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IN-REACTOR MEASUREMENT OF
NEUTRON ABSORBER PERFORMANCE

MASTER

G. W. Hollenberg
J. L. Jackson
J. A. Basmajian

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To be presented at the 1979 Annual Meeting of the American Nuclear Society in Atlanta, Georgia on June 3-8, 1979.

ABSTRACT

An instrumented absorber experiment in the Experimental Breeder Reactor-II yielded data on the helium release of boron carbide at exposures up to 53×10^{20} neutron captures/cm³ and temperatures from 766 to 952°C. Helium release results demonstrated a high rate of helium release initially, and a much lower rate in a secondary region. In addition, the experiment demonstrated the effectiveness of vents in releasing gas from a plenum during irradiation.

ACKNOWLEDGMENTS

An irradiation experiment with the complexity of the BICM-2 Experiment is not designed, constructed and analyzed without talented contributions from many individuals. Of particular note was the pellet fabrication by G. L. Jones and the test assembly fabrication by M. A. Vogel, W. F. Walker, and V. N. Pearson. J. D. Berger, assisted in surveillance of the experiment at EBR-II, and W. E. Hart assisted in assembly and disassembly. A. L. Pitner and D. E. Baker contributed greatly to the postirradiation analysis presented. The guidance and support of R. E. Dahl and D. E. Mahagin through the many years of work is appreciated.

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1.0 INTRODUCTION

Neutron absorber elements in the control and safety rods of fast flux reactors are critical components that must remain functional throughout their operating lifetime. Boron carbide is presently the leading material for use as a neutron absorber material in these reactors, mainly because of its high reactivity worth in fast neutron spectra. The BICM-2 Experiment is one of a long line of experiments aimed at determining the irradiation conditions under which boron carbide's performance is adequate. This report summarizes the results of the BICM-2 Experiment.

Neutron captures in boron carbide are primarily through the $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction; consequently, helium gas is generated. Helium is virtually insoluble in boron carbide, which means that if kinetically possible it will be rejected by the boron carbide matrix, either as small internal pockets of gas within the pellet or as a contribution to the plenum gas. Both effects are key factors in absorber pin performance and design. If released to the plenum, pressure within the absorber element is correspondingly increased and the cladding is subjected to stresses. On the other hand, the retained helium precipitates (bubbles) result in pellet swelling. Swelling, if large enough, adversely affects absorber pin performance because it can also promote high cladding stresses.

The BICM-2 Experiment was an instrumented test designed to continuously measure absorber temperatures and gas pressures during irradiation. Besides yielding time dependent gas release data on high temperature boron carbide performance,

the instrumentation allowed in-reactor evaluation of vent devices which show promise for advanced absorber pin application.

2.0 EXPERIMENTAL DESCRIPTION

In Figure 1, a schematic representation of the BICM-2 subassembly is shown. The subassembly extended upward some 11 meters from the EBR-II core to above the rotary shield plug at the top of the reactor. Above the central shielding, a terminal box contained the pressure transducers and thermocouple connectors. Output from the pressure transducers and thermocouple was provided to the local Data Acquisition System (DAS) computer at EBR-II. The pressure transducers were connected to their respective capsules by long tubes that passed through the bulkhead above the core.

Coaxial with the pressure tubes were the thermocouples that monitored temperatures in the capsules. During the entire 22,700 MWd irradiation, both K and S type thermocouples appeared to function properly. The most convincing evidence of this was the fact that during periods of zero reactor power the thermocouple indications were within a few degrees of the sodium coolant temperature ($366 \pm 7^\circ\text{C}$).

Immediately below the terminal box were located two 250 cm³ vent pressurization tanks. Each tank was connected to a pressure transducer, a valve, and one of the vents located at the bottom of the core. A thermocouple in the tank measured the temperature of the gas. During the experiment, the helium passed through the vent and then to an outlet which allowed the gas to be released into the sodium coolant. As pressure in the tank decreased, the flow rate through the vent was also reduced. When pressure changes reached an insignificant level, the tanks were repressurized to a predetermined level, and a new vent cycle was initiated.

HEDL 7803-152.2

FIGURE 1. Schematic Representation of the BICM-2 Experiment in the EBR-II Core. Insert Features the Location of the Subcapsules.
(Neg. No. 7803462-2)

2.1 The Subcapsules

Figure 1 shows the location of the subcapsules in the EBR-II core. Directly below the bulkhead were two above-core pins which had boron carbide pellet columns 34 cm in length. The rupture of one of these pins during the experiment has been previously reported and is not discussed here.⁽¹⁾ Between the top and center of the EBR-II core were three tiers of boron carbide capsules with 2.5 cm long pellet columns. Beneath these were capsules containing europium oxide. The results obtained from the europium oxide capsules were reported previously.⁽²⁾ Finally, at the bottom of the core were two vent capsules. The top tier of boron carbide capsules contained 92% ^{10}B enriched boron carbide with radial heat transfer paths adjusted so that three different centerline operating temperatures were attained. The three capsules below the 92% ^{10}B capsules had boron carbide with 50% ^{10}B and the three capsules in the tier below that contained natural boron carbide (19.8% ^{10}B). The boron carbide powder used for hot pressing these pellets was quite pure, with all measured impurities being less than 0.02%. The major difference between these capsules was the different heat transfer gaps so that a range of centerline temperatures could be achieved. Cross-sectional views of an above-core pin, an in-core boron carbide capsule and the vent capsules are shown in Figure 2. The test matrix is shown in Table 1.

2.2 Irradiation

The subassembly was irradiated in an EBR-II Row 5 INCOT facility. It accumulated an irradiation exposure of 22,700 MWd (approximately 363 operating days). During that time, there were periodic changes in the core environment as experimental subassemblies were rotated in and out of neighboring positions.

B₄C ABOVE CORE PIN

B₄C CAPSULE

VENT CAPSULES

POROUS PLUG

CAPILLARY VALVE

HEDL 7803-152.1

FIGURE 2. Cross Sectional Views of an Above-Core Pin, a B₄C Capsule and the Vent Capsules. (Reg. No. 7803402-1)

TABLE 1

BICM- 2 EXPERIMENTAL TEST MATRIX

<u>CAPSULE NUMBER</u>	<u>MATERIAL</u>	<u>¹⁰B ENRICHMENT (%)</u>	<u>DENSITY gm/cm³</u>	<u>GRAIN SIZE (μm)</u>	<u>PIN PELLET CAPSULE DIAMETER (cm)</u>	<u>B/C RATIO</u>	<u>CLADDING</u>
1	B ₄ C	92.3	2.17	13.0	.762	4.1	316 SS
2	B ₄ C	92.3	2.17	13.0	.762	4.1	W
3	B ₄ C	92.3	2.17	13.0	.762	4.1	W
4	B ₄ C	50.4	2.28	13.1	.762	4.1	316 SS
5	B ₄ C	50.4	2.28	13.1	.762	4.1	316 SS
6	B ₄ C	50.4	2.28	13.1	.762	4.1	316 SS
7	B ₄ C	19.81	2.31	10.4	.762	4.1	316 SS
8	B ₄ C	19.81	2.31	10.4	.752	4.1	316 SS
9	B ₄ C	19.81	2.31	10.4	.762	4.1	316 SS
15	CAPILLARY VENT	NA	NA	NA	NA	NA	316 SS
16	POROUS PLUG VENT	NA	NA	NA	NA	NA	316 SS
17	B ₄ C	92.3	2.19	13.0	.919	4.1	316 SS
18	B ₄ C	92.3	2.19	13.0	.919	4.1	316 SS

3.0 RESULTS

Much of the pertinent data from the experiment is summarized in Table 2. In the following sections, each column will be explained in detail.

3.1 Temperature

Temperature is an important absorber element design parameter because it directly impacts lifetime limiting processes. For the in-core capsules, temperature was the primary variable between material of the same isotopic content. Time averaged temperatures for most of the capsules are presented in Table 2. In Figure 3, temperatures recorded during reactor operations are compared for three of the capsules.

Most capsule temperatures were relatively constant at full power. The irregularities in the data point out the sensitivity of the instrumented experiment's sensors to the every day occurrences in a reactor that is sometimes assumed to operate isothermally. The intentional increases in the sodium flow rate at approximately 2000 MWd and then again at 4500 MWd, reduced all capsule temperatures about 50°C. After each reactor startup the sodium control valve was readjusted by reactor operations in an attempt to approximate the prior cycle's operating temperatures. In the latter stages of the experiment the sodium flow control valve became inoperable, causing a decline in capsule temperatures between Run 83 and Run 85.

3.2 Burnup

Boron burnup is the primary yardstick for determining how such irradiation damage is accumulated and how much helium is generated in the boron carbide. Burnup values for each capsule were calculated from helium release during postirradiation vacuum fusion and in-reactor pressure measurements.

TABLE 2BICM-2 RESULTS

<u>CAPSULE NUMBER</u>	<u>CENTERLINE TEMPERATURE (°C)</u>	<u>BURNUP 10²⁰ capt/cm³</u>	<u>GAS RELEASE % cm³/cm³</u>
1	952	53.0	25.7 50.7
2	752/1046	62.5*	46.1* 106.9*
3	679/1115	58.1	49.6 107.2
4	812	40.2	17.3 25.9
5	877	40.2	11.9 17.8
6	938	35.8	20.1 26.8
7	766	16.5	30.9 19.0
8	787	16.8	23.2 14.5
9	884	15.9	14.2 8.4
17	744	45.2	19.8 32.4

*Extrapolated Quantities

FIGURE 3. Temperature History of Three Subcapsules in BICM-2.

In Table 2, burnup values for each capsule are presented. Burnup values for the natural boron carbide are the lowest, at approximately 16×10^{20} captures/cm³. Capsules with 50% ¹⁰B boron carbide achieved about 40×10^{20} captures/cm³ because of their higher ¹⁰B content. At the top of the core, the 92% ¹⁰B had a slightly higher burnup (60×10^{20} captures/cm³) than that of 50% ¹⁰B, but it was not a proportional increase with ¹⁰B content because of the lower flux and selfshielding in these capsules.

3.3 Helium Release

The helium that is generated in the boron carbide can be either retained within the B₄C grains or can be released to the plenum. In the design of a sealed absorber pin it is especially crucial that the amount of helium released be well characterized so as to avoid excessive gas pressure buildup. This experiment offered an opportunity to obtain continuous helium release data over a broad range of burnup values.

In Figure 4, helium release results, in terms of the volume of helium released (cm³ STP) per unit volume of B₄C (cm³), were compared for three different subcapsules. During the irradiation, gas pressures were recorded by the DAS system every minute, but the data points plotted here represent time averaged values determined at 200 MWd intervals (approximately three calendar days).

Figure 4 shows that the helium release for the three capsules had two regions; a primary region where the helium release rate, $P(t)$, is quite high and time dependent and a secondary region with a lower but almost steady state rate of release, S . The total helium release, q , was considered to be the integrated helium release rate in the primary region, plus the accrued steady state helium release:

FIGURE 4. Helium Release Versus Burnup in B₄C Capsules. Data Points Are 200 MWd Apart.
(Neg. No. 7813296-2)

HEDL 7811-130.2

HEDL 7809-099

FIGURE 5. The Effect of Temperature on Fractional Helium Release from Boron Carbide. (Neg. No. 7810812-1)

$$q = \int_0^{t_1} P(t) dt + S(t - t_1) \quad \text{Eq. 1}$$

where t_1 is the time at the end of primary gas release

$P(t)$ is the time dependent primary helium release rate.

S is the time independent steady state helium release rate.

Considering only the primary region of helium release, it was found that differences existed in both $P(t)$ and the t_1 that were related to temperature in a complex manner. In the secondary region of helium release, the material at 812°C exhibited almost no release; i.e., $S = 0$. At higher temperatures, 952°C for instance, S was found to be appreciable enough to influence the overall helium release rate.

At the burnup levels above 40×10^{20} captures/cm³ all the data indicated the enhanced release rate that was previously observed.⁽³⁾ At these high burnup levels, severe crystallographic damage was observed and enhancement of the helium release process was anticipated.

In Figure 5, data from a previous instrumented experiment⁽⁴⁾ (BICM-1) when combined with the new BICM-2 data yields an overall view of the effect of temperature on the fractional helium release rate between 690° and 950°C. At the relatively low burnup of 10×10^{20} captures/cm³, the helium release increased from 20% at 700°C to a peak of 60% at about 790°C. At still higher temperatures the helium release values declined to a minimum of 35% at 875°C and then again increased at 960°C. Higher temperature data ($T > 1000^\circ\text{C}$), which are not plotted because of temperature instability, indicated an increase in the gas release values to approximately 45%.

A peak in the fractional helium release at 790°C was previously proposed⁽²⁾ by use of multiparameter regression analysis, but this set of instrumented data emphasized the peak's existence. The utility of an instrumented absorber experiment is that it allows a large number of gas release results to be obtained on samples over a range of burnup levels so that features such as this can be more easily discerned.

At higher burnup levels (see Figure 5), the same general trend of a peak at 790°C and a minimum at 875°C was maintained. However, the amplitude of the maximum and minimum were much less pronounced. In contrast, the slope of the gas release curves at the higher temperatures in Figure 4 appeared to be less sensitive to burnup.

At 10×10^{20} captures/cm³ the high gas release fraction resulted from the first term of Equation 1. At higher burnup levels, the contribution from the secondary term became a more significant factor in the fractional release. Since the gas release rate in the secondary region was low in comparison to that of the primary region, a reduction in the overall gas release fraction developed as burnup in the secondary region was accrued.

In Figure 4, the value of S in the secondary region for the sample at 952°C was obviously higher than that of the one at 877°C. But since the value for the primary region at 877°C was so much greater, the overall fractional gas release at 30×10^{20} captures/cm³ was about the same for both. At 877°C the value of S was almost the same as at 812°C so that the quantity of gas in the primary region determined the difference in helium release between the two temperatures.

In general, the data from these tests demonstrated that the amount of helium released in the primary region, the integral in Equation 1, is greater near 790°C, and hence, caused a maximum in the fraction of helium released. The data also implied that the rate of helium release in the secondary region, S , is more directly related to temperature with no obvious maximum or minimum. Hence, it was apparent that two different processes were operating in the primary and secondary regions of gas release. It was proposed⁽⁴⁾ in the past that the primary region of gas release might be associated with simple long-range diffusion of helium out of the boron carbide grains. The secondary region was thought to be controlled by the formation and growth of helium traps within the boron carbide grains themselves.^(3,4) Evidently the onset of helium trap formation and growth was delayed at 790°C, allowing higher gas release than at other temperatures. The trap formation and growth process, which controls the secondary region, appeared to be related to temperature in a more direct manner.

3.4 Microscopy

The microstructure of the irradiated boron carbide was indicative of the amount of the damage that occurred. In Figure 6, photomicrographs of polished pellets from natural and 50% ^{10}B capsules after irradiation are displayed. The microstructure of the natural boron carbide with a burnup of only 17×10^{20} captures/cm³ remained essentially unchanged during the irradiation. At higher burnup levels (40×10^{20} captures/cm³) some microcracking was observed. The cracked pellets had a bimodal grain size distribution with the finer grains averaging 13 μm in diameter. A few microcracks were scattered between the finer grains, but all of the larger grains had microcracks associated with them; sometimes intragranular as well as intergranular. More microcracking was evident in the pellets which generated higher temperatures (938°C).

In Figure 7, photomicrographs indicate that microcracking permeates the structure at a burnup level of 50 to 60×10^{20} captures/cm³. A more informative view of the structure is the scanning electron fractograph shown in Figure 8. In the material from Capsule 3, microcracks were observed along at least one of the boundaries adjacent to each grain. Intragranular microcracking has been reported to be the result of differential crystallographic swelling.⁽⁵⁾

In addition to microcracks between the grains, the material from Capsule 2 (shown in Figure 8) also exhibited internal cleavage of the grains. This was also observed in the optical micrograph (see Figure 7), under close inspection. In Figure 9, a transmission electron micrograph of the interior of a grain from Capsule 2 displays a large number of helium filled bubbles. These large bubbles would likely contribute to the friable character of the individual grains in material from Capsule 2 (see Figure 8).

Many of the bubbles were longer than 0.1 μm and in some cases appeared to extend to grain boundaries. These bubbles are notably larger than those found in lower temperature material.⁽⁵⁾ Bubble depletion was found around grain boundaries and also near the longer bubbles.

MICROSTRUCTURE OF IRRADIATED B₄C

766°C
 16.5×10^{20} CAPTURES/CM³

787°C
 16.8×10^{20} CAPTURES/CM³

884°C
 15.9×10^{20} CAPTURES/CM³

812°C
 40.2×10^{20} CAPTURES/CM³

877°C
 40.2×10^{20} CAPTURES/CM³

938°C
 35.8×10^{20} CAPTURES/CM³

FIGURE 6. Photomicrographs of Natural and 50% ¹⁰B Boron Carbide After Irradiation. (Neq. No. 7812395-1)

MICROSTRUCTURE OF IRRADIATED B₄C

CAPSULE 1
92% ¹⁰B 952°C
53×10²⁰ CAPTURES/CM³

CAPSULE 2
92% ¹⁰B 752 to 1046°C
62×10²⁰ CAPTURES/CM³

CAPSULE 3 50 μm
92% ¹⁰B 675 to 1115°C
58×10²⁰ CAPTURES/CM³

FIGURE 7. Photomicrographs of 92% ¹⁰B Boron Carbide After Irradiation. (Neg. No. 7812395-3)

CAPSULE 2

92% ^{10}B 752 to 1046°C
 62×10^{20} CAPTURES/ CM^3

CAPSULE 3

92% ^{10}B 679 to 1115°C
 58×10^{20} CAPTURES/ CM^3

10 μM

FIGURE 8. Scanning Electron Fractography of Boron Carbide After Irradiation. Neg. No. 7812395-2)

HEDL 7809-087.4

A horizontal scale bar with the text ".1μm" centered above it.

FIGURE 9. Transmission Electron Micrograph of Boron Carbide with a Total Burnup of 62.5×10^{20} Captures/cm³ at Temperatures from 752 to 1046°C. (Neg. No. 7810731-1)

3.5 Vents

Vented absorber elements hold the promise of eliminating lifetime limits associated with plenum pressure buildup of helium released from the boron carbide. The accurate measurement of venting rates made during this test was the first reported demonstration of the continuous operation and reliability of absorber vents in an irradiation environment.

The porous plug vent was a sintered stainless steel compact with a density somewhat less than theoretical. The capillary tube vent consisted of a small tube filled with ceramic powder and then folded over itself and flattened. As helium flowed through the vents the resulting pressure decay was recorded and later used to compute the flow rate of the escaping helium, R .

$$R = A V_o \left(\frac{\Delta P}{\Delta t} \right) \quad \text{Eq. 3}$$

where A is a temperature factor computed from the initial heat up.

V_o is the volume of the pressurized system.

$\frac{\Delta P}{\Delta t}$ is the rate of pressure drop.

The pressure history of both vents is presented in Figure 10. At the beginning of a pressurization cycle, the helium pressure was raised to a selected value between 0.4 and 1.4 MPa. At these higher pressures, flow rates, and hence pressure changes, were very rapid. As the pressure decreased so did the flow rate until a steady-state pressure level was reached. After attainment of these steady-state levels, new pressurization cycles were initiated.

In Run 81, the capillary tube vent was first pressurized to about 0.4 MPa, but no flow was evident. Subsequent pressure increases to 0.62, 0.79 and 1.03 MPa also failed to start helium flow through the vent. Only when a pressure of 1.4 MPa was reached did venting begin. During the last reactor cycle, gas release in this vent continued until much lower pressures were reached.

HEDL 7802-185.2

FIGURE 10. The History of the Gas Pressure of the Vents in BICM-2. (Fig. No. 7804022-9)

Laboratory testing of vents had previously established that some critical pressure, termed the threshold pressure, must be exceeded in order to initiate flow. Once initiated, the flow of helium continues to very low pressures as observed in the pressure history curves of Figure 10. In several instances, no flow was indicated for a period of time, as if the vent was below the threshold pressure, and then suddenly flow commenced as if the threshold had changed. In some cases the release of gas was triggered by a reactor event, a scram or shutdown for instance, but in other cases no reactor occurrences had been recorded. In all cases, venting was initiated at pressures less than 1.5 MPa.

Figure 11 shows the helium release rate during selected pressurization cycles for the porous plug vent. During each pressurization cycle, the helium release rate was observed to be roughly proportional to the pressure drop across the vents, ΔP . In comparing one run to another, it was apparent that the helium release rate (at equivalent pressures) in the later runs was substantially less than that at the beginning of the experiment. Although the vents were subjected to significant irradiation exposure, the reduction in the gas release rate in later cycles may be more related to time than to irradiation exposure since laboratory investigations exhibited a similar time dependence.

A plot of flow rates versus pressure drop across the capillary tube vent is shown in Figure 12. This vent functioned in much the same manner as the porous plug vent; that is to say, gas release was roughly proportional to pressure drop and there was a trend toward lower helium release rates during the latter cycles. One of the major differences was the observation that the helium release rates were typically less than those of the porous plug vent in this pressure range. Secondly, the operation of the vent was more erratic than that of the porous plug vent. In particular, note the irregularities in the flow rate during Run 78 and Run 80 pressurization cycles. Even more instability was noted when comparing pressurization cycles to each other. Between Run 78 and Run 83 there was a trend toward decreasing helium release rates, but after pressurization for Run 84 a much higher release rate was obtained (see Figure 12). Postirradiation examination of the capillary tube vent yielded no observable changes which might have caused the measured fluctuation in helium release rate.

HEDL 7802-44.1
FIGURE 11. Helium Release Rate During Selected Pressurization Cycles for the Porous Plug Vent.
(Neg. No. 7801477-2)

VENTING BEHAVIOR OF VENT NUMBER 15 (CAPILLARY VALVE VENT)

HEDL 7802-44.2

FIGURE 12. Helium Release Rate During Selected Pressurization Cycles for the Capillary Valve Vent.

4.0 SUMMARY AND CONCLUSIONS

The helium release from boron carbide during irradiation was found to have both a primary and a secondary region. The primary portion exhibited a higher release rate, a definite time dependency, and a maximum at 790°C and a minimum at 875°C. Helium release in the secondary region could be approximated as a steady state process and appeared to be temperature dependent.

Two types of vents were evaluated during the irradiation, a porous plug and a capillary tube vent. During numerous vent pressurization cycles, the venting rate of each vent generally decreased during the latter part of the irradiation. The most important feature to be noted was that the vents continued to function after 22,700 MWd of irradiation, and in no case was a threshold pressure greater than 1.5 MPa observed.

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