

# Irradiation Stability Study on Boron Carbide Reinforced Aluminums Matrix Neutron Absorbing Material

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## Abstract

Boron carbide reinforced aluminums matrix composites are widely applied in spent fuel storage and transportation field.  $^{10}\text{B}$  of boron carbide can capture thermal neutron, and occurring  $^{10}\text{B}(n, \alpha)^7\text{Li}$  nuclear reaction. These nuclear reactions and elastic collisions between neutrons and atoms will lead to radiation damages, which will limit its material life in nuclear material. In order to study the structure evolution and performance change of the material, the full dense 26wt.% $\text{B}_4\text{C}$ -Al composites are fabricated by powder metallurgic method in this study, and irradiation tests were carried out in neutron irradiation environment. The results demonstrated  $\text{B}_4\text{C}$  has good self-healed performance and irradiation stability. 26wt.% $\text{B}_4\text{C}$ -Al composites were put in nuclear reactor and underwent the neutron and  $\gamma$  ray (neutron average dosage is  $5.8\text{E}+19\text{nvt}$  and  $\gamma$  average dosage is  $4.021\text{E}+11\text{ rad}$  ( $E \geq 1\text{MeV}$ )), due to Al irradiation swelling, the dimension and weight of composites decreased slightly while the density increased a little. After neutron bombardment and irradiation hardening happened to Al, inducing the microhardness and yield strength increase slightly; and the areal density had barely changed after irradiation. These results imply that neutron and  $\gamma$  irradiation has only little influence on properties of 26wt.% $\text{B}_4\text{C}$ -Al composites, some properties are even enhanced. In addition, neutron attenuation measurement were adopted to study thermal neutron shielding performance with different thickness of samples, the experimental results revealed that the sample with 3mm thickness can shield 99% thermal neutron, it is agreed with the theoretical value. These research results revealed that 26wt.% $\text{B}_4\text{C}$ -Al composites prepared by hot powder metallurgic possess both excellent thermal neutron absorption property and irradiation stability.

## 1. Introduction

With the speeding up of the construction of nuclear power industry, how to deal with the problem of reactor spent fuel has attracted more and more attention. According to China's nuclear power development goals by 2020, the spent fuel discharged every year in China will exceed one thousand tons. In order to improve the spent fuel storage capacity and facilities to ensure that the dense storage of spent fuel array has enough safety margin, often in the spent fuel storage framework and shipping container set solid neutron absorption material [1]. Boron neutron absorbing materials are the most widely used now, the international commonly used boron neutron absorption materials includes boron steel, boron aluminum composite materials, aluminum, boron, organic boron. The aluminum boron carbide ( $\text{B}_4\text{C}$ -Al) composite materials with light weight, good thermal conductivity, stable size, low expansion coefficient, good ability of neutron absorption, less secondary emission pollution etc, is used in the spent fuel stored, neutron source protection ideal neutron absorption material [2]. In recent years, the domestic relevant research institutes have accelerated the  $\text{B}_4\text{C}$ -Al solid neutron absorption materials research and development process, in order to verify the independent

research and development of B<sub>4</sub>C-Al solid neutron irradiation absorption material performance, the B<sub>4</sub>C-Al solid irradiation test of neutron absorption materials is carried out. This paper studies the use of hot isostatic pressing method 26 wt. % B<sub>4</sub>C-Al neutron absorption materials before and after irradiation, macro, physical properties, mechanical properties, <sup>10</sup>B content change, providing some references for the design, manufacturing, optimization of the B<sub>4</sub>C/Al neutron absorption material.

## 2. Experimental

### 2.1 Material

Sample plate is made by hot isostatic pressing unclad B<sub>4</sub>C/Al sheet. Tensile sample size is shown in figure 1. 6061 aluminum alloy, B<sub>4</sub>C composition and irradiation parameters are shown in table 1, 2.

Table 1 6061 aluminum alloy and B<sub>4</sub>C composition

6061	Composition	Cu	Mn	Mg	Cr	Si	Fe	Al
	wt. %	0.25	0.15	0.8	0.1	0.6	0.7	-
B <sub>4</sub> C	Composition	B	C	Ca	Fe	Si	F	Cl
	wt. %	80.0	16.1	0.3	1.0	0.5	0.25	0.75

Table 2 Irradiation parameters

Material type	Number	γdose/rad	Neutron fluence/nvt	Irradiation temperature/°C
B <sub>4</sub> C/Al	1	-	-	-
	2	4.021×10 <sup>11</sup>	7.185×10 <sup>19</sup>	85

### 2.2 Test method

1. Appearance check of HOT LabHir ROX video microscope (magnification range: ×5~×50);
2. Size measured by HOT Lab NUKE-CMM measurement system, measuring precision 5 μm;
3. Density measured by thermal indoor mettler XS204 type electronic analytical balance;
4. Mechanical tensile test reference to GB/T228.1-2010 standard, in MTS810.10 universal material testing machine, the ambient temperature is room temperature. The loading rate is 0.1 mm/min, and the results come out of the average of three samples.
5. Fractograph observation using spectrometer equipped with Octane pro type FEI, two 450 FEG sem analysis of B<sub>4</sub>C/Al microstructure and fracture characteristics of neutron absorption materials.
6. <sup>10</sup>B surface density (GB/T 3653.1-1988 standard, USE the automatic potentiometric titration analysis to measure the concentration of the sample B, Finngan MAT 262 thermal ionization mass spectrometer analysis to determine the abundance of sample B.

## 3. Results and discussion

### 3.1 Macro morphology before and after irradiation

Before irradiation B<sub>4</sub>C/Al neutron absorption material surface was flat and level, in well-distributed gray, The overall structural integrity sample before and after irradiations show no deformation, crack or scratch. The gray color deepens on the surface of the sample after irradiation. White flocculent corrode spots are distributed over the surface of the sample, some parts of which appears pattern marks. Because the irradiation process is used in water target, therefore in the process of irradiation for B<sub>4</sub>C/Al neutron absorption material has a certain amount of. According to the relevant institutions at home and abroad, corrosion performance of B<sub>4</sub>C/Al neutron absorption materials is studied, After a lot of research confirmed that

corresponds to the flocculation deposition on the surface of the material part, deposition in the process of corrosion floc is in the amorphous form of  $\text{Al}(\text{OH})_3$ .

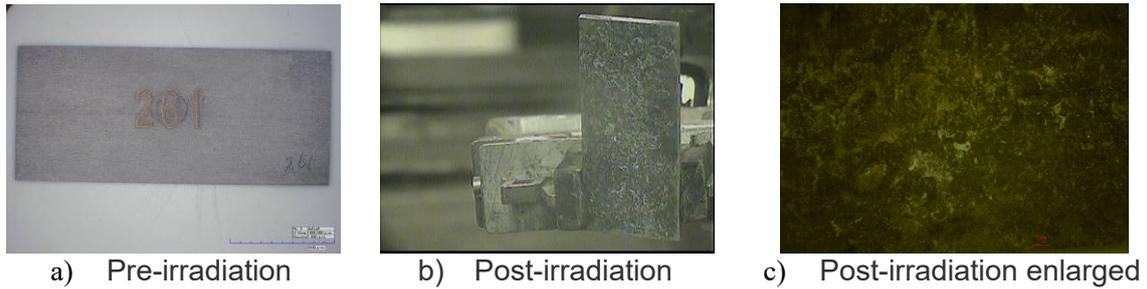


Fig.1  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material, macro photos before and after irradiation

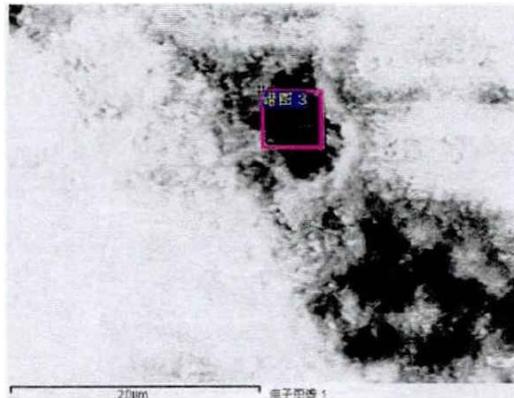


Fig.2  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material in the amorphous form of  $\text{Al}(\text{OH})_3$  flocculated sediment [3]

### 3.2 Physical properties

$\text{B}_4\text{C}/\text{Al}$  neutron absorption material in the process of irradiation. Thermal neutron effect of the  $\text{B}_4\text{C}/\text{Al}$  absorption material, the effect on the properties of matrix component Al negligible. But with  $\text{B}_4\text{C}$  in  $10\text{B} - (n, \alpha)$  reaction cross section is very large, After the  $(n, \alpha)$  reaction, produce He and Li atoms, which will result in swelling of materials. So According to the size and density before and after irradiation, this performance were analyzed.

1) The dimensional

The results of size measurement of  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material before and after irradiation are shown in table 2. The data shows that the sample size changes a little before and after irradiation. the largest growth of the sample length direction is 0.008 mm, The percentage of the change was 0.013% to the maximum; Specimen width direction's maximum growth is 0.004 mm, The maximum percentage of the change is 0.016%. Maximum thickness variation was 0.005 mm. The maximum percentage of the change maximum is 0.224%.

Table 3  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material size measurements

Sample	Before and after irradiation Change/mm			Change the percentage / %			Synthesis of uncertainty	Expanded uncertainty
	Length	Width	Thickness	Length	Width	Thickness		
261	0.008	-0.001	-0.005	0.013	-0.004	-0.224	0.003	0.006
262	-0.002	0.004	0.004	-0.003	0.016	0.179	0.002	0.004
263	0.003	-0.001	0.004	0.005	-0.004	0.182	0.001	0.002

## 2) Density

B<sub>4</sub>C/Al neutron absorption material before and after irradiation size measurement's results are shown in table 4. The data shows that a certain amount of material density decreases after irradiation, but much smaller, the maximum is 0.0094 g/cm<sup>3</sup>.

Table 4 B<sub>4</sub>C/Al neutron absorption material density measurements

material	sample	Pre-irradiation of density /g/cm <sup>3</sup>	Post-irradiation of density /g/cm <sup>3</sup>	Density change /g/cm <sup>3</sup>	Percentage of relative density change/%
26%B <sub>4</sub> C-6061 Al	261	2.6676	2.6582	-0.0094	0.352
	262	2.6679	2.6572	-0.0107	0.401
	263	2.6688	2.6580	-0.0108	0.405

From the above B<sub>4</sub>C/Al neutron absorption material size and density measurements, B<sub>4</sub>C/Al neutron absorption material after irradiation, there did not show irradiation swelling effect, irradiation stability is good.

## 3.3 Mechanical properties

Mechanical performance change before and after irradiation as shown in table 5, shows that after irradiation B<sub>4</sub>C/Al neutron absorption material hardness increased by 5.2 HRB, Yield strength and tensile strength respectively rise 37 MPa and 32 MPa, Breaking elongation rate dropped by 3.6%, the experiment result shows higher strength, plastic drop of materials. The likely cause of the hardness and strength's increasing is that the interface of the Al substrate, B<sub>4</sub>C particles and B<sub>4</sub>C particles with Al changes after irradiation.

Table 5 The mechanical properties of B<sub>4</sub>C/Al material changes before and after irradiation

Material	Number	Neutron Fluence /(nvt)	ΔHRB	ΔYield strength /(MPa)	ΔTensile strength /(MPa)	ΔElongation at break / (%)
B <sub>4</sub> C/Al	1	0	5.2 ↑	37 ↑	32 ↑	3.6 ↓
	2	5.8E+19				

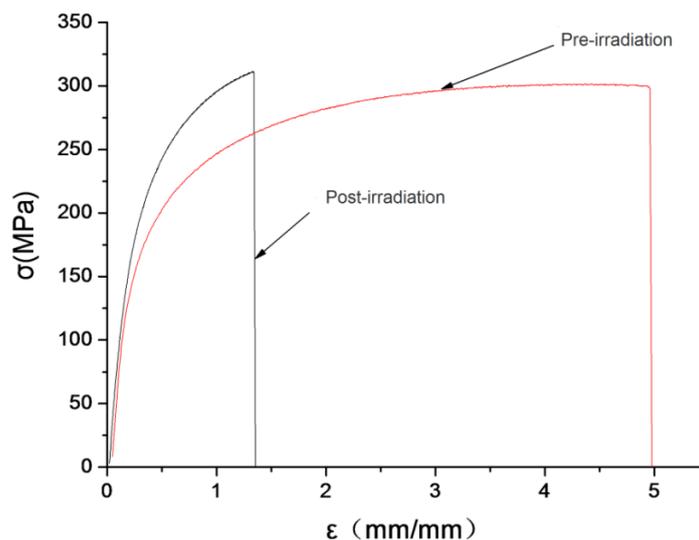
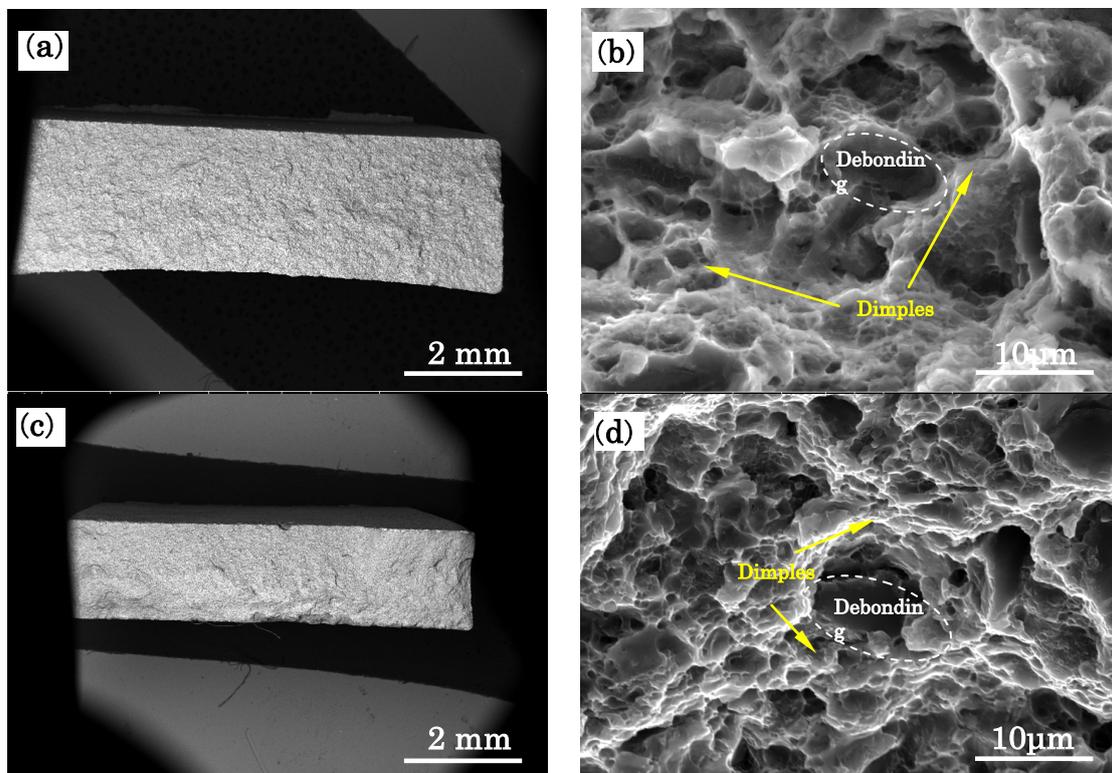


Fig.3 B<sub>4</sub>C/Al material before and after irradiation for typical tensile stress-strain curve.

B<sub>4</sub>C/Al neutron absorption material extending fractures in the macro and micro fractures morphology characteristics were shown in fig.4. The fig.4 shows that B<sub>4</sub>C/Al neutron absorption material's, macro fracture is relatively neat, almost no necking phenomenon

occurred before fracture, which shows it is a fracture, this suggests that the material plasticity is very low. By adding the  $B_4C$  particles, through enhanced phase dislocation strengthening and mechanism of Orowan strengthening, under the action of tensile stress, matrix experiences dislocation and slip deformation, a large number of reinforced phase fault pinning effect hindered the deformation of the aluminum substrate, causing the brittle fracture of materials, due to the  $B_4C$  particles is rigid [4]. It could be seen from the figure 4 the debonding and fracture of aluminum substrate and  $B_4C$  increase phase interface before and after irradiation [5]. The aluminum base around  $B_4C$  has a lot of tenacity, before irradiation material aluminum matrix dimple is a large and deep slurry, after irradiation materials present a large number of small toughening nest. From the morphology of dimple can explain after irradiation  $B_4C/Al$  neutron absorption material strength increased, the plasticity dropped. The  $B_4C/Al$  neutron absorption materials irradiation temperature was  $50\text{ }^\circ\text{C}$ , The influence of irradiation temperature on aluminum alloy has been studied by Jinnan-Yu etal [6], who confirmed that the aluminum combined irradiation temperature below  $100^\circ\text{C}$ .Irradiation damage of serious, The dislocation loop grew led to high strength of aluminum matrix. In terms of  $B_4C/Al$  neutron absorption material, the improvementof mechanical properties of Al matrix also indicates the material mechanical performance improved. Known from the fig.4 fracture morphology, after irradiation  $B_4C/Al$  neutron absorption material aluminum substrate appear a large number of small toughening, this could be an aluminum matrix dislocation strengthening phenomenon. This is because the aluminum matrix in the process of irradiation in the neutron irradiation with fast neutron elastic collision or inelastic collision can producing a lot of vacancy and interstitial atom, form the point defect. Gather near the point defect in fault in node, peg dislocation sources become hardened; Secondary when point defects within the crystal gather, the dislocation loop in the form of a small hardened zone, this is the dot matrix hardening, This two kinds of hardening is a major cause of dislocation strengthening results, which cause the  $B_4C/Al$  neutron absorption material mechanical performance increase the main reasons after the irradiation.



a) Macroscopic fracture before irradiation;b) The micro fracture before irradiation;  
c) Macroscopic fracture after irradiation; d) The micro fracture after irradiation.

Fig.4  $B_4C/Al$  neutron absorption material fracture morphology before and after irradiation

### 3.4 $^{10}\text{B}$ content analysis

The variation in content of  $^{10}\text{B}$  which is one of the important indicators of irradiation stability research about  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material. The change of its content directly affects the neutron absorption ability of the material. Therefore the variation in content of  $^{10}\text{B}$  was analyzed before and after irradiation. Table 6 the measuring results of  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material B abundance before and after irradiation. Table 7 is  $^{10}\text{B}$  surface density calculation results. The data showed that after irradiation  $^{10}\text{B}$  abundance fell slightly,  $^{10}\text{B}$  surface density had little change.

Table 6  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material B abundance of measurement results

Item	Not irradiated samples			Irradiated samples		
	1 <sup>#</sup>	2 <sup>#</sup>	3 <sup>#</sup>	1 <sup>#</sup>	2 <sup>#</sup>	3 <sup>#</sup>
$^{10}\text{B}/^{11}\text{B}$	0.249135	0.248994	0.249391	0.248052	0.248070	0.247807
$^{10}\text{B}$ (Atom) %	19.945	19.936	19.961	19.875	19.876	19.859
$^{11}\text{B}$ (Atom) %	80.055	80.064	80.039	80.125	80.124	80.141

Table 7  $^{10}\text{B}$  surface density calculation results

Item	Not irradiated samples			Irradiated samples		
	1 <sup>#</sup>	2 <sup>#</sup>	3 <sup>#</sup>	1 <sup>#</sup>	2 <sup>#</sup>	3 <sup>#</sup>
$^{10}\text{B}$ surface density ( $\text{g}/\text{cm}^2$ )	$3.31 \times 10^{-02}$	$3.31 \times 10^{-02}$	$3.29 \times 10^{-02}$	$3.31 \times 10^{-02}$	$3.30 \times 10^{-02}$	$3.30 \times 10^{-02}$

### 4. Conclusion

1. 26wt.%  $\text{B}_4\text{C}-\text{Al}$  irradiated neutron absorption material's surface has white flocculent corrode spot, local has traces, pattern has certain corrosion.
2. 26wt.%  $\text{B}_4\text{C}-\text{Al}$  irradiated neutron absorption material's size slightly increase including neutron absorption material's length, width and thickness direction, of which the maximum growth is 0.008 mm, 0.004 mm and 0.005 mm respectively. The density declined slightly, the biggest drop value is  $0.0108 \text{ g}/\text{cm}^3$ , relative density change percentage is 0.405%. The material does not appear obvious irradiation swelling effect.
3. 26wt.%  $\text{B}_4\text{C}-\text{Al}$  irradiated neutron absorption material's yield strength rise 37 MPa, tensile strength rise 32 MPa, elongation at break decreased by 3.6%.
4.  $\text{B}_4\text{C}/\text{Al}$  neutron absorption material's  $^{10}\text{B}$  abundance and surface density has no obvious change before and after irradiation.

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