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RADIATION EFFECTS ON BORON-CONTAINING COMPOUNDS

by

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RADIATION EFFECTS ON BORON-CONTAINING COMPOUNDS

Donald J. Hamman and Paul Schall

Elemental boron, boron carbide, and zirconium diboride were irradiated at reactor ambient temperature (150 F) and elevated temperatures (500 to 750 F) to burnups ranging from 20 to 95 per cent of the boron-10 isotope. Zirconium diboride containing both natural and enriched amounts of boron-10 was evaluated. The amount of helium released during irradiation was measured, and X-ray diffraction, metallographic, and chemical examinations were performed both before and after irradiation.

Zirconium diboride containing natural boron appeared to be the most promising material from a helium-retention standpoint. None of the materials exhibited excessive particle fragmentation or dimensional changes during the course of the experiments even though there was evidence of temperatures much higher than design conditions.

INTRODUCTION

Since helium is produced during the irradiation of boron, the ability of the material to retain this helium is of prime importance. Also important are the dimensional stability and all other properties consistent with reactor operating conditions.

The program reported here on boron-containing compounds was one of several to assist the Knolls Atomic Power Laboratory in the selection of alternate control materials and was supported by the Naval Reactor Branch of the United States Atomic Energy Commission. In the program, elemental boron, boron carbide, and zirconium diboride were irradiated in the MTR at reactor ambient temperature (150 F) and elevated temperatures (500 to 750 F) to burnups ranging from 20 to 95 per cent of the boron-10 isotope. Two capsules containing hafnium diboride are still under irradiation at the ETR.

EXPERIMENTAL PROCEDURES

Selection of Materials

The materials used for this program were powders of known mesh size. This form was chosen to eliminate limitations imposed by dispersions, compacts, etc., or specific geometries of control-rod construction.

Selection of the materials to be studied during the program was based on several factors. First, they were chosen to be representative of the most likely forms of

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boron-containing materials which would be considered for control materials on the basis of chemical and physical properties. The materials selected were elemental boron, boron carbide, and metal borides of both the diboride and hexaboride types. Other metal borides such as the monoborides, tetraborides, and dodecaborides are known but are usually harder to prepare and are relatively unstable compared with the diborides and hexaborides.

The metal borides selected were zirconium and hafnium diborides and dysprosium and europium hexaborides. Of these materials, data have been obtained only on the zirconium diboride. The hafnium diboride is currently being irradiated, and effort on the hexaborides has been limited to preparation of satisfactory materials.

Several different crystal structures are represented by the materials selected for this program. The elemental boron is known to exist in an amorphous state(1) and in two (hexagonal and tetragonal) crystalline states(2). Therefore, boron might undergo changes in either crystal structure or degree of crystallinity during irradiation as the result of displacements of boron atoms and the production of lithium and helium. However, boron is quite stable over a wide range of lattice imperfections, and this property may denote reasonably good radiation stability.

Preliminary experiments indicated that boron carbide was between elemental boron and the metal borides with respect to expected structural radiation stability. The B_4C crystal(3) structure or $B_{12}C_3$ consists of a linear chain of 3 carbon atoms with 12 boron atoms at the points of a nearly regular icosahedron and the whole arranged in a rhombohedral structure.

The metal diborides and metal hexaborides normally have separate and distinct crystalline structures of good stability. The two types of structure vary considerably. The diborides have a primitive hexagonal structure(4) similar to graphite in that they are layered with the boron and metal forming alternate layers. The hexaborides form a complex structure(5) in which the six boron atoms are at the points of a regular octahedron, and each octahedron is linked to six other octahedra, forming a continuous network of cubic symmetry with the metal atoms in the interstices. Radiation-stability differences are suggested by the large volume of the hexaboride octahedra, which may effectively contain the lithium or helium atoms. If there are dimensional changes in the crystal structure, they would be expected to be anisotropic in the case of the diborides and isotropic in the hexaborides.

Nuclear properties were also considered in the selection of the compounds to be studied. Elemental boron has an absorption cross section of 750 barns for the neutrons having a velocity of 2200 m per sec, and, being a 1/V absorber, is effective over a fairly wide neutron-energy range. By using a compound of boron in which the second component is also a strong absorber, especially in the resonance-energy region, its effectiveness as a control material can be significantly improved. On this basis hafnium diboride was selected, and the hexaborides chosen were those of dysprosium and europium.

The results obtained for several boron-containing materials on helium release during irradiation and during postirradiation heat treatment and on structural stability

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(1) References at end of text.

as determined by metallographic examination and X-ray diffraction are presented in the following discussion.

The experimental program consisted of the irradiation of two series of capsules (see Table 1) and preirradiation and postirradiation examinations. There were six capsules in the first series with two each containing elemental boron, boron carbide, and zirconium diboride. The second series consisted of two capsules of zirconium diboride containing natural boron, one capsule of zirconium diboride containing boron enriched 50 per cent in the boron-10 isotope, and one of boron carbide containing natural boron.

TABLE 1. LIST OF CAPSULES

Capsule	Contents[a]	Design Temperature, F	Number of Cycles Irradiated	Where Irradiated	Target Boron Burnup, per cent	Thermocouple
BMI-7-1	B (N)	150	1	MTR	70	No
BMI-7-2	B (N)	150	3	MTR	90	No
BMI-7-3	ZrB_2 (N)	150	1	MTR	70	No
BMI-7-4	ZrB_2 (N)	150	3	MTR	90	No
BMI-7-5	B_4C (N)	150	1	MTR	70	No
BMI-7-6	B_4C (N)	150	3	MTR	90	No
BMI-7-14	ZrB_2 (N)	150	8	MTR	80	No
BMI-7-15	ZrB_2 (E)	500-750	16	MTR	60	Yes
BMI-7-16	ZrB_2 (N)	500-750	19	MTR	80	No
BMI-7-17	B_4C (N)	500-750	20	MTR	80	No
BMI-7-20	HfB_2 (E)	500-750	19[b]	ETR	90	Yes
BMI-7-21	HfB_2 (N)	500-750	19[b]	ETR	90	No

(a) (N) denotes natural boron, (E) denotes boron enriched in the boron-10 isotope.
(b) These capsules are still being irradiated.

The series of six capsules was intended to provide data on the comparative radiation stability of the three materials, and the series of four was intended to provide more complete data on the stability during irradiation at elevated temperatures of boron carbide and zirconium diboride.

In addition to these there are two capsules of hafnium diboride, one containing natural boron and the other containing boron enriched to 55.6 per cent in the boron-10 isotope, being irradiated in the ETR.

Irradiation Conditions

All compounds were irradiated in capsules designed to provide a geometry such that maximum uniformity of burnup would be obtained and still provide enough material for postirradiation examinations. The cavities into which the powders were loaded had a depth of 0.015 in., and calculations indicated that this powder depth would not produce self-shielding effects in excess of 30 per cent from the powder surface to the powder center. These calculations are probably no better than ±25 per cent. Several changes took place in the irradiation-capsule design as the conditions of irradiation progressed from ambient temperature to elevated temperatures maintained by gamma heating and the location of the irradiations changed from the MTR to the ETR.

Figures 1 and 2 show the design drawing and photograph, respectively, of the capsule components for the first series of six capsules designated as BMI-7-1 through BMI-7-6. The powders were contained in the inner aluminum holder which was essentially a split cylinder held together by two drive collars. The cavities were 0.015 in. deep, with the oval cross section being 0.6 in. long by 0.25 in. wide. These capsules were not evacuated. They operated at a maximum temperature calculated to be 150 F in the MTR.

The design drawing and photograph of components for capsules designated as BMI-7-14 through BMI-7-17 are shown in Figures 3 and 4, respectively. The inner core of these capsules was copper and was held in place by copper expansion couplings which also provided an adjustable heat leak. These capsules were designed to operate in the range of 500 to 750 F in the MTR by use of gamma heating, and these temperatures were maintained by evacuating the annulus between the inner and outer cylinders. As the drawing in Figure 3 shows, the powder cavities in these capsules were changed to 0.50 by 0.50 in. The original 0.015-in. depth dimension was retained.

The design drawing and photograph of components of Capsules BMI-7-20 and BMI-7-21, containing HfB_2 (55.6 per cent enriched in boron-10) and HfB_2 (natural), respectively, and currently being irradiated in the ETR, are shown in Figures 5 and 6. In these capsules the inner core is again aluminum to accommodate the higher gamma heating in the ETR, and a bolt through the center of the inner core provides additional clamping action for better powder retention. These capsules were also designed to operate in the range of 500 to 750 F.

Two of the elevated-temperature capsules were fitted with thermocouples as noted in Table 1 to provide a continuous monitor of the irradiation temperature. As is discussed later, the design temperatures were exceeded during at least part of the irradiations. All of the capsules were designed to accommodate a recoil plate next to the boron compound powder. The material used as recoil plates varied with the different capsules.

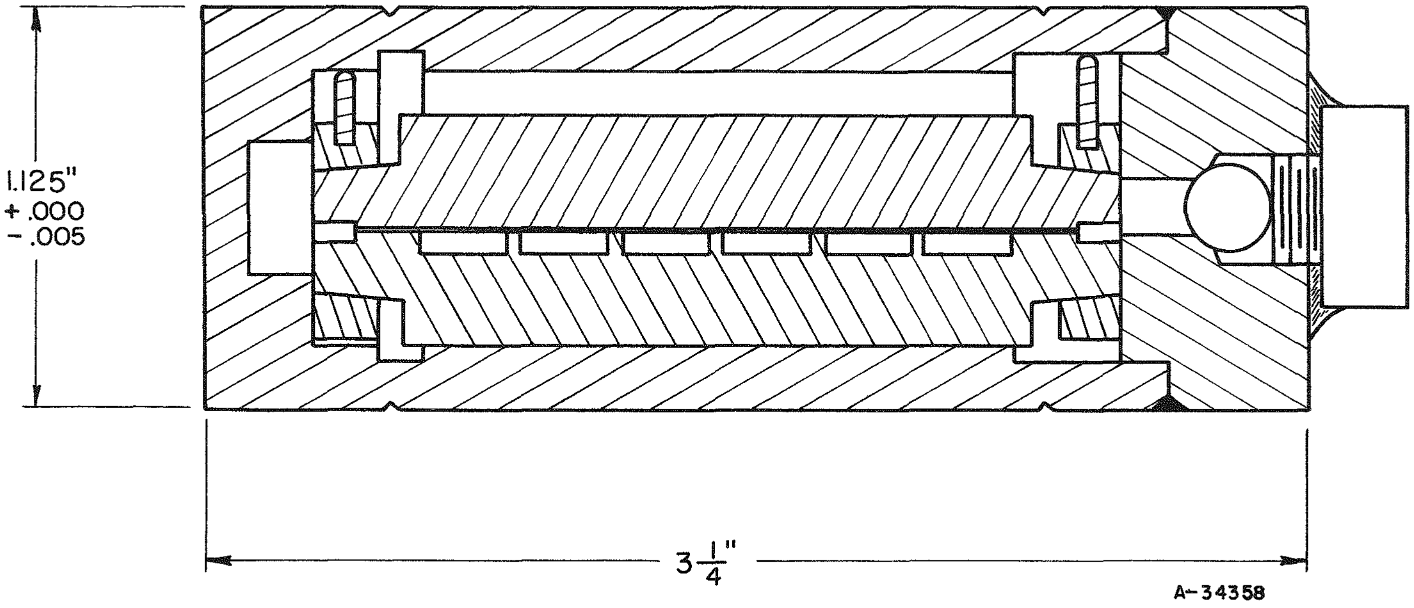

FIGURE 1. DESIGN OF CAPSULES BMI-7-1 THROUGH BMI-7-6

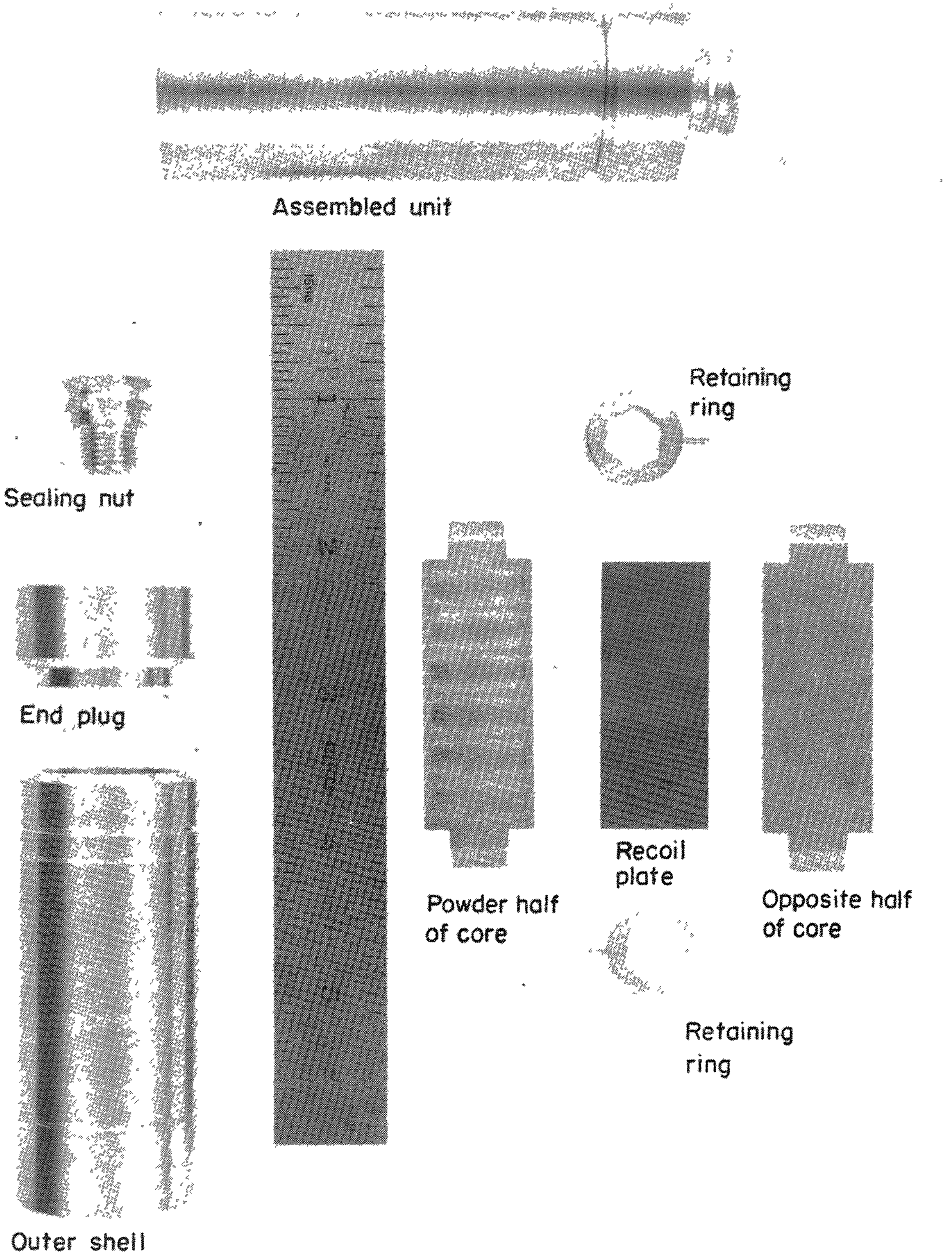

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FIGURE 2. TYPICAL COMPONENTS FOR CAPSULES BMI-7-1 THROUGH BMI-7-6

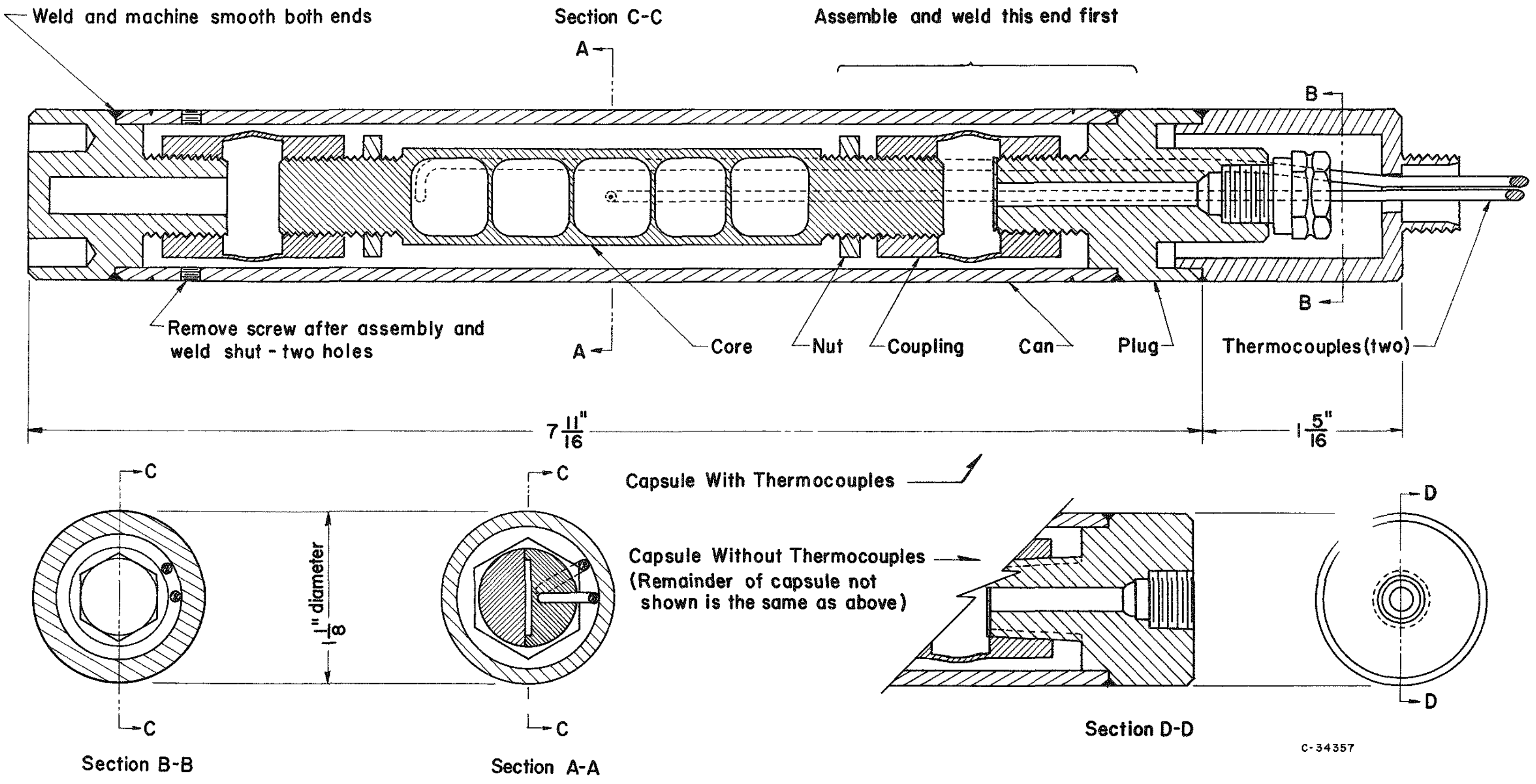

FIGURE 3. DESIGN OF CAPSULES BMI-7-14 THROUGH BMI-7-17

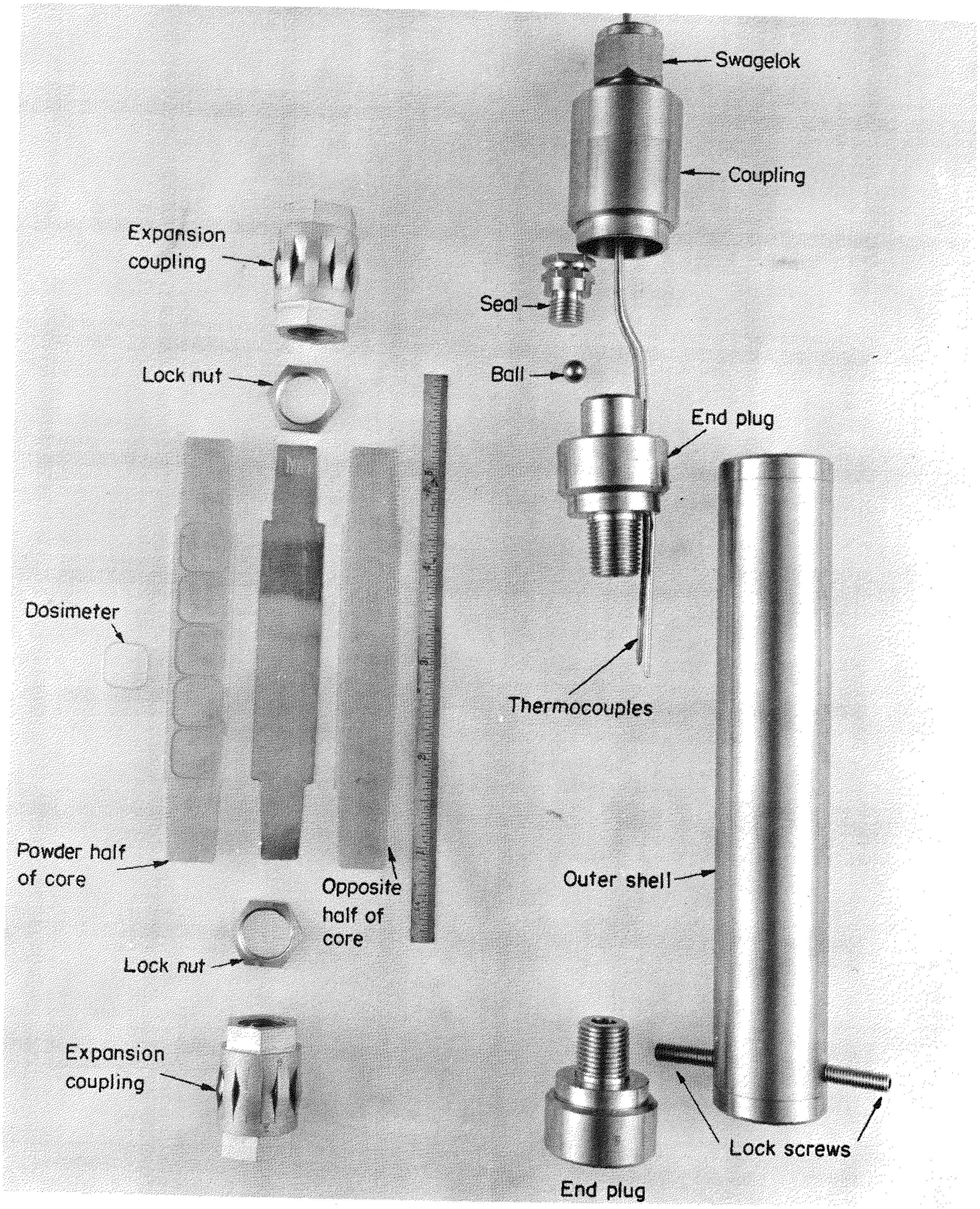

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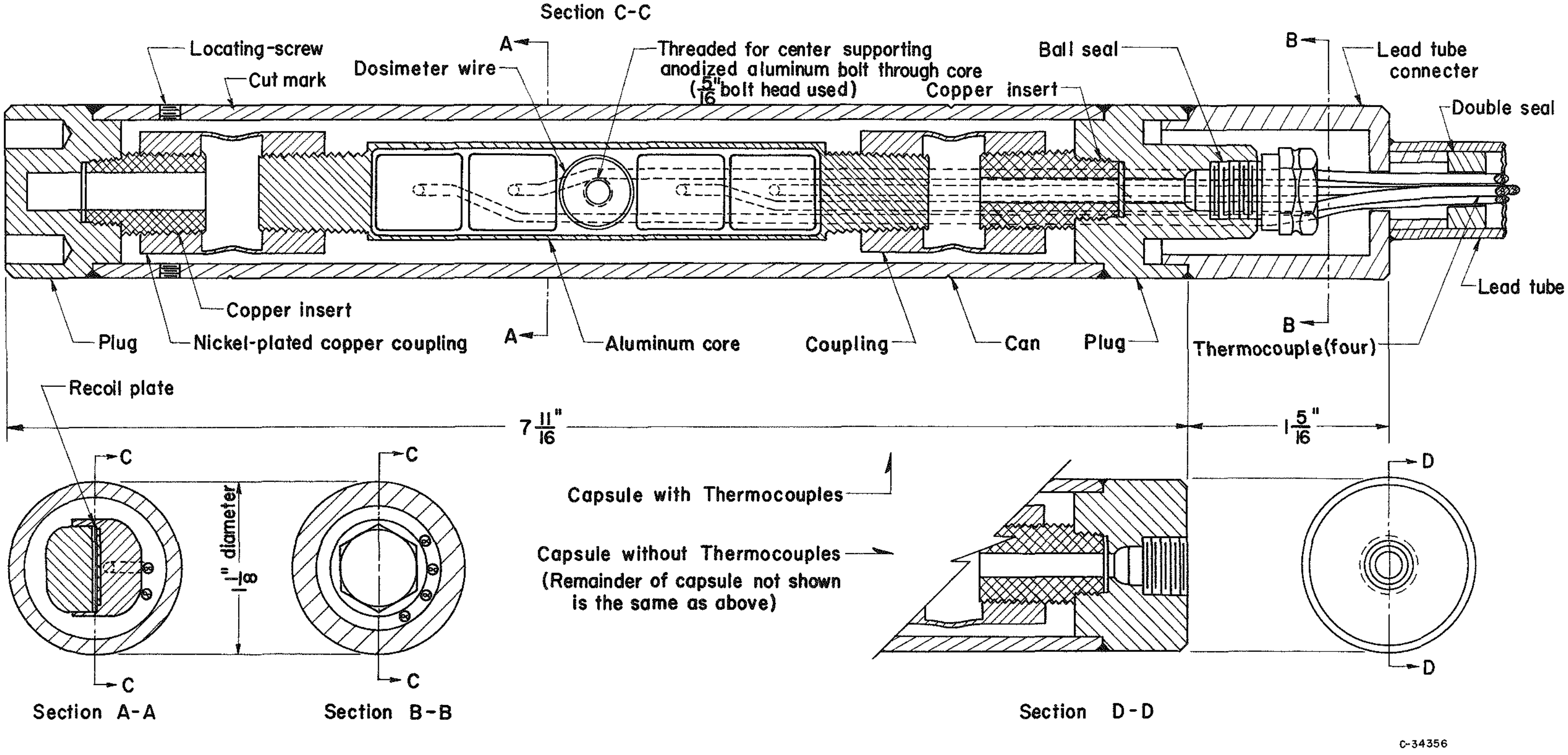
FIGURE 4. TYPICAL COMPONENTS FOR CAPSULES BMI-7-14 THROUGH BMI-7-17



Material: stainless steel unless specified

FIGURE 5. DESIGN OF CAPSULES BMI-7-20 AND BMI-7-21

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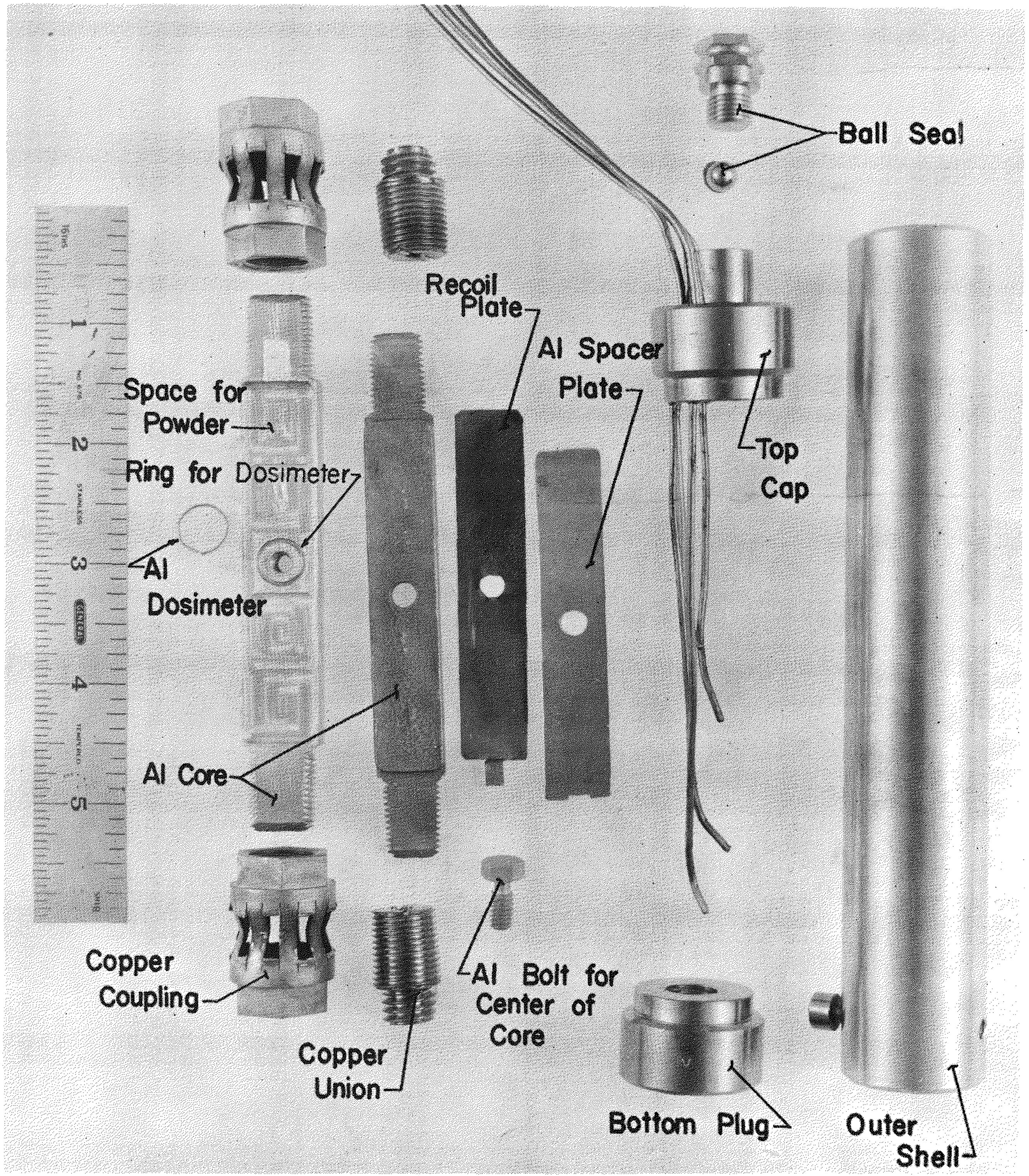

FIGURE 6. TYPICAL COMPONENTS FOR CAPSULES BMI-7-20 AND BMI-7-21

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Preirradiation and Postirradiation Examinations

The examination of the unirradiated powders consisted of:

(1) Chemical and spectrographic analyses to determine purity and composition

(2) X-ray diffraction examination

(3) Metallographic examination.

The postirradiation examination consisted of:

(1) Recovery and analysis of gas released during irradiation

(2) Recovery and analysis of gas released during heat treatment

(3) X-ray diffraction examination

(4) Metallographic examination

(5) Cobalt dosimeter-wire analysis

(6) Analysis for lithium to determine burnup of boron-10

(7) Analysis for retained helium to provide additional check on burnup.

The last three items provide separate means for the determination of the amount of boron-10 burnup. The cobalt-wire technique is considered inferior to the direct analysis for lithium because of the probable 25 per cent uncertainty in calculation of flux depression and attenuation in the powders. The initial analysis for retained helium was accomplished by measuring the amount of helium released after heating the powders at 2200 F until no additional gas release could be detected. This temperature (2200 F) was the maximum attainable with the apparatus used. Since it was not certain that all of the retained helium was released during this treatment, a procedure was developed which involved dissolution of the zirconium diboride powder in molten iron. Since the diboride dissolved in molten iron, fusion of the system could be accomplished at a lower temperature than the melting point of the boride powder.

The flame spectrophotometric method was used to accomplish the lithium analysis using dissolved powder samples. This method was preferred over the analysis for helium since it was assumed that all the helium was not recovered. This was partially verified by the fact that the helium analysis gave lower values of burnup than the lithium analysis in all cases. Accuracy within ±0.5 per cent on analysis of simulated samples of dissolved powder containing known quantities of lithium showed this method to be quite reliable. All of the burnup values quoted in the following discussion are based on the lithium analysis with the exception of the material from Capsule BMI-7-17 which was too radioactive for lithium analysis.

RESULTS

Helium Release

The results on helium release during irradiation and postirradiation heat treatment for the first six capsules designated as Capsules BMI-7-1 through BMI-7-6 and containing boron, boron carbide, and zirconium diboride are summarized in Table 2.

TABLE 2. HELIUM RELEASE FROM IRRADIATED BORIDES IN THE FIRST SERIES OF CAPSULES

Capsule	Contents	Particle Size, U. S. mesh	Burnup of Boron-10, per cent	Helium-Release Data[(a)], per cent		
				As Received[(b)]	1 Week at 700 F[(c)]	2 Weeks at 700 F[(c)]
BMI-7-1	B	-60 +80	20	<0.01	1.0	2.4
BMI-7-2	B	-60 +80	64	34.1	1.2	1.3
BMI-7-3	ZrB_2	-30 +60	19	<6.0	--	<0.4
BMI-7-4	ZrB_2	-30 +60	41	<0.1	0.02	<0.03
BMI-7-5	B_4C	-50 +60	22	<0.1	<0.02	<0.01
BMI-7-6	B_4C	-50 +60	66	5.6	--	--

(a) Per cent of total helium at given burnup.
(b) Capsule as received from the MTR and punctured at room temperature.
(c) Heated under vacuum of less than 5 μ of mercury pressure.

The capsules of lower burnup of each material were irradiated for one MTR cycle and the higher burnups for three MTR cycles. The irradiation temperature of all six capsules was reactor ambient. The material from Capsule BMI-7-6 was exposed to moisture during irradiation and so was not subjected to heat-treatment studies.

Due to the urgency of these irradiations, chemical analyses and X-ray diffraction examinations were not completed prior to encapsulation. Later examination showed that the boron had poor crystallinity. The results obtained, therefore, may not be indicative of the helium-retention capabilities of good crystalline material.

Chemical and spectroscopic analyses, X-ray diffraction studies, and metallographic examinations of the zirconium diboride powder showed that the material had a zirconium-to-boron ratio of approximately unity and that the powder consisted of a zirconium phase and a zirconium diboride phase. The distribution of the phases was such that it is not expected that the zirconium phase had any appreciable effect on the retention of helium and that the results were probably representative of the helium-retention capabilities of the zirconium diboride.

The second series of irradiations included one capsule of zirconium diboride irradiated at reactor ambient temperature to serve as a check on the previous data, one capsule of zirconium diboride irradiated to determine the effect of high-temperature irradiation on gas release, one capsule of zirconium diboride 50 per cent enriched in the boron-10 isotope, and one capsule of boron carbide. The latter three capsules were irradiated at elevated temperature. The data on helium released during these irradiations are summarized in Table 3.

TABLE 3. HELIUM RELEASE DURING IRRADIATION OF BORIDES IN THE SECOND SERIES OF CAPSULES

Capsule	Contents[(a)]	Irradiation Temperature, F	Boron-10 Burnup, per cent	Helium Release During Irradiation, per cent of helium produced
BMI-7-14	ZrB_2 (N)	150	67.8	0.6
BMI-7-15	ZrB_2 (E)	500	33.0	57.0
BMI-7-16	ZrB_2 (N)	500	84.7	5.2
BMI-7-17	B_4C (N)	500	94	67[(b)]

(a) Particle size: minus 80 plus 100 mesh. (N) denotes natural boron, (E) denotes boron enriched in the boron-10 isotope.

(b) This value was calculated from irradiation data obtained from the MTR. All other values were based on lithium analysis of irradiated powder.

The 500 F temperatures given for the last three capsules in Table 3 are the nominal design temperatures. As mentioned earlier the temperatures were to be maintained by reactor gamma heat. Capsule BMI-7-15 was fitted with thermocouples and is known to have maintained a temperature in the range of 475 to 525 F during the first MTR cycle. However, the thermocouples were inadvertently cut at this time and the complete temperature history is not available.

Postirradiation examination of the interior of the capsules revealed reactions between several of the dissimilar metals of the inner capsule, cobalt-aluminum dosimeter wires, and recoil plates. From the observed conditions in the capsule interiors and the known phase diagrams of the various metallic systems, it was concluded that the following maximum temperatures were probably attained during at least part of the irradiation time:

(1) BMI-7-15: close to but not exceeding about 1000 F

(2) BMI-7-16: in the range 1000 to 1200 F

(3) BMI-7-17: in the range 1440 to 1760 F.

Several important points may be noted from the data in Table 3. Natural ZrB_2 exhibits good helium retention even under elevated-temperature irradiation. However, the enriched ZrB_2 exhibits poor helium retention compared with natural material. It should be noted that for both the enriched and natural ZrB_2 irradiated at elevated temperature, the burnups based on total atomic per cent boron are about equal (16.5 and 15.9 per cent, respectively). Furthermore, all evidence from postirradiation examination of the capsule indicates that the natural material attained a higher maximum temperature during irradiation than did the enriched material. This effect of enrichment is in agreement with the results of experiments on dispersions of enriched and natural diboride compounds.(6)

Some of the difference in helium retention of enriched and natural material might be attributed to the higher surface burnup attained for the enriched material for the same average burnup. However, variations in burnup as a function of depth calculated for the 15-mil layer of powders indicate that this effect cannot by itself account for the large observed difference in helium retention.

On the basis of the data in Table 3, no firm conclusion can be drawn with regard to the relative merits of B_4C and ZrB_2 since the B_4C apparently attained a much higher temperature than any of the ZrB_2 specimens. Postirradiation heat treatments of ZrB_2 indicate a "threshold" temperature of approximately 1000 F beyond which the helium release rate rose rapidly.

The results of postirradiation heat treatment of ZrB_2 (natural) from Capsules BMI-7-14 and BMI-7-16 are shown in Table 4. The heat treatment for Capsule BMI-7-14 was conducted under vacuum, and the gas samples were taken by periodically removing gas-sampling bulbs from a manifold external to the furnace. This apparatus did not permit the instantaneous determination of gas-release rate. Observations during the course of the heat treatment of Capsule BMI-7-16 indicated that the gas was released, primarily, in bursts corresponding to microcrack formation, particularly during periods of increasing temperature (see Figure 7). The gas analyses from Capsule BMI-7-14 showed no indication of any gas other than helium. The apparatus used for Capsule BMI-7-16 was modified to permit essentially continuous observation of the gas pressure, but the gas was sampled only at the end of the heat treatment since it was assumed from the previous capsule that no gas other than helium was evolved. Contrary to previous results, analysis of the gas showed that the majority of the gas was not helium. Hydrogen was the major constituent. Helium was present to the extent of 10 volume per cent, and the values given in Table 4 assume that the helium was a constant fraction of the total gas released. The "threshold" temperature is again evident in the heat treatment of Capsule BMI-7-16, although the actual temperature is lower than in Capsule BMI-7-14. There was no thermal cycling during the course of these heat treatments.

Heat treatments were not made on the enriched ZrB_2 from Capsule BMI-7-15 due to lack of sufficient powder or from the B_4C (natural boron) from Capsule BMI-7-17 because of the highly radioactive state of the powder.

The X-ray data obtained on materials from Capsules BMI-7-14, BMI-7-15, and BMI-7-16 are summarized in Table 5.

[redacted]

TABLE 4. POSTIRRADIATION GAS RELEASE ON ZrB_2 CONTAINING NATURAL BORON IN CAPSULES BMI-7-14 AND BMI-7-16

Capsule	Temperature, F	Length of Treatment, hr	Helium Recovered[(a)], per cent
BMI-7-14	Room	--	0.6
	500	72	0.04
	750	140	0.9
	1000	196	1.2
	1200	143	10.0
	1400	96.5	6.0
	1600	98.25	21.1
BMI-7-16	Room	--	5.2
	500	95	2.3
	750	96	1.6
	1000	71	31.4
	1200	71	14.3
	1400	8[(b)]	18.6

(a) Figures given are per cent of total helium produced in the sample based on lithium analysis.

(b) Test halted at this point due to equipment failure.

TABLE 5. RESULTS OF X-RAY DIFFRACTION EXAMINATIONS OF ZIRCONIUM DIBORIDE IRRADIATED IN CAPSULES BMI-7-14, BMI-7-15, AND BMI-7-16

	BMI-7-14 (ZrB_2, Natural)		BMI-7-15 (ZrB_2, Enriched)		BMI-7-16 (ZrB_2, Natural)	
	Preirradiation	Postirradiation	Preirradiation	Postirradiation	Preirradiation	Postirradiation
Unit-Cell Dimension, A						
a_o	3.155	3.220	3.155	3.152	3.155	3.218
c_o	3.508	3.493	3.508	3.496	3.508	3.491
c_o/a_o	1.111	1.084	1.111	1.110	1.111	1.086
Line Broadening, °2θ						
001	--(a)	0.30(c)	--(a)	0.57(c)	--(a)	--(b)
100	--(a)	0.50(c)	--(a)	0.68(c)	--(a)	0.36(c)
101	--(a)	0.44(c)	--(a)	0.60(c)	--(a)	0.62(c)

(a) Line broadening taken as zero for starting material.

(b) Not measurable.

(c) Difference between preirradiation and postirradiation values.

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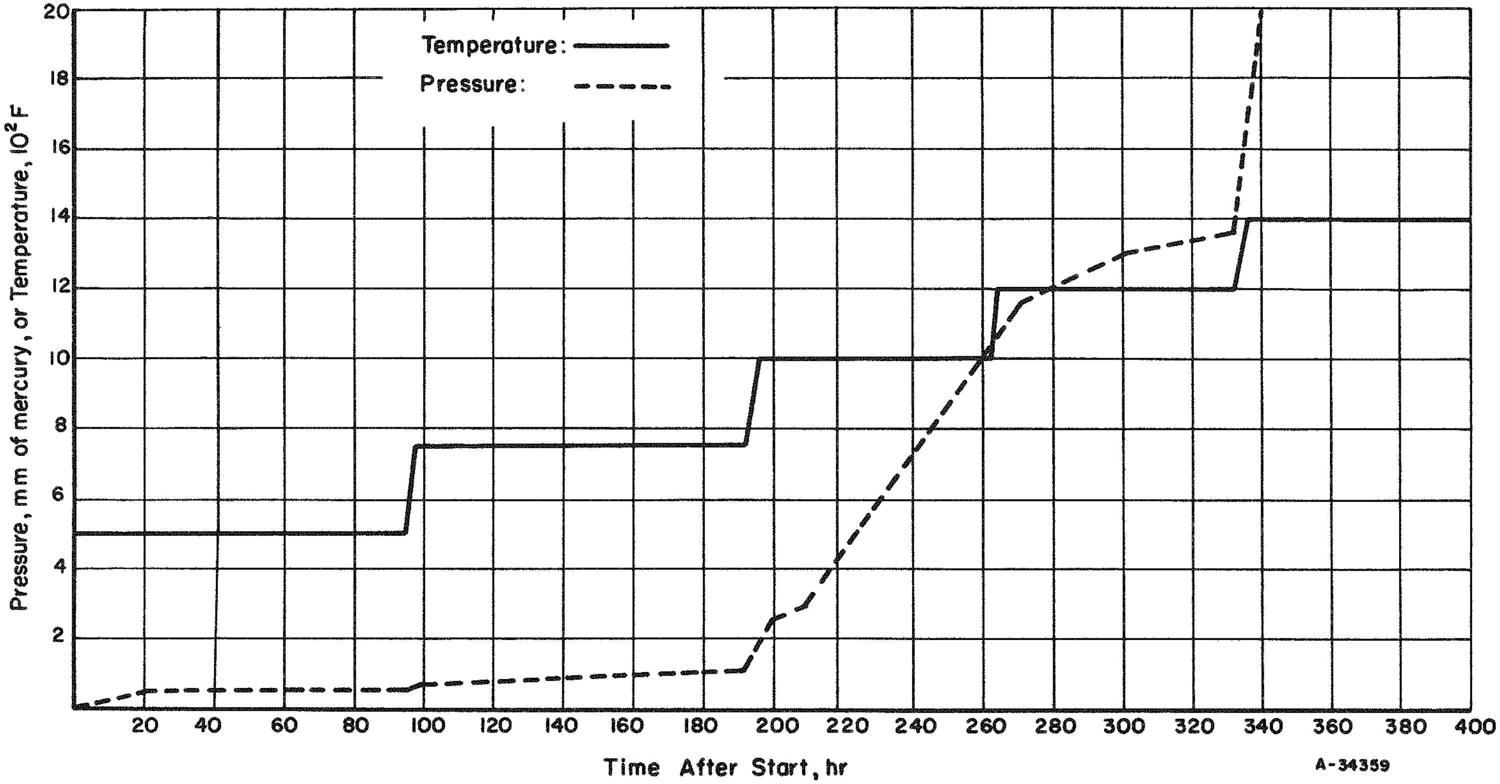


FIGURE 7. GAS RELEASE DURING POSTIRRADIATION HEAT TREATMENT OF NATURAL ZrB_2 IRRADIATED IN CAPSULE BMI-7-16

Of the unit-cell dimensional changes only the changes in the a_o dimension for natural zirconium diboride are considered to be definitely larger than experimental error. The changes in this dimension have been tentatively attributed to a relaxation of the compressive effect of the boron on the zirconium-zirconium interatomic distance (this distance corresponds to the a_o dimension). In zirconium diboride the interatomic distance between zirconium atoms is slightly less than would be predicted for zirconium atoms having a coordination number of 12, while the boron-boron interatomic distance in the boron layer is "normal". It is thought that the reduced zirconium-zirconium distance is due to a compressive effect by the boron layer. This compressive effect would be expected to relax during the weakening of the boron lattice due to burnup of the boron-10. The relaxation of this compressive effect might then allow the zirconium atoms to assume their "normal" interatomic distances, a value very close to the postirradiation a_o dimension for natural zirconium diboride. The agreement in the X-ray data for the two capsules containing the natural zirconium diboride is excellent except for the line-broadening effects on Capsule BMI-7-16. This difference in line broadening between the material in Capsules BMI-7-14 and BMI-7-16 may be due to the higher irradiation temperature of Capsule BMI-7-16. The difference in X-ray data for the natural and enriched materials suggests a possible correlation between dimensional changes and the amount of helium retained during irradiation.

No attempt has been made to obtain a quantitative correlation between line broadening and radiation damage.

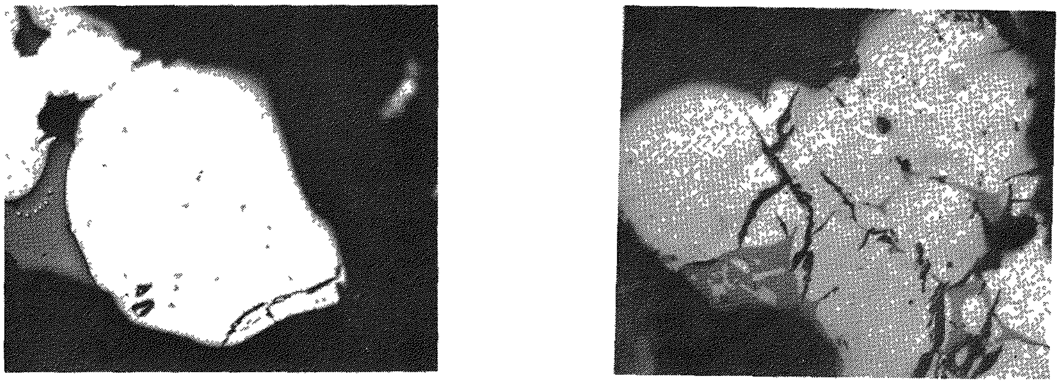
Metallographic Examination

Although both the natural and enriched zirconium diboride showed some microcrack formation during the irradiation, neither of the two materials showed any appreciable fragmentation as evidenced by Figures 8 through 11.

The enriched zirconium diboride in Capsule BMI-7-15 (Figure 9) was considerably more porous, prior to irradiation, than the zirconium diboride containing natural boron. The void volume has been estimated to be about 15 per cent. This porosity may explain, at least in part, the poor behavior of the enriched material from a helium-retention standpoint.

The boron carbide irradiated in Capsule BMI-7-17 was badly permeated with microcracks after irradiation as can be seen in Figure 11. However, again there was no evidence of appreciable particle fragmentation, with most microcracks being less than one-half of the smallest dimension of the particles. The greater degree of cracking no doubt contributes to the increased helium release observed for this material and may have been accentuated by the higher irradiation temperature estimated for this capsule.

Photomicrographs of the recoil plates in Capsules BMI-7-14 and BMI-7-15 are shown in Figures 12 and 13. The edges shown represent a cross section of the face exposed to the boron-containing material. Capsule BMI-7-14 contained zirconium as a recoil material and Capsules BMI-7-15 and BMI-7-16 both contained niobium recoil plates. The recoil plate from BMI-7-17 was not recovered. The zirconium recoil



250X RM10055

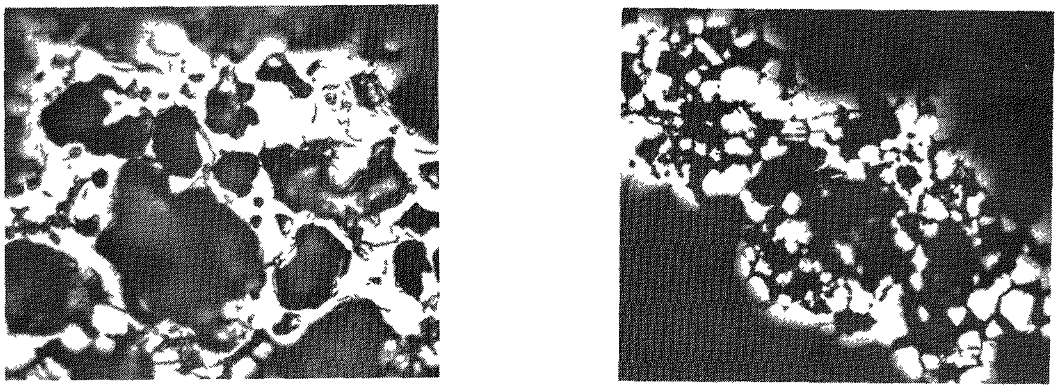
a. Preirradiation

250X HC1336

b. Postirradiation

FIGURE 8. ZrB_2 (NATURAL) FROM CAPSULE BMI-7-14

This material was irradiated at 150 F.



500X RM11750

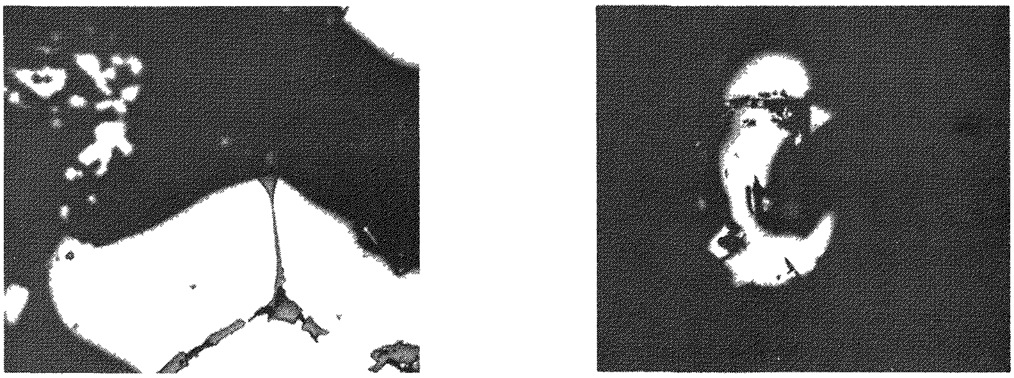
a. Preirradiation

500X RM11743

b. Postirradiation

FIGURE 9. ZrB_2 (50 PER CENT ENRICHED IN BORON-10) FROM CAPSULE BMI-7-15

This material was irradiated at 500 F.



250X RM10055

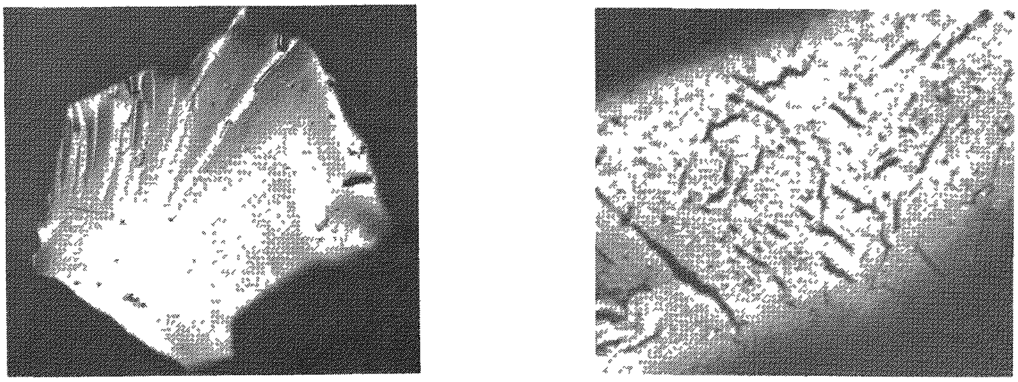
250X RM11748

a. Preirradiation

b. Postirradiation

FIGURE 10. ZrB_2 (NATURAL) FROM CAPSULE BMI-7-16

The irradiation temperature was variable but was greater than 500 F.



250X RM12017

500X HC2273

a. Preirradiation

b. Postirradiation

FIGURE 11. B_4C (NATURAL BORON) FROM CAPSULE BMI-7-17

The irradiation temperature was variable but high.

FIGURE 12. ZIRCONIUM RECOIL PLATE FROM CAPSULE BMI-7-14

The occurrence of what is probably zirconium hydride at the surface (top) of the plate adjacent to the ZrB_2 suggests that hydrogen was released during irradiation.

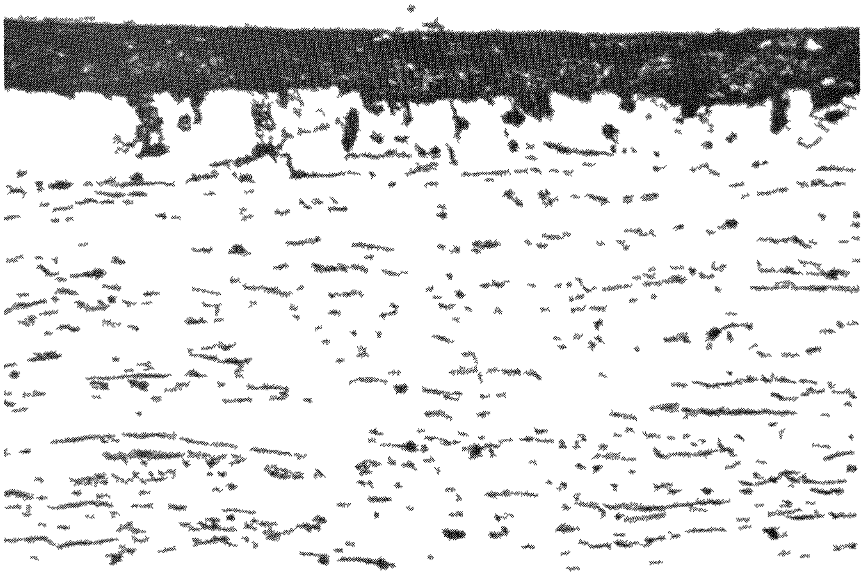


FIGURE 13. NIOBIUM RECOIL PLATE FROM CAPSULE BMI-7-15

The reaction evident on the surface of the plate occurred only on the face next to the ZrB_2 powder.

material from Capsule BMI-7-14 shows considerable variation in microstructure and the edges give the appearance of massive zirconium hydrides with microcracks perpendicular to the edge. It is possible that the powder in this capsule contained hydrogen which was released during irradiation and diffused into the zirconium plate. The niobium plate from Capsule BMI-7-15 shows a pronounced difference at the edges. It can be seen that there is considerable pitting or corrosion at the grain boundaries. There also seems to be a tendency for the grains to change from elongated to equiaxed at the outer edge. The face of the plate away from the powder showed no attack. The niobium recoil plate from Capsule BMI-7-16 had several defects apparently introduced during fabrication, and the information obtained from the photomicrographs had little value other than giving an appearance of some precipitates or pits over both surfaces of the plate.

CONCLUSIONS

The use of boron compounds is more promising than elemental boron as a means of attaining maximum helium retention of the control material. Natural zirconium diboride appears to be better able to withstand high burnup of the boron-10 than either elemental boron, boron carbide, or enriched zirconium diboride. Natural zirconium diboride also withstood temperatures up to approximately 1000 F without exhibiting excessive helium release. The differences observed between natural zirconium diboride and boron carbide may be due at least partially to the maximum temperatures reached during the irradiation.

None of the materials studied exhibited excessive particle fragmentation or dimensional changes during the course of the experiments.

Variability of physical structure and purity of the materials make all the above conclusions subject to some question. The examinations of the unirradiated powders were not normally conducted until after the irradiated powders had been returned from the MTR. Some of the powders were in multiphase metallographic states, some were more porous than others, and most contained several per cent impurities of various kinds.

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