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May 10, 2007

U. S. Nuclear Regulatory Commission  
Washington, DC 20555

**ATTENTION:** Document Control Desk

**SUBJECT:** Calvert Cliffs Nuclear Power Plant  
Unit No. 1; Docket No. 50-317  
Long-Term Coupon Surveillance Program for the Unit 1 Spent Fuel Pool

**REFERENCE:** (a) Letter from Mr. G. S. Vissing (NRC) to Mr. P. E. Katz, dated June 3, 2004, Amendment to Increase the Spent Fuel Pool Maximum Enrichment Limit (TAC No. MB8896)

In 2004, the Nuclear Regulatory Commission imposed a license condition (Reference a) that requires the submission of a long-term coupon surveillance program for the Unit 1 spent fuel pool racks. The long-term coupon surveillance program is necessary to support an approved license amendment which established acceptable soluble boron concentrations in the Unit 1 Spent Fuel Pool. As required by the license condition, we are submitting this program for your approval under the requirements of 10 CFR 50.90. The technical evaluation and no significant hazards consideration is contained in Attachment (1). Enclosure 1 to Attachment (1) contains a technical report from the manufacturer of the spent fuel pool rack neutron absorbing material that provides additional detail on the absorbing material in support of our coupon surveillance program.

**SAFETY COMMITTEE REVIEW**

The Plant Operations Review Committee has reviewed the proposed changes and concurs that operation with the proposed changes will not result in an undue risk to the health and safety of the public.

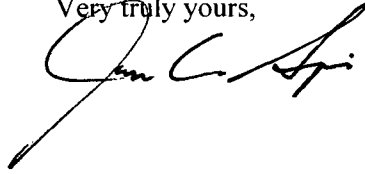
**SCHEDULE**

We are requesting approval of the proposed coupon surveillance program by May 1, 2008. Note that we plan to begin accelerated irradiation of some coupon surveillance samples in 2007 to support the long-term objectives of this program.

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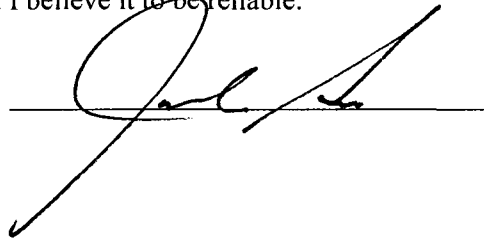
Should you have questions regarding this matter, please contact Mr. Jay S. Gaines at (410) 495-5219.

Very truly yours,

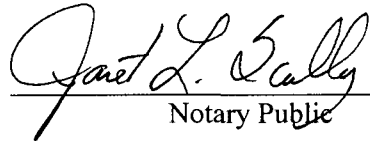
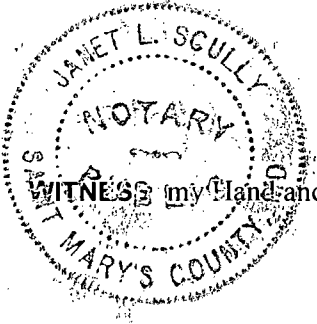


STATE OF MARYLAND :  
  : TO WIT:  
COUNTY OF CALVERT :

I, James A. Spina, being duly sworn, state that I am Vice President - Calvert Cliffs Nuclear Power Plant, Inc. (CCNPP), and that I am duly authorized to execute and file this License Amendment Request on behalf of CCNPP. To the best of my knowledge and belief, the statements contained in this document are true and correct. To the extent that these statements are not based on my personal knowledge, they are based upon information provided by other CCNPP employees and/or consultants. Such information has been reviewed in accordance with company practice and I believe it to be reliable.



Subscribed and sworn before me, a Notary Public in and for the State of Maryland and County of St. Mary's, this 10<sup>th</sup> day of May, 2007.

  
Notary Public

My Commission Expires:

March 1, 2011  
Date

JAS/PSF/bjd

Attachment: (1) Technical Evaluation and No Significant Hazards Consideration

Enclosure (1): Handbook of the Effects of In-Pool Exposure on Properties of Boron Carbide-Resin Shielding Materials

cc: D. V. Pickett, NRC  
S. J. Collins, NRC

Resident Inspector, NRC  
R. I. McLean, DNR

**ATTACHMENT (1)**

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**TECHNICAL EVALUATION AND  
NO SIGNIFICANT HAZARDS CONSIDERATION**

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## ATTACHMENT (1)

### TECHNICAL EVALUATION AND NO SIGNIFICANT HAZARDS CONSIDERATION

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#### **1.0 DESCRIPTION**

In 2004, the Nuclear Regulatory Commission (NRC) imposed a license condition (Reference 1) that requires the submission of a coupon surveillance program for the Unit 1 Spent Fuel Pool (SFP) racks. The coupon surveillance program is necessary to support an approved license amendment which established acceptable boron concentrations in the Unit 1 SFP. Our proposed program is described below. As required by the license condition, we are submitting this program for your approval under the requirements of 10 CFR 50.90.

#### **2.0 PROPOSED CHANGE**

The license condition for Unit 1 states:

"This amendment requires the licensee to develop a long-term coupon surveillance program for the Carborundum samples. This program must verify that the Carborundum degradation rates assumed in the licensee's analyses to prove subcriticality, as required by 10 CFR 50.68, remain valid over the seventy-year life span of the Unit 1 Spent Fuel Pool. The licensee must submit this modified coupon surveillance program to the NRC under the 10 CFR 50.90 requirements for its review and approval."

No changes to the Technical Specifications, Operating Licenses, or Updated Final Safety Analysis Report have been identified that would require review and approval under 10 CFR 50.59. However, we are submitting the modified coupon surveillance program for review and approval in accordance with the license condition.

#### **3.0 BACKGROUND**

The SFP is located outside the Containments in the Auxiliary Building and provides underwater storage of spent fuel assemblies after their removal from the reactor vessels. The pool, designed in two halves, can accommodate 1830 assemblies and one spent fuel shipping cask. The Unit 1 half of the SFP contains storage racks in six 10x10, two 8x10, and one 7x10 array. The storage racks consist of vertical cells grouped in parallel rows (arrays). The pool is constructed of reinforced concrete and lined with stainless steel.

The neutron absorbing material in the Unit 1 SFP storage racks is borated sheets inserted between the walls of the fuel rack cells. The sheets used in the Unit 1 SFP are made of a composite material (carborundum sheet) consisting of a boron carbide ( $B_4C$ ) powder in a fiberglass matrix.

The method of confirming the borated sheets will have sufficient  $B_4C$  to maintain a subcriticality minimum of 5% in the SFP currently consists of an accelerated coupon surveillance program that has been in use since 2004. The program uses periodic testing of the sample coupons that are representative of the material in the SFP racks. The program provides data to support the prediction of  $B_4C$  loss over the life of the racks by monitoring the degradation of the neutron absorbing material.

Until 2004, a long-term coupon surveillance program was also used to determine degradation of the neutron absorbing material. The long-term surveillance program was discontinued in 2004 to support the accelerated coupon surveillance program needed to address the expanded 70 year lifetime of the SFP racks.

#### **4.0 TECHNICAL ANALYSIS**

Our proposed accelerated coupon surveillance program is designed to provide long-term exposure to gamma radiation and borated pool water and allows for samples of the material to be removed from the

## ATTACHMENT (1)

### TECHNICAL EVALUATION AND NO SIGNIFICANT HAZARDS CONSIDERATION

SFP for examination. The coupon surveillance program provides for periodic monitoring of the condition of neutron absorbing material in the SFP.

The coupons are extracted from the SFP for examinations that consist of visual, weight, and dimensional determinations. The visual examination consists of inspecting for evidence of gross changes or deterioration. The weight of each coupon is measured to within 0.001 grams and compared against the sample initial weight using a calibrated scale. The length and width of each sample is measured within 0.001 inch and compared against the sample initial length and width using a calibrated caliper. The loss of mass of the coupons is used as a measure of degradation instead of the areal density because previous experience has indicated that mass loss was more conservative in determining coupon degradation than the change in areal density (Enclosure 1, page 15).

The company that manufactured the carborundum sheets conducted a carborundum qualification program in which test panels were simultaneously exposed to 10E11 rad gamma radiation and borated water, simulating a 40-year lifetime similar to what is expected in the SFP. The results of this program showed that the material exhibited chemical stability, boron retention, and mechanical property changes within design specifications (Enclosure 1 for sheet material).

The Calvert Cliffs Nuclear Power Plant Unit 1 SFP racks contain B<sub>4</sub>C in Carborundum sheets as the neutron absorbing material. Sample coupons of the Carborundum were included on the Long-Term Surveillance Assembly (LTSA) and are included on the Accelerated Surveillance Assembly (ASA). The LTSA coupon tree was placed in a SFP rack location that was to see typical doses from fuel as the fuel aged in the SFP. The ASA coupon tree, however, is placed among freshly discharged fuel following each refueling, and, over time, receives higher exposure than the LTSA coupons.

Unit 1 License Amendment Number 267 included a license condition that requires us to develop a new long-term coupon surveillance program for the Carborundum samples. With only five accelerated sample coupons remaining, the intervals for sampling would have to be increased to allow for a sampling program for the extended life-of-the-plant. Proceeding with these much longer ASA sample intervals (about every eight years) is not an acceptable approach, and therefore the surveillance program is being modified as described below.

The new Carborundum coupon surveillance program will now use the remaining five accelerated samples from the ASA, on a four-year cycle beginning in 2005, followed by the remaining eight long-term samples from the discontinued LTSA, continuing on a four-year cycle through 2053 (end of the 70-year life span of the Unit 1 SFP racks). To make the eight long-term samples available for this new surveillance program, the existing LTSA coupon surveillance program was discontinued in 2004. These eight LTSA samples will now be subjected to accelerated doses so that they are expected to have exposures comparable to the ASA samples when it is time for the first of these eight to be sampled (beginning in 2025). This required the development of a different type of coupon tree to hold these samples.

The existing ASA and LTSA coupon trees are fuel assembly sized items that are placed in the spent fuel storage racks. Each of the four sides of the coupon tree has six coupons attached vertically to the outside of the assembly. The samples on each side of the assembly receive dose primarily from the fuel in the storage rack location adjacent to that side.

However, to accelerate doses to the eight remaining LTSA samples, they are being placed onto new coupon trees that will allow the coupons to see higher doses from fuel. This is possible because the new coupon trees, instead of being placed inside a rack location, will be placed in the space between racks in

**ATTACHMENT (1)**

**TECHNICAL EVALUATION AND NO SIGNIFICANT HAZARDS CONSIDERATION**

the SFP. This will allow the sample coupons to see dose from the fuel that is in the rack locations on both sides of the coupons. These new coupon trees are expected to be installed in 2007.

The schedule for the remaining coupon surveillance withdrawals is given below.

Test Number	Sample Date	Sample Coupon Type
10	2009	Original ASA
11	2013	Original ASA
12	2017	Original ASA
13	2021	Original ASA
14	2025	Accelerated LTSA
15	2029	Accelerated LTSA
16	2033	Accelerated LTSA
17	2037	Accelerated LTSA
18	2041	Accelerated LTSA
19	2045	Accelerated LTSA
20	2049	Accelerated LTSA
21	2053	Accelerated LTSA

**5.0 NO SIGNIFICANT HAZARDS CONSIDERATION**

The proposed change has been evaluated against the standards in 10 CFR 50.92 and has been determined to not involve a significant hazards consideration in operation of the facility in accordance with the proposed amendment:

1. *Would not involve a significant increase in the probability or consequences of an accident previously evaluated.*

The proposed surveillance program supports evaluation of degradation of the neutron absorbing material in the Unit 1 Spent Fuel Pool (SFP). The function of the neutron absorbing material is to provide one means of maintaining criticality safety of the nuclear fuel stored in the SFP.

The postulated accidents for the SFP are basically five types; 1) dropped fuel assembly on top of the storage rack, 2) a misloading accident, 3) an abnormal location of a fuel assembly, 4) loss-of-normal cooling to the SFP, and 5) dilution of boron in the SFP water.

The proposed change in the coupon surveillance program for the Unit 1 SFP racks does not affect any of these previously evaluated accidents. The coupon trees have been evaluated as required by our plant modifications program and have been determined to have no effect on accidents in the SFP.

Therefore, the proposed change does not involve a significant increase in the probability or consequences of an accident previously evaluated.

2. *The proposed change does not create the possibility of a new or different kind of accident from any accident previously evaluated.*

The proposed surveillance program supports evaluation of degradation of the neutron absorbing material in the Unit 1 SFP. The function of the neutron absorbing material is to provide one means of maintaining criticality safety of the nuclear fuel stored in the SFP.

The coupon trees have been evaluated as required by our plant modifications program and do not create the possibility of a new or different kind of accident in the SFP. The surveillance coupons

## ATTACHMENT (1)

### TECHNICAL EVALUATION AND NO SIGNIFICANT HAZARDS CONSIDERATION

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have existed in the SFP since the Unit 1 SFP racks were installed. The form and function of the surveillance coupon trees is not changed because of the need to change the coupon surveillance program. The interaction of the coupons with the spent fuel racks and the SFP is not changed due to the proposed surveillance program change.

The proposed change will not result in any other change in the plant configuration or equipment design. Therefore, the proposed change does not create the possibility of a new or different kind of accident from any previously evaluated.

3. *The proposed change does not involve a significant reduction in a margin of safety.*

The proposed coupon surveillance program supports evaluation of degradation of the neutron absorbing material in the Unit 1 SFP. The function of the neutron absorbing material is to provide one means of maintaining criticality safety of the nuclear fuel stored in the SFP. Evaluation of the coupons as part of an ongoing surveillance program provides assurance that the fuel will remain subcritical under all postulated conditions.

Therefore, the proposed change does not involve a significant reduction in a margin of safety.

#### **6.0 ENVIRONMENTAL CONSIDERATION**

We have also determined that operation with the proposed amendment would not result in any significant change in the types, or significant increases in the amounts, of any effluents that may be released offsite, nor would it result in any significant increase in individual or cumulative occupational radiation exposure. Therefore, the proposed amendment is eligible for categorical exclusion as set forth in 10 CFR 51.22(c)(9). Pursuant to 10 CFR 51.22(b), no environmental impact statement or environmental assessment is needed in connection with the approval of the proposed amendment.

#### **7.0 PRECEDENT**

None

#### **8.0 REFERENCES**

1. Letter from Mr. G. S. Vissing (NRC) to Mr. P. E. Katz, dated June 3, 2004, Amendment to Increase the Spent Fuel Pool Maximum Enrichment Limit (TAC No. MB8896)

**Enclosure (1)**

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**Handbook of the Effects of In-Pool Exposure on Properties of  
Boron Carbide-Resin Shielding Materials**

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# Handbook of the Effects of In-Pool Exposure on Properties of Boron Carbide-Resin Shielding Materials

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A Kennecott Company

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CARBORUNDUM



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## Summary

Two types of resin bonded boron carbide shielding materials have been developed by The Carborundum Company<sup>1</sup> for in-pool shielding on densified spent fuel storage racks. One is a thick rigid plate, the other a thin flexible glass fabric reinforced composite. Both contain granular boron carbide bonded in a phenol formaldehyde matrix. Both of these phenolic bonded boron carbide materials are in service in a number of on-site densified spent fuel storage pools.

Questions have been raised concerning the long term stability of these materials under operating conditions. To answer these questions and to provide assurances concerning this long term stability, The Carborundum Company conducted this test program.

Samples were prepared from lots of typical production materials. They were exposed to extreme levels of gamma radiation, beyond those that might be encountered during projected 40-year in-pool lifetime. Dose rates, and exposure conditions were typical of those that might be encountered in practice.

The data from previous reports (2, 3) are summarized in this and related reports (4, 5). Other reports on related material (6, 7), presentations, and publications (8, 9, 10) will make all of this information generally available.

Exposure of both materials to high energy gamma radiation to doses of  $10^6$  gray ( $10^{11}$  rad) at fluxes of  $10^5$  gray ( $10^8$  rad) per hour, showed chemical stability, boron retention, and mechanical property changes within design specifications.<sup>2</sup>

After  $10^6$  gray ( $10^{11}$  rad) of gamma irradiation exposure, all mechanical design specifications were satisfied. Areal densities of boron-10 exceeded the design minima in all phases of the test. Dimensional changes became significant only at highest levels of irradiation  $5 \times 10^6$  gray ( $5 \times 10^{10}$  rad) and were directly attributable to spalling and physical loss of material rather than intrinsic effects with the materials themselves. No radiation induced  $B_4C$  solubility was seen;

weight changes in both materials were associated primarily with particulate losses through spalling and attrition. Gas evolution rates and compositions corresponded with published results of similar studies.

## Background

Boron carbide is a useful neutron shielding material. As early as 1935, boron carbide, then a relatively new commercial material, was being used as a neutron absorber (11). Boron has highly desirable properties as a neutron absorber. It produces no radioactive daughter products and yields only low energy desintegration products (12).

Boron carbide is a refractory, high-boron material that is crystalline, and resistant to radiation damage. It possesses typical ceramic properties (hard, rigid, brittle) that rule out its use where a ceramic cannot be tolerated.

Shielding in spent fuel storage racks is one such application where monolithic boron carbide parts are unacceptable. First, fabrication of monolithic boron carbide parts requires high temperatures and pressures and is severely size limited. The resulting products are expensive and when fractured, fail catastrophically.

Boron carbide shielding materials must, therefore, be designed using granular boron carbide mechanically supported by more typical engineering material (metal tubes and plates, polymeric or metal bond phases). Introduction of a polymeric bond phase while solving some problems, introduces others. Organic polymers are generally unstable in high energy radiation environments. They do, however, offer excellent corrosion resistance.

Two such polymeric bonded boron carbide materials have been developed. One, referred to here as boron carbide plate material, consists of a homogeneous array of granular boron carbide bonded to and supported by a continuous matrix of a somewhat more flexible phenol formaldehyde resin. The second material is chemically and structurally similar, except that it incorporates a woven glass fabric reinforcement phase. This material, typically thinner than the plate material, is referred to as boron carbide sheet material.

Questions were raised with respect to the long term effects of radioactive degradation of the bond phase during the anticipated in-pool exposures. How would this degradation of the organic

phase affect the strength of these materials? Would the strength continue to meet engineering design specifications?

Along with this, a question was raised as to whether such long term in-pool radiation exposure might conceivably degrade the boron carbide itself, yielding soluble boron species that would reduce the boron areal density and the neutron shielding efficiency.

Another question related to the gas evolution effects of the degradation of the organic phases. How much gas might these materials release, and what might be its composition? Would such high energy radiation induce hydrolytic or oxidation reactions resulting from interactions with water and air in the pool environments?

To answer these questions and to provide confidence in the long term efficacy of the shielding material, an extensive testing program was undertaken. Prior to the initiation of experimental parts of the program, it was reviewed not only with customers, but also with the Nuclear Regulatory Commission (NRC) to determine whether or not the test program might have potential weaknesses, and whether it would satisfactorily answer these questions.

The testing program evaluated the effects of environmental conditions found in spent pools on both boron carbide materials. The materials were subjected to environments typical of those found in commercial spent pools. Emphasis was on the use of a gamma source in the  $10^4$  -  $10^6$  gray ( $10^8$  -  $10^{10}$  rad) per hour to accelerate testing without differing significantly from the dose rates that might be encountered during exposures immediately adjacent to freshly discharged fuel bundles. Water chemistry was typical of that associated with both boiling water reactor (BWR) and pressurized water reactor (PWR) spent fuel pools. The testing program utilized governmental, academic, and industrial facilities.

The single overall objective of the testing program was to obtain, from the NRC, generic approval of the materials for use in densified spent fuel storage racks. Later studies on alternative neutron absorbing materials resulted, but are not presented here.

<sup>1</sup>The Carborundum Company, Boron Products Department, Engineered Systems Group, P.O. Box 414, Niagara Falls, New York 14302.

<sup>2</sup>To conform to present trends, this report is presented in the modern S.I. metric system, with English equivalents where commonly used following in parentheses. Applicable parts of the ASTM metric system (1) are shown in Appendix 1. The reader is advised that these test results were obtained on production lots of materials, engineered and produced to specific design criteria. The results are, therefore, not to be used as engineering data, but are representative only of those specific materials.

**Materials**

**Boron Carbide Plate Material**

The plate material is a mixture of 120 grit nuclear grade boron carbide powder (Table 1) (13) and phenol formaldehyde resin (14).

Plates, typically 85 x 20 x 0.5 cm (33 x 8 x 0.2 in) had an areal density of 0.095 g/cm<sup>2</sup> boron-10. Boric acid having an isotopic content of boron-10 of 18.31% by weight (19.78 atomic percent) (15) was used in the arc furnace manufacture of the boron carbide.

Boron concentration was determined using established analytical procedures (16); Boron-10 areal densities were measured using mass spectrometric (MS) techniques (17). This MS technique yields isotopic ratios within ±0.3 relative percent (17).

Characterizations of a typical component boron carbide lot and manufactured plate material are shown in Tables II and III.

**Boron Carbide Sheet Material**

The sheet material is boron carbide and phenol formaldehyde resin bonded to a woven fiberglass fabric.

The sheet material was 0.122 cm (0.048 in) thick and had an areal density of 0.015 gm per cm<sup>2</sup> of boron-10 as produced. Total boron was determined using established analytical procedures (16), boron-10 isotopic ratios were measured using established mass spectrometric procedures (17).

Characterizations of typical component boron carbide lot and sheet material are shown in Tables II and IV.

**Qualification Testing**

**Samples**

The size of the uniform flux region available for sample irradiation in the High Flux Isotope Reactor (HFIR) at the Oak Ridge National Laboratory (ORNL) spent fuel storage pool was severely limited (8.9 cm diameter, 45 cm high, 3.5 x 18 in.). To maximize the use of this zone, all samples were cut to final dimensions prior to irradiation. Samples 7.5 x 1.25 cm (3 x 0.5 in) were cut from the plate material without regard to orientation; those from the sheet material with their long axis parallel to the continuous direction of the glass fabric.

For statistical purposes, bars prepared from the plate material were grouped in lots, typically consisting of six bars. The test program required a total of 75 lots of samples of plate material — 450 total samples. The sheet material test program required 60 lots of strips — 480 total samples. All test samples were randomly

**Specifications for Nuclear Grade Boron Carbide (13)**

Constituent	Composition Weight (%)	
	Type 1*	Type 2*
Total Boron	— Max 81.0	77.0
	— Min 76.5	70.0
Total Soluble Boron	— Max 0.5	N.D.**
Boric Oxide	— Max 0.2	3.0
Fluorine	— Max 25 ppm	N.D.
Chlorine	— Max 75 ppm	N.D.
Calcium	— Max 0.3	N.D.
Iron	— Max 1.0	2.0
<b>Total Boron Plus</b>		
Total Carbon	— Min 98.0	97.0

\*Type 1 is for in-core application; Type 2 for shielding

\*\*Not Determined

**Table II  
Characterization of Typical Boron Carbide**

Particle Size	Sheet	Plate
+10 mesh	0%	0%
-10 +35	0.1	0
-10 +60	2	1.2
-10 +120	65	42.3
-120	33	57.7

Analysis	Mean	S.D.
Total Boron	77.0%	0.28%
<b>Total Boron plus</b>		
Total Carbon	97.4	0.44
Boric Oxide	0.17	0.04
Iron	0.83	0.15

selected from within a lot and between lots using accepted statistical sampling procedures. Analytical Chemical, x-ray structural analysis, scanning electron microscopy, and mechanical tests were carried out by the analytical laboratories within Carborundum. Gas analyses were done in the Whitmore Laboratory (WL) of the Pennsylvania State University, and in the Lexington (Massachusetts) Laboratories (LEX) of Kennecott Copper Corporation. In addition, scanning electron microscopy (SEM), x-ray and microstructural analyses were performed at the Materials Research Laboratory (MRL) of the Pennsylvania State University.

Boron-10 isotopic analyses were performed by Isotopic Analysis Inc., of Tulsa, Oklahoma.

**Table III  
Typical Plate Characterization**

Composition	
Boron Carbide	73%
Binder	27%
*B Areal Density	.095 g/cm <sup>2</sup>
Physical Properties	
Density	1.8 g/cm <sup>3</sup>
Thickness	0.500 cm (0.2")
Mechanical Properties	
Flexural strength	34.5 MPa (5000 psi)
Compressive strength	69.0 MPa (10,000 psi)
Modulus of Elasticity	11.7 GPa (1.7 x 10 <sup>6</sup> psi)
Thermal Properties	
Coefficient of Thermal Expansion @ 60°C	7.2 x 10 <sup>-6</sup> m/m°C
Specific Heat	0.23 cal/g°C

**Table IV  
Typical Sheet Characterization**

Composition	
Boron Carbide	64%
Binder	18%
Fiberglass	18%
*B Areal Density	.015 g/cm <sup>2</sup>
Physical Properties	
Density	1.4 g/cm <sup>3</sup>
Thickness	0.127 cm (0.050")
Mechanical Properties	
Ultimate Tensile Strength	60.9 MPa (8800 psi)
Modulus of Elasticity	1.5 GPa (2.2 x 10 <sup>6</sup> psi)
Thermal Properties	
Specific Heat of B <sub>4</sub> C	0.23 cal/g°C
Specific Heat of Binder	0.30 cal/g°C

### Physical/Mechanical

Properties, including dimensions, weight, density, strength, and stiffness were determined both at MRL and in the Carborundum labs using standard techniques. Strength and elastic moduli of the plate materials were determined by means of a three point bending test, using a 5 cm (2 in) span, and a strain rate of 0.05 cm per min. (0.02 in per min). An Instron Universal Testing Machine was utilized.

Since the strength of the sheet material is largely derived from the glass fabric reinforcement phase, mechanical tests on this material were made in tension using a modified ASTM fabric test (D-3039) (19). Samples were stressed in tension at a strain rate of 0.05 cm per min (0.02 in per min) using a 2.5 cm (1 in) gage length. An Instron Universal Testing Machine was used as before.

### Irradiation

In a spent fuel storage pool radiation is predominantly in the form of gamma rays derived from the decay of fission products.

Selection of the Oak Ridge irradiation test site was governed by two major factors. A large volume of uniform flux of full spectrum gamma irradiation was required. A high flux, maintained over an extended period, was essential to obtain the  $10^9$  gray ( $10^{11}$  rad) total dose within a reasonable time. At the same time the dose rate must not be too high, otherwise effects not typical of a spent fuel storage pool could be introduced. Such effects might include sample over-heating, and bursting due to rapid gas evolution.

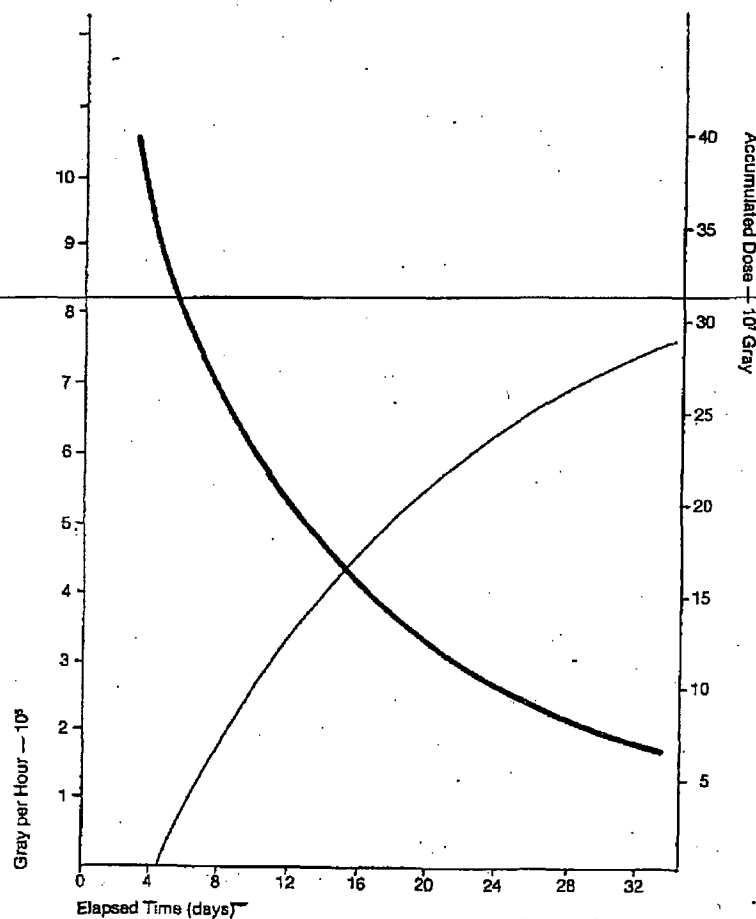
In HFIR highly enriched (93%  $^{235}\text{U}$ ) fuel is used on a 23 day replacement cycle, corresponding to a burn-up of 30% (65%  $^{235}\text{U}$  in spent fuel). Four temporary spent fuel storage pedestals arranged with internal cadmium shielding provide a working zone 9 cm diameter by 46 cm high (3.5 x 18 in) having a  $360^\circ$  exposure to a full spectrum gamma radiation with a minimum neutron flux. Gamma fluxes typically range from  $10^6$  -  $10^7$  gray ( $10^8$  -  $10^9$  rad) per hour. The short fuel cycle maintains these high flux levels over long periods of time.

Dose rate and cumulative dose, shown in Figure 1, as a function of time were provided by ORNL. They are based on calculations associated with the original HFIR design. They were recently confirmed by Compere (20) using both the cerium (IV) sulfate ( $\text{Ce}(\text{SO}_4)_2$ ) and electrical heating techniques.

Using these continuously maintained high levels of radiation it was possible to obtain totals of  $10^9$  gray ( $10^{11}$  rad) in approximately 100 days.

Figure 1

Dose Rate versus Age of Spent Fuel in HFIR Spent Fuel Storage Site.



### Characteristics of the High Flux Isotope Reactor (HFIR)

Design Power:	100 megawatts
Operational Date:	September 1966
Fuel Cycle:	23 days
Fuel Enrichment:	93% Uranium - 235
Fuel Geometry:	Two concentric cylinder
Inner Bundle:	Height - 60.96 cm (24 in)
	ID - 12.70 cm (5 in)
	OD - 26.67 cm (10.5 in)
Outer bundle:	Height - 60.96 cm (24 in)
	ID - 27.94 cm (11 in)
	OD - 43.50 cm (17.125 in)
Core Loading:	Fuel is in curved parallel fins 0.13 cm (0.05 in) thick 9.4 kilograms of enriched Uranium 2.3 grams of Boron
Pool Environment:	Temperature: $35 \pm 1^\circ\text{C}$ ( $95^\circ \pm 2^\circ\text{F}$ ) Chemistry: Deionized water pH 7 nominal

For exposures in a deionized water environment sample bars were systematically arranged vertically in layers in stainless steel baskets stacked into a cylindrical stainless steel holder. This holder was lowered into the uniform flux zone among the central axis of the spent fuel pedestal assembly. Samples were thus exposed to a uniform 360° gamma flux. Deionized water from the pool itself circulated among the samples.

For those samples being irradiated in the borated water environment, a somewhat more complex system was used. Water containing boric acid corresponding to the specified 2500 ppm of boron was circulated through a closed outer stainless steel cylinder in which the test bars were arranged as before. Solution pH was maintained at neutrality.

Finally, those test bars to be exposed in the absence of a water environment were placed within the spent fuel bundle within the gas evolution test apparatus. This will be described in the next section.

After the specified exposures, samples were removed from the baskets and holders, checked by ORNL Health Physics personnel, wrapped in aluminum foil, placed in zip lock polyethylene bags and transported to the appropriate facility for post irradiation characterization, dimensions, mass, strength, elastic modulus and microstructure.

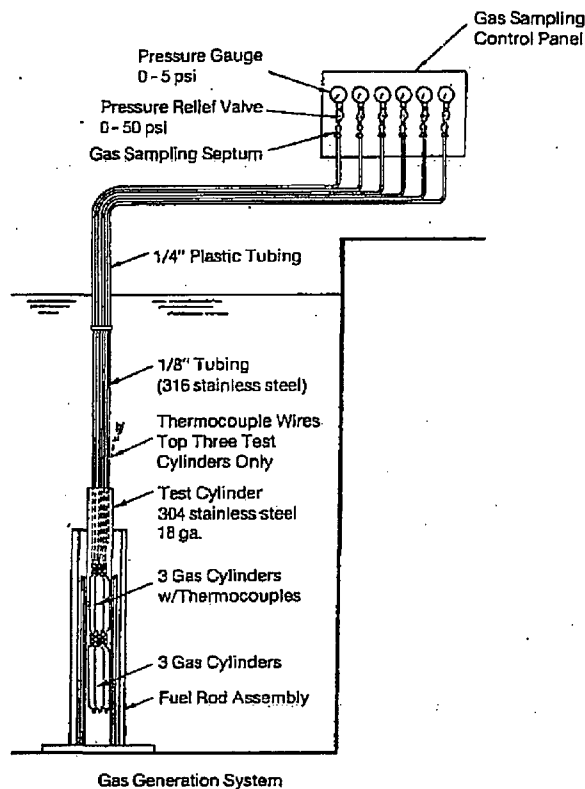
#### Gas Generation

In a separate test, samples, both dry and in the presence of adsorbed moisture, were placed in gas tight stainless steel ampules within the uniform high gamma flux test region. The fixtures, shown schematically in Figure 2 and in photograph (Fig. 3) were so arranged that pressure could be monitored, and gas samples were withdrawn from a position adjacent to and above the pool, without having to remove or otherwise disturb the apparatus.

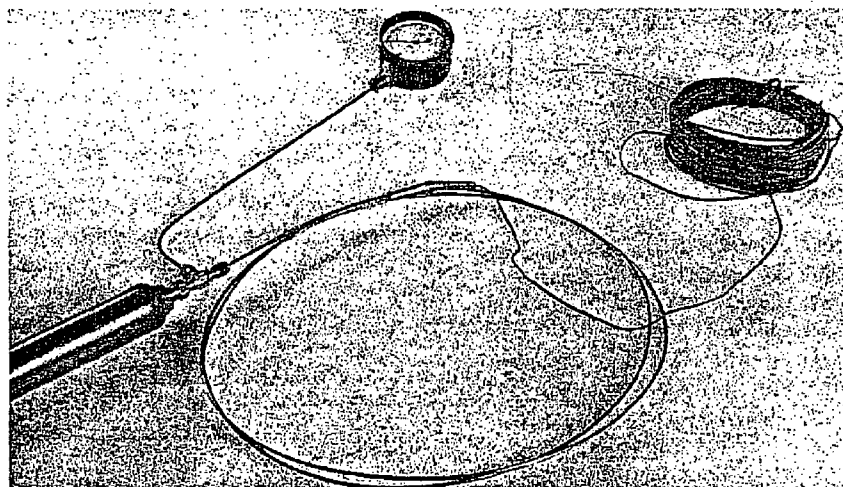
In this way, by monitoring pressures, by venting known volumes of gas, and by removing known volumes of gas for analysis, it was possible to determine both the incremental and total volume of gas produced and its composition as a function of the unit exposure and also as a function of the total exposure.

Dose rates of  $10^8$  gray ( $10^8$  rad) per hour and cumulative dose levels of  $10^7$  -  $10^8$  gray ( $10^7$  -  $10^{10}$  rad) were used. Each type of material was irradiated in two modes, as prepared together with small amounts of adsorbed water (normal) and after drying at  $105^\circ\text{C}$  for 12

**Figure 2**  
Schematic of Gas Generation Test Assembly.



**Figure 3**  
Assembly for Exposure of Samples for Temperature or Gas Evolution Measurements



hours (dry). Prior to gas generation testing, typical samples in gas sample ampules were lowered into the uniform flux region and their temperature was monitored until constant.

Samples of plate material sandwiched around a thermocouple were tested at  $2 \times 10^7$  and  $8 \times 10^7$  gray ( $2 \times 10^7$  and  $8 \times 10^7$  rad) per hour dose rates. The sheet material was tested only at the lower level. These results are shown in Figure 4. The thermal stability level of the material was not approached, even at the higher dose rates.

Samples of the gas were collected in gas tight, evacuated stainless steel cylinders. They were transported to one of the two laboratories (WL, LEX) for analysis by gas chromatography, using helium as the carrier gas. Measuring gas pressures on each cell, and knowing the volume of the cell assembly, together with the volumes and pressures of gas removed for analysis or by venting permitted the calculation of the total volume of gas produced in each test.

Measurements were made in parallel using polyethylene as a reference material in one cell. Showing that gas volumes and compositions evolved from the polyethylene control were in agreement with the numerous data reported in the published literature assured that the flux level was as expected, and that analytical procedures were adequate. One complication arose from the fact that the exposure conditions, namely the ambient atmosphere present within the sample container changed during the test. Initially, the ambient contained the essentially pure air sealed into the container with the samples.

This air and with it the reactive oxygen was preferentially removed with the evolved gas species in early phases of the test. In later stages after the air had become a minor constituent, this effect was reversed, and reaction products were removed preferentially. Nevertheless, each sample withdrawn further reduced the amount of residual air, and consequently the absolute oxygen concentration within the test environment.

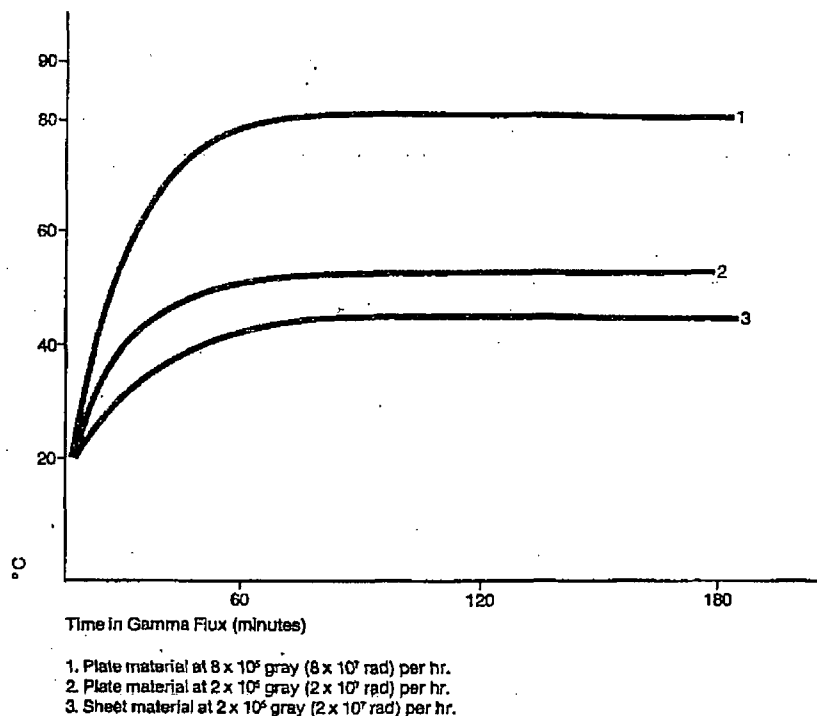
#### Leachability

In order to determine the effect of gamma irradiation on the leachability of boron species, samples of boron carbide materials in deionized water were exposed to gamma radiation levels over  $10^6$  gray ( $10^{10}$  rad). Periodically samples of the water were withdrawn by temporarily removing the sample container from the pool.

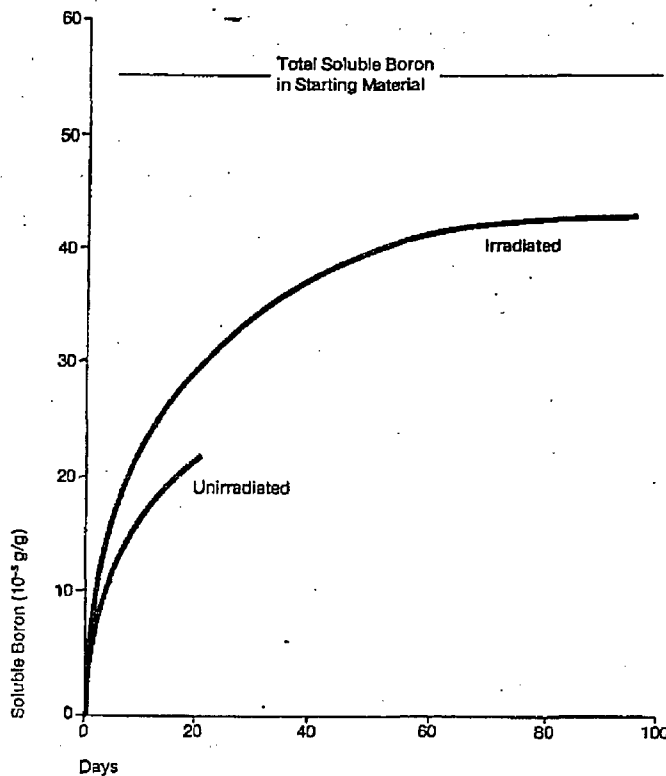
Due to the low levels of boron dissolved in the water from the samples, these tests could only be carried out in the deionized water environment. Deionized water would be expected to be the more effective one for boron dissolution.

In order to isolate radiative effects, additional samples of the plate material were immersed in deionized water in the absence of applied radiation. Differences between these baseline data, and those from the in-pool leaching tests may be attributed to the effects of radiation (Figure 5).

**Figure 4**  
Temperature of Samples Exposed in HFIR Spent Fuel Storage Site.



**Figure 5**  
Boron Leachability from Plate Material Under Irradiated and Non-Irradiated Conditions.



Data and results from each of these portions of the test program are summarized below without comment or discussion as a series of tables and figures. Discussion as to the procedures used can be found in previous sections, while those relating to observations concerning data and other ancillary observations are reserved for the discussion section which follows.

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Weight	XIV
<b>Borated Water Environment</b>	
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Weight	XVII
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Plate Material, Normal	XX & XXI
Plate Material, Dry	XXII & XXIII
Sheet Material, Normal	XXIV
Sheet Material, Dry	XXV
Specific Gas Generation	XXVI

**Table V**  
**Flexural Strength and Elastic Modulus of Plate Material Exposed in Deionized Water Environment\***

Dose ( $10^4$ gray $\gamma$ ) ( $10^{10}$ rad $\gamma$ )	MOR				MOE			
	$10^7$ Pa***		$10^3$ psi		$10^6$ Pa		$10^3$ psi	
	X	SD	X	SD	X	SD	X	SD
0**	5.09	0.72	7.38	1.05	12.00	2.21	1.74	0.32
0.1	3.85	0.52	5.59	0.76	12.07	2.48	1.75	0.36
1	3.38	0.37	4.90	0.54	8.75	2.00	1.27	0.29
1	3.12	0.28	4.53	0.41	8.76	2.00	1.27	0.29
3	3.01	0.32	4.37	0.47	7.03	0.55	1.02	0.08
5	2.19	0.41	3.17	0.59	4.41	0.90	0.67	0.13
8	2.44	0.37	3.54	0.54	4.62	0.76	0.53	0.11
10	1.94	0.26	2.82	0.37	3.65	0.41		0.06

\*Except in the unirradiated condition in which 24 specimens were used, each number was derived from measurements on 12 specimens.

\*\*As produced specimens, received no in-pool exposure.

\*\*\*All data converted to S.I. units, psi x 6894 = Pa.



**Table VI**  
**Dimensions of Plate Material Exposed in Deionized Water Environment\***

	Dose (10 <sup>5</sup> gray γ) (10 <sup>20</sup> rad γ)	Dimensions							
		Initial**				Final			
		(cm.)***		(in.)		(cm.)***		(in.)	
		X	SD	X	SD	X	SD	X	SD
Length	0.1	7.615	0.023	2.998	0.009	7.620	0.023	3.000	0.009
	1	7.612	0.013	2.997	0.005	7.645	0.015	3.010	0.006
	1	7.615	0.023	2.998	0.009	7.666	0.038	3.018	0.015
	3	7.633	0.025	3.005	0.010	7.666	0.023	3.018	0.009
	5	7.617	0.028	2.999	0.011	7.747	0.030	3.050	0.012
	8	7.635	0.140	3.006	0.055	7.732	0.036	3.044	0.014
	10	7.633	0.033	3.005	0.013	7.757	0.036	3.054	0.014
Width	0.1	1.280	0.013	0.504	0.005	1.280	0.013	0.504	0.005
	1	1.280	0.010	0.504	0.004	1.285	0.010	0.506	0.004
	1	1.285	0.010	0.506	0.004	1.298	0.013	0.511	0.005
	3	1.278	0.010	0.503	0.004	1.283	0.020	0.505	0.008
	5	1.273	0.010	0.501	0.004	1.293	0.010	0.509	0.004
	8	1.267	0.010	0.499	0.004	1.278	0.010	0.503	0.004
	10	1.270	0.018	0.500	0.007	1.285	0.015	0.506	0.006
Thickness	0.1	0.546	0.023	0.215	0.009	0.546	0.018	0.215	0.007
	1	0.541	0.010	0.213	0.004	0.544	0.010	0.214	0.004
	1	0.536	0.028	0.211	0.011	0.538	0.025	0.212	0.010
	3	0.549	0.013	0.216	0.005	0.544	0.013	0.218	0.005
	5	0.538	0.015	0.212	0.006	0.549	0.015	0.216	0.006
	8	0.541	0.015	0.213	0.006	0.549	0.018	0.216	0.007
	10	0.544	0.020	0.214	0.008	0.549	0.023	0.216	0.009

70 % of y  
 + .020  
 + .434  
 + .670  
 + .433  
 + 1.707  
 + 1.270  
 + 1.625

\*Each number derived from measurements on 12 specimens.

\*\*As produced specimens, receiving no in-pool exposure.

\*\*\*All data converted to S.I. units, in. x 2.54 = cm.

**Table VII**  
**Weight of Plate Material Exposed in Deionized Water Environment\***

Dose (10 <sup>5</sup> gray γ) (10 <sup>20</sup> rad γ)	Weight					
	Initial		Final		Loss (Gain) (g)	Loss (Gain) (%)
	X	SD	S	SD		
0.1	9.584	0.304	9.596	0.302	(0.014)	(0.15)
1.0	9.566	0.308	9.527	0.318	0.039	0.41
1.0	9.281	0.444	9.232	0.049	0.049	0.53
3.0	9.710	0.279	9.552	0.296	0.158	1.63
5.0	9.456	0.249	9.239	0.269	0.217	2.29
8.0	9.562	0.231	9.097	0.272	0.465	4.86
10.0	9.482	0.337	8.961	0.399	0.521	5.49

\*Each number derived from data on 12 specimens

**Table VIII**  
**Boron Leachability from Plate Material Exposed in Deionized Water Environment**

Dose (10 <sup>7</sup> gray γ) (10 <sup>22</sup> rad γ)	Exposure (days)	Total Boron Leached (10 <sup>-5</sup> g/g)	Incremental Boron Leached (10 <sup>-5</sup> g/g)
None	0.08	1.21	1.21
	1.0	4.04	2.83
	3.0	8.38	4.34
	7.0	12.7	4.32
	15.0	19.3	6.60
			56.9
0.1	0.16	2.15	2.15
3.4	5.16	17.7	15.2
5.5	9	22.8	5.1
9.6	19	26.2	3.1
13.0	30	30.9	4.7
14.4	36	35.2	4.3
19.7	58	42.3	7.1
30.1	106	42.3	0

\*Total soluble boron, based on analysis of starting material

**Table IX**  
**Flexural Strength and Elastic Modulus of Plate Material Exposed in Borated Water Environment\***

Dose (10 <sup>3</sup> gray γ) (10 <sup>20</sup> rad γ)	MOR				MOE			
	10 <sup>7</sup> Pa***		10 <sup>3</sup> psi		10 <sup>8</sup> Pa		10 <sup>6</sup> psi	
	X	SD	X	SD	X	SD	X	SD
0**	5.09	0.72	7.38	1.05	12.00	2.21	1.74	0.32
0.1	3.61	0.55	5.23	0.08	12.20	2.83	1.77	0.41
1	3.49	0.21	5.06	0.31	9.72	1.31	1.41	0.19

\*Except in the unirradiated condition in which 24 specimens were used, each number was derived from measurements on 12 specimens.

\*\*As produced specimens, receiving no in-pool exposure.

\*\*\*All data converted to S.I. units, psi x 6.894 x 10<sup>3</sup> = Pa.

**Table X**  
**Dimensions of Plate Material Exposed in Borated Water Environment\***

	Dose (10 <sup>3</sup> gray γ) (10 <sup>20</sup> rad γ)	Dimensions							
		Initial** (cm.)***				Final (cm.)***			
		X	SD	X	SD	X	SD	X	SD
Length	0.1	7.620	0.020	3.000	0.008	7.033	0.023	3.005	0.009
	1	7.607	0.018	2.995	0.007	7.630	0.018	3.004	0.007
Width	0.1	1.283	0.008	0.505	0.003	1.285	0.013	0.506	0.005
	1	1.288	0.010	0.507	0.004	1.293	0.013	0.509	0.005
Thickness	0.1	0.549	0.015	0.216	0.006	0.549	0.015	0.216	0.006
	1	0.549	0.015	0.216	0.006	0.549	0.015	0.216	0.006

\*Each number derived from measurements on 12 specimens.

\*\*As produced specimens, receiving no in-pool exposure.

\*\*\*All data converted to S.I. units, in. x 2.54 = cm.

**Table XI**  
**Weight of Plate Material Exposed in Borated Water Environment\***

Dose (10 <sup>3</sup> gray γ) (10 <sup>20</sup> rad γ)	Weight							
	Initial (g)		Final (g)		Loss (Gain) (g)		Loss (Gain) (g)	
	X	SD	X	SD				
0.1	9.571	0.286	9.634	0.283	(0.063)		(0.66)	
1.0	9.638	0.231	9.729	0.253	(0.091)		(0.94)	

\*Each number derived from data on 12 specimens.

**Table XII**  
**Ultimate Tensile Strength and Elastic Modulus of Sheet Material Exposed in Deionized Water Environment\***

Dose (10 <sup>4</sup> gray γ) (10 <sup>23</sup> rad γ)	UTS				MOE			
	10 <sup>7</sup> Pa***		10 <sup>3</sup> psi		10 <sup>8</sup> Pa		10 <sup>6</sup> psi	
	X	SD	X	SD	X	SD	X	SD
0**	6.09	0.79	8.84	1.14	1.52	0.21	0.22	0.03
0.1	5.83	0.92	8.46	1.33	1.10	0.14	0.16	0.02
1	4.53	0.61	6.59	0.89	1.17	0.28	0.17	0.04
	5.63	0.41	8.16	0.60	1.58	0.14	0.23	0.02
3	2.19	0.26	3.17	0.33	1.58	0.14	0.23	0.02
5	1.91	0.24	2.77	0.35	1.72	0.14	0.25	0.02
8	1.90	0.33	2.76	0.48	1.38	0.21	0.20	0.03
10	2.11	0.17	3.06	0.25	1.58	0.14	0.23	0.02

\*Each number derived from data on several specimens: unirradiated 24; 0.1 x 10<sup>4</sup>, 14; 1 x 10<sup>3</sup>, 15 and 12; others 12.

\*\*As produced specimens, received no in-pool exposure.

\*\*\*All data converted to S.I. units, psi x 6.894 x 10<sup>3</sup> = Pa.

**Table XIII**  
**Dimensions of Sheet Material Exposed in Deionized Water Environment\***

Parameter	Dose (10 <sup>3</sup> gray γ) (10 <sup>20</sup> rad γ)	Dimensions							
		Initial**				Final			
		(cm.)		(in.)		(cm.)		(in.)	
X	SD	X	SD	X	SD	X	SD		
Length	0.1	7.668	0.112	3.019	0.044	7.630	0.061	3.004	0.024
	1.0	7.617	0.033	2.999	0.013	7.617	0.036	2.999	0.014
	1.0	7.569	0.084	2.980	0.033	7.577	0.079	2.983	0.031
	3.0	7.557	0.043	2.975	0.017	7.564	0.046	2.978	0.018
	5.0	7.577	0.071	2.983	0.028	7.574	0.069	2.982	0.027
	8.0	7.579	0.061	2.984	0.024	7.577	0.061	2.983	0.024
	10.0	7.549	0.056	2.972	0.022	7.539	0.056	2.968	0.022
Width	0.1	1.354	0.030	0.533	0.012	1.367	0.033	0.538	0.013
	1.0	1.354	0.043	0.533	0.017	1.354	0.041	0.533	0.016
	1.0	1.306	0.023	0.514	0.009	1.308	0.023	0.514	0.009
	3.0	1.298	0.018	0.511	0.007	1.293	0.018	0.509	0.007
	5.0	1.313	0.038	0.517	0.015	1.306	0.033	0.514	0.013
	8.0	1.303	0.025	0.513	0.010	1.285	0.081	0.506	0.007
	10.0	1.298	0.046	0.511	0.018	1.265	0.030	0.498	0.012
Thickness	0.1	0.119	0.003	0.047	0.001	0.122	0.003	0.048	0.001
	1.0	0.119	0.003	0.047	0.001	0.124	0.003	0.049	0.001
	1.0	0.119	0.005	0.047	0.002	0.122	0.003	0.048	0.001
	3.0	0.124	0.005	0.049	0.002	0.122	0.003	0.048	0.001
	5.0	0.122	0.005	0.048	0.002	0.114	0.005	0.045	0.002
	8.0	0.122	0.005	0.048	0.002	0.109	0.028	0.043	0.011
	10.0	0.122	0.003	0.048	0.001	0.107	0.003	0.042	0.001

*7% Change*

- .496  
 0  
 - .106  
 + .093  
 - .040  
 - .026  
 - .132  
 + .960  
 0  
 0  
 - .385  
 - .533  
 - 1.381  
 - 2.542  
 +  
 +  
 +  
 - 1.613  
 - 6.557  
 - 10.656  
 - 12.30

\*Numbers were derived from measurements on several samples; 0.1, 14; 1.0, 15 and 12; others, 12.

\*\*As prepared specimens, no in-pool exposure.

**Table XIV**  
**Weight of Sheet Material Exposed in Deionized Water Environment\***

Dose (10 <sup>3</sup> gray γ) (10 <sup>20</sup> rad γ)	Weight					
	Initial		Final		Loss (g)	Loss (%)
	(g)		(g)			
X	SD	X	SD			
0.1	1.761	0.059	1.757	0.059	0.004	0.23
1.0	1.787	0.098	1.767	0.096	0.020	1.10
1.0	1.687	0.049	1.660	0.049	0.027	1.60
3.0	1.718	0.061	1.653	0.060	0.065	3.78
5.0	1.735	0.086	1.511	0.049	0.224	12.91
8.0	1.704	0.055	1.399	0.042	0.305	17.90
10.0	1.675	0.066	1.337	0.067	0.338	20.18

\*Numbers were derived from measurements on several specimens; 0.1, 14; 1.0, 15 and 12; others, 12.

**Table XV**  
**Ultimate Tensile Strength and Elastic Modulus of Sheet Material Exposed in Borated Water Environment\***

Dose (10 <sup>3</sup> gray γ) (10 <sup>20</sup> rad γ)	UTS				MOE			
	10 <sup>7</sup> Pa***		10 <sup>3</sup> psi		10 <sup>8</sup> Pa		10 <sup>6</sup> psi	
	X	SD	X	SD	X	SD	X	SD
0**	6.09	0.79	8.84	1.14	1.52	0.21	0.22	0.03
0.1	5.63	0.69	8.16	1.00	1.17	0.34	0.17	0.05
1	5.31	0.62	7.70	0.90	1.24	0.21	0.18	0.03

\*Each number derived from data on several specimens; unirradiated, 24; 10<sup>3</sup>, 10<sup>20</sup> 12.

\*\*As produced specimens, received no in-pool exposure.

\*\*\*All data converted to S.I. units, PPSix 6.897 x 10<sup>3</sup> = Pa.

**Table XVI**  
**Dimensions of Sheet Material Exposed in Borated Water Environment\***

	Dose (10 <sup>3</sup> gray γ) (10 <sup>3</sup> rad γ)	Dimensions							
		Initial**				Final			
		(cm.)		(in.)		(cm.)		(in.)	
		X	SD	X	SD	X	SD	X	SD
Length	0.1	7.623	0.066	3.001	0.026	7.695	0.066	3.006	0.026
	1	7.610	0.030	2.996	0.012	7.617	0.028	2.999	0.011
Width	0.1	1.348	0.028	0.530	0.011	1.349	0.025	0.531	0.010
	1	1.326	0.033	0.522	0.013	1.326	0.036	0.522	0.014
Thickness	0.1	0.122	0.005	0.048	0.002	0.122	0.005	0.048	0.002
	1	0.122	0.003	0.048	0.001	0.122	0.003	0.048	0.001

\*Each number derived from data on 12 specimens.  
 \*\*As produced specimens, received no in-pool exposure.

**Table XVII**  
**Weight of Sheet Material Exposed in Borated Water Environment\***

Dose (10 <sup>3</sup> gray γ) (10 <sup>3</sup> rad γ)	Weight				Loss (g)	Loss (%)
	Initial**		Final			
	X	SD	X	SD		
0.1	1.777	0.057	1.773	0.057	0.004	0.23
1	1.738	0.073	1.733	0.071	0.005	0.29

\*Each number derived from data on 12 specimens.  
 \*\*As produced specimen, before in-pool exposure.

**Table XVIII**  
**Gas Evolution from Polyethylene Reference Material**

Dose (10 <sup>7</sup> gray γ) (10 <sup>6</sup> rad γ)	Total Specific Gas Evolution (cm <sup>3</sup> /g)	Specific Gas Evolution (10 <sup>-7</sup> cm <sup>3</sup> /g gray) (10 <sup>-6</sup> cm <sup>3</sup> /g rad)
0.065	3.3	0.51
0.20	9.5	0.48
0.22	10.2	0.46
0.31	13.1	0.42
0.44	19.6	0.45

**Table XIX**  
**Composition of Gas Evolved from  
 Polyethylene Reference Material (%)**

Component	Exposure Level (10 <sup>7</sup> gray γ) (10 <sup>6</sup> rad γ)		
	0.065	0.31*	0.31**
H <sub>2</sub>	N.D.***	65.9	54
O <sub>2</sub>	22	1.75	4.8
N <sub>2</sub>	78	31.3	37
CO <sub>2</sub>	N.D.	N.D.	3.2
CO	N.D.	N.D.	0.8
CH <sub>4</sub>	N.D.	N.D.	N.D.
Total	100	98.9	99.8

\*Analyzed at Lexington Development Center  
 \*\*Analyzed at Pennsylvania State University  
 \*\*\*Residual Air

**Table XX**  
**Gas Evolution from Plate Material Exposed in Normal State**

Dose (10 <sup>7</sup> gray γ) (10 <sup>6</sup> rad γ)	Total Volume of Gas Evolved (cm <sup>3</sup> /g)	Average Specific Gas Evolution (10 <sup>-7</sup> cm <sup>3</sup> /g gray) (10 <sup>-6</sup> cm <sup>3</sup> /g rad)	Incremental Rate of Gas Evolution (10 <sup>-7</sup> cm <sup>3</sup> /g gray) (10 <sup>-6</sup> cm <sup>3</sup> /g rad)
1.44	0.808	0.56	0.56
2.95	1.69	0.57	0.54
3.62	1.92	0.53	0.34
5.54	2.76	0.50	0.44
6.45	3.01	0.47	0.27

**Table XXI**  
**Composition of Gas Evolved from Plate Material Exposed in Normal State**

Component	Exposure Level (10 <sup>7</sup> gray γ) (10 <sup>6</sup> rad γ)									
	1.44		2.95		3.62		5.54		6.77	
	LDC*	PSU**	LDC*	LDC*	PSU**	PSU**	LDC*	LDC*		
H <sub>2</sub>	13	37	50	69	59	77	75	71		
O <sub>2</sub>	16	5	3	1	2	1	1	2		
N <sub>2</sub>	68	51	38	18	27	12	10	11		
CO <sub>2</sub>	2	6	7	11	11	9	12	14		
CO	N.D.	Tr	Tr	Tr	Tr	Tr	Tr	Tr		
CH <sub>4</sub>	Tr	N.D.	Tr	Tr	Tr	Tr	N.D.	Tr		
Total	100	100	99	100	100	100	100	100		

\*Analyzed at Lexington Development Center  
 \*\*Analyzed at Pennsylvania State University

**Table XXII**  
Gas Evolution from Plate Material Exposed in Dry State

Dose ( $10^7$ gray $\gamma$ ) ( $10^6$ rad $\gamma$ )	Total Volume of Gas Evolved ( $\text{cm}^3/\text{g}$ )	Average Rate of Gas Evolution ( $10^{-7}$ $\text{cm}^3/\text{g}$ gray) ( $10^{-9}$ $\text{cm}^3/\text{g}$ rad)	Incremental Rate of Gas Evolution ( $10^{-7}$ $\text{cm}^3/\text{g}$ gray) ( $10^{-9}$ $\text{cm}^3/\text{g}$ rad)
1.44	0.78	0.54	0.54
2.95	1.50	0.51	0.48
3.62	1.74	0.48	0.36
5.54	2.45	0.44	0.37
6.45	2.68	0.42	0.25

**Table XXIV**  
Gas Evolution from Sheet Material Exposed in Normal State

Dose ( $10^7$ gray $\gamma$ ) ( $10^6$ rad $\gamma$ )	Total Gas Evolved ( $\text{cm}^3/\text{g}$ )	Average Rate of Gas Evolution ( $10^{-7}$ $\text{cm}^3/\text{g}$ gray) ( $10^{-9}$ $\text{cm}^3/\text{g}$ rad)	Incremental Rate of Gas Evolution ( $10^{-7}$ $\text{cm}^3/\text{g}$ gray) ( $10^{-9}$ $\text{cm}^3/\text{g}$ rad)
1.44	1.00	0.69	0.69
3.62	2.07	0.57	0.49
6.45	2.75	0.42	0.24

**Table XXV**  
Gas Evolution from Sheet Material Exposed in Dry State

Dose ( $10^7$ gray $\gamma$ ) ( $10^6$ rad $\gamma$ )	Total Gas Evolved ( $\text{cm}^3/\text{g}$ )	Average Rate* of Gas Evolution ( $10^{-7}$ $\text{cm}^3/\text{g}$ gray) ( $10^{-9}$ $\text{cm}^3/\text{g}$ rad)	Incremental Rate** of Gas Evolution ( $10^{-7}$ $\text{cm}^3/\text{g}$ gray) ( $10^{-9}$ $\text{cm}^3/\text{g}$ rad)
1.44	0.54	0.38	0.38
6.45	1.35	0.21	0.04

\*Calculated by dividing Total Gas Evolved per gram by the Total Dose.

\*\*Calculated by dividing the difference between the last two Total Gas Volumes by the difference between the last two Dose Levels.

**Table XXVI**  
Specific Gas Generation by Polyethylene, and by Plate and Sheet Materials

Dose (gray $\gamma$ ) (rad $\gamma$ )	P.E.	G( $10^{-7}$ $\text{cm}^3/\text{g}$ gray) G( $10^{-9}$ $\text{cm}^3/\text{g}$ rad)			
		Plate		Sheet	
		Normal	Dry	Normal	Dry
$4 \times 10^6$	4.5				
$6 \times 10^6$		0.275	0.253	0.240	0.162

**Table XXIII**  
Composition of Gas Evolved from  
Plate Material in Dry State

Component	Exposure Level ( $10^7$ gray $\gamma$ ) ( $10^6$ rad $\gamma$ )		
	1.44	2.95	3.62
H <sub>2</sub>	2	43	39
O <sub>2</sub>	19	2	5
N <sub>2</sub>	76	44	43
CO <sub>2</sub>	2	9	12
CO	N.D.	Tr	N.D.
CH <sub>4</sub>	Tr	N.D.	N.D.
Total	99	98	99

\*Analyzed at Lexington Development Center  
\*\*Analyzed at Pennsylvania State University

### Deionized versus Borated Water Environment

#### Environment

As originally designed, the experimental program consisted of two parallel series of tests, one consisting of exposures in a deionized water environment, the other in borated water. This was done to simulate the chemistry found in the spent fuel storage pools of both PWR and BWR systems, with and without dissolved boric acid.

It soon became apparent that this duplication would be not only needlessly time consuming, but unnecessary. Exposures to as little as  $10^8$  gray ( $10^{20}$  rad) of gamma radiation were sufficient to show that adverse radiation effects of both strength reduction and weight loss of plate and sheet materials were consistently as great or greater in the deionized water environment as they were in borated water (Tables XXVII through XXIX).

This increased severity of the deionized water environment, together with the item savings brought about by reducing the test scope, and the inability to carry out meaningful leaching tests in the presence of added boron, all led to the elimination of further testing in borated water environments.

On this basis, further long range testing beyond the  $10^8$  gray ( $10^{20}$  rad) level in a borated water environment was eliminated and all such tests were confined to the deionized water environment.

#### Wet Strength of Materials

In order to be able to reproduce exact test conditions, one test criteria specified that all samples be dried at  $105^\circ\text{C}$  prior to testing. While this method provided reproducible conditions, equivalent to those before exposure, it imposed an unrealistic factor. In actual use the materials are exposed to continual saturation. Thus, determination of strength in a saturated condition would be a more practical measure of in-pool capabilities of these neutron absorbing materials.

Accordingly, groups of sheet material samples were broken in both the saturated and after-drying conditions. Although all of the literature pertaining to strength properties of glass fiber reinforced composites report a decrease in observed strength on wetting, all data from the present program show the opposite. Samples in the as-tested, water

Strength of Plate Material Exposed in Borated and Deionized Water Environments

Dose ( $10^8$ gray $\gamma$ ) ( $10^{20}$ rad $\gamma$ )	MOR				Strength Loss (%)	
	Borated		D.I.			
	$10^7$ Pa	$10^3$ psi	$10^7$ Pa	$10^3$ psi	Borated	D.I.
0	5.09	7.38	5.09	7.38		
0.1	3.61	5.23	3.85	5.59	29.1	24.3
1.0	3.49	5.06	3.38	4.90	31.4	33.6
			3.12	4.53		38.8

Table XXVIII

Ultimate Tensile Strength of Sheet Material Exposed in Borated and Deionized Water Environments

Dose ( $10^8$ gray $\gamma$ ) ( $10^{20}$ rad $\gamma$ )	U.T.S.				Strength Loss (%)	
	Borated		D.I.			
	$10^7$ Pa	$10^3$ psi	$10^7$ Pa	$10^3$ psi	Borated	D.I.
0	8.09	8.84	6.09	8.84	—	—
0.1	5.63	8.16	5.83	8.46	7.6	4.3
1.0	5.31	7.70	5.63	8.16	12.8	7.9
			4.53	6.59		25.5

Table XXIX

Weight Loss During Exposure in Borated and Deionized Water Environments

Dose ( $10^8$ gray $\gamma$ ) ( $10^{20}$ rad $\gamma$ )	Weight Loss %			
	Plate		Sheet	
	D.I.	Borated	D.I.	Borated
0.1	(0.15)*	(0.66)	0.23	0.23
1.0	0.41	(0.94)	1.10	0.29
1.0	0.53		1.60	

\*Parentheses indicate a weight gain.

Table XXX

Effect of Drying on Ultimate Tensile Strength of Irradiated Boron Carbide Sheet Material

Radiation ( $10^8$ gray $\gamma$ ) ( $10^{20}$ rad $\gamma$ )	Ultimate Tensile Strength			
	Wet (As Exposed)		Dry (1 hr. @ $105^\circ\text{C}$ )	
	$10^7$ Pa	$10^3$ psi	$10^7$ Pa	$10^3$ psi
5	3.51	5.09	1.91	2.77
8	3.20	4.64	1.90	2.76
10	4.10	5.94	2.11	3.06

saturated condition were appreciably stronger, by a factor of nearly two, than corresponding samples after drying at  $105^\circ\text{C}$  ( $220^\circ\text{F}$ ).

Numerous inquiries to persons specializing in glass fiber composites failed to provide a clear explanation for this increased observed wet strength. One logical hypothesis (21) put forth was that the presence of a liquid within the organic matrix might allow a certain (though small) degree of motion of one phase with respect to the other.

These slight displacements could permit fiber alignment such that a greater fraction of the reinforcing phase fibers become stressed before appreciable fiber failure causes the specimen to break. In other words, the stress on a number of fibers would be more nearly equal, as opposed to the other extreme in which certain fibers would reach their failure stress levels without other fibers receiving a significant load.

These strength data are shown in Table XXX.

### Plate Material Strength

The plate material, though bonded by an organic polymer, forms a rigid structure which deforms elastically, and fails without significant plastic or non-recoverable deformation.

Irradiated samples showed a rapid initial decrease in strength. Two factors, radiation damage and moisture, were suspected. To separate these effects, a series of samples, both plate and sheet, were immersed in deionized water in the absence of radiation. To accelerate the test, it was carried out at 93°C.

After only one day, strength of the plate was reduced by 42%, and after three days, 55%. This level remained constant throughout the remainder of the 16 day test (Figure 6).

### Elastic Modulus

As shown in Table V the elastic modulus of plate material showed a continual decrease with increasing radiation level. After  $10^9$  gray ( $10^{11}$  rad) the elastic modulus had decreased to  $3.6 \times 10^9$  Pa ( $0.5 \times 10^8$  psi). To put this in perspective, this final elastic modulus is approximately the same as that of unreinforced phenolic resin as formulated.

### Dimensions

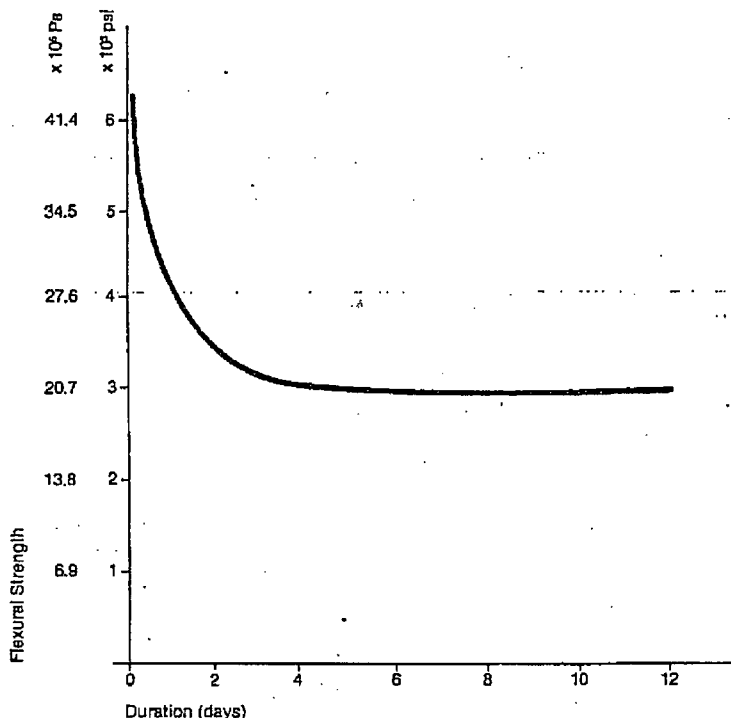
Dimensions of plate material during in-pool radiation exposures up to  $10^9$  gray ( $10^{11}$  rad) showed slight increases. At the highest level of exposure, length increased by 1.6% with a standard deviation of 0.4 - 0.5% on both the original and final measurements. Changes in dimensions in either the width or thickness directions were less than the standard deviation of either the initial or final measurements and as such are not significant.

### Weight Loss/Boron Leachability

These studies were not designed with the purpose in mind of determining mechanisms, but instead to show the overall effects of radiation on gas evolution, strength, weight loss and boron loss. For this reason, data obtained are not sufficient to separate and quantify the several mechanisms which may be contributing to weight losses. These may include gas evolution, solution, transmutation and attrition.

Evolution of gaseous species resulting from radiation degradation of the polymeric bond phase contributes to weight losses. Since the volume and composition of the gas are known, the weight can be calculated at least to a first approximation. These calculations are shown in Appendix 2. A weight loss of 0.2% can be accounted for by gas evolution.

Figure 6  
Strength of Plate Material as a Function of Water Soak Duration (D.I. Water 93°C).



Solution effects are more complicated. Soluble species include soluble boron species, such as those present in the original boron carbide powders, incompletely polymerized polymer fractions and soluble degradation products arising from degradation of the polymer phase. Only one of these, soluble boron, is determinable from the data.

It has been reported (23) that intense radiation of phenolic polymers produces not only swelling, hardening, and embrittlement, but also yields a water soluble degradation product, "which causes the material to disintegrate in water."

Long term leaching data, seen graphically in Fig. 5 showed that under non-radiative conditions boron losses amounted to 0.025% over a period of 25 days. The leachability boron under gamma radiation ( $1.3 \times 10^8$  gray,  $1.3 \times 10^{10}$  rad) amounted to 0.047% in 30 days.

Transmutation, the conversion of boron atoms to soluble lithium atoms and helium gas results from neutron exposure. Since the zone in which the gamma radiation exposures were carried out is cadmium shielded, it is safe to assume that neutron-related transmutation effects are negligible. As an order of magnitude  $10^{10}$  absorbed neutrons per cubic centimeter would assume only about  $10^{-10}$ % of the available boron-10 atoms, or  $2 \times 10^{-11}$ % of the total boron present.

Another mechanism, and in fact probably the most important one is weight loss due to attrition. Embrittlement and weakening of the polymeric bond phase reduces the tenacity with which the boron carbide particles are held. After exposures at the higher levels of radiation, surfaces showed a tendency to "rub off." During removal of samples, handling, wrapping, shipping and testing various amounts of material were lost. Test conditions were such that losses during each of the steps after removal from the pool could not be quantitatively determined. All that can be said with certainty is that losses of weight in the near static in-pool environment are significantly less than the observed total weight losses.

In summary, weight losses from each of these mechanisms may be approximated.

Gas evolution	0.2%
Boron Solubility	0.05%
Transmutation	negligible
Total weight loss	5.5%
Attrition (by difference)	5.25%

The plate material was manufactured with an excess of 10% boron over the specified areal density. Samples of plate both before and after irradiation were analyzed for boron by destructive methods. Results before and after show a final boron retention exceeding that for which it was designed.

### Gas Evolution

Organic molecules are not stable high energy radiation such as that found in spent fuel pools. The energy of this gamma radiation is greater than even the strongest, most stable organic bonds. When a bond is ruptured any of several things can happen.

First, two free radicals can be formed. These can rearrange, cross link, split off hydrogen, split off functional groups or any of a myriad of possible free radical reactions. The presence of oxygen or moisture is known to have a very strong effect on the stability of free radicals in synthetic organic chemistry. They produce, through interactions with free radicals, oxidation and hydrolysis, with possible intermediate formation of ozonide and peroxide linkages. These last react to form oxidation products and hydroxyl groups. Within the plate material, and more particularly within the resin phase, water and oxygen will not be as available, hence the scission, polymerization, and cross linking reactions would be expected to predominate.

The tests were designed to demonstrate gas evolution rate and total volume, as well as composition. They were not designed to differentiate among various possible mechanisms. In a general way they suggest that internal polymerization, scission, can cross linking predominate, although hydrolysis cannot be ruled out. Each of these could contribute to hydrogen produced.

Oxidation clearly is another contributing mechanism. Not only is the most logical non condensable oxidation product ( $\text{CO}_2$ ) present in increasing concentrations, but the nitrogen-oxygen ratio increases far above that in air (4.0), indicating that oxygen is being consumed. This change in nitrogen/oxygen ratio could also be explained on the basis of reaction between the hydrogen formed and residual oxygen. Were this the case, and recognizing the violence of hydrogen oxygen reaction, damage in the form of distorted cans and broken shielding might well be anticipated. This was not seen, however.

The fact that gaseous products from the polyethylene control material were of the quantity and composition predicted from published data lend credence to the fact that the test conditions and analytical techniques were as expected.

### Sheet Material

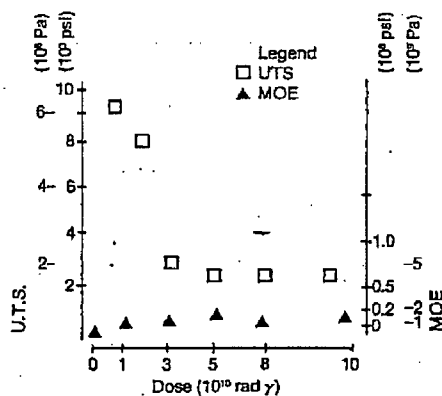
#### Strength

Mechanical behavior of sheet material exhibits certain basic differences from that of plate material. Strength and

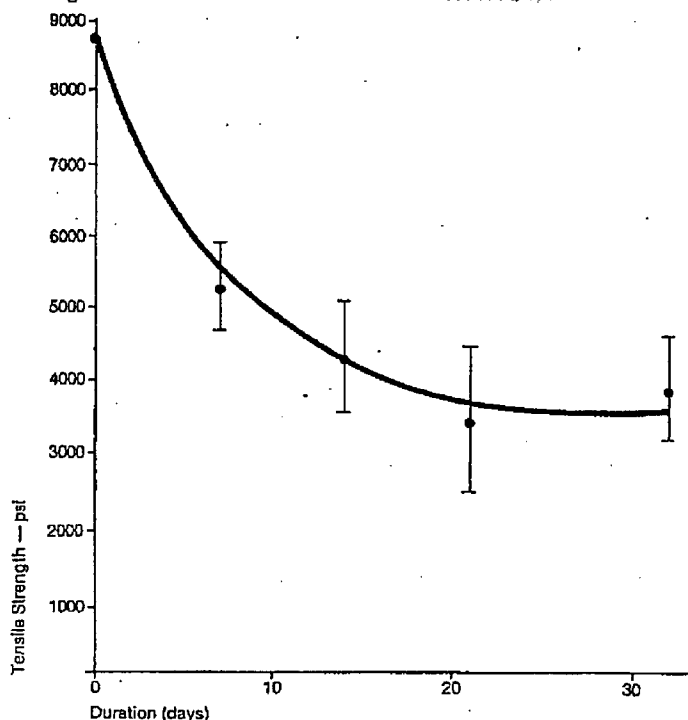
elastic modulus are primarily to the presence of the woven glass fabric rather than to the resin and boron carbide components. Strength of sheet material, measured as ultimate tensile strength showed rapid initial decrease followed by a nearly constant plateau. (Figure 7). The initial rapid decrease may be ascribed to the initial soaking. Typical strength of glass fiber reinforced composites when exposed to water shows up as rapid decrease during the initial soak, thereafter remaining constant through further water exposure.

In order to determine what portion of the initial strength loss is due to water infiltration, and not radiation, samples were exposed to deionized water under non-radiative conditions. The results (Figure 8) show clearly that a major

**Figure 7**  
Ultimate Tensile Strength (UTS) and Modulus of Elasticity (MOE) of Sheet Material as a Function of Dose in D.I. Water Environment.



**Figure 8**  
Strength of Sheet Material as a Function of Water Soak.



portion of the initial strength loss is due to water, and not to effects of radiation.

Data obtained from samples exposed in borated water, though limited, showed similar trend.

The observed elastic moduli of glass fiber composites, other than those containing fibers all of which are oriented uniaxially, are far lower than would be expected on the basis of the volume fraction of fibers present.

Observed values of the elastic moduli of sheet material samples were all approximately  $1.4 \times 10^8$  Pa ( $0.2 \times 10^6$  psi), and remained unchanged, within statistical limits of significance throughout the test exposures.

### Dimensions

After  $10^8$  gray ( $10^{14}$  rad) of gamma irradiation, neither of the dimensions parallel to the glass fabric (length, width) had changed significantly. Dimensional changes were all within one standard deviation.

Up to  $8 \times 10^8$  gray ( $8 \times 10^{10}$  rad) exposure level observed thickness decreases were likewise within one standard deviation. This lack of statistical significance belies the fact that losses of material had actually occurred, and thicknesses were similarly decreasing.

At  $10^9$  gray ( $10^{11}$  rad) a statistically significant thickness decrease (12.5%) was observed. These thickness changes were at least in part associated with physical attrition and loss of particulate material from the surface. This is discussed in more detail in the section addressing weight loss.



### Weight Loss/Boron Leachability

Possible mechanisms to which weight losses could be ascribed are enumerated and discussed in an earlier section. One additional mechanism, insignificant by comparison with others, is the solubility and leaching of components in the glass fabric reinforcing phase.

Weight losses in the sheet material reached 20% at 10<sup>9</sup> gray (10<sup>11</sup> rad) exposure. While it is not possible to quantify losses due to each mechanism, physical losses due to spalling and surface abrasion quite obviously account for the major portion. Finger and forceps were usually soiled with particulate material after handling highly irradiated specimens.

As in the case of the plate material the recorded weight losses were unrealistically high. Major fractions of these losses were associated with specimen handling and were not losses incurred within the pool environment.

Analysis of the sheet material after exposure to 10<sup>9</sup> gray (10<sup>11</sup> rad) showed a reduction in boron content of 15%, compared to the noted 20% weight loss of the material.

The amount of gas evolution data obtained with the sheet material was considerably less than that obtained using plate. The results both in quantity of gas evolved and its composition, are similar to those obtained from plate experiments.

### Correlation with Electron Beam Experiments

To facilitate and speed testing, early exposure experiments were carried out using electron beam (EB) radiation to simulate in-pool results. These earlier EB data are compared with gamma irradiation on plate material (Table XXXI). Although the two sets of data are not strictly comparable, they suggest that strength reductions are somewhat less severe, for given dose level with EB than with gamma radiation.

Table XXXI

Effect of Electron Beam versus Gamma Radiation on Strength of Boron Carbide Plate Material

Dose (10 <sup>9</sup> gray) (10 <sup>10</sup> rad)	Gamma		EB	
	(10 <sup>7</sup> Pa)	(10 <sup>9</sup> psi)	(10 <sup>7</sup> Pa)	(10 <sup>9</sup> psi)
0	5.09	7.38		
1.0	3.12	4.53		
1.0	3.38	4.90		
2.0			3.00	4.35
3.0	3.01	4.37		
4.4			2.74	3.97
5	2.19	3.17		
8	2.44	3.54		
10	1.94	2.82	2.38	3.45
15			2.08	3.01
20			1.97	2.86

## Appendix 1

### Glossary

Borated Water — A solution containing 2500 ppm boron in water

Boron Carbide Materials — either of the two material systems described below:

1. Boron Carbide Plate — A fabricated two phase material composed of boron carbide grain and phenolic binder
2. Boron Carbide Composite — A fabricated, three phase material composed of boron carbide grain, a reinforcing fabric and phenolic binder

Gray - (gy) = joules (j)/kg; 1 gy = 100 rad

HFIR - High Flux Isotope Reactor at the Oak Ridge National Laboratory

LEX - Kennecott Copper Corporation's research facility in Lexington, Massachusetts

N

Mean ( $\bar{X}$ ):  $(1/N) \sum_{i=1}^N X_i$

where N = number of observations  
X<sub>i</sub> = individual observations

MRL - Materials Research Laboratory, Pennsylvania State University

Pascal - (Pa) = kg. m/sec<sup>2</sup>; 6894 Pa = 1 lb/in<sup>2</sup>

$$SD = \sqrt{\left[ \frac{1}{N-1} \right] \sum_{i=1}^N (X_i - \bar{X})^2}$$

N = number of observations  
X<sub>i</sub> = individual observations  
 $\bar{X}$  = mean of N observations

SS - stainless steel (Type 304 unless otherwise specified)

## Appendix 2

Calculation of weight loss of plate material due to gas evolution.

Assume:

- a. Specific gas formation (see Table XXVI)  
2.5 x 10<sup>-7</sup> cm<sup>3</sup>/g gy (2.5 x 10<sup>-9</sup> cm<sup>3</sup>/g rad)
- b. Gas composition (see Table XIX); 86% H<sub>2</sub>, 14% CO<sub>2</sub>
- c. No condensation of gaseous degradation products.

$$(0.25 \times 10^{-7} \text{ cm}^3/\text{g gy}) \times 10^9 \text{ gy} \times 1 \text{ g} = 25 \text{ cm}^3$$

$$\frac{0.86}{2} = 0.43 \text{ MOL (Volume) Fraction H}_2$$

$$\frac{0.14}{44} = 0.003 \text{ MOL (Volume) Fraction CO}_2$$

$$\frac{0.43}{0.43} \times 100 = 99\% \text{ H}_2$$

- d. Neglect species other than Hydrogen  
[25 cm<sup>3</sup>/22,400 cm<sup>3</sup>/MOLE x 2 g/MOLE  
= 0.0022 g H<sub>2</sub>/g Plate @ 10<sup>9</sup> gr  
= .22% weight loss as H<sub>2</sub> @ 10<sup>9</sup> gy (10<sup>7</sup> rad)

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