

**ATTACHMENT 5**

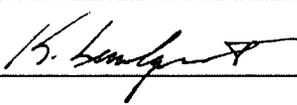
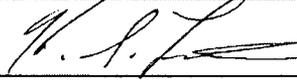
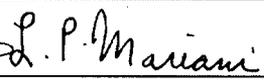
**Northeast Technology Corp. Report No. NET-259-03,  
"Material Qualification of Alcan Composite for Spent Fuel Storage," Revision 5**

# MATERIAL QUALIFICATION OF ALCAN COMPOSITE FOR SPENT FUEL STORAGE

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## 1.0 Introduction and Summary

The purpose of this topical report is to demonstrate that aluminum/B<sub>4</sub>C sheet produced from DC (direct chill) cast rolling billets supplied by Rio Tinto Alcan Inc. is a suitable material for use as a neutron absorber in spent nuclear fuel storage applications and in particular it is a suitable material from which to fabricate NETCO-SNAP-IN<sup>®</sup> neutron absorber inserts. The NETCO-SNAP-IN<sup>®</sup> neutron absorber insert is installed in existing spent fuel storage racks to restore the reactivity hold-down capability of racks with degraded Boraflex. Once installed, these neutron absorber inserts become permanently affixed to the storage racks.

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The suitability of Rio Tinto Alcan Inc. material as demonstrated herein is based upon:

- detailed comparison with highly similar material with a successful record of industry-wide, in-service performance
- accelerated corrosion testing in simulated BWR and PWR spent fuel pool environments
- evaluating and testing of mechanical properties to verify acceptability of installed insert retention force
- measurement of B<sub>10</sub> areal density to confirm satisfactory neutron absorption capability
- short term and long term in-situ coupon surveillance programs.

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These evaluations are detailed in the various sections of this report.

The Alcan material is supplied as 6x6 inch rectangular DC cast rolling billets that are hot rolled to final gage. The material is designated as aluminum boron carbide composite W1100N.XYB where XY is the boron carbide content which can range from 16 to 30

volume percent. The reinforcing phase of the composite is boron carbide powder containing at least 76 w/o boron and with an average particle size of  $7.5 \pm 2 \mu\text{m}$  ( $D_{50}$ ).

As stated above, one particular application of the W1100N.XYB composite in spent fuel pools is the NETCO-SNAP-IN<sup>®</sup> neutron absorber insert. The NETCO-SNAP-IN<sup>®</sup> is proprietary to NETCO and is protected by U.S. Patent No. 6,741,669 B2.<sup>[1-1]</sup> The first use of NETCO-SNAP-IN<sup>®</sup> absorber inserts will be at Exelon's LaSalle Unit 2 Station. Other applications of the W1100N.XYB composite include newly fabricated spent fuel storage racks and dry spent fuel storage and transportation casks. With respect to the latter use, this material has been used in dry storage/transportation in the U.S. and extensively in Europe.

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Recent guidance has been published for the qualification and acceptance of new boron based metallic neutron absorbers for storage and transportation casks.<sup>[1-2]</sup> Using this document as a guide, the qualification process described in this report consists of the following elements:

- Review of the composition and manufacturing process of the W1100N.XYB composite and a detailed comparison with the composition and manufacturing process for BORAL<sup>™</sup>, a neutron absorber material that has been successfully used extensively worldwide for spent nuclear fuel storage racks for the last 40 years.
- An accelerated corrosion program has been completed in both demineralized water and boric acid (2500 ppm as boron). The program ran for one year in duration. Interim and final results are reported.
- A fast start surveillance coupon program has been initiated (March 08) at LaSalle Unit 2 to provide in-service performance data on the W1100N.16B composite in the actual proposed service environment. This will provide performance data that will always lead the installed NETCO-SNAP-IN<sup>®</sup> inserts in both time of exposure and absorbed gamma dose. The fast start coupon program consists of a string of 24 coupons connected by stainless links. The coupons have been precharacterized with respect to dimensions, dry weight, density and boron-10

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areal density. Periodically coupons will be removed from the string and sent to a qualified laboratory for post exposure testing and inspection.

- A long term surveillance assembly will be placed in the LaSalle pool prior to the installation of the first NETCO-SNAP-IN<sup>®</sup> inserts. These coupons will differ from the "Fast-Start" and, in particular, will be composed of 17 vol-% B<sub>4</sub>C instead of 16 vol-% B<sub>4</sub>C material. This modification is due to a manufacturing revision of the NETCO-SNAP-IN<sup>®</sup> inserts intended to ensure compliance with minimum areal density requirements. The tree will hold the following types of coupons:
  - unclad Alcan W1100N.17B composite coupons
  - W1100N.17B composite coupons with 304L stainless steel, In-718 and Zircaloy samples
  - W1100N.17B composite bend coupons

Periodically coupons will be removed from the assembly and sent to a qualified laboratory for testing and inspection.

The following sections of this report describe:

- NETCO-SNAP-IN<sup>®</sup> and Installation Tooling
- Manufacturing process and quality control used for NETCO-SNAP-IN<sup>®</sup> inserts
- Composition and physical properties of the W1100N.XYB composite
- Description of accelerated corrosion testing and interim results
- Comparative evaluation of W1100N.XYB composite and BORAL<sup>™</sup>
- Anticipated performance of W1100N.XYB in spent fuel pools
- Detailed description of the "fast start" coupon surveillance program
- Detailed description of long term coupon surveillance program
- Conclusions

#### References Section 1

- 1-1. Lindquist, K. O., U.S. Patent No. 6,741,669 B2, "Neutron Absorber Systems and Method for Absorbing Neutrons," May 25, 2004.

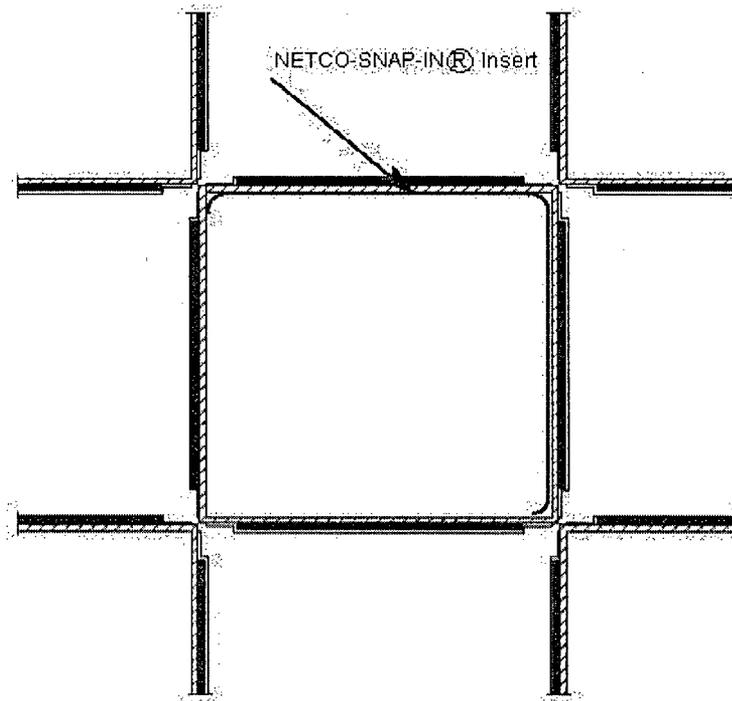
1-2.ASTM C 1671-07, "Standard Practice for Qualification and Acceptance of Boron Based Metallic Neutron Absorbers for Nuclear Criticality Control for Dry Cask Storage Systems and Transportation Packaging."

## 2.0 Description of the NETCO-SNAP-IN<sup>®</sup> and Installation Tooling

Neutron absorber materials are incorporated in spent fuel storage racks to permit the safe storage of LWR fuel assemblies in close proximity to each other. One or two panels of a neutron absorber material are placed between each face of every fuel assembly in order to maintain the stored fuel in a sufficiently sub-critical condition.

One neutron absorber material used for this purpose, Boraflex, has been observed to experience in service degradation well in advance of its design service life. As degradation proceeds, the matrix intended to retain the neutron absorber (boron carbide) dissolves and the boron carbide slumps to the bottom of the pool. As this occurs, less and less of the neutron absorber is in place to maintain the fuel in a sub-critical condition.

The NETCO-SNAP-IN<sup>®</sup> insert mitigates the boron carbide loss from Boraflex by inserting a thin chevron-shaped metallic sleeve into the fuel storage cell of the rack. The sleeve is fabricated from an aluminum/boron carbide composite. When installed, this sleeve, or insert, abuts two adjacent faces of the rack wall. It is intended that an insert be installed in all the storage cells of a given module as shown in Figure 2-1. With each insert installed in the same configuration, every face of all fuel assemblies will have neutron absorber material between it and one face of the adjacent fuel assemblies. Since the inserts are fabricated of a neutron absorbing material, replacement of the initial reactivity hold-down system is effectively achieved.

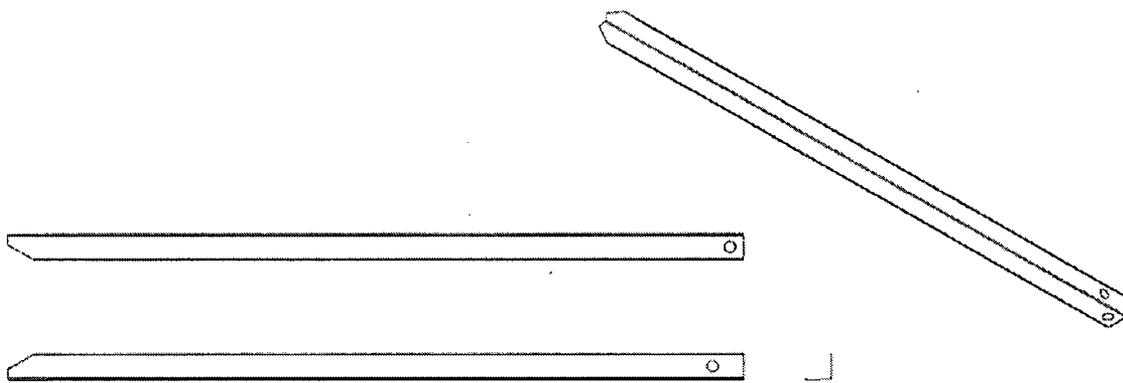


*Figure 2-1 NETCO-SNAP-IN® Insert Installed in a Spent Fuel Storage Rack*

Figure 2-2 shows a typical NETCO-SNAP-IN®. The insert has a length equivalent to the length of the fuel storage cell and the lower end is tapered to facilitate insertion into the fuel storage cell. The chevron is formed with a central bend angle along its length greater than 90°. The width of each wing of the chevron is slightly less than the minimum inside dimension of the fuel storage cell. Each edge of the wing is roll formed and it is this feature that accommodates cell to cell variations in inside dimensions. Near the top of the NETCO-SNAP-IN® is a hole in each wing that engages the installation tool.

It is noted that the chevrons are formed with a greater than 90° bend angle and this causes compression of the insert as it is “pushed” into the rack cell and assumes the

90° angle between adjacent rack cell walls. The insert is designed to become an integral part of the fuel rack once it has been installed. This is achieved through the elastic deformation of the insert bearing against the rack cell wall and the associated friction force. The force exerted due to this deformation is predicted by the effective spring constant of the insert, which is described in detail elsewhere. The force between the insert wings and the rack cell walls in conjunction with the static friction between these surfaces serves to retain the NETCO-SNAP-IN<sup>®</sup> and make it a permanent part of the rack once it is installed.



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Figure 2-2 NETCO-SNAP-IN<sup>®</sup> Insert

The installation tool with a NETCO-SNAP-IN<sup>®</sup> engaged is shown in Figure 2-3. At the top of the tool is a bail that replicates the bail on a BWR fuel assembly. As such the installation tool can be engaged with a fuel grapple or with the refueling mast. The bail is attached to an anvil assembly that provides a bearing surface on the top edge of the insert. Immediately below the anvil assembly is the head assembly. The head assembly contains two spring loaded cylinders, that engage the insert while it is being moved to the storage cell into which it is destined for installation. When, during installation, the cylinders come into contact with the rack cell wall they retract, thus allowing full insertion of the insert. The curvature of the upper edge of each cylinder is so configured that when the insert is fully installed, upward movement of the tool allows the cylinder to clear the engagement holes in the insert, leaving the insert fully seated in the rack cell.

Again referring to Figure 2-3, a counterweight is suspended from three rods below the head assembly. In addition to partially providing downward insertion force, the counterweight, which contributes to insert stability during installation, lowers the center of gravity of the tool. The insertion tool is constructed entirely of stainless steel and weighs less than 1290 lbs.

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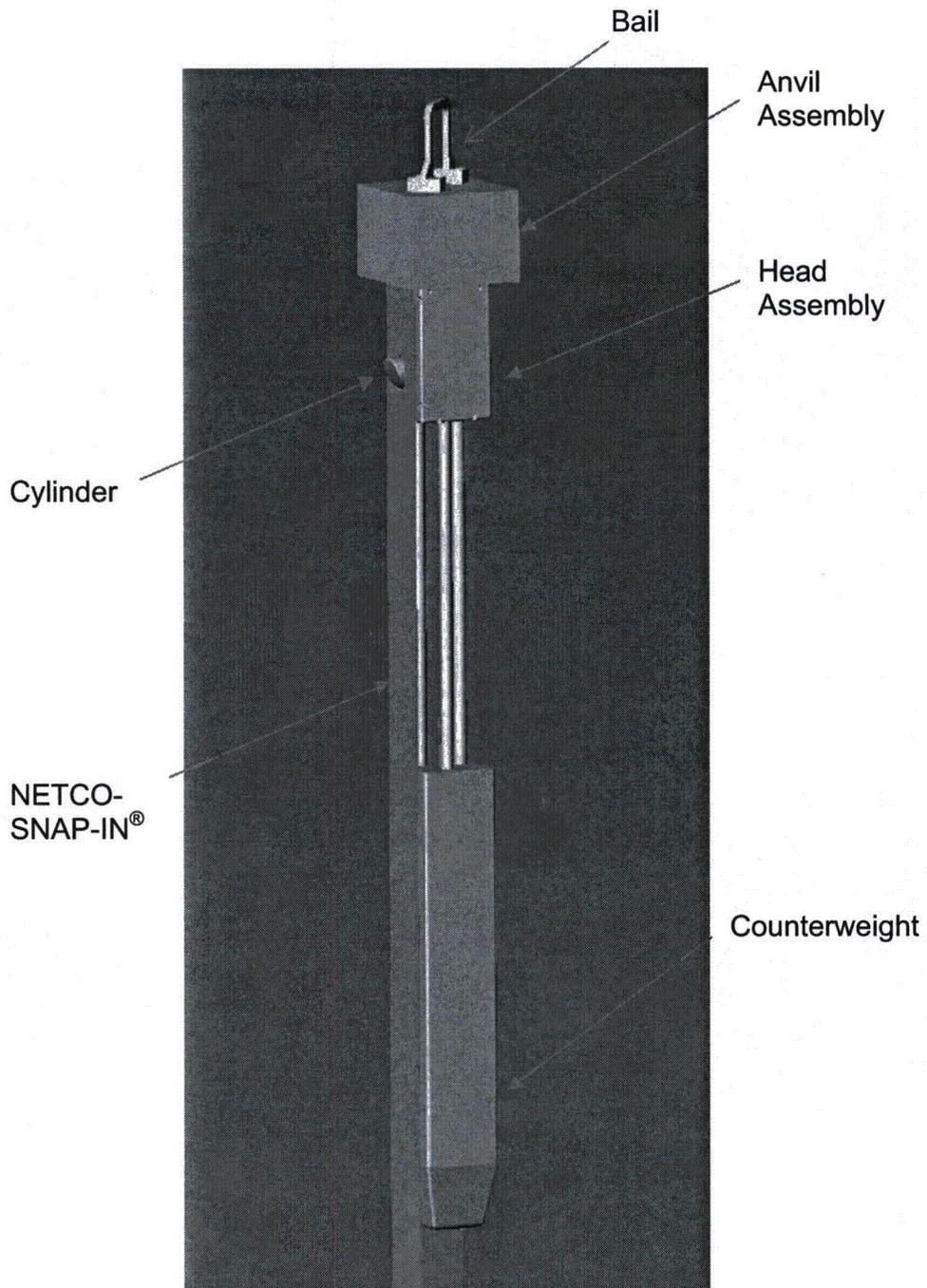


Figure 2-3 Installation Tool

## 3.0 Manufacturing the NETCO-SNAP-IN<sup>®</sup> Neutron Absorber Insert

### 3.1 Billet Production

There are two basic methods for producing aluminum/boron carbide metal matrix composites: powder metallurgy and liquid metal mixing. In the case of powdered metallurgy, atomized metal powder is blended with boron carbide particles, compacted and sintered to form a billet for further processing. The billet is generally extruded to produce rectangular preforms for rolling to final gage. This method has proved to be expensive due to the high cost of atomized metal powders and time consuming processing steps. In addition, wear products from the extrusion die on the surface of the preform need to be removed by cleaning or machining so as not to result in galvanic corrosion in wet storage applications.<sup>[3-1]</sup> Furthermore, and depending on the process used to produce the billets, the final rolled sheet may have limited ductility making the sheet difficult to form by bending.<sup>[3-2]</sup>

Alcan has developed a liquid mixing process for producing aluminum/boron carbide composites that use mechanical stirring to mix the powdered B<sub>4</sub>C in the molten aluminum. As this mixing process is conducted at temperatures well over the melting point of aluminum, significant aluminum and boron carbide interactions can occur that can result in degraded mechanical and physical properties.<sup>[3-3]</sup> A significant physical property effect can be the agglomeration of B<sub>4</sub>C particles resulting in a non-uniform boron distribution in the composite. Alcan has found that by adding small amounts of Ti (< 2.5%) to the molten aluminum, the B<sub>4</sub>C particles become stable in the molten aluminum, eliminating particle clusters, and a uniform blend is achieved. It is thought that a Ti rich zone forms around each boron carbide particle, preventing Al/B<sub>4</sub>C interactions.

The molten aluminum/boron carbide blend is direct chill cast into 6"x6" rectangular billets. The length of the billets is determined by the size and gage of the final rolled

product. The rectangular billets can be rolled directly without an intermediate extrusion step and the potential problems and cost associated with extrusion.

### **3.2 NETCO-SNAP-IN<sup>®</sup> Production**

The Alcan billets are heated to ~ 950°F and hot rolled to final gage. After one transverse rolling the billet is rotated 90° and reduced to final gage in 33 passes in the rolling mill. The rolled sheet is trimmed on a shear to final blank size.

Once the blanks have been produced, the final fabrication steps required to produce the finished NETCO-SNAP-IN<sup>®</sup> inserts are as follow. The two long edges are trimmed on a shear to provide a tapered lead-in at the bottom of the inserts to facilitate installation. The inserts are then formed on a press brake to an angle somewhat larger than 90° and the two remaining long edges roll formed. The holes that engage the installation tool can be formed by stamping or water jet cutting.

### **3.3 Applicable Codes, Standards and Regulatory Guidance**

The following codes, standards and practices are used as applicable for the design and manufacture of the NETCO-SNAP-IN<sup>®</sup> inserts.

- ANSI/ANS 8.1 - Nuclear Criticality Safety in Operations with Fissionable Materials Outside Reactors.
- ANSI/ANS 8.12 - Nuclear Criticality Control and Safety of Plutonium - Uranium Fuel Mixtures Outside Reactor.
- ANSI/ANS 8.17 - Criticality Safety Criteria for the Handling, Storage, and Transportation of LWR Fuel Outside Reactors.
- ANSI/ANS 57.2 - Design Requirements for Light Water Reactor Spent Fuel Storage Facilities at Nuclear Power Plants.
- ANSI N45.2.1 - Cleaning of Fluid Systems and Associated Components during Construction Phase of Nuclear Power Plants - 1973 (R.G. 1.37).
- ANSI N45.2.2 - Packaging, Shipping, Receiving, Storage and Handling of Items for Nuclear Power Plants - 1972 (R.G. 1.38).

- American Society for Nondestructive Testing SNT-TC-1A, June 1980, Recommended Practice for Personnel Qualifications and Certification in Nondestructive Testing.
- ASTM C750 - Standard Specification for Nuclear-Grade Boron Carbide Powder.
- ASTM C992 - Standard Specification for Boron-Based Neutron Absorbing Material Systems for Use in Nuclear Spent Fuel Storage Racks.
- ASME NQA-1 - Quality Assurance Program Requirements for Nuclear Facilities.
- ASME NQA-2 - Quality Assurance Requirements for Nuclear Power Plants.
- General Design Criterion 62, Prevention of Criticality in Fuel Storage and Handling.
- Memorandum from L. Kopp, SRE, to Timothy Collins, Chief, Reactor Systems Branch, Division of Systems Safety and Analysis, "Guidance on the Regulatory Requirements for Criticality Safety Analysis of Fuel Storage at Light Water Reactor Power Plants," August 19, 1988.
- "OT Position for Review and Acceptance of Spent Fuel Storage and Handling Applications," dated April 14, 1978, and the modifications to this document of January 18, 1979.
- 10CFR21 - Reporting of Defects and Non-compliance.
- 10CFR50 Appendix B - Quality Assurance Criteria for Nuclear Power Plants and Fuel Reprocessing Plants.
- 10CFR50.68 - Criticality Accident Requirements.
- USNRC Standard Review Plan, NUREG-0800, Section 9.1.1, New Fuel Storage and Section 9.1.2, Spent Fuel Storage.
- USNRC Regulatory Guide 1.13, Spent Fuel Storage Facility Design Basis, Rev. 2, December 1981.

### **3.4 Quality Assurance**

The NETCO-SNAP-INS<sup>®</sup> are designed and fabricated under control and surveillance of NETCO's Quality Assurance Program<sup>[3-4]</sup> that conforms to the requirements of 10CFR50 Appendix B. Since the NETCO-SNAP-INS<sup>®</sup> are used for reactivity control of fuel assemblies stored in close proximity, they are classified as nuclear Safety Related (SR).

As such, and as required by NETCO's Quality Assurance Program<sup>[3-4]</sup>, they are designed and fabricated to preclude the use of any material or manufacturing process that deviates from a rigorous set of specifications established by the NETCO design team. Process controls for materials and fabrication are established to preclude the incidence of errors and inspection steps are implemented to ensure that all critical attributes, as identified by the design team, for the feed material and rolled sheet are met in the final product.

The raw materials including AA1100, B<sub>4</sub>C and Ti used to make the cast billets are obtained by Rio Tinto Alcan from qualified suppliers. The material certifications supplied with the feed material are independently confirmed. An independent mass spectroscopic measurement of boron-10 fraction is performed on each lot of boron carbide powder used. Each cast of B<sub>4</sub>C and aluminum is chemically analyzed to assure that the composition conforms to the design specification for weight fraction of boron, Al and Ti. Permanent records of these analyses are retained in NETCO's quality assurance files. Each completed NETCO-SNAP-IN<sup>®</sup> has a unique identifying number etched along the inside upper surface and this number is fully traceable to the billet, cast and feed material lots.

For these purposes, coupons are cut from each rolled insert blank, which is of sufficient size to manufacture two NETCO-SNAP-INS<sup>®</sup>. Samples from the coupons are subjected to neutron attenuation testing to verify as-manufactured boron-10 areal density and mechanical testing to assure sufficient ductility to permit forming.

Quality Assurance procedures are enforced on the fabrication shop floor that provide all controls necessary to comply with all quality assurance requirements. One hundred percent final inspection at the shop includes dimensions, formed angle, bend, twist, cleanliness, identifying markings and freedom from imperfections.

A summary table of critical characteristics and qualification tests performed in support of those characteristics is listed below:

Table 3-1 Insert Quality Assurance Testing Summary

Critical Characteristic	Qualification Testing Performed	Acceptance Criteria
Minimum B-10 Areal Density	Neutron Attenuation Testing	> 0.0087 g B10/cm <sup>2</sup>
Material Composition	ICP Analysis	Boron, Carbon, Titanium and Aluminum within specification limits
Mechanical Properties	Tensile and Bend Testing	Tensile Strength >10 ksi Elongation > 3% Bend Test to support design specification for insert retention force.

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## References Section 3

- 3-1. "Qualification of METAMIC for Spent-Fuel Storage Application," EPRI Report No. 1003137, Prepared for EPRI by Northeast Technology Corp., Kingston, NY, 10/2001.
- 3-2. "Handbook of Neutron Absorber Materials for Spent Nuclear Fuel Transportation and Storage Applications, 2006 Edition," EPRI Report No. 1003721, Prepared by Northeast Technology Corp., Kingston, NY, 10/2006.
- 3-3. Z. Zhazy, A. Charlette, R. Ghomusheki, X.-G Chen, "Effect of Titanium on Solidification Microstructure of A-16% B<sub>4</sub>C Composites," *Light Metals*, 2005, Calgary, Alberta, Canada.
- 3-4. Quality Assurance Manual, Rev. 1, Northeast Technology Corp., 2007.

## 4.0 Engineering Evaluation of the Alcan Composite

The Alcan composite is very similar in composition to another neutron absorber material, BORAL™, that has been used extensively for more than 40 years for both wet and dry storage applications. The in-service performance of BORAL™ has been good. In this section the composition, physical properties and mechanical properties of both materials are compared and the industry experience with the BORAL™ neutron absorber reviewed.

### 4.1 Comparison of the Alcan Composite with BORAL™

#### Composition

Both of these neutron absorber materials utilize AA1100 as the base alloy for the metal matrix that retains the boron carbide. The compositions of the alloy matrices are compared in Table 4-1. With the exception of the addition of Ti to the Alcan composite, as noted previously, the compositions are almost identical. In fact, the Alcan requirement for other elements is somewhat more stringent than the BORAL™ requirement.

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Table 4-1

## Comparison of Aluminum Alloy Matrices

Property	AA1100 UNS A91100 Temper O	BORAL Metal Matrix Material Spec	ALCAN Composite Metal Matrix Material Spec	ALCAN Composite Vol 16% B <sub>4</sub> C Typical Properties	ALCAN Composite Vol 17% B <sub>4</sub> C Typical Properties
Al	99.00% min	99.00% min	99.00% min	82.7%	82.0%
Si & Fe	0.95% max	1.00% max	0.45% max	0.38%	0.39%
Cu	0.05-0.20%	0.05-0.20%	0.05-0.20%	0.11%	0.11%
Mn	0.05% max	0.05% max	0.05% max	< 0.01%	< 0.01%
Zn	0.10% max	0.10% max	0.10% max	<0.01%	0.01%
Mg	---	---	0.05% max	< 0.01%	<0.01%
Ti	---	---	1.00% - 2.50%*	1.85%	2%
B <sub>4</sub> C	---	---	---	15.3%	15.9%
Other Elements	0.15% total 0.05% max each	0.15% max each	0.15% total 0.05% max each	0.08%	0.08%
Tensile	11 ksi to 15.5 ksi	10 ksi	Not Specified	17 ksi	17 ksi
Yield	3.5 ksi min	---	Not Specified	10 ksi	10 ksi
Elongation	30% min	0.1	Not Specified	5% - 8%	5% - 8%

\*Titanium is added during mixing of the B<sub>4</sub>C and not part of the matrix material specification.

The boron carbide specifications are compared in Table 4-2. The Alcan specification is somewhat tighter in terms of allowable impurities and requires a much smaller particle size. With respect to the latter, the smaller particle size results in a more homogeneous absorber, less potential for neutron streaming and a more effective neutron absorber material.

Table 4-2  
Comparison of Boron Carbide

<b><u>BORAL™</u></b>	<b><u>Constituent</u></b>	<b><u>Alcan Composite</u></b>
70.0 min	Total Boron	76 w/o min
3.0 max	Boric Oxide	0.03 % Typ.
2.0 max	Iron	0.075% Typ.
94.0 min	Total Boron & Carbon	99.6% Typ.
75 - 250 $\mu\text{m}$	Particle Size	17.5 $\pm$ 2 $\mu\text{m}$

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#### Manufacturing Process Physical Form

The manufacturing processes for BORAL™ and the Alcan composite are compared in Figure 4-1. The manufacture of BORAL™ starts with the complete blending of atomized AA1100 powder and boron carbide. An AA1100 rectangular box ~ 12 to 15 inches on a side and a few inches high depending on the thickness of the finished product is filled with the blended powder. The walls of the box are ~ 1 inch thick. After a top is welded on the box, the billet is ready for hot rolling to final gage.

The production process for the Alcan material differs from the BORAL™ process in that the boron carbide powder is blended into molten aluminum and a rectangular billet formed by direct chill casting. Hot rolling is used to produce the final sheet.

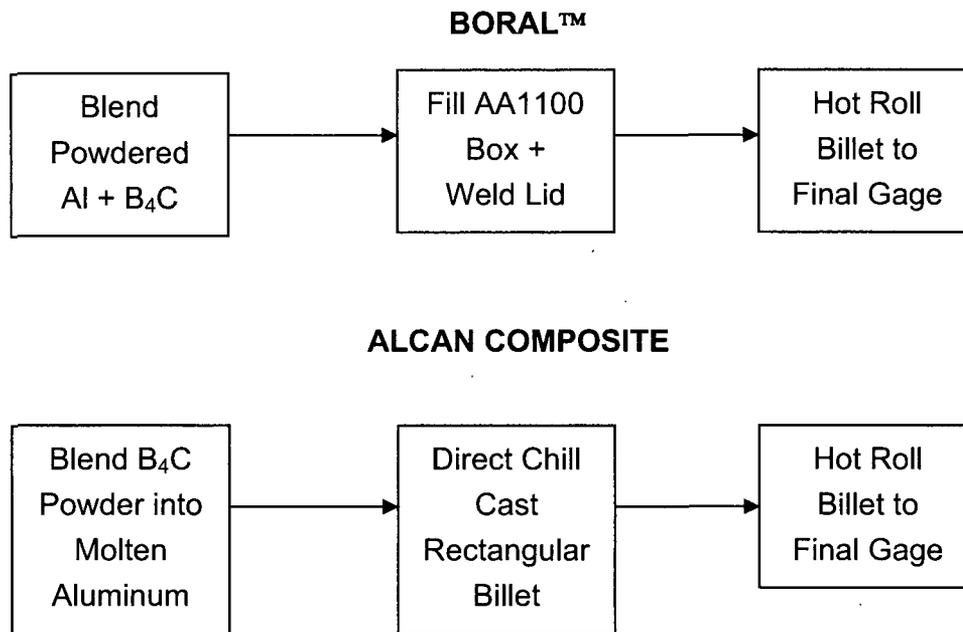


Figure 4-1: Comparison of Manufacturing Processes

In its finished form, BORAL™ consists of 1) a core of uniformly mixed and distributed boron carbide and alloy AA1100 aluminum particles; and 2) an AA1100 surface cladding on both sides of the core, serving as a solid barrier. Figure 4-2 is a micro photograph of the edge of a BORAL™ sample showing the core and clad region. BORAL™ has been produced with the core containing anywhere between 35 w% and 65 w% boron carbide. For most cores produced recently, the core contains about 50 w% boron carbide. In addition, the core is not fully dense and contains as much as 5% open and interconnected porosity.

The Alcan composite, on the other hand, in its final form is a fully dense homogeneous mixture of fine boron carbide particles embedded in the matrix aluminum alloy. As such it contains no porosity that can allow water intrusion and potential problems associated with internal moisture.

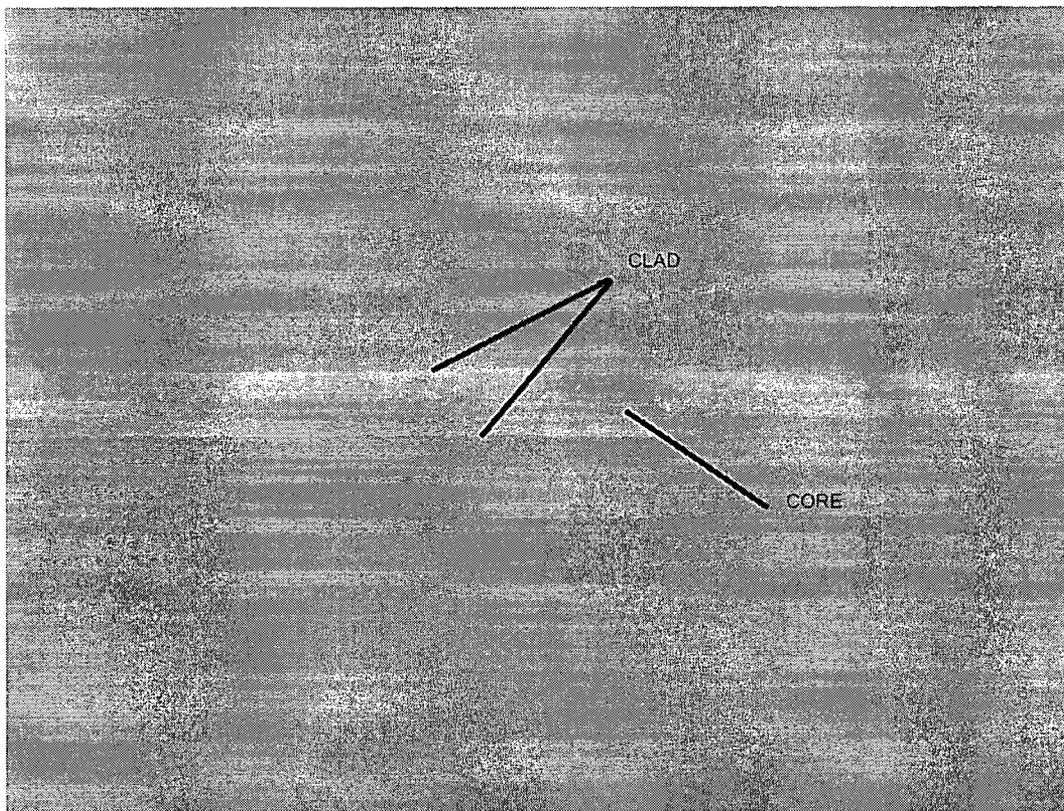


Figure 4-2: Micro Photograph of BORAL™

### Mechanical Properties

The mechanical properties of BORAL™ and the Alcan composite are compared in Table 4-3.

Table 4-3

Room Temperature Mechanical Properties of BORAL™ and the Alcan Composite

<b><u>BORAL™</u></b>		<b><u>Alcan Composite</u></b>
10	Tensile Strength, ksi	10
	Ultimate Strength, ksi	17
0.1	Elongation, %	7.0

This comparison shows that the tensile properties of the two materials are similar but the Alcan composite has improved ductility.

## Stress Relaxation

During installation, the absorber inserts are compressed from an initial bend angle greater than 90° to the square dimensions of the rack cell interior. Once installed, the inserts maintain a fixed strain within the rack storage cell that may be susceptible to relaxation over time. An analysis of stress relaxation in aluminum alloys has been performed to establish the expected performance of the inserts in this regard.

As shown above, the Rio Tinto Alcan W1100N.16 B alloy had similar mechanical characteristics to 6061 aluminum alloy based Boral material. Reference 4-1 details stress relaxation performance of 6060-T6 alloy after 1000 hours at various temperatures. The data shows approximately 15% stress relaxation after 1000 hours at 100° C.<sup>[4-1]</sup>

Average bulk pool temperatures within the LaSalle spent fuel pool are approximately 85° F. Stress relaxation at this temperature is expected to be significantly lower than 15% over 1000 hours. As an upper limit, however, data for AA1100-H112 series aluminum<sup>[4-2]</sup> was analyzed to estimate total stress relaxation after 20 years of service. The results of that analysis showed that the AA1100 series aluminum was, based upon extrapolated data, expected to have experienced an approximate stress reduction of 50% over 20 years. Given the reduced elongation of the Rio-Tinto Alcan composite in comparison with AA1100 series aluminum, this stress relaxation is likely an upper limit for the performance of the W1100N series material.

Typical breakaway withdrawal forces were measured and are typically several hundred pounds. At the 15% relaxation predicted for the 6061-T6 alloy, a reduction in retention force between 45 and 90 lbf after 1000 hours at 100° C would be expected. At the limiting case of a 50% reduction in retention force over 20 years, the inserts would still maintain greater than 150 lbf of retention within the cell if there were no other mitigating factors. These values are adequate to maintain the inserts in their configuration during fuel movement operations provided the fuel bundles are qualified for use in those

locations (i.e. they fit within the specified dimensions). However, the following factors would tend to mitigate the stress relaxation effects:

1. Stress relaxation in boron carbide reinforced aluminum will be less than for the pure alloy;
2. The formation of an oxide film on the surface of the inserts will increase the stress (by increasing the spacing between the rack wall and the insert) as well as the coefficient of friction between the insert and the cell wall.

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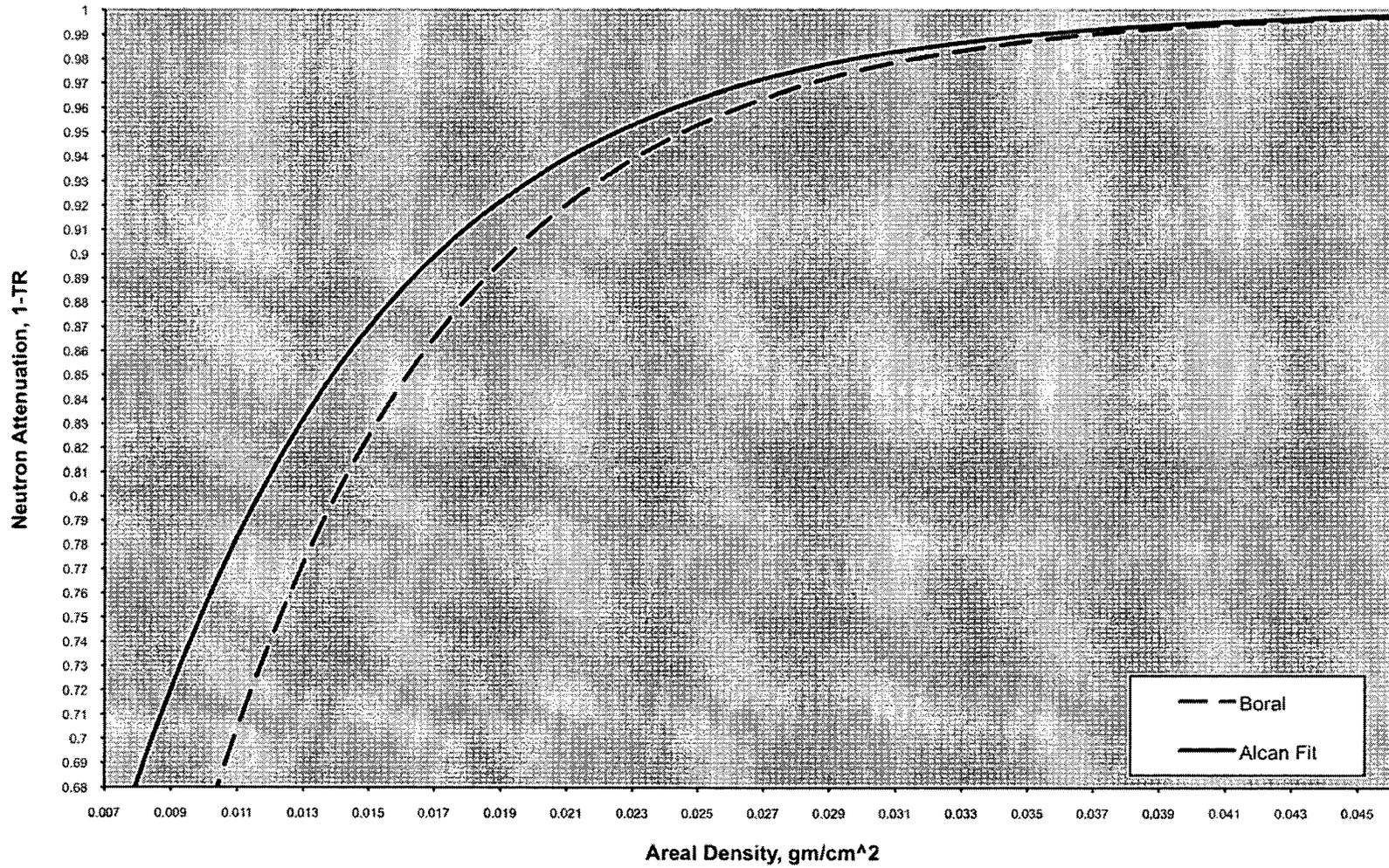
### Neutronic Properties

It has been previously noted that the average particle size of boron carbide in BORAL™ is 85 microns and individual particles can range up to 250 microns. Particles of these dimensions introduce self shielding effects that can diminish the neutron absorption effectiveness. NETCO has measured the neutron attenuation characteristics of BORAL™ and the Alcan composite, the latter material with average boron carbide particle size of 17.5 microns.

Figure 4-3 compares the neutron attenuation characteristics of BORAL™ and the Alcan composite. The neutron attenuation characteristics are measured using a collimated thermal energy neutron beam. A sample of a neutron absorber is placed in this neutron beam and the intensity of the beam incident on the absorber,  $I_i$ , is measured. The intensity of the beam transmitted through the material,  $I_t$ , is also measured and the neutron attenuation characteristic, NA, is calculated as:

$$NA = 1 - I_t/I_i$$

Figure 4-3 shows that for the same areal density BORAL™ absorbs fewer neutrons than the Alcan absorber. This illustrates the importance of neutron channeling effects in absorbers with relatively large particles when a normal mono-directional neutron beam is incident on the absorber.



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Figure 4-3: Neutron Attenuation Comparison: BORAL™ vs. Alcan Aluminum Matrix Absorber

## **4.2 In-Service Performance of Aluminum Matrix Neutron Absorber Material**

BORAL™ has been used for nuclear applications for almost 45 years starting in 1964 when it was used for reactivity control in the Yankee Rowe spent fuel racks. Nuclear applications include control elements for test reactors, fuel storage racks for spent nuclear fuel and in dry fuel storage and transportation casks. Table 4-4 contains a partial listing of research reactors where BORAL™ has been used. Table 4-5 contains a partial list of LWR plants where BORAL™ has been used in spent fuel storage racks. It is noted that LaSalle Unit 2 sister unit currently has some 43,000 lbs of BORAL™ in its racks. Table 4-6 is a partial list of plants where BORAL™ has been used for reactivity control in dry storage casks.

For dry storage applications, it is noted that the Alcan composite is now approved for use in the NUHOMS dry storage system as well as the Transnuclear metal cask storage system. The Alcan composite is being used at Peach Bottom, Limerick and St. Lucie as well as in Europe.

Table 4-4

Partial Listing of Research and Test Reactors Where BORAL™ Has Been Used

<b>Research and Test Reactors</b>
AE-6 (USAEC)
BORAX-5 (USAEC)
Brookhaven Medical Research Reactor
JEN-1 (Spain)
Philippine Research-1
Rhode Island Reactor
Triga Mark II (Italy, Austria, etc.)
University of Kansas Reactor
University of Wisconsin Reactor
Venezuela-1
Washington State University Reactor

Table 4-5

Partial List of LWRs Where BORAL™ Has Been Used in Spent Fuel Storage Racks

Pool	Plant Type	Manufacturer	Storage Locations	Country
BEAVER VALLEY 1	PWR	Holtec	1621	USA
BELLEFONTE 1	PWR	Westinghouse	1058	USA
BRAIDWOOD 1&2	PWR	Holtec	2984	USA
BROWNS FERRY 1	BWR	GE	3471	USA
BROWNS FERRY 2	BWR	GE	3471	USA
BROWNS FERRY 3	BWR	GE	3471	USA
BRUNSWICK 1	BWR	Holtec	1839	USA
BYRON 1&2	PWR	Holtec	2984	USA
CALLAWAY	PWR	Holtec	1302	USA
COMANCHE PEAK 1	PWR	Holtec	222	USA
COMANCHE PEAK 2	PWR	Holtec	219	USA
CONN YANKEE	PWR	Holtec		USA
COOK 1&2	PWR	Holtec	3613	USA
COOPER	BWR	NES		USA
CRYSTAL RIVER 3	PWR	Westinghouse	932	USA
DAVIS BESSE 1	PWR	Holtec	1624	USA
DRESDEN 1	BWR	CECO	3537	USA
DRESDEN 2	BWR	CECO	3537	USA
DRESDEN 3	BWR	CECO	3537	USA
DUANE ARNOLD	BWR	PAR	1898	USA
DUANE ARNOLD	BWR	Holtec	1254	USA
FERMI 2	BWR	Holtec	559	USA
FITZPATRICK	BWR	PAR	2797	USA
FITZPATRICK	BWR	Holtec		USA
FT. CALHOUN	PWR	Holtec	160	USA
HARRIS 1	PWR	Holtec	484	USA
HATCH 1	BWR	GE	5830	USA
HATCH 2	BWR	GE	2765	USA
HOPE CREEK	BWR	Holtec	3998	USA
HUMBOLDT BAY 3	BWR	Unknown		USA
INDIAN POINT 3	PWR	UST&D	1340	USA

Table 4-5 (con't.)

## Partial List of LWRs Where BORAL™ Has Been Used in Spent Fuel Storage Racks

KEWAUNEE	PWR	Holtec	215	USA
KOEBERG 1	PWR	Holtec		South Africa
KOEBERG 2	PWR	Holtec		South Africa
KORI-4	PWR	Holtec		South Korea
KUOSHENG 1	BWR	ENSA	1578	Taiwan
KUOSHENG 2	BWR	ENSA	1578	Taiwan
LAGUNA VERDE 1	BWR	Holtec		Mexico
LAGUNA VERDE 2	BWR	Holtec		Mexico
LASALLE 1	BWR	UST&D	4029	USA
LIMERICK 1	BWR	Holtec	2500	USA
LIMERICK 2	BWR	Holtec	2766	USA
MAINE YANKEE	PWR	PAR	1464	USA
MCGUIRE 1	PWR	Holtec	286	USA
MCGUIRE 2	PWR	Holtec	286	USA
MILLSTONE 3	PWR	Holtec	1104	USA
MONTICELLO	BWR	GE	2229	USA
NINE MILE POINT 1	BWR	Holtec	3496	USA
OYSTER CREEK	BWR	Holtec	390	USA
PERRY 1	BWR	PAR	2400	USA
PERRY 2	BWR	PAR	1620	USA
PILGRIM	BWR	Holtec	1539	USA
SALEM 1	PWR	ENC	1117	USA
SALEM 1	PWR	Holtec	1117	USA
SALEM 2	PWR	ENC	1139	USA
SALEM 2	PWR	Holtec	1139	USA
SEABROOK 1	PWR	Westinghouse	576	USA
SEQUOYAH 1	PWR	Westinghouse	2091	USA
SEQUOYAH 2	PWR	Holtec		USA
SEQUOYAH 2	PWR	PAR	2091	USA
SIZEWELL B	PWR	Holtec	1901	United Kingdom
SUMMER 1	PWR	Holtec	1712	USA
SUSQUEHANNA 1	BWR	PAR	2840	USA
SUSQUEHANNA 2	BWR	PAR	2840	USA

Table 4-5 (con't.)

## Partial List of LWRs Where BORAL™ Has Been Used in Spent Fuel Storage Racks

THREE MILE ISLAND 1	PWR	Holtec	1284	USA
TURKEY POINT 3	PWR	Holtec	131	USA
TURKEY POINT 4	PWR	Holtec	131	USA
ULCHIN 1	PWR	Holtec	1000	South Korea
VERMONT YANKEE	BWR	UST&D	2860	USA
VOGTLE 1	PWR	Unknown	1476	USA
WATERFORD 3	PWR	Holtec	2232	USA
WATTS BAR 1	PWR	Holtec	1610	USA
WATTS BAR 2	PWR	Holtec	1610	USA
YANKEE ROWE	PWR	PAR	721	USA
YONGGWANG 1	PWR	Holtec	1152	South Korea
YONGGWANG 2	PWR	Holtec	1152	South Korea
ZION 1	PWR	Holtec	3012	USA
ZION 2	PWR	Holtec	3012	USA
ANGRA 1	PWR	Holtec	1252	Brazil
CATTENOM-1	PWR	Framatome	2520	France
CATTENOM-2	PWR	Framatome	2520	France
CATTENOM-3	PWR	Framatome	2520	France
CATTENOM-4	PWR	Framatome	2520	France
BELLEVILLE-1	PWR	Framatome	1260	France
BELLEVILLE-2	PWR	Framatome	1260	France
NOGENT-1	PWR	Framatome	1260	France
NOGENT-2	PWR	Framatome	1260	France
PENLY-1	PWR	Framatome	1260	France
PENLY-2	PWR	Framatome	1260	France
GOLFECH-1	PWR	Framatome	1260	France
GOLFECH-2	PWR	Framatome	1260	France

Table 4-6

Partial Listing of LWRs Where BORAL™ Has Been Used in Dry Storage Casks

Plant	Type	Supplier	Current Inventory	Module Capacity	Absorber Type
ARKANSAS 2	Hi-Storm 100(MPC-32)	Holtec	416	32	BORAL
CATAWBA 1	UMS-24	NAC		24	BORAL
DIABLO CANYON 1	Hi-Storm 100(MPC-32)	Holtec			BORAL
DIABLO CANYON 2	Hi-Storm 100(MPC-24)	Holtec			BORAL
DRESDEN 2	Hi-Storm 100(MPC-68)	Holtec	1632	68	BORAL
DUANE ARNOLD	NUHOMS-61BT	Transnuclear	610	61	BORAL
FITZPATRICK	Hi-Storm 100(MPC-68)	Holtec	204	68	BORAL
HADDAM NECK	MPC-24	NAC	651	24	BORAL
HATCH 2	Hi-Storm 100(MPC-68)	Holtec	1496	68	BORAL
MAINE YANKEE	UMS-24	NAC	1440	24	BORAL
PALO VERDE 1	UMS-24	NAC	624	24	BORAL
PEACH BOTTOM 2	TN-68	Transnuclear	1632	68	BORAL
PRAIRIE ISLAND 1	TN-40	Transnuclear	680	40	BORAL
SEQUOYAH 2	Hi-Storm 100(MPC-32)	Holtec	96	32	BORAL
TROJAN	MPC(24)-Only	Holtec	816	24	BORAL
SUSQUEHANNA 1	NUHOMS-61BT	Transnuclear	183	61	BORAL
* as of mid 2005					

#### In-Service Experience

##### BORAL™ Plate and Sheet in Wet Storage

It has been noted that in conventional storage racks, once BORAL™ is installed in fuel racks, it is not accessible for inspection to determine its in-service performance. Accordingly, the NRC has, in the past, required utilities to initiate a coupon surveillance program when new racks were installed. A coupon surveillance program consists of a series of small coupons either in a shroud (simulating the manner in which the BORAL™ is encapsulated) or bare. The coupons are generally attached to a surveillance assembly, which is placed in a spent fuel rack storage cell.

The surveillance assembly is generally surrounded by recently discharged fuel assemblies to accelerate the rate at which the coupons accumulate gamma exposure. Prior to placing the assembly in service, the coupons are generally characterized with respect to:

- visual appearance
- dry weight
- dimensions
- specific gravity and density
- boron-10 areal density

Periodically, coupons are removed from the surveillance assembly and sent to an independent laboratory for testing. The post-irradiation test results generally mirror the pre-irradiation test results. As the surveillance coupons are prepared from BORAL™ coupons cut from panels taken from the same production lot(s) used in the racks, the performance of the coupons should be indicative of the performance of the material in the racks.

NETCO maintains laboratory facilities and offers inspection and testing services of neutron absorber surveillance coupons. In that capacity, NETCO has inspected hundreds of aluminum matrix surveillance coupons, many of them BORAL™, from spent fuel pools around the world. It has been observed during testing that some surveillance coupons can be subject to a generalized corrosion, that includes the development of a uniform oxide film. This film, once it forms, tends to be self passivating and prevents further corrosion. Depending on pool conditions, other coupons can be susceptible to localized pitting corrosion. It should be noted that while these corrosion effects can occur in aluminum matrix neutron absorbers, to date this in-service corrosion has not resulted in any detectable decrease in the boron-10 areal density. It is therefore concluded that the aluminum alloy matrix serves as suitable matrix to retain the boron carbide in spent fuel storage racks. Additional qualification testing has been performed to further demonstrate the corrosion resistance of the Alcan W1100.XYB material in

BWR and PWR spent fuel pool applications. This testing is described in Section 5.0 of this report.

### **4.3 NETCO Credentials**

NETCO has been evaluating, specifying and qualifying neutron absorber material for storage systems and transportation packaging for more than a quarter of a century. In this capacity, NETCO has become an internationally recognized expert in assessing the in-service performance of this class of materials.

In 1987, the Electric Power Research Institute (EPRI) retained NETCO to evaluate the instances of unanticipated performance of one neutron absorber, Boraflex, at two Midwest plants. NETCO's first report on this phenomenon concluded the observed shrinkage of the sheets of absorber would be expected when cross linking polymer was exposed to gamma radiation. NETCO notified EPRI that the BISCO materials qualification program did not adequately test the synergistic effects of gamma radiation and long term exposure to the pool water.

These projections subsequently proved to be remarkably accurate and formed the bases for NETCO's development of BADGER and RACKLIFE. BADGER (Boron-10 Areal Density Gage for Evaluating Racks) is a non-destructive test method that measures the residual boron-10 in spent fuel racks. BADGER has now been used in some 35 test campaigns to assess the reactivity hold down capability of both Boraflex racks and racks with other neutron absorbers.

RACKLIFE is a comprehensive computer program that tracks the performance of each and every Boraflex panel (as many as 4,000) in a typical rack installation. RACKLIFE is based on first principles and on the mass balance of soluble silica as it dissolves from the degraded Boraflex matrix and gradually migrates to the bulk pool volume. NETCO tested samples of irradiated Boraflex and measured the rate of dissolution as a function of both absorbed dose and temperature in its laboratory. This experimental data serves as the basis for the RACKLIFE dissolution model. This model and the RACKLIFE

software upon which it is based as verified by BADGER measurements, serve to assure that spent fuel pool criticality limits are met.

At the Penn State Breazeale Research Reactor laboratory, NETCO routinely tests neutron absorber surveillance coupons. These tests include Alcan composite material, BORAL™, Boraflex, borated stainless steel, METAMIC, Talbor, Carborundum and ESK borated graphite and nano steel.

NETCO was selected by Reynolds Metals Company to conduct qualification testing of its new neutron absorber material, METAMIC. In this test sequence, NETCO conducted accelerated radiation testing, accelerated corrosion testing and elevated temperature testing to qualify this material for use in spent fuel racks and storage and transportation casks. The resulting test report has been accepted by the NRC for both wet and dry applications. A second qualification test sequence for another new neutron absorber BorTec™ was completed by NETCO for DWA Technologies, the manufacturer of BorTec™. As such NETCO is the only organization to have successfully qualified new neutron absorber materials for wet storage applications since BORAL™ was qualified some 40 years ago.

NETCO was retained by EPRI, ENRESA and AAR (former BORAL™ manufacturer) to evaluate clad blister formation under cask drying conditions. Laboratory testing by NETCO simulating cask drying condition lead to recommended changes in the AAR rolling schedule and lead to an improved BORAL™ product that is largely blister resistant.

**References Section 4:**

- 4-1 K. Farrell, "ORNL/TM-13049 Assessment of Aluminum Structural Materials for Service Within the ANS Reflector Vessel," Oak Ridge National Laboratory, August 1995
- 4-2 John Gilbert Kaufman, Properties of Aluminum Alloys, ASM International, 1999

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## 5.0 Accelerated Corrosion Testing

### 5.1 Test Description

The accelerated corrosion test program has been designed to determine the susceptibility of the Rio Tinto Alcan composite to general (uniform) and localized (pitting) corrosion in PWR and BWR spent fuel pools. Two sets of coupons have been tested at the NETCO laboratory; one set in deionized water, simulating BWR pool conditions and one set in deionized water containing 2500 ppm boron as boric acid, simulating PWR pool conditions. Both tests were conducted at 195°F (90.5°C) to accelerate any corrosion effects, which might occur after the 8000 hour (~ 1 year) test period. The tests are accelerated by testing at elevated temperatures relative to typical temperatures in the actual service environment. Typically, spent fuel pools are operated in the temperature range of 80 to 100°F (27 to 38°C) with short term excursions to 130°F (54°C) during refueling outages.

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Coupons from each environment were removed after approximately 2000, 4000, 6000 and 8000 hours and subjected to testing. Prior to testing the coupons were precharacterized with respect to thickness, weight and boron-10 areal density. After testing, the coupons were subjected to post-test characterization of these same attributes. The testing after 2000, 4000, 6000 and 8000 hours has been completed. This document represents the final report for this accelerated corrosion test.

### 5.2 Test Matrices and Coupon Description

The coupon test matrix for the accelerated corrosion test is shown in Table 5-1. A total of 168 coupons have been tested; 84 in deionized water and 84 in 2500 ppm boron as boric acid. As shown in Table 5-1, coupons with two levels of boron carbide loadings were tested. The coupons with an intermediate loading contain 16 vol% boron carbide. The coupons with the maximum boron carbide loading contain 25 vol% boron carbide.

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Table 5-1

## Coupon Test Matrix for Accelerated Corrosion Testing

Type of Coupon	Number of Coupons	
	16 vol % B <sub>4</sub> C	25 vol % B <sub>4</sub> C
General	12	12
Bend	12	12
Galvanic (bi-metallic)*	18	18
Subtotal	42	42
Total	84	

\*Note: For the galvanic bi-metallic coupons, there are 3 subtypes. These are SS-304L, Zircaloy and Inconel 718, each separately in combination with the Alcan composite.

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At each of the scheduled test campaigns specific coupons were removed from the baths and subjected to testing. The number and type of coupons tested at 2000 and 6000 hours is summarized in Table 5-2. The number and type of coupons tested at 4000 and 8000 hours is summarized in Table 5-3. Three types of coupons at two boron carbide loadings have been tested as described in Tables 5-2 and 5-3.

Table 5-2

## Number and Type of Coupons per Bath Tested After 2000 and 6000 Hours

Type of Coupon	Boron Carbide Loading	
	16 vol %	25 vol %
General (G)	3	3
Bend (B)	3	3
Total	12	

Table 5-3

Number and Type of Coupons per Bath Tested After 4000 and 8000 Hours

Type of Coupon	Boron Carbide Loading	
	16 vol %	25 vol %
General (G)	3	3
Bend (B)	3	3
Bi-Metallic (BSS)	3	3
Bi-Metallic (BZ)	3	3
Bi-Metallic (BI)	3	3
Total	30	

Since the NETCO-SNAP-INS<sup>®</sup> are to be used with a mill finish absorber material, that is the finish of the coupons tested. For each coupon type the corrosion rates are determined. The three coupon types are described below.

#### *General*

General coupons are rectangular (nominally 4 in. x 2 in. in length and width). A test objective of the general coupons is to determine the rate at which a uniform oxide film forms. The rate of oxide build-up is determined by changes in the coupon weight and thickness. Post test exposure, the coupons are subject to precision weighing prior to testing and after a sequence of nitric acid washes and drying after testing.

#### *Bend*

Coupons with press brake formed bends are included in the test matrix. These coupons have been deformed to the same bend angle and bend radius used for the NETCO-SNAP-IN<sup>®</sup>. The test objective of the bend coupons is to determine whether or not bend deformation and stress adversely affect the corrosion susceptibility of the Alcan material. These will be subject to the same pre and post testing as the general coupons. In addition the inner and outer bend radius will be subject to microscopy before and after acid cleaning.

### *Galvanic (Bi-Metallic) Coupons*

In conventional spent fuel racks the neutron absorber material is enclosed in 304L stainless steel wrapper plate, thus the potential for aluminum/stainless steel galvanic corrosion exists. In the NETCO-SNAP-IN<sup>®</sup> application this material is used unsheathed so that it could be in contact with LWR fuel assemblies. Of the materials in LWR fuel assemblies supplied by U.S. fuel manufacturers, only stainless steel, Inconel and Zircaloy could contact the surface of fuel racks. Accordingly bi-metallic coupons have been prepared with Alcan composite and:

304L stainless steel

Inconel 718

Zircaloy

The test objective of the galvanic coupon is to evaluate the potential for galvanic corrosion. The above alloy coupons are nominally 2 in. x 4 in. x 0.065 in. thick. A piece of 2 in. x 4 in. Alcan composite forms the other piece of each couple. The two metals comprising each couple are fastened to each other mechanically with AA1100 wire. Inspection of the galvanic coupons is via optical microscopy, thickness and dry weight measurements. Post test acid cleaning is used depending on the depth of any oxide films.

### **5.3 Water Chemistry**

The laboratory tap water was processed by first passing it through two universal ion exchange columns and then through two research grade ion exchange columns. The typical quality of the deionized water used for both the BWR and PWR corrosion baths and make up water is shown in Table 5-4.

Table 5-4

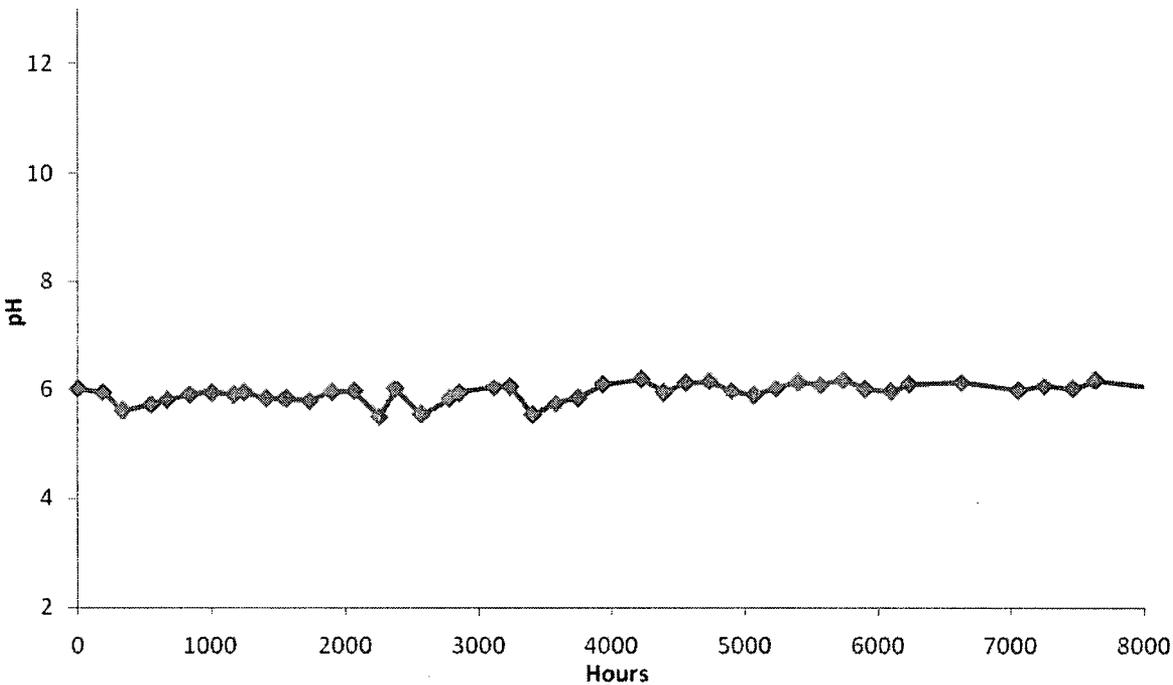
## Water Specification for Corrosion Baths

pH	5.75 @ 20°C
Conductivity, : $\mu\text{s}/\text{cm}$	0.5
Resistivity, : $\Omega/\text{cm}$	2.0
Aluminum	< 0.010 ppm
SiO <sub>2</sub>	< 0.100 ppm
Cl	< 0.010 ppm
Na	< 0.030 ppm

To the PWR bath, sufficient reagent grade boric acid was added to bring the boron concentration to ~ 2500 ppm. This increased the initial conductivity from < 1.0  $\mu\text{s}/\text{cm}$  to ~ 40  $\mu\text{s}/\text{cm}$  @ 20.0°C and decreased the initial pH from 5.75 @ 20.0°C to ~ 4.76 at 20.0°C.

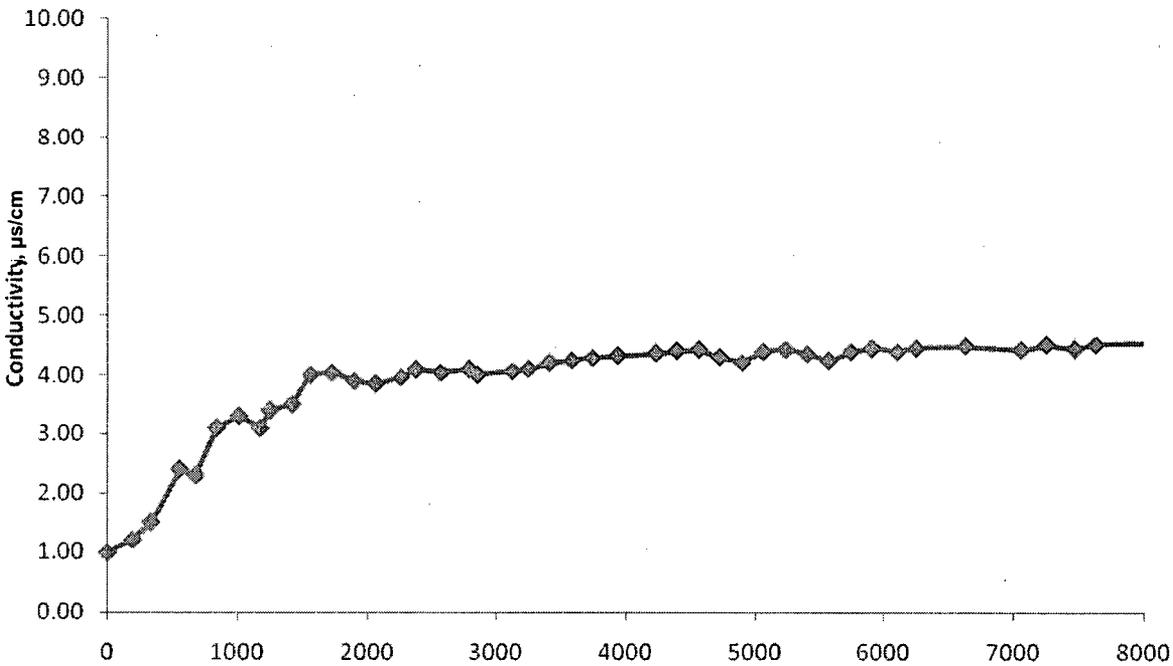
The conductivity and pH of each of the baths was measured at an approximate frequency of once per week. Figures 5-1 and 5-2 are plots of the measured pH and conductivity of the BWR bath versus time. The plots of the measured pH and conductivity versus time of the PWR bath are shown in Figures 5-3 and 5-4, respectively.

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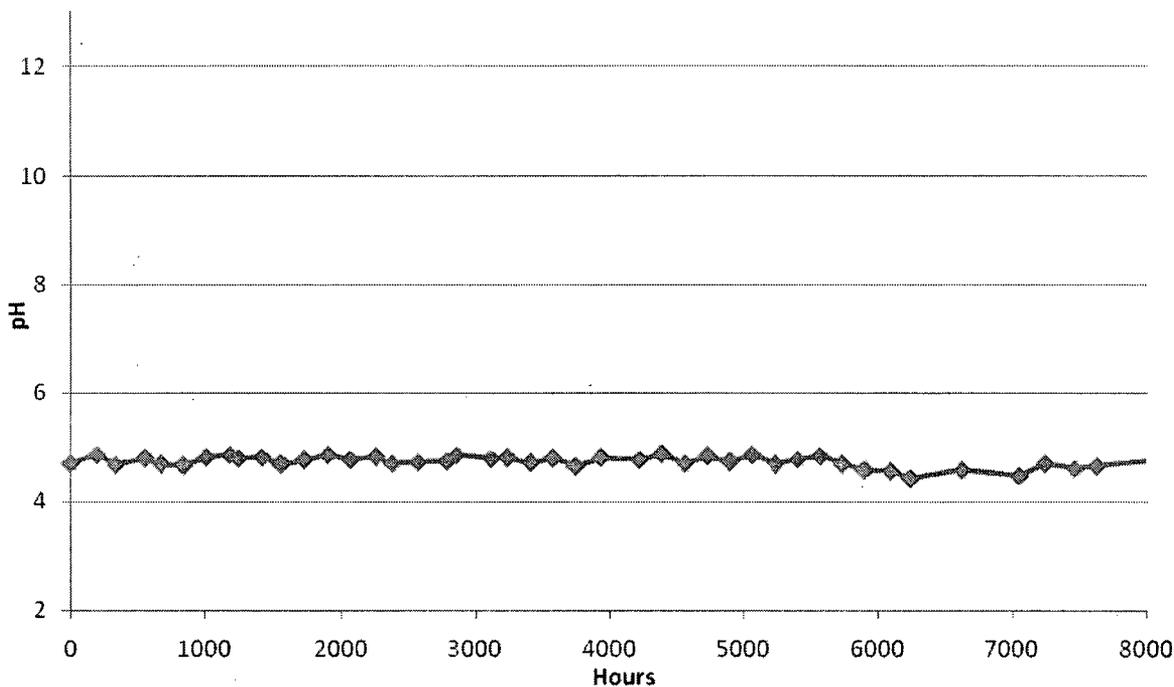
Figure 5-1: pH versus Time: BWR Accelerated Corrosion Test



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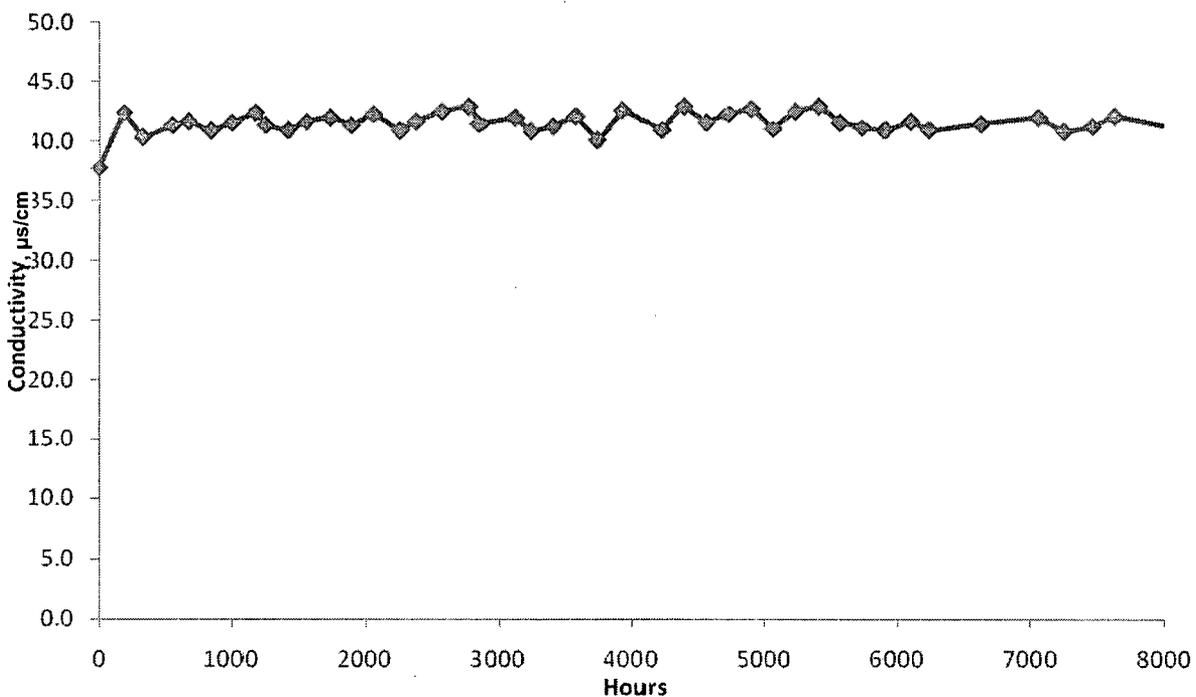
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Figure 5-2: Water Conductivity versus Time: BWR Accelerated Corrosion Test



4

Figure 5-3: pH versus Time: PWR Accelerated Corrosion Test



1

4

Figure 5-4: Water Conductivity versus Time: PWR Accelerated Corrosion Test

## **5.4 Corrosion Test Results**

### Visual Inspection and Microscopy

All coupons removed from the BWR and PWR accelerated corrosion tests after 2000, 4000, 6000 and 8000 hours were subjected to visual inspection. Some of the coupons were subjected to optional microscopy. In addition, high resolution macro photographs were taken both upon removal from the test solutions and after air drying. Optical microscopy was also performed to verify that the oxide films were substantially removed prior to determining coupon weight loss and prior to inspecting for any anomalies along the outer bend radii of the bend coupons. The coupons and the digital data files of all macro photographs, microphotographs and photo micrographs are in permanent storage.

A sampling of photographs and photomicrographs are included in this report to illustrate the pre and post test appearance of the coupons. Photographs and photomicrographs from the 4000 hour and 8000 hour inspections are compared to illustrate little change in coupon surface appearance once an initial oxide film forms. This suggests that the initial oxide film is largely self-passivating, limiting the rate of subsequent oxidation of the base metal.

Figure 5-5 is a macrophotograph that compares the appearance of the 16 vol % coupons exposed to the BWR and PWR test conditions for 4000 hours with an as-fabricated archive coupon. The archive coupon is a somewhat darker grey color than pure AA1100 aluminum. This appearance is characteristic of mill finish aluminum that is darkened somewhat by numerous small black boron carbide particles embedded in its surface. Figure 5-6 contains a similar macrophotograph for coupons inspected after 8000 hours.

The coupons exposed to the BWR environment have a more or less uniform white oxide coating with some larger black areas where the boron carbide areas have been exposed. The areas of exposed boron carbide are larger than on the archive material. This may be due to boron carbide that was near the surface but covered by a thin layer

of aluminum in the as-produced material. Once oxidized, the thin oxide layer has insufficient strength to adhere to the underlying boron carbide and is loosened by the circulating bath water.

The coupons exposed to the PWR environment appear somewhat different. The background color is a light grey with smaller areas of exposed boron carbide on the surface. Examination of the surface under a microscope revealed the surface is covered with a uniform translucent film showing some of the color of aluminum through the film. Randomly interspersed are smaller dark areas of exposed boron carbide particles. The surface appearance is similar after 4000 hours and 8000 hours.

Figures 5-7 and 5-8 compare the post test coupons with archive material for the composite with 25 vol % boron carbide loading after 4000 and 8000 hours, respectively. It is noted that the archive material has a darker appearance than the archive material with 16 vol % due to the higher B<sub>4</sub>C loading. Similarly, the post test coupons are somewhat darker for the same reason. The oxidized surfaces of these post test coupons are similar to the surfaces of the 16 vol % post test coupons and the appearances maintain their similarity after 4000 and 8000 hours exposure.

Figures 5-9 and 5-10 are microphotographs that compare the appearance of the 16 vol % post test coupons after 4000 and 8000 hours, respectively, with archive material at 8X magnification. Figures 5-11 and 5-12 are microphotographs of 25 vol % coupons exposed to demineralized water and boric acid after 4000 hours and 8000 hours, respectively.

Figures 5-13 and 5-14 are photomicrographs of the surfaces of 16 vol % and 25 vol % boron carbide composite, respectively, in the as-fabricated condition. These can serve as a reference when evaluating the surface condition of the composites after 8000 hours exposure to demineralized water and boric acid.

Figures 5-15 and 5-16 are photomicrographs of the 16 vol % B<sub>4</sub>C composite after 8000 hours in demineralized water and boric acid, respectively. The surface appearance in Figure 5-15 is characteristic of areas of between locally heavy surface boron carbide (see e.g. Figure 5-10).

Figures 5-17 and 5-18 are photomicrographs of the 25 vol % B<sub>4</sub>C composite after 8000 hours in demineralized water and boric acid, respectively.

The photograph and microphotographs contained in Figures 5-5 through 5-18 serve to illustrate that all coupons develop a more or less uniform oxide film on all surfaces. The appearance of the coupons after 4000 hours exposure compared to their appearance after 8000 hours exposure show that they are essentially identical. This suggests that the oxide film, once formed, is self-passivating and retards subsequent corrosion of the base metal. This conclusion is further supported by the quantitative corrosion rate measurements described subsequently.

The difference in appearance of the coupons exposed to demineralized water and boric acid suggests the different pHs of the baths result in different forms of the oxide. Subsequent acid cleaning of the oxide to measure weight loss further supports this hypothesis. For the coupons exposed to demineralized water, the oxidized layer is readily removed by one short soak in nitric acid. For the coupons exposed to the boric acid solution, it required several longer soak periods in order for oxide removal and the coupons to achieve constant weight. It is postulated that at lower pH in the boric acid condition,  $\alpha$ -alumina (Al<sub>2</sub>O<sub>3</sub>) forms, which is less soluble in nitric acid than the oxide formed at a higher pH in demineralized water. The latter form of the oxide formed at the higher pH in demineralized water is thought to be a hydrated form or Gibbsite (Al<sub>2</sub>O<sub>3</sub>·3H<sub>2</sub>O). Optical microscopy of the inside and outside radius of the bend coupons before and after acid cleaning revealed no cracks or other anomalous corrosion behavior.

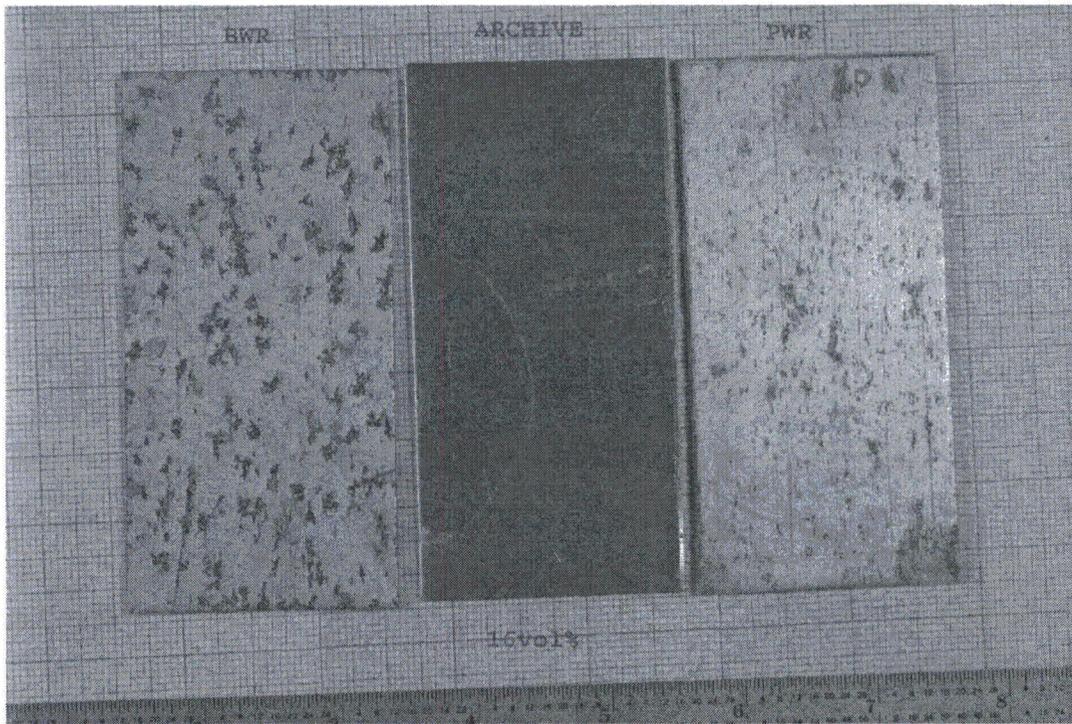


Figure 5-5: Comparison of Post-Test Coupon Appearance with Archive Material: 16 vol % Boron Carbide Loading after 4000 Hours

4

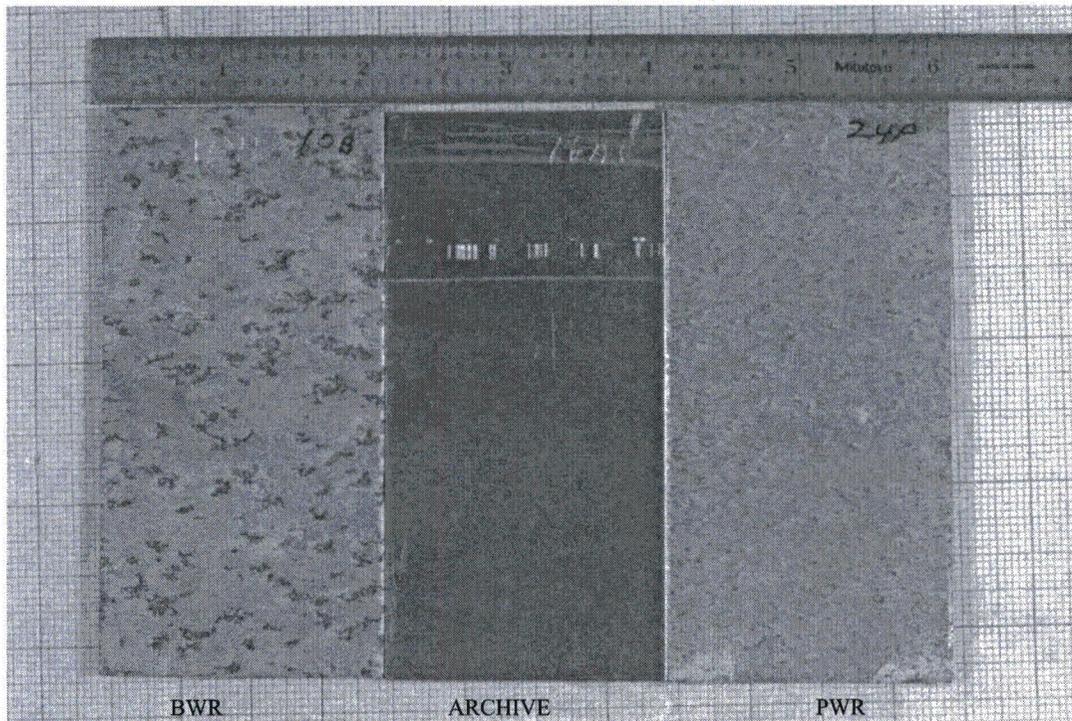


Figure 5-6: Comparison of Post-Test Coupon Appearance with Archive Material: 16 vol % Boron Carbide Loading after 8000 Hours

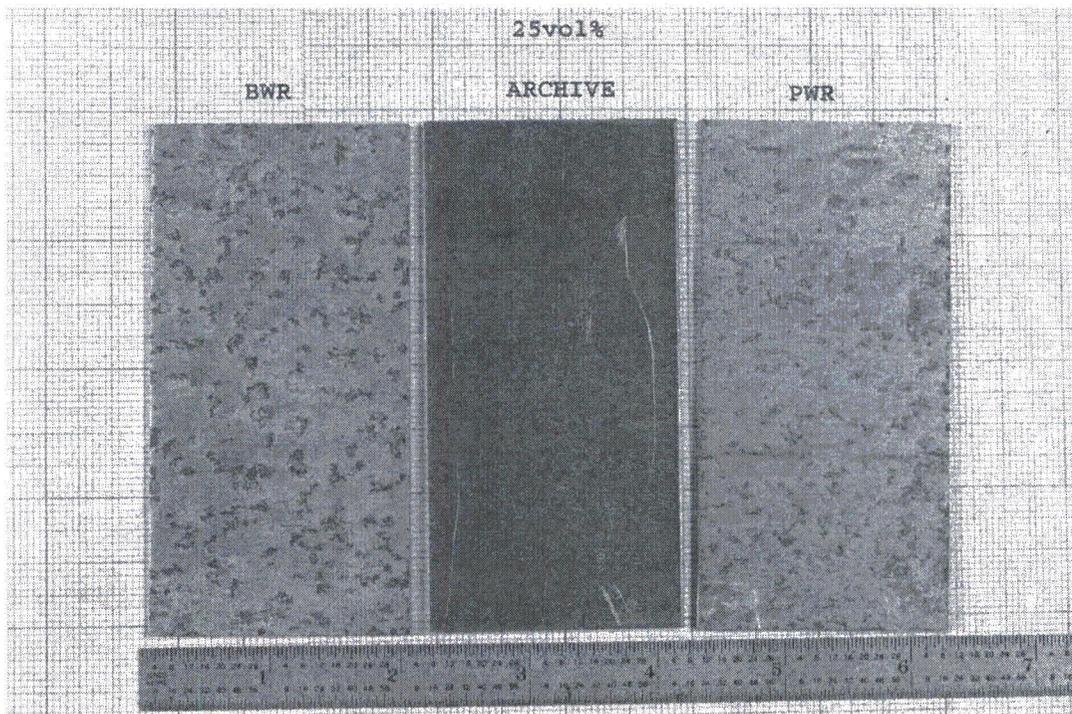


Figure 5-7: Comparison of Post-Test Coupon Appearance with Archive Material: 25 vol % Boron Carbide Loading after 4000 Hours

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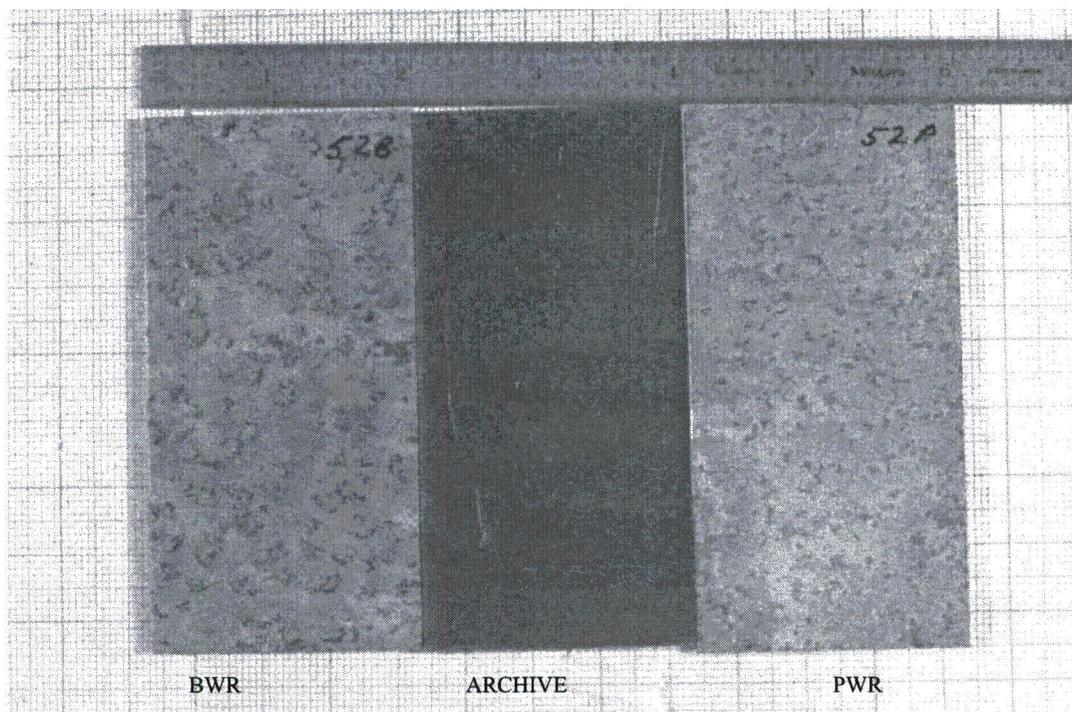


Figure 5-8: Comparison of Post-Test Coupon Appearance with Archive Material: 25 vol % Boron Carbide Loading after 8000 Hours

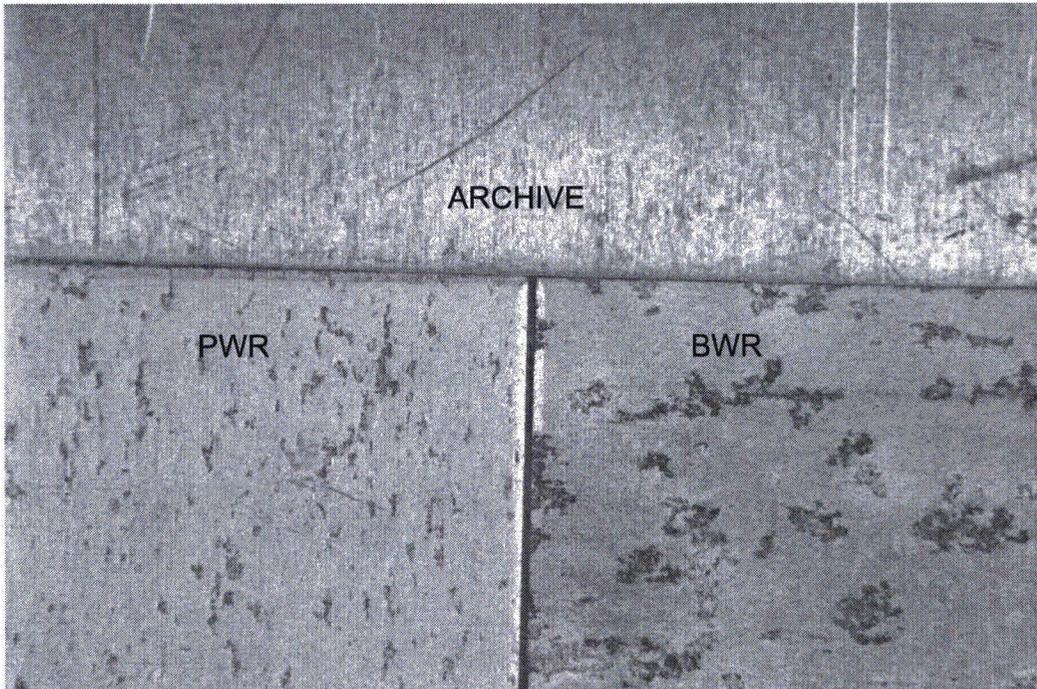


Figure 5-9: Comparison of Post-Test Coupon Appearance with Archive Material: 16 vol % Boron Carbide Loading at 8X Magnification after 4000 Hours

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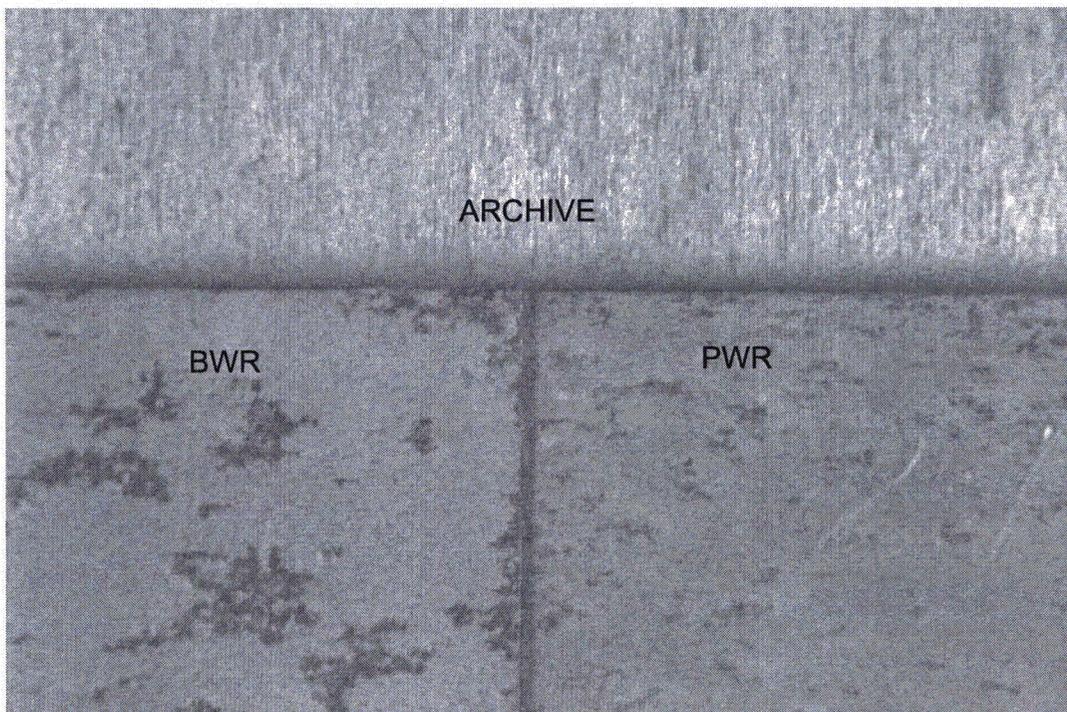


Figure 5-10: Comparison of Post-Test Coupon Appearance with Archive Material: 16 vol % Boron Carbide Loading at 8X Magnification after 8000 Hours

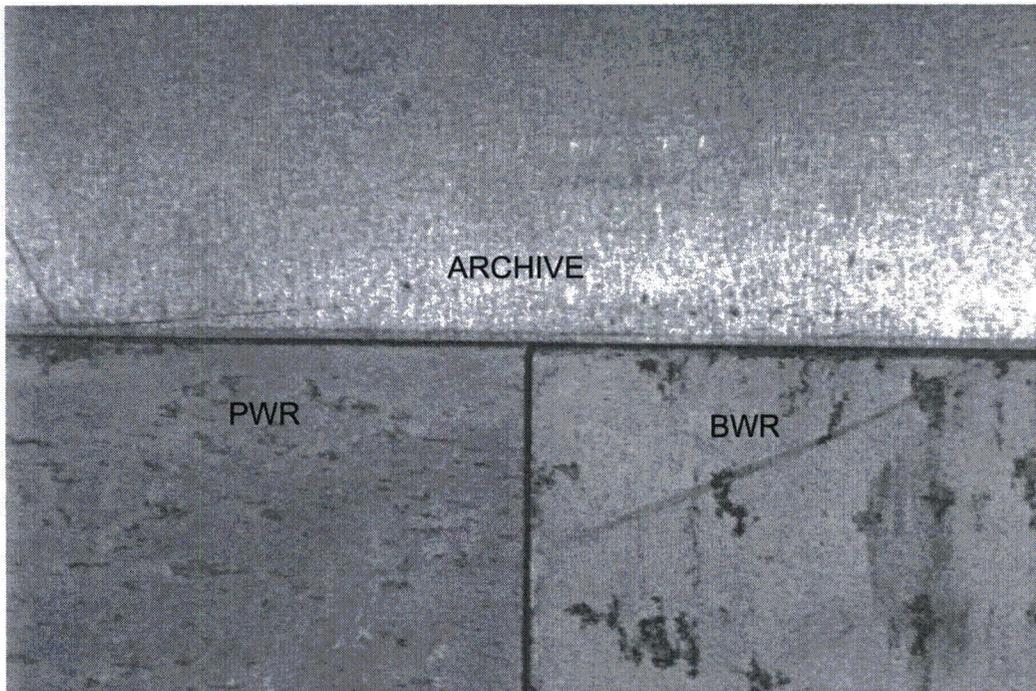


Figure 5-11: Comparison of Post-Test Coupon Appearance with Archive Material: 25 vol % Boron Carbide Loading at 8X Magnification after 4000 Hours

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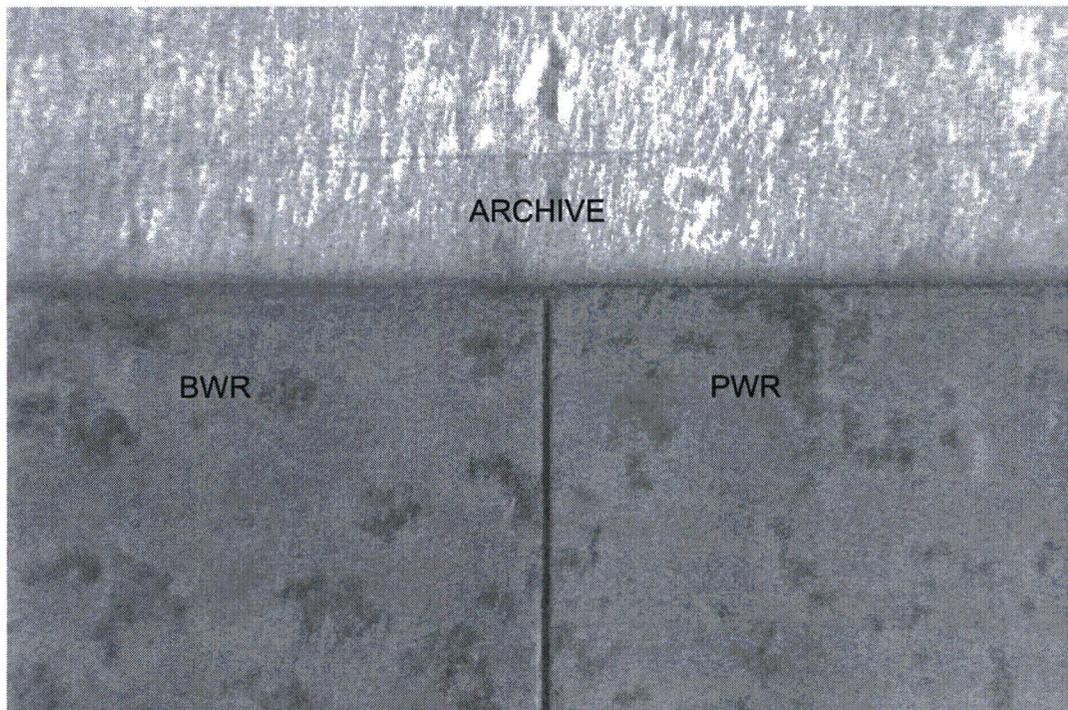


Figure 5-12: Comparison of Post-Test Coupon Appearance with Archive Material: 25 vol % Boron Carbide Loading at 8X Magnification after 8000 Hours



Figure 5-13: Photomicrograph of 16 vol % B<sub>4</sub>C Composite As-Fabricated (45X)

4



Figure 5-14: Photomicrograph of 25 vol % B<sub>4</sub>C Composite As-Fabricated (45X)



Figure 5-15: Photomicrograph of 16 vol % B<sub>4</sub>C Composite after 8000 Hours in Demineralized Water (45X)

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Figure 5-16: Photomicrograph of 16 vol % B<sub>4</sub>C Composite after 8000 Hours in Boric Acid (45X)



Figure 5-17: Photomicrograph of 25 vol % B<sub>4</sub>C Composite after 8000 Hours in Demineralized Water (45X)

4

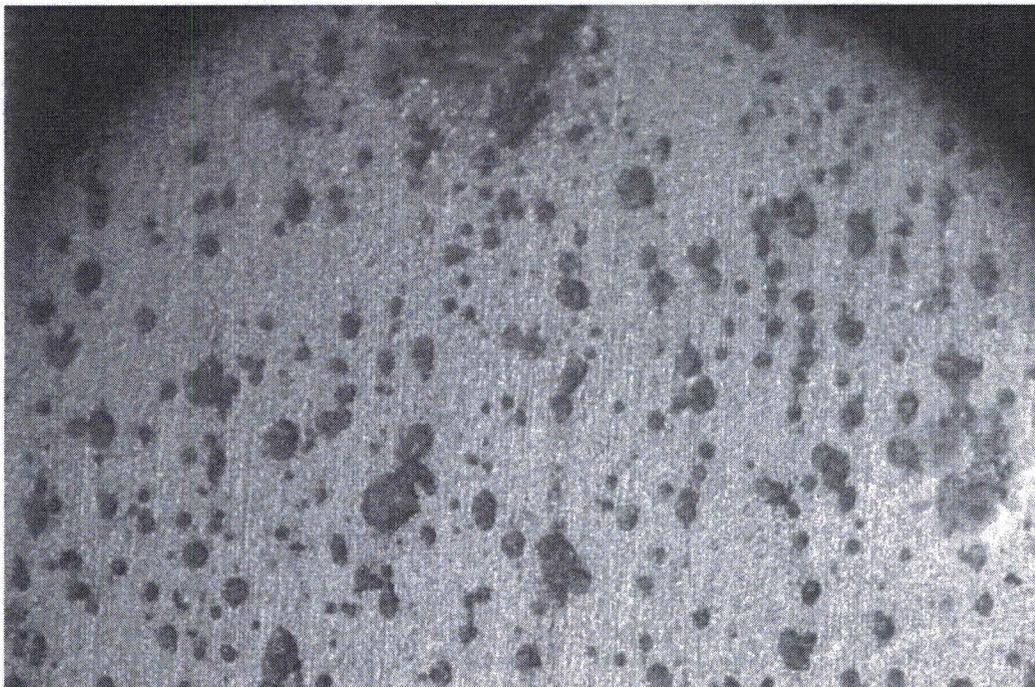


Figure 5-18: Photomicrograph of 25 vol % B<sub>4</sub>C Composite after 8000 Hours in Boric Acid (45X)

### Quantitative Corrosion Measurements

The change in coupon thickness can provide a semi-quantitative measure of the extent and progression of corrosion. The change in coupon weight, on the other hand, can provide a more accurate measure of the extent and progression of corrosion. As the aluminum base metal on the surface of the coupons is converted to the oxide (either  $\text{Al}_2\text{O}_3$  or  $\text{Al}_2\text{O}_3 \cdot n \text{H}_2\text{O}$ ), the volume of the oxide exceeds the volume of the original base metal consumed. Concurrent with this conversion is an increase in thickness and increase in weight.

The pre-test thickness of each general coupon was measured at nine locations. The pre-test thickness of each bend coupon was measured at six locations. The post-test thickness was measured at the same nine and six locations on the general and bend coupons, respectively. The values reported are the average of either nine or six measurements for each coupon. The pre and post test coupons' weights were measured for each coupon after a one hour drying at  $110^\circ\text{C}$  to remove surface moisture.

Figures 5-19 and 5-20 contain the results of the average coupon thickness changes versus exposure times for the BWR and PWR coupons, respectively. The data show that, with one exception, the coupons show a measurable increase in thickness. The scatter in the data is likely attributable to measuring small changes in thickness in relatively thin samples. To place this data in perspective, a 5% increase in thickness on a coupon of initial thickness of 0.080 inches is 0.004 inches or 4 mils. If it is assumed that both sides of the coupon contribute equally then the increase is 0.002 inches per side. After 8000 hours, the average change in the thickness of the coupons exposed to accelerated BWR conditions is 4.2%; the corresponding value for the PWR coupons is 4.1%. Figures 5-21 and 5-22 contain plots of coupon weight change versus exposure time.

At the 4000 hour and 8000 hour tests, eighteen of the galvanic corrosion couples were removed from each test bath and were subjected to testing. These included three 304L stainless steel couples, three Inconel 718 couples and three Zircaloy coupons at each

B<sub>4</sub>C loading. No couples were tested at 2000 hours and 6000 hours. Figures 5-23 and 5-24 show the weight change of the BWR and PWR couple samples after coupon drying.

In accordance with ASTM G-31-72<sup>[1]</sup> and ASTM G-1-03<sup>[2]</sup>, the coupons were cleaned in 1.42 sp.gr. nitric acid to remove the corrosion products. The intent of cleaning is to remove all of the corrosion products but none of the base metal so that the weight of the corrosion products can be determined. This weight change can subsequently be used to determine the corrosion rate in mils/year.

As noted previously, the oxide on coupons in the BWR test was easily removed by one or two ten-minute soaks in nitric acid at room temperature. For the PWR coupons it required several successive soak periods. After each cleaning cycle, the coupons were dried and reweighed. The cleaning proceeded until the coupons achieved constant weight or visual and/or microscopic examination indicated the oxide film had been removed.

Figures 5-25 and 5-26 show the average change in coupon thickness after acid cleaning for the BWR and PWR general and bend coupons, respectively. Figures 5-27 and 5-28 show the change in coupon thickness after acid cleaning for the BWR and PWR galvanic coupons, respectively, after 4000 hours and 8000 hours. These values represent the thickness of oxide removed by acid cleaning. The average change in thickness by coupon type are summarized in Table 5-5. The data in Table 5-5 illustrate the difficulty in obtaining accurate changes in coupon thickness when the specimens are very thin. This is further evidenced by the variability in the coupon thickness data.

The coupon weight changes as a result of acid cleaning are shown in Figures 5-29 and 5-30 for the BWR and PWR general and bend coupons, respectively. The coupon weight changes as a result of acid cleaning are shown in Figures 5-31 and 5-32 for the BWR and PWR galvanic coupons, respectively. The average change in coupon weight by coupon type are summarized in Table 5-6. This table shows the weight changes

exhibit far less variability than the thickness changes in Table 5-5. Accordingly, the weight change data are used subsequently to compute corrosion rates.

Table 5-5

## Summary of Coupon Thickness Changes by Coupon Type

<b>Coupon Types</b>	<b>Thickness Change, %</b>			
	<b>2153 Hours</b>	<b>4019 Hours</b>	<b>5871 Hours</b>	<b>8119 Hours</b>
<b>BWR General and Bend</b>				
16 vol % B <sub>4</sub> C	-1.14% ± 0.59%	-3.08% ± 0.64%	-1.40% ± 1.07%	-0.55% ± 0.88%
25 vol % B <sub>4</sub> C	-0.72% ± 0.89%	-2.09% ± 1.40%	0.08% ± 0.44%	-0.94% ± 0.79%
<b>BWR Galvanic</b>				
16 vol % B <sub>4</sub> C		-0.07% ± 0.68%		-0.91% ± 0.35%
25 vol % B <sub>4</sub> C		-1.05% ± 0.35%		-0.98% ± 0.81%
<b>PWR General and Bend</b>				
16 vol % B <sub>4</sub> C	-0.03% ± 0.51%	-2.73% ± 1.14%	-0.70% ± 0.92%	-3.32% ± 4.64%
25 vol % B <sub>4</sub> C	-0.55% ± 0.46%	-1.24% ± 1.05%	0.44% ± 1.10%	-0.13% ± 0.82%
<b>PWR Galvanic</b>				
16 vol % B <sub>4</sub> C		-1.04% ± 0.81%		-2.55% ± 3.25%
25 vol % B <sub>4</sub> C		-1.24% ± 0.27%		-0.40% ± 0.88%

Table 5-6

## Summary of Coupon Weight Changes by Coupon Type

<b>Coupon Types</b>	<b>Weight Change, %</b>			
	<b>2153 Hours</b>	<b>4019 Hours</b>	<b>5871 Hours</b>	<b>8119 Hours</b>
<b>BWR General and Bend</b>				
16 vol % B <sub>4</sub> C	-0.15% ± 0.12%	-0.28% ± 0.22%	-0.68% ± 0.19%	-0.50% ± 0.17%
25 vol % B <sub>4</sub> C	-0.29% ± 0.27%	-0.36% ± 0.38%	-0.68% ± 0.25%	-0.69% ± 0.75%
<b>BWR Galvanic</b>				
16 vol % B <sub>4</sub> C		0.05% ± 0.2%		-0.34% ± 0.19%
25 vol % B <sub>4</sub> C		-0.24% ± 0.27%		-0.81% ± 0.28%
<b>PWR General and Bend</b>				
16 vol % B <sub>4</sub> C	-0.12% ± 0.04%	-0.07% ± 0.09%	-0.10% ± 0.12%	-0.26% ± 0.37%
25 vol % B <sub>4</sub> C	-0.04% ± 0.07%	-0.02% ± 0.05%	-0.17% ± 0.12%	-0.16% ± 0.09%
<b>PWR Galvanic</b>				
16 vol % B <sub>4</sub> C		-0.64% ± 0.77%		-0.89% ± 0.46%
25 vol % B <sub>4</sub> C		-0.79% ± 0.98%		-0.66% ± 0.46%

Boron-10 Areal Density

The boron-10 areal density of the general coupons was measured using neutron attenuation testing. The results of the post-test measurements were compared with similar testing of archive coupons. Figures 5-33 and 5-34 contain plots of the change in boron-10 areal density versus exposure time for the BWR and PWR coupons, respectively. To place these measurements in perspective, the average areal density of the 16 vol % coupons is 0.010 gms B-10/cm<sup>2</sup>; the corresponding areal density of the 25 vol % coupons is 0.0176 gms B-10/cm<sup>2</sup>. The variation in areal density changes are within ± 1.0 to 2.0% of zero change, which is within the absolute uncertainty of the areal density measurements.

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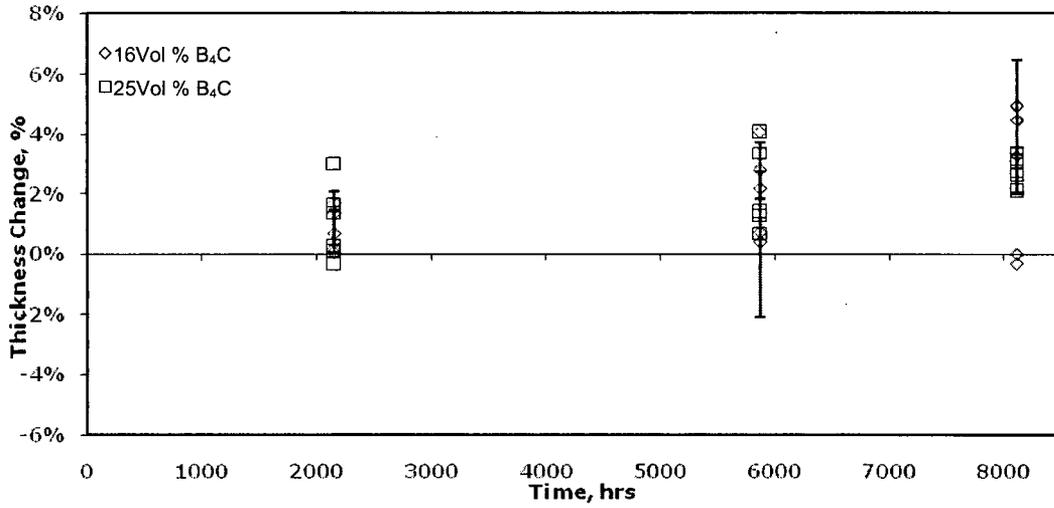


Figure 5-19: BWR Thickness Change (Pre-Test vs. Post-Test)

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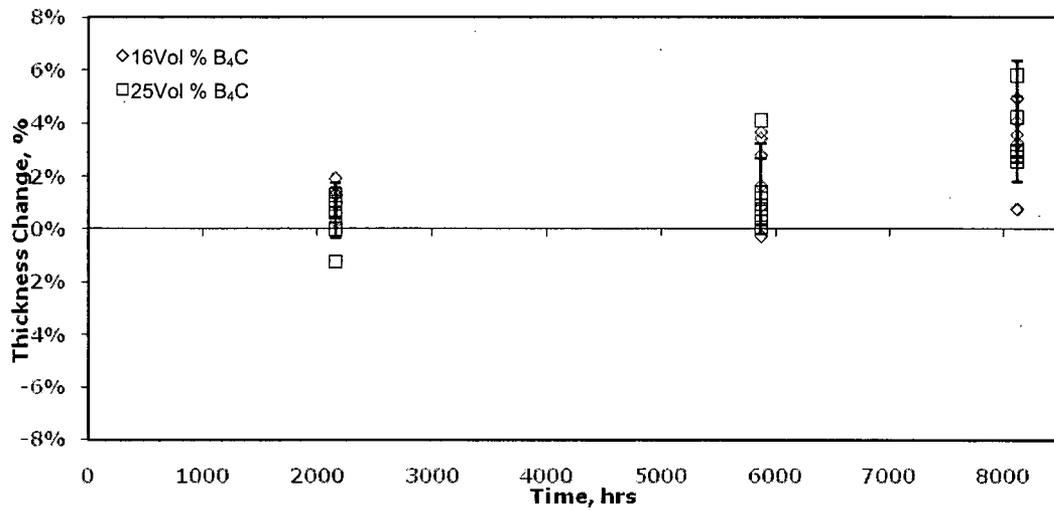


Figure 5-20: PWR Thickness Change (Pre-Test vs. Post-Test)

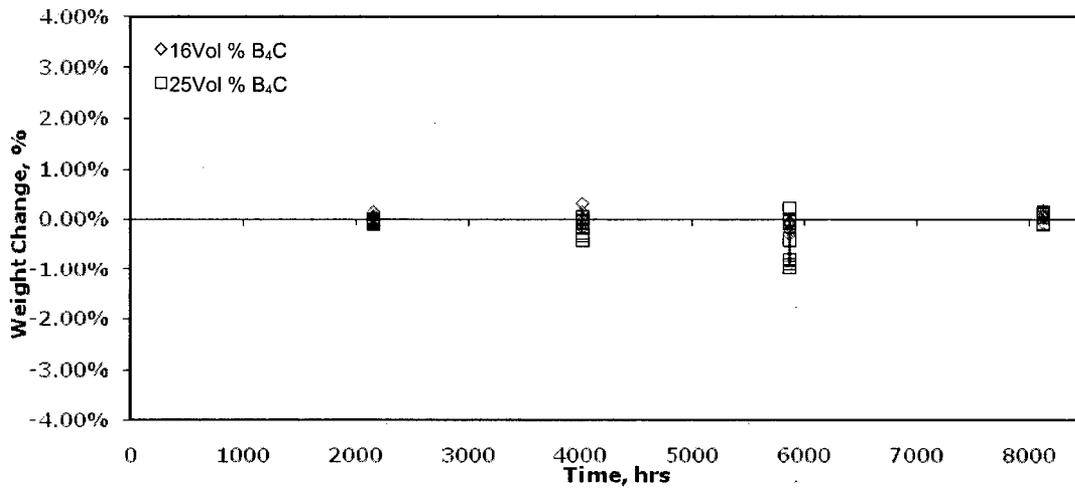


Figure 5-21: BWR Weight Change (Pre-Test vs. Post-Test)

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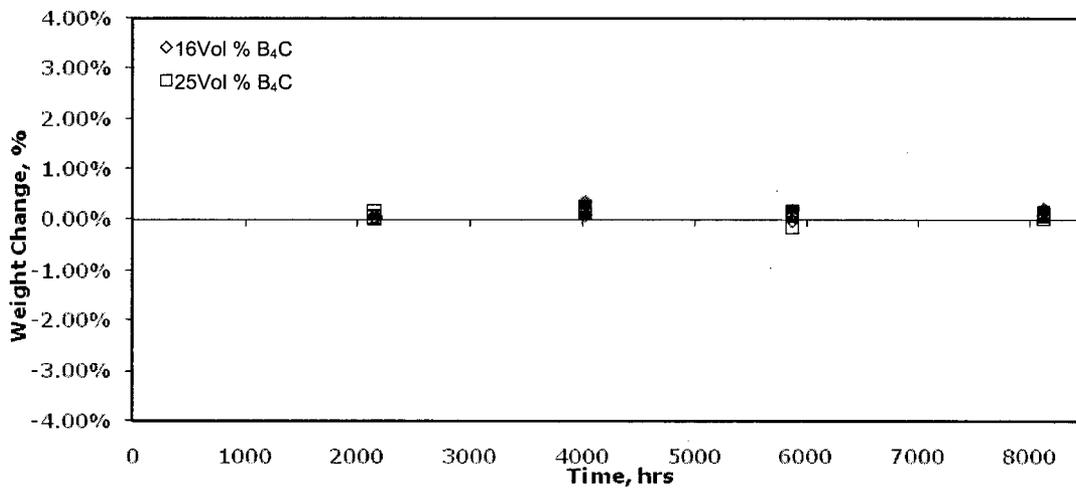


Figure 5-22: PWR Weight Change (Pre-Test vs. Post-Test)

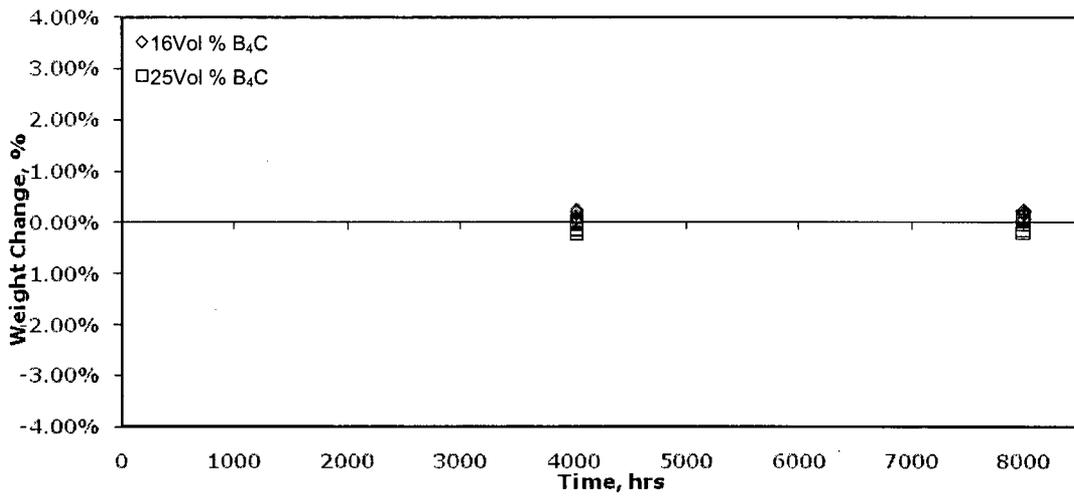


Figure 5-23: BWR Coupon Weight Change versus Time: Galvanic Couple Coupons

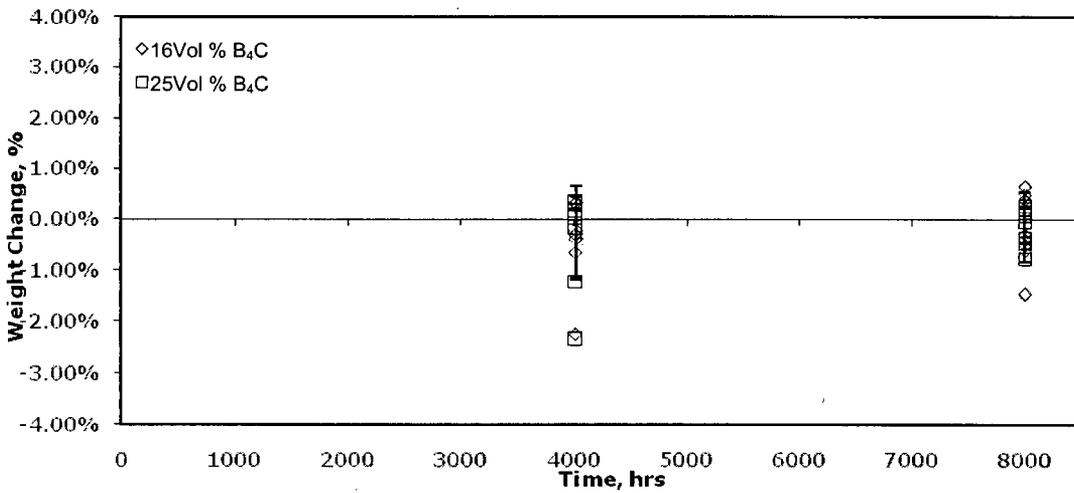


Figure 5-24: PWR Coupon Weight Change versus Time: Galvanic Couple Coupons

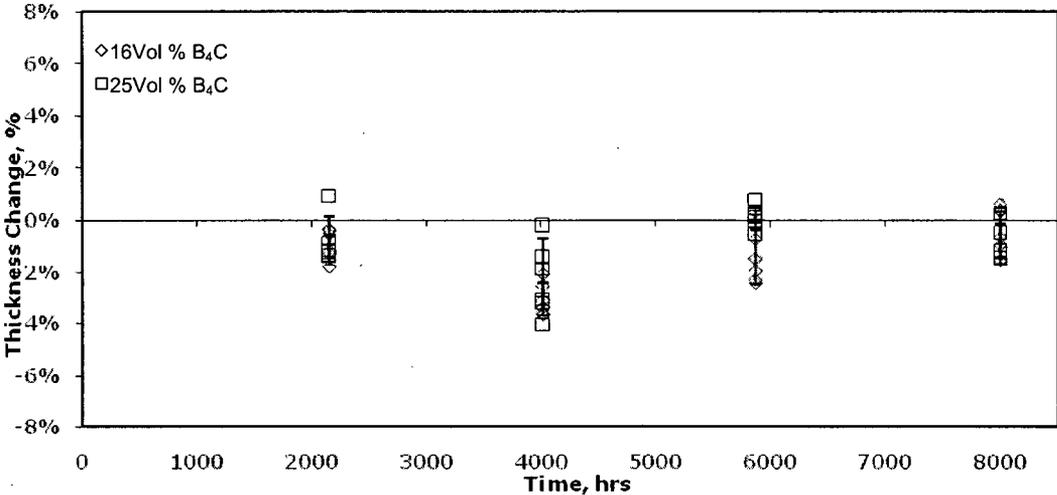


Figure 5-25: BWR Thickness Change (Pre-Test vs. Post-Test) After Acid Cleaning: General and Bend Coupons

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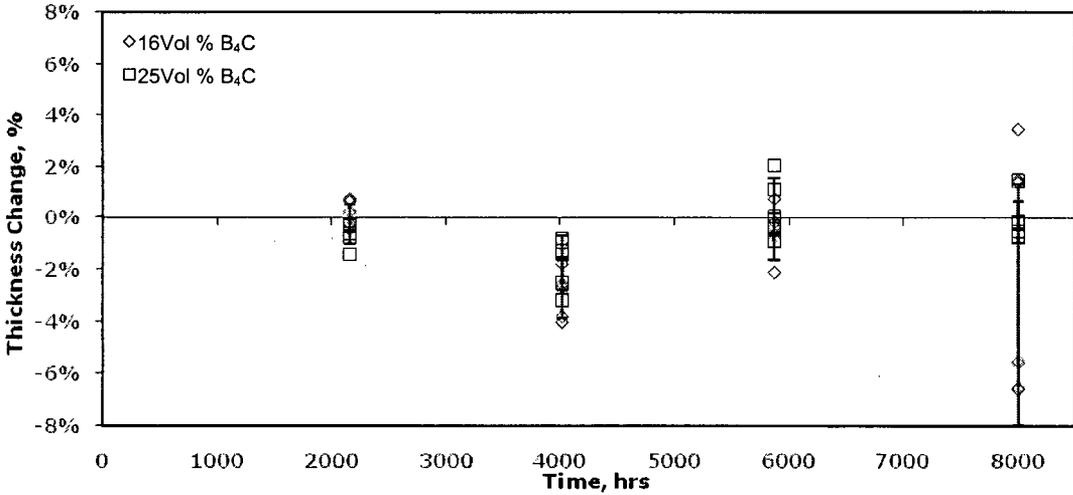


Figure 5-26: PWR Thickness Change (Pre-Test vs. Post-Test) After Acid Cleaning: General and Bend Coupons

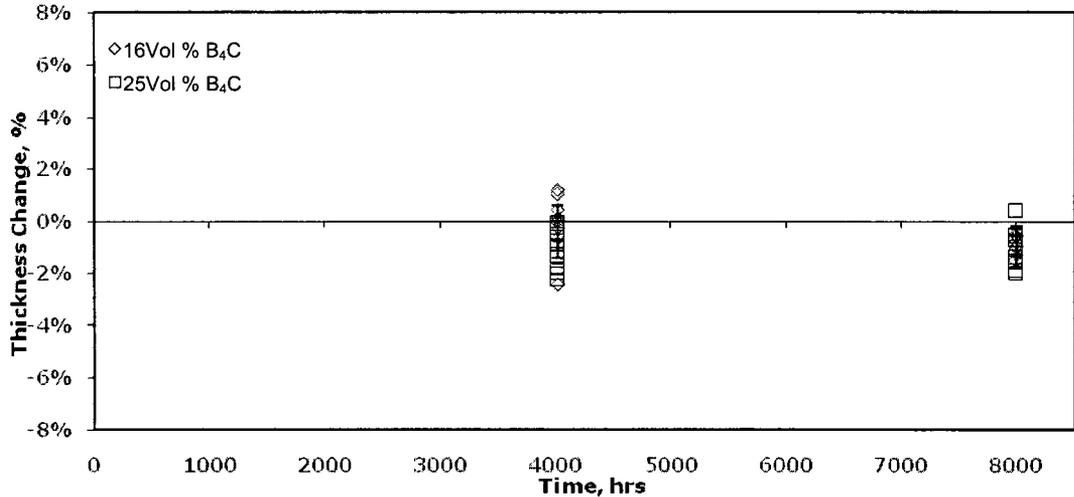


Figure 5-27: BWR Galvanic Coupons After Acid Cleaning: Coupon Thickness Change

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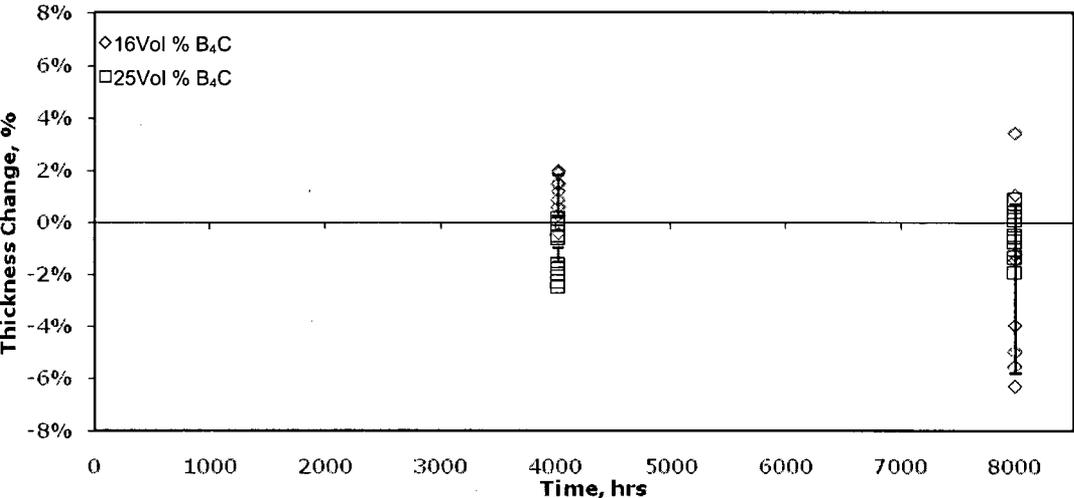


Figure 5-28: PWR Galvanic Coupons After Acid Cleaning: Coupon Thickness Change

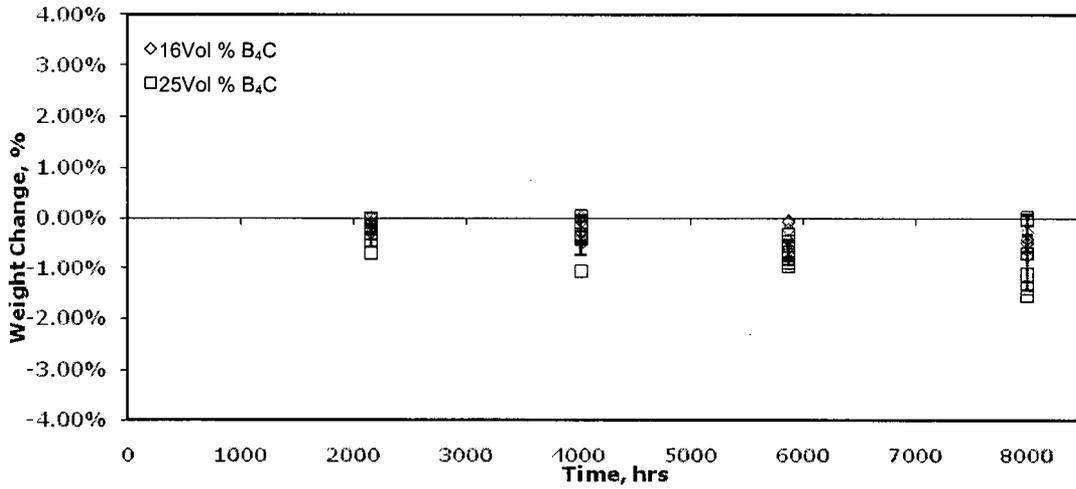


Figure 5-29: BWR Coupon Weight Change (Pre-Test vs. Post-Test) After Acid Cleaning: General and Bend Coupons Only

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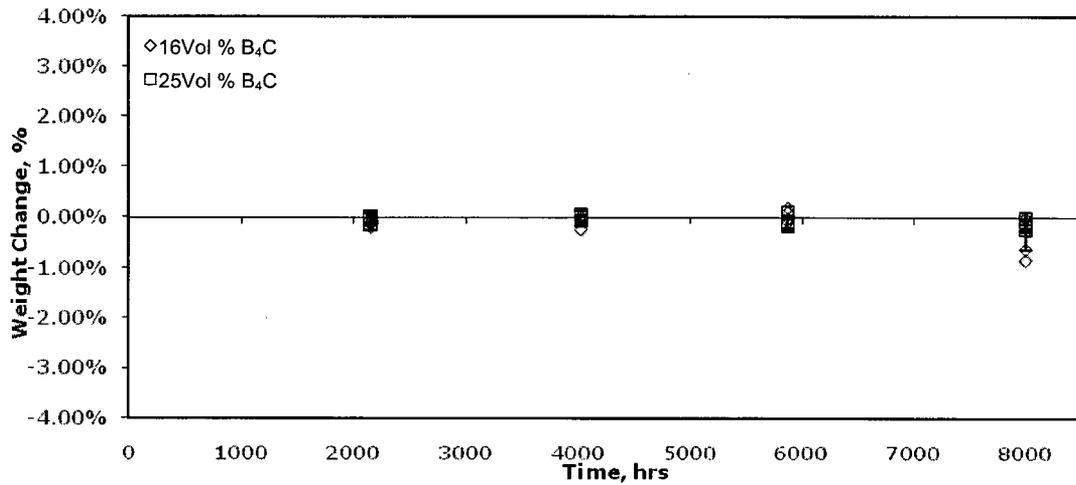


Figure 5-30: PWR Coupon Weight Change (Pre-Test vs. Post-Test) After Acid Cleaning: General and Bend Coupons Only

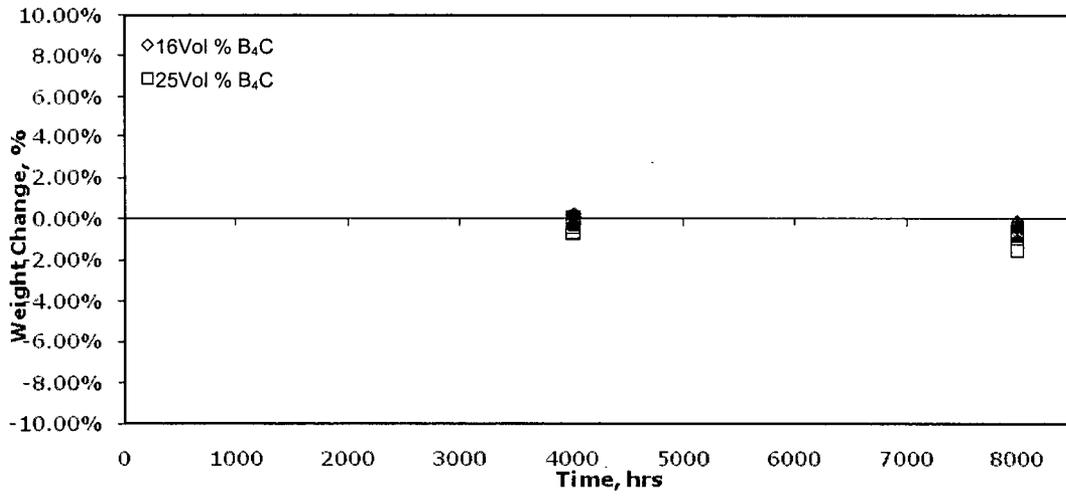


Figure 5-31: BWR Galvanic Coupon Weight Change versus Time

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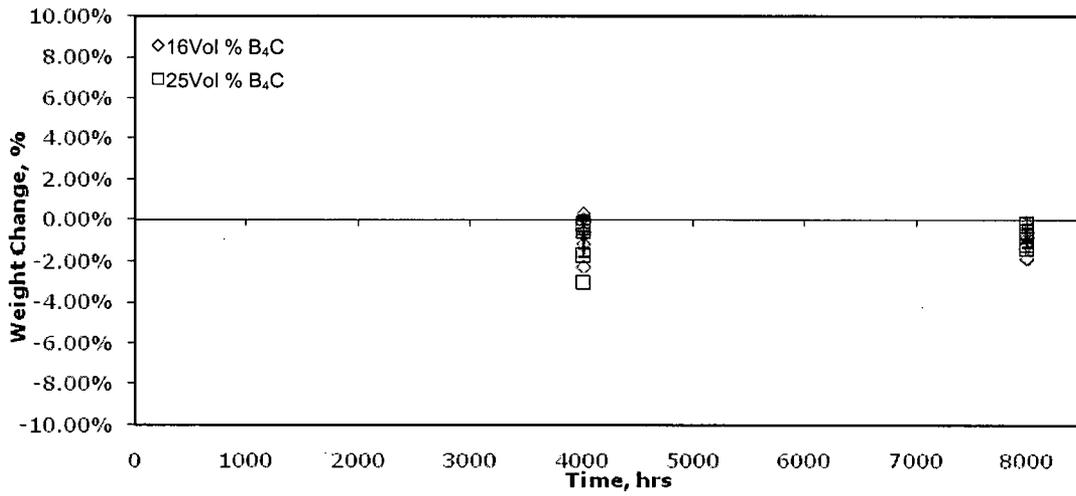


Figure 5-32: PWR Galvanic Coupon Weight Change versus Time

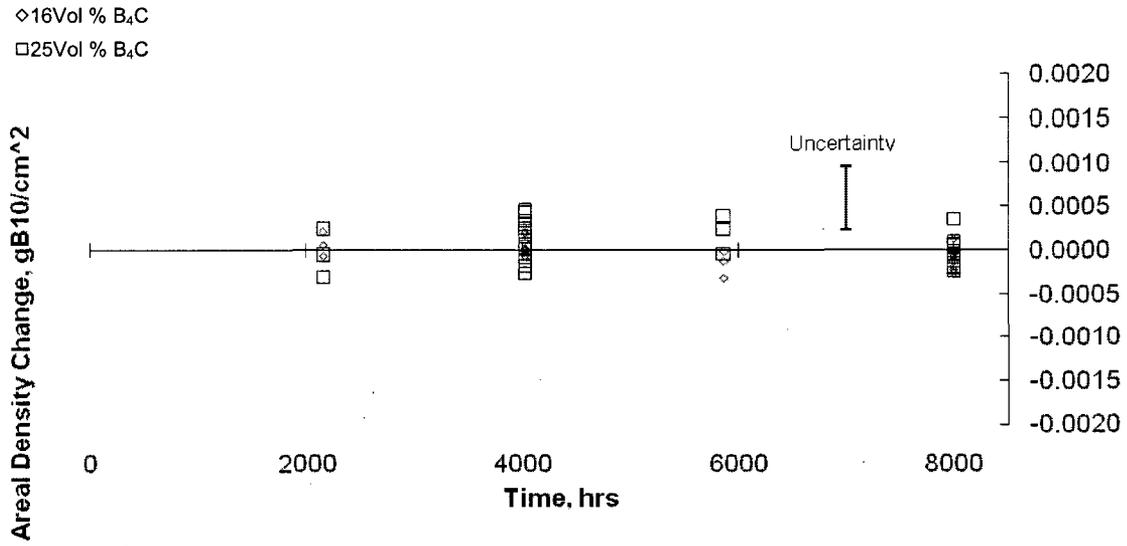


Figure 5-33: BWR Coupon Areal Density Change versus Time

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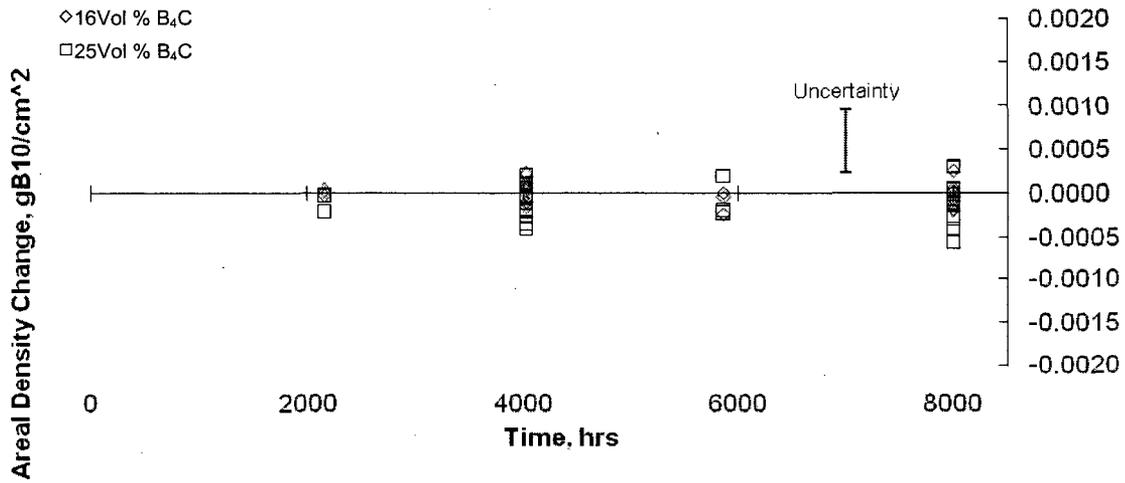


Figure 5-34: PWR Coupon Areal Density Change versus Time

### 5.5 Discussion of Test Results and Conclusions

Following the guidance provided in Reference 1, the post-test coupon weights after acid cleaning can be compared with pre-test weights to compute the test environment corrosion rate (mils/year) of the various coupon types. Using corrosion data in Reference 3 for AA1200 series aluminum in water at 122° F and 392° F, it is possible to compute the corresponding in-service corrosion rates at typical spent fuel pool conditions and water temperature (typically 80° F). These calculations are contained in the Appendix and the so calculated in-service corrosion rates are summarized in Table 5-7. Table 5-8 contains the equivalent in-service exposure times for each of the four test intervals.

Table 5-7: Average Corrosion Rates

<u>Coupon Types</u>	<u>In-Service Corrosion Rates, mils/year</u>			
	<u>2153-Hour Test</u>	<u>4019-Hour Test</u>	<u>5871-Hour Test</u>	<u>8119-Hour Test</u>
BWR General and Bend				
16 vol % B <sub>4</sub> C	-0.01	-0.01	-0.02	-0.01
25 vol % B <sub>4</sub> C	-0.02	-0.02	-0.02	-0.02
BWR Galvanic				
16 vol % B <sub>4</sub> C		0.01		-0.01
25 vol % B <sub>4</sub> C		-0.01		-0.02
PWR General and Bend				
16 vol % B <sub>4</sub> C	-0.01	-0.01	-0.03	-0.01
25 vol % B <sub>4</sub> C	-0.01	-0.02	-0.01	-0.01
PWR Galvanic				
16 vol % B <sub>4</sub> C		-0.02		-0.02
25 vol % B <sub>4</sub> C		-0.04		-0.01

Table 5-8: Equivalent Exposure Time

<u>Test Hours @ 195° F</u>	<u>In-Service Hours @ 80° F</u>
2153	39050
4019	72911
5871	107447
8119	148605

The computed in-service corrosion rates shown in Table 5-7 are extremely low and in each instance are based on the average weight loss of several coupons. A corrosion rate of -0.02 mils/year can be interpreted to mean that after 100 years an oxide film 2 mils thick would be expected on all surfaces. The reason for this extremely low corrosion rate is that once an oxide film forms on all surfaces, the film tends to be self-passivating; that is, it tends to retard further corrosion. This property of the oxide film formation leads to the excellent corrosion resistance of AA1100 aluminum alloy and the performance of the Rio-Tinto Alcan material shows similar performance. This has been observed in other aluminum boron carbide composites tested by NETCO.<sup>[4]</sup>

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It is further noted that for both the 16 vol % and the 25 vol % coupons, there has been no measurable change in the B-10 areal density, nor has any local corrosion (pitting) or cracking been detected. Optical microscopy of inside and outside radius of bend coupons revealed no cracks or anomalous corrosion behavior. These observations apply to both the BWR and the PWR test environments.

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Once installed, the inserts assume a constant strain condition within the rack cell. This compression leads to internal stresses, especially at the bend, that might make the inserts susceptible to stress corrosion cracking. An examination of the literature on this subject<sup>[5-5],[5-6]</sup>, indicates that "In general, high-purity aluminum and low-strength aluminum alloys are not susceptible to SCC."<sup>[5-5]</sup> However, surveillance bend coupons to be placed in the pool prior to the installation of the inserts will be maintained under the same strain conditions to provide indication of any unexpected crack phenomena.

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Notwithstanding the low measured corrosion rates, corrosion itself does not result in any loss of boron carbide. After the corrosion film forms, the boron carbide remains tightly bound in the corrosion layer. This was confirmed by the neutron attenuation measurements for boron-10 areal density, which were performed prior to acid cleaning to remove corrosion products.

As determined by the testing sequences described herein, the low measured corrosion rates under accelerated corrosion test conditions as well as the constancy of boron-10 areal density, recommends that the AA1100/boron carbide composite produced by Rio Tinto Alcan is a highly suitable neutron absorber material for use in spent fuel storage racks.

References Section 5:

- 5-1 ASTM G-31-72 (Reapproved 2004), Standard Practice for Laboratory Corrosion Testing of Metals.
- 5-2 ASTM G-1-03, Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens.
- 5-3 Godard, Epton, Bothwell and Kane, The Corrosion of Light Metals, John Wiley & Sons, Inc., New York, 1967.
- 5-4 Qualification of METAMIC® for Spent-Fuel Storage Applications, Electric Power Research Institute Report 1003137 by Northeast Technology Corp., Kingston, NY. October 2001.
- 5-5 Davis, J.R. Corrosion of Aluminum and Aluminum Alloys. ASM International. November 2000. Pg. 108
- 5-6 Bauccio, Michael. ASM Metals Reference Book, Third Edition. ASM International. April 2003. Pg. 408

## **6.0 Fast Start Coupon Surveillance Program Description**

The fast start coupon surveillance program consists of a series of 24 coupons cut from extra Alcan composite produced for the LaSalle demonstration. These coupons are 2 x 4 inches in width and length and have two 0.25 inch diameter holes along the top and bottom edge as shown in Figure 6-1. Their thickness is nominally 0.065 inch and each coupon contains 16 vol% boron carbide. The purpose of the fast start program is to provide early performance data on the Alcan composite in the LaSalle Unit 2 pool environment.

Each of the coupons will be connected to the next coupon with a stainless steel link clip. The string of 24 coupons will be attached to a short piece of stainless steel chain, which in turn will be attached to a head assembly (See Figure 6-2). The head assembly contains a hook so that it can be remotely lowered into a rack storage cell in the LaSalle Unit 2 pool. When in place the head piece will rest on top of a storage cell with the string of coupons suspended in the cell below. The length of the connecting chain between the head piece and the string of coupons was adjusted so that all 24 coupons are within the active fuel region of the eight surrounding fuel assemblies.

At each refueling outage the fast start coupons will be in a cell surrounded in all eight locations with freshly discharged fuel. In this manner the gamma energy disposition and temperatures of the coupons will be maximized. Two coupons will be removed every six months from the string and sent to a qualified laboratory for testing and inspection. The coupons have been subjected to pre characterization and will be post test characterized. Table 6-1 contains the pre and post test inspections and measurements.

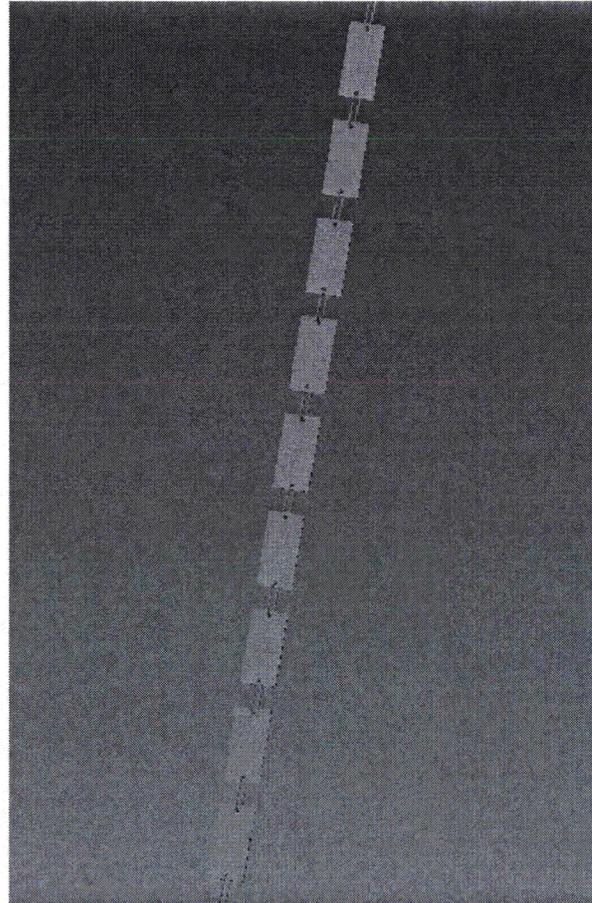
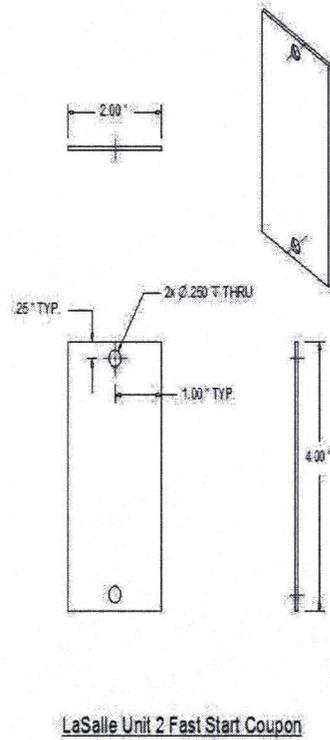


Figure 6-1: Fast Start Surveillance Coupon

Figure 6-2: Fast Start Coupons String

Table 6-1

## Pre and Post Test Coupon Characterization

Test	Pre Characterization	Post Characterization
Visual (Hi resolution digital photo)	√	√
Dimension	√	√
Dry Weight	√	√
Density	√	√
Areal Density	√+	√
Acid Cleaning		√
Weight Loss		√
Corrosion Rate		√
Microscopy		√*

+ On select coupons

\*as-required

A prime objective of this fast start program is to provide some early data as to the actual corrosion rates anticipated under actual LaSalle spent fuel pool conditions.

## 7.0 Long-Term Surveillance Program

The long-term surveillance program will consist of a specially designed surveillance tree to which a series of surveillance coupons are attached. The long-term surveillance tree will be placed within the pool as part of the first installation campaign of NETCO-SNAP-INS<sup>®</sup> and will reside there as long as the fuel racks continue to be used. Periodically, coupons will be removed and sent to a qualified laboratory for testing.

### 7.1 Tree and Coupon Description

The surveillance tree will be a four-sided structure with 18 - 2" x 4" coupons attached to each side and 12 - 2" bend coupons abutting adjacent faces. The bend coupons will be maintained under a fixed strain in the fixture. They will be manufactured at the same initial angle as the inserts and bent to the square angle of the rack cells. All coupons will contain 17 vol% boron carbide. The types and numbers of coupons included in the program are shown in Table 7-1.

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Table 7-1

Long-Term Surveillance Coupons

Coupon Type	Number	Objective
General	48	Quantify long-term corrosion
Bend	24	Track effects along bend radii including stress relaxation and stress corrosion cracking
Galvanic (bi-metallic)	24	Trend galvanic corrosion with 304SS, In and Zirc coupons

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### 7.2 Coupon Inspection and Testing

Specific coupons will be removed from tree on a frequency schedule as described subsequently. The general coupons will be subject to pre and post examination according to Table 7-2.

Table 7-2

## Long-Term Surveillance General Coupon Characterization

Test	Pre Characterization	Post Characterization
Visual (Hi resolution digital photo)	√	√
Dimension	√	√
Dry Weight	√	√
Areal Density	√+	√
Acid Cleaning		√
Weight Loss		√
Corrosion Rate		√
Microscopy		√*

+ On select coupons

\*as-required

4

The bend and galvanic coupons will be subject to the tests in Table 7-3.

Table 7-3

## Long-Term Surveillance Bend and Galvanic Coupon Characterization

Test	Pre Characterization	Post Characterization
Visual (High Resolution Digital Photo)	√	√
Thickness	√	√
Dry Weight	√	√
Bending Stress (Bend Coupons Only)	√	√
Acid Cleaning		√
Weight Loss and Corrosion Rate		√
Microscopy		√*

\*as-required

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**7.3 Frequency for Coupon Inspection**

The frequency for coupon inspection will depend on the coupon type and results of previous inspections. The frequency for inspection is shown in Table 7-4.

Table 7-4  
Frequency for Coupon Testing

<b>Coupon Type</b>	<b>First Ten Years</b>	<b>After 10 Years with Acceptable Performance</b>
General	2 coupons every 2 years	2 coupons every 4 years
Bend	1 coupon every 2 years	1 coupon every 4 years
Galvanic Couples 304 Stainless Zirc In	1 couple every 6 years " "	

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