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Westinghouse Astronuclear Laboratory

EXPERIMENT DESIGN FOR GTR-20

RADIATION EFFECTS TEST OF

SHIELD MATERIAL, 37/W411
(U)

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43

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~~Confidential Restricted Data~~
~~7/2/68~~
 Authorized Classifier Date

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by

117.0 TIC, date SEP 11 1973

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DETERMINATION	DBW	U	4-8-82
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ACKNOWLEDGMENTS

The author wishes to acknowledge and thank Miss M. A. Capo, Mr. K. L. Rieke, and Mr. E. E. Menge of the Thermal and Nuclear Design Department for their assistance in supplying the nuclear and thermal environments of this material, Mr. W. H. Horton of the Reliability Department for his assistance in the statistical design and analysis, Mr. R. P. Shogan and Mr. R. E. Hough of the Materials Department and Mr. F. L. Shubert of the NERVA Fuel and Material Control Department for their assistance in supplying materials information.

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WANL-TME-1818

July 10, 1968

OUTLINE

ACKNOWLEDGMENTS

- I. Experiment Purpose
- II. Experimental Objectives
- III. Analysis Procedures
- IV. Requirements for Experimental Data
 - A. Accuracy Requirements
 - B. Prediction of Results
- V. Experimental Procedures
 - A. Specimen Irradiation Fluence and Temperature
 - B. Tensile Testing (Instron Machine)
 - C. Metallography
 - D. Data Reduction
 - E. Pedigree Specimens
 - F. Disposition of Spare Specimens
 - G. Dimensional Analysis

REFERENCES

APPENDIX A

APPENDIX B

FIGURES

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July 10, 1968

I. Experimental Purpose

The shield material is intended to be used for the central and extension shield in the R-1 design. A proposed design is shown in Figure 1. This shield material is a recently developed product of the NERVA program. There exist several different designs for the shield at this time, some of which have the shield forward of the core support plate instead of aft of it as shown in Figure 1. The environment for the shield material consists of fast neutron fluences ($E > 1 \text{ MeV}$) between 1×10^{16} and 3×10^{18} nvt (Reference 1) and temperatures between 300 and 800°R (Reference 2). The fluences are based on a ground test having 100 minutes of equivalent time at full power.* The uncertainty in fast fluence as determined by Thermal and Nuclear Design is $\pm 50\%$. The first known irradiation of this material has been accomplished recently in the Plum Brook Reactor in a water environment at ambient temperatures, but material properties have not yet been measured.

* 75 minutes of full power and 25 minutes allowed for ramps to full power

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II. Experimental Objectives

Following are the objectives for 37/W411. To determine:

1. 0.2% tensile yield strength
2. 0.2% compressive yield strength
3. hydrogen loss characteristics
4. the ultimate tensile and compression strengths
5. elongation
6. reduction of area

As a function of:

1. test temperature from 140 to 1140°R
2. fast neutron fluence from 0 to 1×10^{19} nvt
3. extrusion A or B
4. annealing temperatures between 540 and 740°R (at a fast neutron fluence of 5×10^{18} nvt)
5. annealing times between 10 and 1000 minutes
6. notched versus unnotched tensile specimens

Total length measurements will be made after irradiation for all specimens to be tested at room temperature and above.

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July 10, 1968

III. Analysis Procedures

Standard techniques to perform an analysis of variance will be used to determine the material properties as a function of the independent variables discussed in Section II and the random variability of the specimens. These analyses will be performed by the WANL Reliability Department, and references as to their use may be found in many statistics text books, such as References 3 and 4. The matrices for each group of specimens used in the analysis of variance are given in Appendix A. A fractional factorial design is used. Results will be given in the form of functional equations expressing the property of interest as a function of the variables considered in the experiment plans. The standard deviation associated with the random variability of each property will also be determined.

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WANL-TME-1818
July 10, 1968

IV. Requirements for Experimental Data

A. Accuracy Requirements

The neutron fluence for specimens with the same fluence requirement in any given group shall be within $\pm 5\%$. Whenever a group has two required fluences (for example, group A-I has 24 specimens that require 5×10^{18} nvt and 12 specimens that require 1×10^{19} nvt) the ratio of high to low fluence shall be $2.00 \pm 10\%$. Variation in fluence between groups shall be within $\pm 30\%$.

The gamma dose of all specimens shall be reported to within $\pm 50\%$ to comply with the NERVA Trend Data Retrieval Program.

The Instron machine load shall be recorded within $\pm 0.5\%$.

All elongation measurements shall be made to $\pm 0.5\%$.

Dimensional measurements of length shall be to the nearest 0.5 mils.

Measurements of diameter shall be to the nearest 0.1 mil.

Reduction of area measurements shall be to $\pm 1\%$.

Room temperature shall be recorded to the nearest $^{\circ}\text{R}$.

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July 10, 1968

IV. (Continued)

The Instron machine grip temperatures shall be monitored with thermocouples, and they should be recorded to within $\pm 1^\circ\text{R}$. The specimen temperature shall be accurate to $\pm 5^\circ\text{R}$.

Annealing temperatures shall be accurate to $\pm 2^\circ\text{R}$ in the cryogenic range and to $\pm 5^\circ\text{R}$ at temperatures $\geq$ room temperature.

The annealing times shall be accurate to $\pm 1\%$.

B. Prediction of Results

This is a newly developed material that has never been irradiation tested at cryogenic temperatures. No predictions of its physical properties as a function of neutron irradiation are available. Some unirradiated data is available, however. Average mechanical properties at 540°R and 140°R have been determined for three compositions of the material. As expected, yield and ultimate strengths increase as the proportion of aluminum in the composite is increased. At 140°R , test samples containing up to 20 v/o TiH_2 and B_4C show yield strength (0.2% offset) of up to 20,000 psi and ultimate tensile strength of 29,000 psi. Elongations ranged from 0.5% to 6% in one inch, averaging about 3% at 140°R .

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V. Experimental Procedures

A. Specimen Irradiation Fluence and Temperature

All specimens shall be irradiated at 140°R in LN₂. The following table shows the required neutron fluence for each shield material specimen to be irradiated and its group. These group numbers correspond to those in Appendix A, the tabulation of specimen groups.

<u>Specimen Group</u>	<u>Number of Specimens To Be Irradiated</u>	<u>Required Fast Neutron Fluence (nvt)</u>
D-I	14 (Including 2 spares)	5×10^{18}
D-I	14 (Including 2 spares)	1×10^{19}
D-II	26 (Including 8 spares)	5×10^{18}
D-III	8 (Including 2 spares)	5×10^{18}
D-IV	16 (Including 4 spares)	5×10^{18}

The specimens in group D-I scheduled to receive the high dose of 1×10^{19} nvt shall be placed on the inner surface of the dewar nearest the North face of the core within a 5 inch radius circle, as shown in Figure 2.* The center of the circle represents the projection of the core center. The specimens scheduled to receive the low fluence of 5×10^{18} nvt shall be placed on a spherical surface (as shown in Figure 3) that is 5 inches North of the inner surface of the dewar on a horizontal line through the core center perpendicular to the core North face and intersects a 10 inch radius

* Based on estimated flux distribution provided by General Dynamics, Fort Worth.

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Astronuclear
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WANL-TME-1818
July 10, 1968

V. (Continued)

circle on the inner surface of the dewar. This surface may be modified slightly and its deviation from spherical defined as a result of further dosimetry analysis and calculation of the attenuation from the high fluence specimens.

The neutron fluence for specimens with the same fluence requirement in any given group shall be within $\pm 5\%$. For group D-I, which has 14 specimens that require 5×10^{18} nvt and 14 specimens that require 1×10^{19} nvt, the ratio of high to low fluence shall be $2 \pm 10\%$. Variation in fluence between groups shall be within $\pm 30\%$.

Specimen dimensions are approximated in the following table defining the space each specimen will occupy. The table also gives the drawing numbers associated with each specimen. These drawings are Figures 4 and 5.

Specimen	Approximate Specimen Dimensions (Inches)			Drawing Number(s)	Number of Specimens at Indicated Neutron Fluence (nvt)		Number of Unirradiated Control Specimens
	Length	Width	Thickness or Diameter		Fast Fluence		
					5×10^{18}	1×10^{19}	
BATH Shield Tensile	3.6	1.0	0.26	801C377	48	14	22
BATH Shield Compression	1.06	---	0.36	650A584	16		16

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V. (Continued)

B. Tensile Testing (Instron Machine)

Specimen test temperature shall be controlled with LN_2 , $LN_2 + N_2$ or a Marshall tube furnace. Each specimen shall be held at its planned test temperature for 15 minutes before starting the crosshead. This time is approximate and may be changed by the WANL monitor at the time of testing. The Instron machine crosshead speed shall be 0.005 inches/min. for all shield material tensile and compression testing. Alignment checks shall be made intermittently during tensile testing program. This shall be accomplished with three strain gages mounted 120° apart around the circumference of the gage section of a dummy specimen. The WANL monitor will determine whether the alignment of the system is adequate.

C. Metallography

Macrographs with a magnification of 2 to 4 (determined by WANL monitor) shall be taken of the fractured surface of specimens numbered 134, 136, 141, 143, 161, 169, 199, 200, 203, 204, and 214. (See Appendix A for specimen numbering.)

Electron fractographs (having suggested magnification 6000) of the fractured surface and microphotographs (having magnification 100 and 500) of the polished longitudinal area including the fracture shall be taken of specimens numbered 141, 143, 169, and 177. WANL shall receive replicas of the electron fractographs.

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WANL-TME-1818

July 10, 1968

V. (Continued)

D. Data Reduction

The data to be taken shall be that outlined in Sections A, C, H, and I plus the following: The ultimate and 0.2% offset tensile yield strength, the Instron chart elongation, compressive ultimate strength, the plastic elongation at maximum stress, the total plastic elongation obtained with bench measurement, and reduction of area using photography methods and/or micrometer measurements. Data shall be based on actual WANL reported gage length. Original or legible reproduced copies of all load-deflection curves, temperature recording curves, and raw data tabulations are to be made available to WANL as soon after testing as possible.

E. Pedigree Specimens

Twenty four unirradiated shield material specimens will be tested at WANL at various temperatures after fabrication. Four will be plain tensile, four will be notched tensile, and 16 will be compression specimens. These specimens will be from the same batch as those that will be shipped to GD/FW.

The fabrication procedure, density, chemical composition, and metallography are given as background information on this material. The pedigree specimens and the specimens for GD/FW are both made from extrusion bars 8 and 9.

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V-4

V. (Continued)

The titanium hydride powders are obtainable in small particle sizes and sufficient purity from the Ventron Corporation. Similarly, the boron carbide powders are manufactured by the Electro Minerals Division of the Carborundum Company. ALCOA and others supply the aluminum powders.

From mixtures of these powders, rods to $2\frac{1}{2}$ inches in diameter have been extruded successfully by conventional extrusion techniques. After charge preparation, the powders are mixed together in a Vee blender for sufficient time to insure homogeneity. Following this, the composite mixture is isostatically pressed to a billet as shown in Figure 6. The vendor has capacity to press to 94% of theoretical density with a finished compact up to 11 inches in diameter. To date, the work has been done by Sylvania Electric Products, Inc. at the Towanda, Pa. Plant. The billets were pressed at 60,000 psi to a density of about 95% of theoretical.

In the next step, the billets are machined to the desired or uniform diameter prior to extrusion to the required shape. Extruding hot at an extrusion ratio greater than 15 to 1 produces a sound billet with theoretical density greater than 97 percent. The extrusion temperature is critical in that sufficient heat is needed to permit the material to be moved through the die; and, yet, time

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WANI-TME-1818

July 10, 1968

V. (Continued)

at temperature and temperature itself must be at a minimum to guard against loss of hydrogen during the heating or extruding of the billet. Although various extrusion speeds are practical, best results are obtained with a maximum application of force and speed. Whittaker Corporation and ALCOA have fabricated the round bar successfully. Other vendors with the equipment to develop unit pressures greater than 60,000 psi should be satisfactory. The following shows the actual press practice followed.

EXTRUSION PRESS PRACTICE FOR SHIELD MATERIAL

<u>Extrusion Number</u>	<u>Composition, v/o Al, TiH₂, B₄C</u>	<u>Unit Pressure psi</u>	<u>Billet Temperature °F</u>	<u>Cylinder Temperature °F</u>
8	70 24 6	78,700	700	760
9	70 24 6	80,500	690	760

Billets were heated to 700°F nominal and extruded after applying maximum force and maximum ram speed. In each case, a 2½ inch rod about 18 feet in length was produced. Two typical rods are shown in Figure 7.

Property data to date on this series of billets and extruded rods are limited to hydrogen, chemistry of major constituents in the shield material, and densities. The following table shows the composition of samples from the compacted billet material. Converting the weight

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V. (Continued)

percent values obtained to volume percent indicates that the billets had the specified composition.

CHEMISTRY OF COMPACTED BILLETS

<u>Identity</u>	<u>Composition, v/o</u>			<u>Analysis w/o</u>			
	<u>Al</u>	<u>TiH₂</u>	<u>B₄C</u>	<u>H₂</u>	<u>Ti</u>	<u>B₄C</u>	<u>Al</u>
1-T-1	80	16	4	0.71	19.4	3.3	76.4
				0.83	19.5	3.3	
				0.89			
				0.84			
				0.74			
				0.84			
1-T-3	70	24	6	1.17	29.7	5.6	63.4
				1.24	29.7	5.7	
				1.33			
				1.30			
				1.31			

Single hydrogen and density determinations have been completed on the extruded rods. The values are shown in the following table for rods 8 and 9.

HYDROGEN PRESENT AND DENSITY OF EXTRUDED SHIELD MATERIALS

<u>Extrusion</u>	<u>Composition, v/o</u>			<u>H₂, w/o</u>	<u>Density gm/cc</u>	<u>Density % of Theoretical</u>
	<u>Al</u>	<u>TiH₂</u>	<u>B₄C</u>			
8	70	24	6	1.17	2.926	99.3
9	70	24	6	1.24	2.933	99.4

V. (Continued)

In calculating the density, a theoretical density of 2.95 gm/cc was used. The densities determined are consistent with the microstructure which shows very little porosity present.

Microstructural studies show a matrix of aluminum with uniformly dispersed particles of TiH_2 and B_4C . Soundness is evident with only minor porosity which is consistent with measured densities of 97 percent or greater. Microstructures are shown in Figures 8 and 9 for extruded bars 8 and 9 respectively

F. Disposition of Spare Specimens

Spare specimens will be used to replace damaged specimens if there are any. If spares are not used to replace damaged specimens they will be used for replication at the most stringent test temperature.

G. Hydrogen Analysis

Selected compression specimens shall be analyzed for hydrogen concentration after irradiation by gravimetric combustion techniques at Westinghouse, Waltz Mill. Appendix B gives the procedure for hydrogen determination as well as that for determining Ti, B_4C , and Al. Some control specimens taken from the same region as were the compression specimens will also be analyzed for hydrogen and compared to the irradiated specimens.

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July 10, 1968

V. (Continued)

H. Dimensional Analysis

A dimensional analysis shall be performed on the shield material specimens that are tested at room temperature, by measuring lengths and diameters of selected specimens.

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WANL-TME-1818
July 10, 1968

REFERENCES

1. M. A. Capo, "R-1 Radiation Environment," DRM-1005, May 17, 1968
(A CRD Document).
2. "Engineering Data and Design Concepts," WANL-TME-1798, Volume 3,
June, 1968 (A CRD Document).
3. O. L. Davies, Design and Analysis of Industrial Experiments,
Hafner Publishing Company, New York City.
4. R. L. Anderson and T. A. Bancroft, Statistical Theory in
Research, McGraw-Hill Book Company, New York City.
5. "NERVA Quarterly Progress Report for April, May, and June,"
PRJ-050, Project NE-2800 Shield Materials Development Program.

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July 10, 1968

APPENDIX A

The following pages give tabulation of all
shield material specimens, the group number
of each group, and the independent variables.

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July 10, 1968

D-I Shield (BATH) - Plain Tensile

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Extrusion</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
141	A	0	140
152			340
134			540
159			740
164			940
169			1140
144		5 x 10 ¹⁸	140
155			340
139			540
173			740
179			940
177			1140
143		10 ¹⁹	140
150			340
136			540
175			740
176			940
161			1140

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July 10, 1968

D-I Shield (BATH) - Plain Tensile
(Continued)

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Extrusion</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
148	B ↓	0	140
154			340
133			540
158			740
166			940
162		↓	1140
147		5×10^{18}	140
151			340
137			540
163			740
172			940
167		↓	1140
146		10^{19}	140
149			340
138			540
168			740
171			940
157		↓	1140

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July 10, 1968

Spare Specimens

D-I Shield - Plain Tensile

<u>Specimen No.</u>	<u>Extrusion</u>	<u>Fluence (nvt)</u>
445	A	0
446	↓	5×10^{18}
447	↓	10^{19}
448	B	0
449	↓	5×10^{18}
450	↓	10^{19}

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D-II Shield (BATH) - Plain Tensile - Annealing Study

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Extrusion</u>	<u>Annealing Temperature (°R)</u>	<u>Annealing Time (min.)</u>
182	A ↓	340	10
190		↓	100
191		↓	1000
187		540	10
188		↓	100
189		↓	1000
185		740	10
197		↓	100
194		↓	1000
196		340	10
193	B ↓	↓	100
183		↓	1000
181		540	10
198		↓	100
186		↓	1000
184		740	10
192		↓	100
195		↓	1000

Spare Specimens

D-II Shield - Plain Tensile - Annealing Study
5 x 10¹⁸ nvt

<u>Specimen Number</u>	<u>Extrusion</u>
451
452	
453	
454	
455	
456	
457	
458	

WANL-TME-1818
July 10, 1968

D-III Shield (BATH) - Notched Tensile

(To Be Combined With Some Data of I.)

Test Conditions

<u>Specimen No.</u> <u>&</u> <u>Test Order</u>	<u>Fluence (Fast)</u> <u>(nvt)</u>	<u>Test</u> <u>Temperature (°R)</u>
142	0	140
153		340
135		540
160		740
165		940
170		1140
145	5 x 10 ¹⁸	140
156		340
190		540
174		740
180		940
178		1140

Spare Specimens

D-III Shield - Notched Tensile

<u>Specimen No.</u>	<u>Fluence (nvt)</u>
459	0
460	0
461	5 x 10 ¹⁸
462	5 x 10 ¹⁸

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WANL-TME-1818

July 10, 1968

D-IV Shield (BATH) - Compression

Test Conditions

Specimen No. & Test Order	Extrusion	Fluence (Fast) (nvt)	Test Temperature (°R)
203	A	0	140
208			340
200			540
219			740
211			940
214		↓	1140
204		5 x 10 ¹⁸	140
210			340
199			540
220			740
216			940
212	↓	↓	1140
206	B	0	140
209			340
201			540
218			740
213			940
217		↓	1140
205	↓	5 x 10 ¹⁸	140

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WANL-TME-1818

July 10, 1968

D-IV Shield (BATH) - Compression
(Continued)

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Extrusion</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
207	B	5×10^{18}	340
202			540
222			740
221			940
215			1140

Spare Specimens

D-IV Shield - Compression

<u>Specimen No.</u>	<u>Extrusion</u>	<u>Fluence (nvt)</u>
463	A	0
464		0
465		5×10^{18}
466		5×10^{18}
467	B	0
468		0
469		5×10^{18}
470		5×10^{18}

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July 10, 1968

APPENDIX B

The procedures for determining hydrogen concentration by gravimetric combustion and for determining Ti, B_4C , and Al concentrations are given here.

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July 10, 1968

RECOMMENDED PROCEDURE

DETERMINATION OF HYDROGEN CONTENT OF SHIELDING ALLOY

1. SCOPE

This procedure provides for the determination of hydrogen content of shielding alloys and metallic hydrides.

2. SUMMARY OF METHOD

This determination is made by igniting the shielding alloy or metallic hydride with a high frequency induction furnace, oxidizing the evolved hydrogen to water, collecting the water on magnesium perchlorate and weighing.

3. SIGNIFICANCE

This procedure is intended for shielding alloys containing metallic hydride.

4. INTERFERENCES

H₂O and hydrocarbons.

5. APPARATUS

5.1 Analytical balance, precision at least ± 0.2 mg.

5.2 High frequency induction furnace.

5.3 Zircon cups (Leco type).

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WANL-TME-1818
July 10, 1968

- 5.4 Graphite-quartz enclosed crucibles (Leco type)
- 5.5 U-tube absorption bulbs - conventional - double stoppered
- 5.6 Gas purification train including flowmeter
- 5.7 Resistance tube furnace (Hevi-Duty Electric Company Model 4838-201)
- 5.8 Nickel tube (about 15" long x 1" O.D.)
- 5.9 Variac (Powerstat Type 136B) - to control high frequency induction furnace, if needed
- 5.10 Variac (Adjusti-Volt Type 500B) - to control CuO oxidizing furnace, keeping temperature at 600°C.

6. REAGENTS

- 6.1 Argon gas
- 6.2 Ascarite (for gas purification train)
- 6.3 Magnesium perchlorate - reagent grade (purification train and U-tube)
- 6.4 Concentrated sulfuric acid (purification train)
- 6.5 Copper oxide shot - reagent grade

7. SAFETY

- 7.1 All safety switches on electrical equipment must be operational.
- 7.2 Induction coil must be shielded.
- 7.3 All connections should be secure and tight.

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July 10, 1968

8. SAMPLING

- 8.1 The sample is mechanically blended to ensure homogeneity (for powder only)
- 8.2 The sample is accurately weighed so as to evolve hydrogen equivalent to about 100 mg of water.

9. CALIBRATION AND STANDARDIZATION

- 9.1 The balance must be standardized using weights with accuracy conforming to Class S tolerances established by the National Bureau of Standards.
- 9.2 Prepurge system with argon and heat copper oxide furnace overnight at 600°C.
- 9.3 Argon flow rate of overnight purge about 0.1 liters per minute
- 9.4 Pre-ignite combustion chamber with quartz-graphite loaded crucible for about 15 minutes before running blanks or sample. This is to remove moisture trapped within the combustion tube.
- 9.5 Control used is any suitable metallic hydride which has known hydrogen content.

10. PROCEDURE

- 10.1 Weigh sample to obtain about 100 mg weight as water.
- 10.2 Place sample in zircon cup, insert cup into graphite-quartz enclosed crucible, and seal in combustion chamber.
- 10.3 Adjust argon flow rate to 1.0 liter per minute.

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- 10.4 Purge system for 2 minutes. Attach pre-weighed magnesium perchlorate U-tube using nylon swage nuts to end of CuO oxidation furnace.
- 10.5 Ignite sample to 700°C (heating above 700°C does not increase efficiency or results but causes argon to ionize)
- 10.6 Ignite sample for 15 minutes, then cool for 5 minutes for a total of 20 minutes.
- 10.7 Disconnect U-tube, and allow 20 minutes for cooling and equilibration on bench near balance or in balance.
- 10.8 Reweigh bulb. Weight gain is recorded as water.

11. CALCULATIONS

The total hydrogen of the sample is calculated as follows:

$$\% \text{ hydrogen} = \frac{(A - B) \times 0.1115}{W_s} \times 100$$

where:

- A = Total weight increase of weighing bulb (grams)
- B = Blank (grams)
- 0.1115 = Conversion factor from water to hydrogen
- W_s = Total sample weight in grams

12. PRECISION AND ACCURACY

Based on eleven (11) determinations of the control (TiH_2) and analysis of individual samples, the coefficient of variation is 0.93.

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July 10, 1968

NOTES ON THE ANALYSIS OF SHIELDING ALLOYS FOR TITANIUM, BORON CARBIDE,
AND ALUMINUM CONTENT

Dissolve about 2 grams of the sample in 15 milliliters of concentrated hydrochloric acid. Use a cool water bath to slow down reaction. After the reaction is slowed down, add an additional 25 milliliters of concentrated hydrochloric acid.

Place sample on hot plate and heat to $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 16 hours. Use 125 milliliter Erlenmeyer flasks covered with watch glasses.

Cool the sample slowly and filter through a medium porosity glass crucible. Wash with 4 - 10 milliliter portions of concentrated hydrochloric acid.

Transfer filtered solution of a 200 milliliter volumetric flask using concentrated hydrochloric acid. The final volume of 200 mls will consist approximately of 150 milliliters of concentrated hydrochloric acid and 50 milliliters of demineralized water. Store solutions in poly bottles.

Rewash glass crucibles with 4 - 10 milliliter portions of demineralized water. Vacuum dry them overnight at room temperature.

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July 10, 1968

Reweigh the previously weighed crucibles, the difference is boron carbide. The aliquot that was stored in the poly bottles is taken and to it is added 10 milliliters of perchloric acid. Fume. Remove from heat and cool. Place in 50 milliliter volumetric and dilute to 40 milliliters. Add 5 mls of 3% hydrogen peroxide and dilute to volume.

The solution is placed in 1 cm cells and read on a Beckman Du Spectrophotometer at 425 mm. A calibration curve is drawn, ranging from 0 - 3500 micrograms of titanium using standards.

The aluminum is calculated as the difference of the total sample weight versus the amount of titanium, hydrogen, boron carbide found.

The spectrographic survey is also done to scan for any impurities in the starting material and the finished products that would tend to bias the individual results either toward the high side or the low side.

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FIGURE 1 R-1 Reactor Assembly

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FIGURE 2 Loading of High Fluence Specimens

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FIGURE 3 Loading of Low Fluence Specimens

FIGURE 4 Drawing of Shield Material Tensile Specimens

FIGURE 5 Drawing of Shield Material Compression Specimens

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FIGURE 6 Isostatically Pressed Billet

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FIGURE 7 Extruded Rods

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100X

500X

FIGURE 8 Microstructures of Bar 8 at 100 and 500X

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100X

500X

FIGURE 9 Microstructures of Bar 9 at 100 and 500X

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