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IRRADIATION OF TANTALUM AND VIBRATORY-COMPACTED
 B_4C IN THE EBR-II - - FIRST DISCHARGE

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IRRADIATION OF TANTALUM AND VIBRATORY-COMPACTED

B₄C IN THE EBR-II — FIRST DISCHARGE

INTRODUCTION

Boron carbide and tantalum have been designated as the primary candidate control materials for the Fast Flux Test Facility (FFTF). However, little or no irradiation data have been accumulated on these materials under projected FFTF operating conditions.⁽¹⁾ The bulk of irradiation testing on B₄C has been performed in thermal reactors at relatively low temperatures (~ 500°F). In a fast neutron spectrum, the absorption cross section of B₄C is greatly reduced, and reaction occurs more homogeneously within the material than in a thermal flux, where the reaction is concentrated near the surface. At FFTF control rod temperatures, >1100°F, the kinetics of helium gas release (formed from the B¹⁰ (n,α) Li⁷ reaction) will likely be different than at the lower temperatures previously investigated. Also, radiation-induced damage to the lattice from the large fraction of high-energy neutrons in a fast flux spectrum probably will cause B₄C to behave differently than in a thermal reactor spectrum (increased swelling rate, for example). The prime candidate material for low-worth regulating rods in the FFTF is tantalum. Again, irradiation data applicable to the operating conditions of the FTR control rods are lacking. For these reasons, the behavior of FFTF control materials can not be reliably predicted from the available data, and it is necessary to test the candidate materials in a fast reactor system under conditions typical of the FFTF.

A seven-pin non-instrumented experimental subassembly containing four boron-carbide capsules and three tantalum capsules was irradiated in a Row 7 position of the EBR-II. The B₄C capsules, designated BC-1, BC-2, BC-4, and BC-5, each contained a six-inch column of vibratory compacted B₄C (80% TD) in a 1/2 inch dia. 304 stainless steel tube. The cladding thickness of BC-1 and BC-2 was 0.065 in., while that of BC-4 and BC-5 was 0.035 in. Two cladding thicknesses were chosen to determine the relative effectiveness in restraining any swelling of the B₄C that might occur. The tantalum capsules, designated

TA-1, TA-2, and TA-3, each contained a single tantalum sample 1/4 in. dia. x 3 in. long. After six months of irradiation, the subassembly was discharged from the reactor and two each B₄C and tantalum capsules, BC-1, BC-4, TA-1, and TA-2, were removed and destructively examined. They were replaced in the subassembly by similar capsules, and the irradiation is presently continuing. This report presents the results obtained from the four capsules that were disassembled after the partial discharge.

SUMMARY

Two capsules containing vibratory-compacted boron carbide (80% TD) and two capsules each containing a tantalum rod sample were removed from an EBR-II seven-pin experimental subassembly and disassembled after 1.58 atom% B¹⁰ burnup in the B₄C and 6.8×10^{21} nvt ($E > 0.1$ MeV) exposure on the tantalum. The irradiation temperature of the B₄C samples was about 1100°F, while that of the tantalum specimens was ~1200°F. The B₄C was contained in 1/2 in. dia. 304 stainless steel cladding, the wall thickness being 0.065 in. in one capsule and 0.035 in. in the other. No significant cladding deformation was measurable in either capsule. There was some unrestrained axial growth of the powders (~1/2%), but it is uncertain whether this was a result of radiation induced swelling of the B₄C, or was caused by jostling of the capsules during shipment and handling. Approximately 30% of the helium gas generated in the irradiation from the B¹⁰ (n,α) Li⁷ reaction was released from the B₄C. Although the boron carbide powder samples had a sintered appearance and were hard to remove from the capsules during disassembly, a sieve-analysis showed that the particles actually broke up during irradiation, particularly those in the 10-20 mesh range. Metallography of the cladding suggested there may have been some slight poison-clad interaction in the thin-walled capsule only. This capsule probably ran ~40°F hotter than the thick-walled capsule because of the larger quantity of B₄C present.

Length measurement of the tantalum samples after irradiation showed a 0.12% growth. Densitometer measurements of the specimens disclosed a density decrease of 0.56% (16.73 g/cc → 16.63 g/cc).

MATERIALS

The boron carbide samples were in the form of vibratory-compacted powder samples of 80% TD. The powders were obtained from the Carborundum Company, and are designated as their "High Boron" grade. A chemical analysis of the material is given in Table I. Several mixtures of particle sizes were investigated, and the formula which yielded the highest packing density is given in Table II.

TABLE I
Chemical Analysis of Boron Carbide

98% Total B_4C	
76% Boron	
Fe	0.3%
B_2O_3	0.2 - 0.4%
Al	Trace
Si	Trace
Mg	Trace
C	1.3%

TABLE II
Mix Formula for Vibratory-Compacted B_4C Powder Samples

<u>Mesh</u>	<u>Wt. %</u>
10- 20	45
30- 30	10
200-325	21
-325	24

Moisture content was determined on a portion of the boron carbide used in the capsules. Weight loss was measured by two methods. One large sample weighing 24.4134 grams was heated to 500°C in a vacuum of 3×10^{-5} mm Hg for three hours. The weight loss of the sample was 0.03%. Thermogravimetric analysis was made on a second sample weighing 0.3048 gram. After heating to 520°C and holding for seven hours in a vacuum of 10^{-3} mm Hg, the sample lost 0.0001 gm (0.03%).

Density of the particles in the 10-20 and 30-60 mesh ranges was measured by the sink-float method and found to be 95% of theoretical on the average. The 5% difference from theoretical was accounted for by metallographic and electron microprobe examinations. These revealed respectively porosity in the particles and the presence of a second phase identified as graphite.

The tantalum samples were in the form of 0.25 in. dia. x 3 in. long rods. Several tantalum protrusions were welded on the circumference to keep the sample centered in the capsule and maintain gas gap required for heat transfer. The tantalum was 99.9% pure, and a chemical analysis is given in Table III. Hardness of the tantalum was determined using Rockwell "F" scale. The average value of unannealed samples was 72.5. After annealing at 1600°C in a vacuum for one-half hour, the average hardness was measured to be 74.5. The tantalum samples used in the capsules were not annealed. Densitometer measurements of the tantalum gave a density of 16.67 gm/cc.

Type 304 stainless steel was used exclusively for all capsule components.

TABLE III
Chemical Analysis of Tantalum

C	38 ppm
O	22 "
N	21 "
H	<5 "
W	150 "
Nb	240 "
Zr	<10 "
Mo	<10 "
Ti	<10 "
Fe	20 "
Ni	<10 "
Si	<10 "
Mn	<10 "
Ca	<10 "
Al	<10 "
Cu	<10 "
Sn	<10 "
Cr	<10 "
V	<10 "
Co	<10 "
Mg	<10 "

EXPERIMENTAL

The irradiation was conducted in the EBR-II utilizing a Mark B-7 experimental subassembly. This subassembly consists of seven non-instrumented 304 stainless steel capsules, each with dimensions of 0.806 in. dia. x 0.035 in. wall x 58 in. long. All samples were doubly encapsulated within these outer pins. Four of the capsules contained boron carbide and three contained tantalum samples. The subassembly, designated X-042, was irradiated in the Row 7 position 7D5. After six months in the reactor, two B_4C capsules and two tantalum capsules were removed from the subassembly and disassembled. They were replaced in the subassembly by similar capsules, and the irradiation is continuing.

The boron carbide samples were in the form of six-inch long columns of powder, vibrationally compacted to 80% TD in 1/2 in. dia. 304 stainless steel tubes. Two thicknesses of cladding were used to provide a comparison of restraint on the radial expansion of the B_4C powder. Capsules BC-1 and BC-2 utilized 0.065 in. thick cladding wall, while BC-4 and BC-5 used 0.035 in. cladding. A gas plenum of approximately 100 cc above the B_4C samples was included in the inner capsule to contain the helium gas released from the B_4C during irradiation. The powder sample was held in place by a spring-loaded graphite plug. Because the spring constant was low, however, any radiation-induced axial growth of the sample would be essentially unrestrained. Flux and melt wires were contained in the bottom plug of the inner capsule. The melting temperatures of the melt wires were 1071, 1148, 1310, and 1434°F. Flux wires were Fe and Ni. A spacer tube below the inner capsule positioned the sample so that it was centered in the reactor core within the outer .806 in. dia. capsule. Gas atmosphere in both inner and outer capsules was helium. Figure 1 is a photograph showing a boron carbide capsule and its components.

The tantalum capsules, TA-1, TA-2, and TA-3, each contained a single sample 0.25 in. dia. x 3 in. long. A drawing of the tantalum capsule is shown in Figure 2. Again the samples were doubly encapsulated, the inner 304 stainless steel capsule being 3/8 in. OD x 0.035 in. wall. In order to

keep the sample centered in the tube and maintain the gap required for heat transfer, tantalum protrusions were welded on the surface at three places equally spaced on each end. Melt wires were contained in a 304 stainless steel holder located directly below the sample. Melting temperatures of the four wires were 898, 1148, 1310, and 1434°F. Gas atmosphere in inner and outer capsules was helium.

The design of the capsule configuration required to achieve the desired operating temperatures was accomplished with the aid of a computer heat transfer program.⁽²⁾ Heating values used in the calculations were 0.8 watt/gram for gamma heating, 6 watts/gram from the $B^{10}(n,\alpha)Li^7$ reactions, and 0.7 watt/gram for neutron absorption in the tantalum. Post-irradiation examination of melt wires indicated that these were realistic values.

Burnup of the B^{10} in the B_4C was determined from the B^{10}/B^{11} ratio found by mass spectrometry measurements on the irradiated samples. Integrated neutron exposure on the tantalum samples was determined from analysis of Fe and Ni dosimeter wires.

The unirradiated length of the boron carbide column in each capsule was determined by x-ray radiography of the assembled capsules. The length of the column after irradiation was found by neutron radiography of the capsules. This was performed by Argonne National Laboratory at the TREAT reactor facility at Idaho Falls. After disassembly the length was also physically measured by inserting a rod in the opened capsule until it contacted the top of the B_4C column.

Sieve analysis of the B_4C powder was accomplished by brushing the material through different meshes of screen. Using a brush to filter the material through the screens yielded better reproducibility than did using a shaker to vibrate the powder through the screens. The results of the sieve analysis on the unirradiated B_4C agreed well with the formula used in mixing the powder samples.

The cladding diameters of the B_4C capsules were measured by two separate techniques. Micrometer measurements were made along the cladding

length at one-inch intervals and at three orientations 120° apart. Profilometer measurements were also taken on each capsule at three orientations. All the measurements were indexed so they could be reproduced in the same locations after irradiation.

The amount of gas released in each B_4C capsule was found by puncturing the capsule and allowing the gas to escape into an evacuated, calibrated system and measuring the resultant pressure. Internal void volume was determined similarly. After puncturing, the capsule was backfilled with air to atmospheric pressure, and again the gas was permitted to escape into the calibrated system where the pressure was measured.

Removal of the boron carbide samples from their cladding after irradiation proved to be quite difficult. The material was fixed solidly in the capsules, and attempts to pick it out in the hot cells were not very successful. The best method for removing the material was found to be the use of a sharp-pointed vibratool.

Length measurements on the tantalum samples were made with a micrometer. The ends of the specimens were rounded to facilitate this operation. Densities were determined by immersion weighing the samples in a bath of distilled water with a wetting agent added. An unirradiated specimen was measured as a standard when the irradiated samples were measured to insure that the operation was performed correctly.

IRRADIATION RESULTS

The burnup of B^{10} in the boron carbide sample was found by mass spectrometry to be 1.58 atom%. This was the average value of ten samplings. Standard deviation of the ten measurements was 0.22 atom%.

Visual examination of the melt wires during disassembly showed that only the 1071°F wire in both B_4C capsules had melted. The next highest melting temperature of a wire was 1148°F, indicating an irradiation temperature somewhere between these values. Because BC-4 had a thinner cladding wall and consequently more B_4C , it is expected to have operated ~ 30-40°F higher than BC-1. In any case, both samples apparently had irradiation temperatures near 1100°F.

After removal of the subassembly from the reactor, all four boron carbide capsules were neutron-radiographed to determine whether any axial growth of the vibratory-compacted B_4C may have occurred. In addition, the B_4C column heights of capsules BC-1 and BC-4 were determined by measuring how deep a rod could be inserted from the top of the opened capsule. The results of these measurements are given in Table IV. The accuracy of the neutron radiograph measurements is estimated to be $\pm 1/64$ in. ($\pm 0.26\%$), while that of the rod measurements is considered to be $\pm 1/32$ in. ($\pm 0.5\%$).

TABLE IV			
Axial Growth of B_4C Powders Columns			
Capsule	Clad Thickness	Neutron Radiograph	Rod Measurement
BC-1	0.065 in.	0.26%	0.26%
BC-2	0.065 in.	0.51%	
BC-4	0.035 in.	1.03%	0.51%
BC-5	0.035 in.	0.26%	

Consequently the apparent disagreement of the growth values for BC-4 can be at least partly resolved by the uncertainties in the measurements. In any event the length changes are small, and could be easily attributable to loosening of the powder during handling and shipping rather than to radiation-induced swelling.

Gas pressure measurements after the $1100^\circ F$ irradiation showed that 27.8% of the helium gas was released from the boron carbide in BC-1, while 29.7% was released in BC-4. These values were determined by calculating the total quantity of helium generated at 1.58% burnup of the B^{10} . The chemical composition of the gases collected from capsules, as determined from mass-spectroscopic analyses, is shown in Table V. Gases other than helium that were detected are natural air gases that apparently found their way into the sampling either during capsule assembly or when the gas was collected from the capsule.

TABLE V
Chemical Composition of Released Gases in B₄C Capsules

	<u>BC-1</u>		<u>BC-4</u>	
He	96.4	Mol%	99.9	Mol%
N ₂	2.8	"	0.10	"
O ₂	0.74	"	0.02	"
Ar	0.037	"	0.001	"
CO ₂	0.017	"	0.009	"

The cladding diameter of each capsule was measured with a micrometer at one-inch intervals along the length and at three indexed orientations 120° apart, just as before irradiation. The results are given in Table VI. The measurements should be accurate to at least 0.001 inch. The largest differences between pre - and post - measurements occur near the end plug (#6 position). Because the welding of the end plug to the capsule likely caused some deformation to the tubing in that area, measurements would be hard to reproduce and the dimensional changes indicated are probably not realistic. If these end measurements are neglected and the remainder averaged, then the micrometer measurements indicate the cladding diameter of BC-1 did not change, while that of BC-4 contracted 0.16%. This dimensional change is within the experimental error of the measurement, so cladding deformation can be said to be negligible. Certainly no shrinkage of the 304 stainless steel cladding would be expected from the irradiation. Profilometer measurements supported the lack of cladding distortion. The profiles of the capsules after irradiation were similar to those measured prior to irradiation. Using the average values of cladding diameter indicated from the profilometer measurements, comparison of pre - and post-irradiation measurements indicates a 0.02% cladding contraction for BC-1, and no change for BC-4. Again the 0.02% value is well within experimental error.

When the capsules were opened to remove the B₄C powder, the material had a sintered appearance. After removing the boron carbide from the cladding, it was noted that some of the particles were indeed adhered to one another. However, when these particle clumps were disturbed, they broke up easily. A sieve analysis was performed on the material after

TABLE VI
Pre-Irradiation and Post-Irradiation Cladding Diameters of B₄C Capsules

	<u>Axis</u> [*]	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
BC-1 Pre-Irradiation	1	0.4977	0.4978	0.4979	0.4978	0.4980	0.4976
	2	0.4978	0.4978	0.4977	0.4976	0.4975	0.4970
	3	0.4978	0.4977	0.4977	0.4978	0.4977	0.4976
BC-1 Post-Irradiation	1	0.4980	0.4980	0.4979	0.4976	0.4979	0.4989
	2	0.4973	0.4976	0.4979	0.4978	0.4970	0.4970
	3	0.4979	0.4979	0.4980	0.4979	0.4979	0.4989
BC-4 Pre-Irradiation	1	0.4993	0.4994	0.4998	0.4997	0.4997	0.4994
	2	0.4998	0.4993	0.4992	0.4995	0.4994	0.4996
	3	0.5004	0.4999	0.4991	0.4993	0.4996	0.4998
BC-4 Post-Irradiation	1	0.4990	0.4989	0.4989	0.4989	0.4989	0.4979
	2	0.4990	0.4980	0.4979	0.4989	0.4989	0.4999
	3	0.4989	0.4989	0.4987	0.4987	0.4988	0.4998

^{*}Axes are 120° apart

irradiation, and the results are shown on Table VII. Rather than sintering, it actually appears that generally the particle sizes decreased. Whereas 43% of the particles were in the 10-20 mesh size before irradiation, only 25% of the powder fell in this range after irradiation. The corresponding increase in the finer particle fraction can be seen in the table. While only 24% of the unirradiated material was -325 mesh, 33% of the irradiated B₄C was comprised of these fine particles.

TABLE VII
Sieve Analysis of B₄C

<u>Screen Mesh</u>	<u>Unirradiated</u>	<u>Irradiated</u>
10	3.5%	2.4%
20	43.0	25.3
28	0.4	4.4
60	10.7	15.2
200	0.6	9.0
325	17.9	10.9
-325	23.9	32.8
	100.0	100.0

A metallographic examination was performed on a cladding section from each capsule. It appeared that there was some interaction between the boron carbide and the cladding in BC-4, but it was not severe. No evidence of interaction could be detected in BC-1. It is possible that the slightly higher irradiation temperature of BC-4 ($\sim 40^\circ\text{F}$) could have promoted some interaction which did not occur at the BC-1 temperature.

Microhardness tests were taken across the walls of the cladding to help determine whether any interactions occurred. The results are shown in Table VIII. Measurements were taken from the inside of the cladding to the outside at approximately equal spacings with a 100 gm load. There is considerable scatter in the test results, with no firm indication of any interaction. The low value for the first position of BC-1 is likely due to offset of the indentation into the ID of the cladding.

TABLE VIII
Microhardness (Knoop) of B_4C Cladding

Position	BC-1	BC-4
1	111.4	211.6
2	290.4	206.5
3	216.9	201.7
4	240.0	175.7
5	282.3	211.6
6	259.8	259.8
7	139.5	
8	259.8	
9	253.0	

Examination of the melt wires in the tantalum capsules TA-1 and TA-2 disclosed that only the low temperature (898°F) wire melted in each capsule. The next highest temperature wire was 1148°F , which did not melt. These results indicate that the 304 stainless steel melt wire holder experienced a temperature of $\sim 1100^\circ\text{F}$, and imply that the tantalum samples operated at $\sim 1200^\circ\text{F}$. These results agree reasonably well with the heat transfer calculations. Integrated neutron exposure on the tantalum as determined from Fe and Ni dosimeter wire analysis was 6.8×10^{21} ($E > 0.1 \text{ MeV}$).

Length change of each tantalum sample was + 0.12%. Density measurements by the immersion technique yielded densities of 16.632 g/cc and 16.633 g/cc for the TA-1 and TA-2 samples, respectively. A simultaneous measurement of an unirradiated piece showed a density of 16.727 g/cc, indicating decreases in density of 0.57% and 0.56% for the irradiated samples. These values do not agree with a density change of -0.36% inferred from the dimensional growth measurements. Also, the tantalum densities indicated by the densitometer measurements are higher than the value of 16.6 quoted in the handbooks. Nonetheless, the densitometer measurements of the irradiated and unirradiated samples were performed identically and at the same time, and the relative change in density indicated should be accurate.

CONCLUSIONS

Boron carbide in the form of vibratory-compacted powder was irradiated in a fast neutron spectrum (EBR-II) at $\sim 1100^\circ\text{F}$ to 1.58 atom% burnup of the B^{10} . Two thicknesses of 1/2 in. dia. 304 stainless steel cladding were employed to contain the B_4C : 0.035 in. and 0.065 in. The irradiation caused no detectable deformation of either cladding. Some small unrestrained axial growth was noted in the B_4C poison columns ($\sim 1/2\%$), but it is possible this was a result of loosening the powder compact during handling and shipping before radiation.

Nearly 30% of the helium gas formed from the $\text{B}^{10}(\text{n},\alpha)\text{Li}^7$ reaction was released during irradiation. This release fraction is considerably higher than has been observed in thermal flux irradiations at lower temperatures. Valovage⁽⁴⁾ found that B_4C powder (72% TD) irradiated at 270°F to 5% B^{10} burnup released only $\sim 6\%$ of the helium gas formed during irradiation, and released an additional 3% of the gas when subsequently heated to 900°F . Pellets of 96% TD released $\sim 5\%$ of the gas during irradiation at 500°F to 36% burnup, and released $\sim 13\%$ more of the gas when heated to 1500°F . Hoyt and Zimmerman⁽⁵⁾ irradiated a 70% TD B_4C powder sample to 85% burnup and at 750°F and observed 18% gas release. A 100% TD pellet irradiated to 37% burnup at 480°F released 11% of the gas during irradiation, and a total of 30% cumulative gas release was seen after annealing at

1025°F. The latter gas release fraction agrees well with that seen in the present study at approximately the same temperature, but the burnup was much higher. It is believed that the high fraction of gas release witnessed in the current irradiation test is due primarily to two factors: the relatively higher temperatures ($\sim 1100^\circ\text{F}$) which would expedite the diffusion of helium through the material, and the high percentage of fine mesh (-325) particles in the sample mix. The latter feature would reduce the path length required for helium atoms to diffuse out of the particles, and probably also contribute to the amount of helium released directly from the recoil of α - particles in the $\text{B}^{10}(\text{n},\alpha)\text{Li}^7$ reaction. It is also possible that lattice dislocations by fast neutrons play a role in the helium release mechanism, an effect that would not be as prominent in thermal reactor irradiations.

Metallographic examination of the 304 stainless steel claddings suggested that some interaction with the B_4C may have occurred in BC-4, the thin-walled capsule. This capsule probably experienced temperatures about 40°F higher than BC-1 which did not show any interaction. Other compatibility studies⁽⁶⁾ between B_4C and stainless steels imply that the temperature threshold for interaction with 304 stainless steel may be in the neighborhood of 1100°F , and the slightly higher temperature of BC-4 could have been enough to promote some interaction.

Tantalum samples irradiated in the EBR-II at $\sim 1200^\circ\text{F}$ to an exposure of 6.8×10^{21} nvt ($E > 0.1$ MeV) showed length changes of +0.12% and density decreases of 0.56%. This is considered to be a very high swelling rate. Discharge of an additional tantalum capsule scheduled for autumn 1969 will provide data at about the same exposure level, but at temperatures from 1100 to 1600°F . After approximately 2-1/2 years of further irradiation, the subassembly will be discharged from the reactor with maximum tantalum exposures of $\sim 5 \times 10^{22}$ nvt and B^{10} burnups of 10%.

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Figure 1.

FIGURE 2
EBR II Tantalum Capsule