	.0102
60	599.34	450	.0102
70	599.34	450	.0102
80	599.34	450	.0102
90	596.34	452	.0103
100	596.34	450	.0103
110	595.34	450	.0103
120	594.34	450	.0103

NOTE: Start-up Time: 20 min.
 Cooldown Time: 40 min.

TABLE 4-2 (CONTINUED)

Specimen A85910 - GH_2

<u>Time (min.)</u>	<u>P_{in} (PSIA)</u>	<u>T_{out} (°F)</u>	<u>$\dot{W}$ (lb/sec.)</u>
0	613.29	418	.0099
10	612.29	421	.0100
20	612.29	440	.0101
30	610.29	450	.0102
40	610.29	455	.0102
50	609.29	455	.0103
60	610.29	455	.0103
70	609.29	455	.0103
80	610.29	455	.0103
90	610.29	455	.0105
100	609.29	455	.0105
110	609.29	455	.0105
120	608.29	455	.0105

NOTE: Start-up Time: 20 min.
Cooldown Time: 40 min.

Specimen A85913 - GN_2

0	614.54	469	.0096
10	614.54	469	.0095
20	614.54	469	.0096
30	614.54	472	.0096
40	614.54	472	.0097
50	614.54	474	.0097
60	614.54	474	.0096
70	614.54	475	.0096
80	614.54	477	.0096
90	614.54	477	.0096
100	614.54	477	.0096
110	614.54	476	.0097
120	619.54	479	.0096

NOTE: Start-up Time: 2 hrs. 40 min.
Cooldown Time: 2 hrs. 50 min.

TABLE 4-2 (CONTINUED)

Specimen A85914 - GN₂

<u>Time (min.)</u>	<u>P_{in} (PSIA)</u>	<u>T_{out} (°F)</u>	<u>Ẇ (lb/sec.)</u>
0	614.25	476	.0098
10	613.25	481	.0098
20	613.25	478	.0098
30	613.25	481	.0098
40	610.25	476	.0098
50	611.25	471	.0098
60	611.25	468	.0098
70	611.25	459	.0098
80	611.25	459	.0098
90	613.25	455	.0098
100	613.25	460	.0098
110	613.25	460	.0098
120	613.25	460	.0098

NOTE: Start-up Time: 1 hr. 19 min.
 Cooldown Time: 2 hrs. 15 min.

Specimen A 85915 - GN₂

0	611.47	478	.0098
10	611.47	478	.0099
20	611.47	463	.0099
30	610.47	459	.0099
40	611.47	459	.0098
50	611.47	459	.0098
60	611.47	459	.0098
70	611.47	457	.0099
80	610.47	459	.0099
90	610.47	457	.0098
100	610.47	459	.0098
110	610.47	458	.0099
120	610.47	459	.0098

NOTE: Start-up Time: 1 hr. 45 min.
 Cooldown Time: 1 hr. 22 min.

TABLE 4-2 (CONTINUED)

Specimen A85916 - GN₂

Time (min.)	P _{in} (PSIA)	T _{out} (°F)	Ẇ (lb/sec.)
0	609.58	482	.0101
10	609.58	474	.0103
20	609.58	474	.0103
30	609.58	467	.0103
40	609.58	467	.0103
50	610.58	465	.0104
60	611.58	465	.0105
70	611.58	465	.0105
80	611.58	464	.0107
90	611.58	464	.0107
100	610.58	466	.0106
110	610.58	466	.0104
120	611.58	466	.0103

NOTE: Start-up Time: 1 hr. 18 min.
Cooldown Time: 2 hrs.

Specimen A 85917 - GN₂

0	613.6	482	.0101
10	610.6	471	.0103
20	611.6	471	.0103
30	611.6	472	.0103
40	611.6	468	.0102
50	611.6	466	.0102
60	611.6	466	.0102
70	611.6	470	.0102
80	611.6	452	.0102
90	611.6	452	.0102
100	612.6	452	.0102
110	612.6	449	.0103
120	612.6	446	.0102

NOTE: Start-up Time: 1 hr. 26 min.
Cooldown Time: 2 hrs.

TABLE 4-3
PRE-AND POST-TEST SAMPLE WEIGHT

<u>Sample No.</u>	<u>Pre Test Wt. (gms)</u>	<u>Post Test Wt. (gms)</u>
A85906	18.782	18.783
A85907	18.820	18.819
A85908	18.841	18.841
A85909	18.640	18.640
A85910	18.796	18.795
A85913	18.823	18.823
A85914	18.796	18.795
A85915	18.768	18.767
A85916	18.639	18.638
A85917	18.740	18.738

NOTE: Weights are average of 3 readings. Uncertainty on balance is $\pm .002$ gm.

TABLE 4-4

PRE-AND POST-TEST SPECIMEN CHEMISTRY

The chemical analysis of these specimens was performed by the Analytical Service Laboratory/Westinghouse ARD, Waltz Mill Site under IWR-10977.

Hydrogen Analysis

<u>Tested Specimen</u>		<u>Control Specimen</u>	
<u>No.</u>	<u>% H₂</u>	<u>No.</u>	<u>% H₂</u>
A85906	1.155	A85912	1.153
A85907	1.157		
A85908	1.153		
A85909	1.157		
A85910	1.153		

The above analysis was obtained by employing the "Inert Gas Combustion" method.

Nitrogen Analysis

<u>Tested Specimen</u>		<u>Control Specimen</u>	
<u>No.</u>	<u>PPM</u>	<u>No.</u>	<u>PPM</u>
A85913	664	A85912	609
*A85914	585		
*A85915	540		
*A85916	641		
*A85917	623		

*The samples from these specimens were analyzed by the same technique as A85912 and A85913, but the samples were obtained from the specimens by a different manner.

The above analysis yields parts-per-million of total nitrides by the Kjeldahl method. A complete discussion of this method and the inert gas combustion method used for hydrogen analysis can be obtained from DRM No. 51229, Rev. G.

5.0 ANALYSIS

Test specimens were analyzed for hydrogen and nitrogen content. Control specimens were also analyzed for comparative purposes.

Analysis for nitrogen was by the Kjeldahl method. Hydrogen was analyzed by the inert gas combustion method. A discussion of the analysis can be found in DRM 51229G.

In summary, analysis of the hydrogen and nitrogen contents from above disclosed that Bartlett's test for homogeneity of variances was not significant between the specimens subjected to H_2 or to N_2 , and between the control specimens and the specimens subjected to H_2 or N_2 . Also, analysis of variance disclosed no significant differences in the means. Therefore, based on 99% probability at 95% confidence level, there was no significant change in hydrogen or nitrogen content of the specimens due to exposure to flowing hydrogen, or nitrogen at $900^{\circ}R$.

6.0 CONCLUSIONS

Test results indicate the following:

- 1) Corrosion or decomposition of the specimens or holders did not occur.
- 2) Generation of debris or spalling of particles greater than .007 inch in maximum dimension did not occur.
- 3) Weight change greater than $\pm .01\%$ did not occur during the test.
- 4) There was no significant change in hydrogen or nitrogen content of the specimens due to exposure to flowing hydrogen or nitrogen at 900° R.

Therefore, BATH shield material in combination with 6061-T61 Aluminum and Type 304 Stainless Steel structural materials should meet the requirements of Paragraph 3.3.6 when exposed to the environment specified on Paragraph 3.1.2.4.2.3, Thermal Environment of ECC-677562.

TITLE Shield Element Corrosion Development Test Report	
	DOCUMENT NO. WANL-TME-2723	DATE September, 1970
ABSTRACT Specimens of BATH shield material were tested to determine their resistance to hydriding, dehydriding, nitriding, and contact corrosion with structural material when exposed to flowing hydrogen or nitrogen gas at a pressure of 600 psia and a temperature of 900°R for a period of two hours. Tests results indicated no corrosion or decomposition of the specimens or holders and there was no significant change in hydrogen or nitrogen content after 2 hour exposure at the above conditions.		
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