IRRADIATION BEHAVIOR OF BORON CARBIDE*

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ABSTRACT

Boron carbide is the prime candidate material for neutron absorbers in fast breeder reactors and the Fast Test Reactor. Important data required for design of control rods for these reactors are swelling, gas release, and structural integrity of boron carbide under the expected operating conditions. This paper presents data from irradiations of boron carbide powders in a thermal reactor and powders and pellets in a fast flux reactor. These data are interpreted and discussed in terms of expected performance in a fast reactor. The most important variable in determining irradiation behavior appears to be the mobility of the helium produced relative to the rate it is produced. This precludes extrapolation of data obtained in thermal reactors to predict performance in fast reactors.

We conclude that the only accurate way to predict the performance of boron carbide pellets in a fast reactor is to conduct irradiation tests for the same temperatures, times, and burnup levels expected in service.

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INTRODUCTION

Boron carbide is the prime candidate material for neutron absorbers in liquid metal cooled fast breeder reactors and the Fast Test Reactor. Although the material has been used extensively in thermal reactors, only limited data are available on the performance of boron carbide under fast reactor conditions. The capture of a neutron by ^{10}B results in the formation of ^7Li plus a helium nucleus. Once the compatibility of the control rod materials has been established, the prime requirements for design are the amount of swelling and the amount of helium release to be expected. We are currently conducting tests in the Experimental Breeder Reactor-II (EBR-II) to obtain values for these design parameters for several material and testing variables.

This paper presents some results from (1) irradiation tests of boron carbide powders in the Oak Ridge Research Reactor (ORR) which has a primarily thermal spectrum, (2) evaluation by transmission electron microscopy and x-ray diffraction of powders irradiated in EBR-II by Hanford Engineering Development Laboratory (HEDL), and (3) evaluation of boron carbide pins from the EBR-II Higher Worth Control Rod. We have interpreted these data and have related these results to expected performance under fast reactor operating conditions.

LATTICE PARAMETER CHANGES

The unit cell of boron carbide is rhombohedral with icosahedral units on the corners and a three-atom chain along the diagonal. The structure

as first determined (1,2) was assumed to be composed of boron atoms in the icosahedra and carbon atoms in the chain with a composition of $B_{12}C_3$ based on the unit cell. More recent x-ray work (3,4) and nuclear magnetic resonance (NMR) work (5,6) has shown that the center atom in the chain is often or always a boron atom. Carbon atoms are assumed to occupy sites on the icosahedra, leading to a unit cell composition designated $(B_{11}C)CBC$. Hoard and Hughes (3) present evidence to show that the only completely ordered compound is $(B_{12})CBC$ (or $B_{13}C_2$). The wide range of compositions possible may be a result of substitution of boron and carbon atoms for each other or as interstitials. The structure of boron carbide may then be pictured as in Fig. 1. The central atom in the chain is often if not always a boron atom and carbon substitutes for boron in some of the rhombohedral sites on the icosahedra. Recent results at our laboratory (7) on the lattice parameters as a function of chemical composition indicate that the boron carbide structure exists as a single phase at $2000^\circ C$ from less than 4.8 at. % C to about 20 at. % C. The behavior of the lattice parameters suggests that for boron content in excess of the composition $B_{13}C_2$ the boron atoms may occupy interstitial sites.

The lattice parameters for boron carbide in equilibrium with graphite are about $a = 5.162 \text{ \AA}$ and $\alpha = 65.69^\circ$. The crystal structure is often referred to the hexagonal system in which the three-atom chain is parallel with the c axis and the parameters are about $a = 5.601 \text{ \AA}$ and $c = 12.07 \text{ \AA}$. The effect of irradiation on these parameters is discussed below. All of these parameters were determined using Cr-K α (V-filtered) x radiation which gives good precision in the back-reflection region.

Thermal Spectrum Tests

We have irradiated boron carbide powders at about 350°C in the ORR which has a primarily thermal flux spectrum for periods of three and six weeks. The lattice parameter changes after three weeks irradiation (about 8% ^{10}B burnup or 16×10^{20} n, α/cm^3) are presented in Table I.

The unit cell showed a volume increase in both powders through an increase in the rhombohedral edge, a , and a decrease in the angle α in one case and an increase in α for the other. Powders irradiated six weeks showed too much disruption of the crystal structure for lattice parameter measurements. Single crystals (mostly twinned) irradiated in the ORR and in the target region of the High Flux Isotope Reactor (HFIR) have yielded x-ray patterns which are a composite of sharp Bragg maxima (unbroadened relative to the unirradiated crystal) and diffuse reflections located at or near the sharp maxima (8). The unit cell volume calculated from the sharp peaks shows an increase, whereas the unit cell volume calculated from the diffuse peaks shows a decrease. These results are included in Table I. Yakel suggests that the crystals may be a composite of regions containing different types of defects. Thus, a polycrystalline powder sample would yield Debye-Scherrer patterns which were some combined average of the two types of effects.

Fast Spectrum Tests

We have examined both powders and pellets irradiated in the EBR-II as irradiated and after annealing at various temperatures from 400 to 1900°C.

We examined powders from capsules BC-2 and BC-8 irradiated by HEDL in EBR-II experiment X042 (refs. 9 and 10). Capsules BC-2 and BC-8 were irradiated at 650 and 730°C to burnups of 10 and 7.7×10^{20} n, α /cm³, respectively. The lattice parameters of BC-2 as irradiated and after annealing 1 hr at various temperatures are shown in Figs. 2 and 3. The unit cell volume has decreased as irradiated by a shrinkage of the edge a with little change in the angle α . During annealing at progressively higher temperatures, a first increases to above the original value, then decreases to slightly below it. The angle α shows a very sharp decrease during annealing at the lower temperatures, then increases sharply to about the original value. This different behavior at the different temperatures may be due to the predominance of separate types of defects annealing out at the high and low temperatures.

Boron carbide pellets irradiated in EBR-II as part of the Higher Worth Control Rod, assembly L-4008S, showed unit cell volume increases through an increase in a and little change in α . During annealing, the parameters showed a behavior similar to the X042 powders in that a first increased, then decreased, and the angle α first decreased and then increased sharply. As shown in Fig. 4, the unit cell volumes for both the powders and pellets changed uniformly with annealing temperature and returned to their original value at about 1250°C. The magnitude and direction of the unit cell volume change appear to depend primarily on temperature. The rather limited unit cell volume change data we have

generated is shown as a function of irradiation temperature in Fig. 5. More data are needed to test this effect of temperature. At the lower temperatures, the reaction products may tend to remain as interstitials or very small localized defects. At higher temperatures, the agglomeration of the reaction products may exert a compressive strain on the lattice.

MICROSTRUCTURAL CHANGES

Optical Metallography

The powders irradiated in the ORR were examined before and after irradiation by conventional metallography and scanning electron microscopy. No changes were visible after irradiation. Examination of the pellets from the EBR-II Higher Worth Control Rod revealed a couple of interesting changes due to irradiation. The pellets in general appeared to be in very good condition. A few small cracks were observed. These, in general, were along grain boundaries and linked graphite particles or other inclusions as shown in Fig. 6. The microstructure of the unirradiated control sample is typical of hot-pressed boron carbide showing twinning, intergranular and intragranular porosity, some metallic appearing inclusions, and some free graphite. We were not able to electrolytically etch the as-irradiated samples because they would not conduct electricity. However, annealing at 1150°C for 1 hr restored electrical conductivity sufficiently to etch the samples. One hour at 1600°C restored the electrical conductivity to about the unirradiated value. The anneals did not affect the as-polished appearance. The etched microstructure in Figs. 7 and 8 shows

an apparent lack of twins. Twins are observed in transmission electron microscopy after irradiation and after annealing as discussed below.

The voids on the grain boundaries in Fig. 8 are an etching effect. They were not observed when the sample was repolished and more lightly etched.

Transmission Electron Microscopy

We have examined irradiated powders and a slice of a high-density pellet from the ORR (11), powders from the X042 experiment (10), and pellets from the EBR-II Higher Worth Control Rod by transmission electron microscopy. The powders were dispersed in aluminum by vacuum hot pressing so that they could be thinned by ion bombardment and examined. The pellets were sliced with a diamond saw, ground, and then thinned by ion bombardment.

The powders and a slice from a high-density pellet irradiated three weeks in the ORR at 350 to 400°C to 8% ^{10}B burnup show a high density of black-spot damage similar to that observed in metals after irradiation to high fast fluences at low temperature. The black spots, indicative of lattice strain, were uniformly distributed throughout the samples. No change in damage density was observable near grain boundaries, twin boundaries, intragranular voids, or particle edges. A grain boundary is shown in Fig. 9 and a particle edge is shown in Fig. 10. The defects were not resolvable at magnifications up to 140,000x.

Particles irradiated in the EBR-II capsules BC-2 and BC-8 to burnups of 4.8 and 3.7% ^{10}B at temperatures of 650 and 730°C showed discrete defects

accompanied by complex strain fields. These are shown in Figs. 11 and 12. The powders in Fig. 12 have been annealed at 1000°C for 1 hr and show a denuded zone adjacent to the grain boundary. The appearance of the defects was unchanged by the anneal. The same damage was observed in pellets from the EBR-II Higher Worth Control Rod. As shown in Fig. 13, the defects are much smaller than those in the higher burnup, higher temperature powders from BC-2 and BC-8. These defects have been identified as platelike or ribbonlike cavities filled with gas at a pressure in excess of the surface tension (12). The largest face of these cavities is most frequently parallel to the trace of the (111) plane and less frequently parallel to {100} and {110} traces, indexed on the rhombohedral unit cell. There is a denuded region near grain boundaries, particle edges, and the edges of intragranular voids.

Annealing at or above 1450°C eliminated the lattice strains around the cavities. The lattice parameters also returned to essentially their original value during this anneal. The cavities increased in size and decreased in number density during the anneal. The crystallographically faceted bubbles resulting from annealing are shown in Fig. 14. The largest faces of the slab-shaped bubbles are parallel to (111), {110}, and {100} planes, as was the case for the as-irradiated cavities. At higher temperatures, the bubbles tended to become more equiaxed, as shown in Fig. 15. The denuded zone near grain boundaries increased in width during annealing. Bubbles were very seldom observed on grain boundaries, indicating that once the helium diffuses to the grain boundaries it leaves the sample relatively quickly. The apparent volume increase due to the growth of the bubbles during annealing is about 2.4% for a pellet sample of 1.6% ^{10}B burnup.

SWELLING

We presently have only limited data on the swelling of boron carbide during irradiation in fast reactors. Pellets in pins G and H of the EBR-II Higher Worth Control Rod swelled 0.28 and 0.38%, respectively, at an average burnup of 1.5% of ^{10}B . This is in reasonable agreement with swelling results from thermal reactor experiments (13). However, neutron radiographs from our EBR-II experiment X099, now being evaluated, indicate that the swelling is considerably higher at higher temperatures.

GAS RELEASE

In our short-time (three to six weeks) thermal irradiation tests of boron carbide powders, gas release averaged about 5.5% for burnup up to 10% at temperatures of 200 to 500°C. Temperature within this range had very little effect. Particle size, the presence of intraparticle voids, and boron-to-carbon ratios in the range 3.6 to 4.2 also had no appreciable effect on gas release. The presence of elemental boron in boron carbide powders significantly increased gas release. Powders prepared with elemental boron contents of 0.61, 2.2, and 11.6 wt % and irradiated for three weeks under similar conditions had gas releases of 7.6, 11.6, and 29.9%, respectively.

Under fast reactor conditions, temperature appears to have a very pronounced effect on gas release. For example, HEDL measured gas releases of 17 and 48% for X042 capsules BC-2 and BC-8 irradiated at 650 and 730°C, respectively (14). Our retained helium analyses by vacuum fusion of

powders from these capsules indicated gas releases of 20 ± 4 and $42 \pm 3\%$, respectively, which is in reasonable agreement with the HEDL values. Our plenum gas measurements for pin F from the EBR-II Higher Worth Control Rod indicated only 1.6% gas release for these high-density pellets irradiated at about 450°C. Preliminary results of vacuum fusion for retained helium indicate that $8 \pm 4\%$ of the gas generated was released from the Higher Worth Control Rod pellets.

STRUCTURAL INTEGRITY

We observed no fracturing of particles during our low-temperature thermal reactor irradiations of boron carbide powders. Fracturing of bulk samples during thermal irradiations does occur due to the extreme burnup gradients (13, 15).

Under fast reactor conditions, fracturing appears to be influenced greatly by irradiation temperature. No cracking was observable under 10X visual examination of pellets from the EBR-II Higher Worth Control Rod and only very minor cracking was observed metallographically. In the X042 experiment (650 to 730°C), particle fracturing was evidenced by a decrease in the average particle size in the rods (9). We are presently examining capsules from our EBR-II experiment X099. These capsules achieved about 3% burnup during 155 full-power days (at 62.5 MW) at design temperatures of 430 and 700°C. Neutron radiography of these capsules reveals extensive fragmentation of the high-temperature high-density pellets with much less cracking in the low-temperature pellets.

Scanning electron micrographs of fractured surfaces from the Higher Worth Control Rod pellets are shown in Fig. 16. The as-irradiated pellets fractured transgranularly as shown by the presence of the polyhedral voids which lie in the centers of the grains. After annealing at 1150°C for 1 hr, the pellets fractured intergranularly.

DISCUSSION OF RESULTS

These results indicate that the relative mobility of the helium produced will have a profound influence on the behavior of boron carbide pellets during irradiation. Thus, irradiation temperature is probably the single most important operating parameter. We discuss the expected performance of boron carbide pellets in a fast reactor in terms of operating temperature and several materials variables which may be expected to significantly affect the performance.

Operating Variables

Based on these rather limited irradiation tests and annealing results, we feel that the irradiation performance may be discussed in terms of three distinct temperature ranges, the limits of which are not defined accurately at the present time.

1. $T < T_1$ (about 500°C)

- (1) No bubbles form that reach about 50 Å in size,
- (2) Platelike defects containing helium will be formed, but their size will remain small,
- (3) Lattice parameters show a volume increase in the unit cell,

- (4) Gas release will be relatively low, at least to some undetermined burnup level,
- (5) No fragmentation will occur below some undetermined burnup level.

2. $T_1 < T < T_2$ (about 1000°C)

- (1) Helium bubbles will form and produce high internal stresses within the grains,
- (2) Lattice parameters will show a unit cell volume decrease,
- (3) Gas release will be higher due to a widening denuded zone adjacent to the grain boundaries in which most of the helium generated will diffuse to the grain boundaries and out of the pellet,
- (4) At some burnup level, cracking and pellet fragmentation will occur due to high internal stresses resulting in spurts of gas release and an increase in effective surface area for diffusion of helium from the pellet.

3. $T_2 < T$

- (1) Large bubbles will form, leading to increased bulk swelling,
- (2) Gas release will increase due to widening denuded zones,
- (3) Cracking may or may not be a problem, depending on whether self-diffusion in the boron carbide is fast enough to prevent high stress buildup around bubbles,
- (4) Lattice parameters will show little or no change in unit cell volume.

4. Effect of Thermal Transients

The effect of a thermal transient in a pin operating at relatively low temperature may be an initial burst of gas release. The transient could initiate bubble formation which would increase bulk swelling but would decrease the post-transient gas release rate since the bubbles could act as traps for helium. However, if the transient is severe enough to result in cracking, the post-transient gas release rate may be increased due to the additional surface area.

Materials Variables

The presence of elemental boron is expected to increase the gas release in fast reactors as it did in thermal reactor tests. Generally though, hot-pressed boron carbide of near-B₄C composition will not contain any elemental boron. The pellet grain size will probably influence gas release due to the increase in relative volume adjacent to grain boundaries as grain size decreases. Decreased grain size will tend to increase gas release but may decrease bulk swelling in the temperature regions where bubbles are formed. Intragranular voids and inclusions may tend to act as traps for gas and lower the gas release in the intermediate temperature range.

Another factor which may influence observed behavior is ¹⁰B enrichment. Increasing the ¹⁰B enrichment to obtain burnup in a shorter time effectively lowers the diffusion rate of helium relative to the rate it is produced. This change in relative helium diffusion rate may be expected

to influence gas release, bulk swelling, and the tendency to form cracks.

Boron-to-carbon ratio may not be expected to have a pronounced effect on irradiation performance since the same crystal structure is maintained over a wide range of stoichiometry. However, the larger unit cell as boron content is increased may either provide more space for reaction products as interstitials or, alternatively, the more open lattice may speed up diffusion of helium from the material. The excess boron may also help stabilize the crystal structure as boron atoms are depleted during irradiation. In compositions where excess carbon is present, the free graphite particles could induce cracking due to the irradiation-induced dimensional changes in the graphite.

CONCLUSIONS

The longer times for equivalent burnup at generally higher temperatures in a fast reactor as compared to a thermal reactor precludes the extrapolation of thermal spectrum data to predict performance of boron carbide in a fast reactor. The results presented here show that the relative diffusion of the helium produced during irradiation is probably the most important variable controlling the performance of boron carbide. Thus, we feel the only way to accurately predict the performance of boron carbide pellets in a fast reactor is to conduct irradiation tests for the same temperatures, times, and burnup levels expected in service. If severe thermal transients are expected in service, the effects of these on gas release, solid swelling, and pellet fragmentation need to be investigated experimentally.

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Table I. Effects of Irradiation on Lattice Parameters
of Boron Carbide Powders and Single Crystals

	Parameters				
	Hexagonal			Rhombohedral	
	$\frac{a}{\text{\AA}}$	$\frac{c}{\text{\AA}}$	Volume ($\text{\AA}^3$)	α ($^\circ$)	$\frac{a}{\text{\AA}}$
Norbide 325F Powders					
As-received	5.601	12.076	328.07	65.66	5.163
Irradiated three weeks, 350°C, in ORR (0.56% $\Delta V/V$)	5.620	12.062	329.92	65.90	5.167
Irradiated three weeks, 350°C, in ORR (0.42% $\Delta V/V$)	5.596	12.14	329.22	65.41	5.178
MORTON M103 Single Crystals					
As-received ^a	5.625 (0.007)	12.15 (0.02)	333 (1)	65.61 (0.05)	5.192 (0.005)
Irradiated three weeks, ~ 400°C, in ORR					
Sharp	5.660	12.067	334.7	66.20	5.182
Diffuse	5.645	11.973	331.5	66.50	5.156
Irradiated 1 hr, 55°C, in HFIR					
Sharp	5.6408	12.140	334.5	65.77	5.194
Diffuse	5.6285	12.103	332.0	65.81	5.180

^aUnweighted means of 15 crystals. Numbers in parentheses below the parameters are unweighted average deviations from the means.

LIST OF FIGURES

Fig. 1. Crystal Structure of Boron Carbide, Showing the Rhombohedral Cell and Relation to Icosahedral Structure. On the icosahedra, light circles designate rhombohedral sites and dark circles designate equatorial sites. The two large light circles designate carbon atoms at the ends of the three-atom chain, and the large dark circle designates the atom at the center of the chain (often or always a boron atom). Reprinted from ref. 6.

Fig. 2. Rhombohedral a Parameter for BC-2 Powders As-Irradiated and After Annealing 1 hr at Temperature.

Fig. 3. Rhombohedral Angle α for BC-2 Powders As-Irradiated and After Annealing 1 hr at Temperature.

Fig. 4. Unit Cell Volume Change for Fast-Reactor-Irradiated Powders and Pellets and After Annealing 1 hr at Temperature.

Fig. 5. Unit Cell Volume Change Versus Irradiation Temperature.

Fig. 6. Microstructure of EBR-II Higher Worth Control Rod Pellet H-13 As-Irradiated. As-polished.

Fig. 7. Microstructure of EBR-II Higher Worth Control Rod Pellet H-13 Annealed 1 hr at 1150°C After Irradiation; Electrolytically Etched.

Fig. 8. Microstructure of EBR-II Higher Worth Control Rod Pellet H-13 Annealed 1 hr at 1600°C After Irradiation; Electrolytically Etched.

Fig. 9. Transmission Electron Micrograph Showing Grain Boundary in Boron Carbide Irradiated at 400°C in the ORR.

Fig. 10. Transmission Electron Micrograph of Edge of Boron Carbide Particle Irradiated at 350°C in the ORR.

Fig. 11. Transmission Electron Micrograph of BC-2 Powders As-Irradiated in EBR-II. 80,000x.

Fig. 12. Transmission Electron Micrograph of Grain Boundary in BC-8 Powders As-Irradiated and Annealed at 1000°C. Defect appearance was not changed by the anneal. 40,000x.

Fig. 13. Transmission Electron Micrograph of EBR-II Higher Worth Control Rod Pellet As-Irradiated. (a) Defect clusters and grain boundary denuded regions at low magnification (20,000x). (b) Contrast to show complex strain fields (100,000x). (c) Contrast to show helium cavities (100,000x).

Fig. 14. Transmission Electron Micrograph of BC-8 Powders After Annealing 1 hr at 1450°C.

Fig. 15. Transmission Electron Micrograph of Higher Worth Control Rod Pellet After Annealing 1 hr at 2000°C.

Fig. 16. Scanning Electron Micrographs of Fracture Surfaces of EBR-II Higher Worth Control Rod Pellets. (a) As-irradiated, and (b) after postirradiation anneal for 1 hr at 1150°C.

Fig. 1

Fig. 2

Fig. 3

VOLUME CHANGE OF UNIT CELL (%)

0.6

0.4

0.2

0

-0.2

-0.4

-0.6

O - OPR POWDERS

□ - EBR-II HIGH WIDTH
CONTROL ROD

△ - X042 POWDERS

Fig 5

TEMPERATURE (°C)

○

○

□

△

△

400

500

600

700

800

72827
B4C H-13A
H5-1220.

500x

Fig. 6

72881

ANN 1450°C
550x

Fig. 7.

72884
H-13-P-2
MM 1600C 500X

Fig. 8

Fig. 9

Fig. 10

Fig. 11

Fig. 12

STRUCTURE IN IRRADIATED BORON CARBIDE (EBR-II)

(a) Defect Clusters and Grain Boundary Denuded Regions at Low Magnification (20,000X)

(b) Contrast to Show Complex Strain Fields (100,000X)

(c) Contrast to Show Helium Cavities (100,000X). Magnified structure showing strain fields associated with small helium cavities.

Fig. 13

Fig. 14

Fig. 15

Fig. 16