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Westinghouse Astronuclear Laboratory

EXPERIMENT DESIGN FOR GTR-20

RADIATION EFFECTS TEST OF

COPPER-BORON-10, 37/W409
(U)

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EXPERIMENT DESIGN FOR GTR-20
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I. Experimental Purpose

Copper-boron-10 will be used in the control vanes and in the poison plates in the R-1 reactor in a proposed design shown in Figure 1.

The control vanes will utilize 24 volume % boron-10, while the poison plates (cluster plates) utilize 12 volume % boron-10.

Figure 1 shows these two components in the R-1 reactor.

Copper-boron-10 is a newly developed material that has never been used in a reactor; therefore, it must be qualified. The environment for control vane copper-boron-10 in the R-1 reactor consists of fast neutron fluences ($E > 1 \text{ MeV}$) between 1.5×10^{17} and 6.0×10^{18} nvt (Reference 1) and temperatures between 200 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 the neutron fluence is $\pm 50\%$ as defined by Thermal and Nuclear Design, while that in temperature is $\pm 100\%$. Hence, a knowledge of 24% Cu-B¹⁰ material properties is needed for fast neutron fluences between 7.5×10^{16} and 9.0×10^{18} nvt and for temperatures between 200 and 1600°R. The environment for poison plate copper-boron-10 consists of fast neutron fluences between 1.9×10^{18} and 3.7×10^{18} nvt (Reference 1) and a maximum temperature of 360°R (Reference 2).

II. Experimental Objectives

Following are objectives for 37/W409. (Unless otherwise stated, the boron-10 concentration is 12% by volume.):

For group B-I determine:

1. 0.2% tensile strength
2. ultimate tensile strength
3. elongation and reduction of area

As a function of:

1. test temperature from 140 to 1540°R
2. longitudinal or transverse specimen orientation
3. fast neutron fluence from 0 to 1×10^{19} nvt

For group B-II determine the effect of annealing on:

1. 0.2% tensile yield strength
2. ultimate tensile strength
3. elongation and reduction of area

As a function of:

1. test temperature from 540 to 940°R
2. annealing time from 10 to 1000 minutes
3. longitudinal or transverse specimen orientation

For group B-III determine for notched specimens:

1. notched tensile strength
-
-
-

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II. (Continued)

As a function of:

1. test temperature from 140 to 540°R
2. fast neutron fluence from 0 to 5×10^{18} nvt

For group B-IV (24% by volume boron-10) determine:

1. 0.2% tensile yield strength
2. ultimate tensile strength
3. elongation and reduction of area

As a function of:

1. test temperature from 140 to 1340°R
2. fast neutron fluence from 0 to 5×10^{18} nvt
3. longitudinal or transverse specimen orientation

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

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 can 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 given.

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 B-I has 20 specimens that require 5×10^{18} nvt and 10 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 $^{\circ}\text{R}$.

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 $\pm 5^{\circ}\text{R}$.

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IV. (Continued)

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

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

B. Prediction of Results

This is a new material that has never been tested. No predictions of its physical properties as a function of neutron irradiation are made.

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 Cu-B¹⁰ specimen to be irradiated and the group it is in. 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>
B-I	20 (Including 4 spares)	5×10^{18}
B-I	10 (Including 2 spares)	1×10^{19}
B-II	22 (Including 6 spares)	5×10^{18}
B-III	6 (Including 2 spares)	5×10^{18}
B-IV	10 (Including 2 spares)	5×10^{18}

The specimens 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 dashed lines indicate that these specimens are directly underneath (or slightly farther away than) the Inconel-718 specimens. 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

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

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V. (Continued)

dewar on a horizontal line through the core center perpendicular to the core North face and intersects a 10 inch radius 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 $\pm 5\%$. Whenever a group has two required fluences (for example group B-I has 20 specimens that require 5×10^{18} nvt and 10 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 less than $\pm 30\%$.

Specimen dimensions are approximated in the following table to give an idea of the space each specimen will occupy. The table also provides the drawing number associated with the specimens for the exact dimensions. This drawing is Figure 4.

Specimen	Approximate Specimen Dimensions (Inches)			Drawing Number	Number of Specimens at Indicated Neutron Fluence (nvt)		Number of Unirradiated Control Specimens
	Length	Width	Thickness		Fast Fluence		
			or Diameter		5×10^{18}	1×10^{19}	
Copper-Boron-10	3.50	1.0	0.1	799C877	58	10	26

V. (Continued)

B. Tensile Testing (Instron Machine)

Specimen test temperature shall be controlled with LN₂, N₂ + LN₂, or 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 0.05 inches/min. for all copper-boron tensile testing.

C. Metallography

Macrographs with a magnification of 2 to 4 shall be taken of the fractured surface of specimens numbered 227, 230, 242, 252, 525, 526, 531, and 534. (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 227, 230, 242, and 243. WANL shall receive replicas of the electron fractographs.

D. Data Reduction

The data to be taken shall be that outlined in Sections A, C, and G plus the following: The ultimate tensile and 0.2% offset yield strength, the Instron chart elongation, the plastic elongation at

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V. (Continued)

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, the raw data tabulations are to be made available to WANL as soon after testing as possible.

E. Pedigree Specimens

Sixteen unirradiated Cu-B¹⁰ specimens will be tested at WANL at various temperatures after fabrication. Ten of these will be the 24% boron-10, and the remaining six will be the 12% boron-10. These specimens will be from the same batch as those that will be shipped to GD/FW. The fabrication process, the specimen machining, chemical composition, density, and metallography are given here as background information for the material to be tested.

For the 12 volume % boron-10 material the powder is mixed and then compacted isothermally at 60,000 psi. It is sectioned into 3 billets, which are flame sprayed with 10-20 mils of copper. The billets are heated to 800°C and extruded using a Dynapac. The extrusions are then cut into 2 pieces giving a total of six sections. Each section is cross rolled at 150°F to a strip approximately 4.75"

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V. (Continued)

wide using various anneals at 600°C and a 10% rolling reduction per pass. This is continued until the thickness is $0.100 \pm .001$ inches.

The 24% boron-10 material is fabricated the same way except the billets are canned in copper after compaction. Then they are extruded, cut, and partially rolled before the can is etched off.

The specimen machining is accomplished by laying blank specimens out on the sheet. These blanks are then sheared out, and holes are punched in the specimen. The gage section is punched and then finish ground. The 24% specimens were painted green prior to machining as a means of classification.

The ranges of densities for the two materials are given below.

<u>Cu-B¹⁰ Material</u> <u>(% B¹⁰)</u>	<u>Range of Densities</u> <u>(gm/cm³)</u>
12	7.85 - 7.95
24	6.97 - 7.14

The boron in these materials is 95.7% B¹⁰, and the copper is 99.6% pure. More complete chemical analyses will be issued after they have been performed.

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V. (Continued)

Unirradiated metallography of both materials is given in Figure 5 for a magnification of 100X.

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 (140°R or as determined by the WANL monitor).

G. Dimensional Analysis

A dimensional analysis shall be performed on the copper-boron-10 specimens that are tested at room temperature, by measuring lengths and widths of selected specimens.

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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.

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APPENDIX A

The following pages give tabulation of all copper-boron-10 specimens, the group number of each group, and the independent variables.

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B-I Cu-12% B¹⁰ - Plain Tensile

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Orientation</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
227	Long.	0	140
224			540
252			1140
243			1540
230		5 x 10 ¹⁸	140
238			340
225			540
261			740
239			940
260			1140
246			1340
242			1540
237		10 ¹⁹	340
241			740
240			940
259			1340
235	Trans.	0	340
258			740
248			940

B-I Cu-12% B¹⁰ - Plain Tensile

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Orientation</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
244	Trans.	0	1340
229	↓	5 x 10 ¹⁸	140
233		↓	340
223			540
255			740
256			940
251			1140
249			1340
262			1540
232		10 ¹⁹	140
226		↓	540
257			1140
247			1540

Spare Specimens

B-I Cu-12% B¹⁰ - Plain Tensile

<u>Specimen Number</u>	<u>Orientation</u>	<u>Fluence (nvt)</u>
427	Long.	0
428	↓	5 x 10 ¹⁸

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Spare Specimens

B-I Cu-12% B¹⁰ - Plain Tensile

<u>Specimen Number</u>	<u>Orientation</u>	<u>Fluence (nvt)</u>
429	Long.	5×10^{18}
430	↓	10^{19}
431	Trans.	0
432	↓	5×10^{18}
433	↓	5×10^{18}
434	↓	10^{19}

B-II Cu-12% B¹⁰ - Plain Tensile - Annealing Study

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Orientation</u>	<u>Annealing Temperature (°R)</u>	<u>Annealing Time</u>
270	Long.	540	5 min (1)
273			10 (2)
274			100 (3)
267		↓	1000 (4)
277		940	5 (1)
276			10 (2)
275			100 (3)
266		↓	1000 (4)
271	Trans.	540	5 (1)
272			10 (2)
265			100 (3)
264		↓	1000 (4)
268		940	5 (1)
263			10 (2)
278			100 (3)
269		↓	1000 (4)

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Spare Specimens

B-II Cu-12% B¹⁰ - Plain Tensile - Annealing Study

<u>Specimen Number</u>	<u>Orientation</u>	<u>Fluence (nvt)</u>
435	Long.	5×10^{18}
436	<div>↓</div> <div>Trans.</div> <div>↓</div>	5×10^{18}
437		5×10^{18}
438		5×10^{18}
439		5×10^{18}
440		5×10^{18}

B-III Cu-12% B¹⁰ - Notched Tensile
(To Be Combined With Some Data From I.)

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Orientation</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
236	Long.	0	340
245		0	1340
231		5 x 10 ¹⁸	140
261		5 x 10 ¹⁸	1140
228	Trans.	0	140
253		0	1140
234		5 x 10 ¹⁸	340
250		5 x 10 ¹⁸	1340

Spare Specimen

B-III Cu-12% B¹⁰ - Notched Tensile

<u>Specimen Number</u>	<u>Orientation</u>	<u>Fluence (nvt)</u>
441	Long.	0
442	Long.	5 x 10 ¹⁸
443	Trans.	0
444	Trans.	5 x 10 ¹⁸

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B-IV Cu-24% B¹⁰ - Plain Tensile

Test Conditions

<u>Specimen No.</u> & <u>Test Order</u>	<u>Orientation</u>	<u>Fluence (Fast)</u> <u>(nvt)</u>	<u>Test</u> <u>Temperature (°R)</u>
526 (8)	Long.	0	140
527 (9)	↓	↓	340
522 (4)			540
531 (13)			1340
525 (7)			140
530 (12)		↓	340
521 (3)			540
534 (16)			1340
524 (6)			140
528 (10)		↓	340
520 (2)			540
533 (15)			1340
523 (5)			140
529 (11)		↓	340
519 (1)			540
532 (14)			1340

Spare Specimens

B-IV Cu-24% B¹⁰ - Plain Tensile

<u>Specimen Number</u>	<u>Orientation</u>	<u>Fluence (Fast) (nvt)</u>
535	Long.	0
536	Long.	5×10^{18}
537	Trans.	0
538	Trans.	5×10^{18}

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FIGURE 1 R-1 Reactor Assembly

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FIGURE 2 Loading of High Fluence Specimens

FIGURE 3 Loading of Low Fluence Specimens

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③ ① TYPE FP-1

⑥ ⑤ ③ ② TYPE FN-1

FIGURE 4 Copper-Boron-10 Specimen Drawing

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12% B¹⁰

24% B¹⁰

FIGURE 5 Cu-B¹⁰ Metallography at 100X

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