Study on the microstructural characteristics of Al-B₄C composites based on the B₄C content

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1. Introduction

2. Experimental procedure

In nuclear power plant, the inevitable occurrence of spent nuclear fuel necessitates urgent expansion of storage and transportation facilities due to the imminent saturation of storage facilities domestically. In storage facilities, neutron absorbers are employed, wherein the kinetic energy of neutrons is absorbed during the process of generating new particles due to collisions with neutrons, within the reinforcing material of the neutron absorber. There are borated stainless steel, boron aluminum alloys, and aluminum boron carbide composites as neutron absorbers. Research and development efforts for the production and manufacturing technology of neutron-absorbing aluminum composite materials are actively underway abroad, with some companies commercializing and utilizing such materials. Representative examples of neutron-absorbing aluminum composite materials include MAXUS, BORAL, and MEATMIC [1]. The main production methods of Al-B₄C Metal Matrix Composite (MMC) are primarily made by powder metallurgy [2]. These methods include extrusion processing, which applies high pressure to the metal material and pushes it through the holes of a mold to shrink the cross-section, and compaction processing, which compresses the metal above its recrystallization temperature. MMC is produced by mixing high-purity B₄C powder during compaction processing. Most Al-B₄C composite materials produced through powder metallurgy face challenges due to their expensive materials, high process costs, and long, complex manufacturing processes. Additionally, neutron absorbers are considered strategically important materials, and technology transfer from abroad poses significant challenges for future endeavors. Consequently, domestic research on absorber material development is underway. Efforts are being made to develop low-cost cast-based materials to address these shortcomings. Typically, Al-B₄C with 18% and 30% content was analyzed, such as Al6061+18% B₄C and Al1100+30% B₄C. In this study, the influence of B₄C content on the corrosion properties of Al-B₄C composite materials was obtained through analysis of morphology of precipitated particles and electrochemical corrosion test.

2.1 Material preparation

 B_4C powder was added to Al melted at 700°C and mixed at a constant rotation speed to ensure uniform distribution of B_4C particles. The casting material was homogenized using HIP (hot isostatic pressing) to ensure uniformity and mitigate defects. The removal of unnecessary portions and machining of the slab casting in mold is conducted to refine its shape and dimensions after casting. Following that, the slab undergoes heat treatment and hot rolling. The chemical composition of Al6061/B₄C, with a B₄C content of 18%, is shown in Table 1.

The specimen was precisely processed through electrical discharge machining to achieve a high-quality surface finish. Electrical discharge machining induces electrical discharges between the specimen and the electrode, leading to high temperatures and pressures, which in turn erode and anneal the surface of the workpiece. The specimens were polished to achieve a surface finish of 1 micrometer in preparation for SEM analysis. Subsequently, the specimens underwent immersion in an etching solution 70°C for 40 seconds, followed by rinsing in cold water according to ASTM E407. The etching solution comprised 25% HNO₃ and 75% H₂O [3].

The microstructure of the Al- B_4C specimens was analyzed using both an optical microscope (OM) and a Scanning Electron Microscope (SEM) equipped with a Focused Ion Beam (FIB) instrument. Additionally, the chemical composition of the specimens was investigated via Energy Dispersive Spectroscopy (EDS).

Table I: Chemical composition (wt.%) of 18% B4C/6061Al and 30% B4C/1100Al

•	18% B ₄ C/6061Al
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Si	Fe	Cu	Mn	Mg	Cr
0.65	0.7	0.25	0.15	0.9	0.07
Zn	Ti	Al		В	С
0.25	0.15	Bal.	+	82.13	17.87

•	30%	$B_4C/11$	00A1
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Si	Fe	Cu	Mn	Mg	Zn
0.074	0.132	0.041	0.052	0.005	0.022
Al				В	С
Bal.			+	82.13	17.87

2.2 Electrochemical test

The electrochemical tests and polarization tests were conducted using electrode system. The corrosion rate was calculated following Equation (1) specified in the ASTM G59-97 standard [4]. In this equation, the corrosion current density, extracted from the polarization curves, was used as a parameter.

$$CR = 3.27 \times 10^{3} \times \frac{i_{corr} EW}{\rho}$$
(1)

$$CR = Corrosion \ rate(cm/year)$$

$$i_{corr} = corrosion \ current \ density \ (\mu A \ cm^{-2})$$

$$EW = equivalent \ weight \ (g)$$

$$\rho = density \ (g \ cm^{-3})$$

3. Results and Discussion

3.1 Microstructure of Al-B₄C MMC

Figure. 1 shows optical images of 18% B₄C mixed with Al6061 at different magnifications x10, and x50. From Figure. 1(a), it is apparent that dense black particles appeared on the specimen. B₄C particles distributed homogeneously in the matrix. The size of B₄C particles, ranging from several hundred nanometers to several microns, is not uniform. Some white particles can also be identified on the specimen in Fig. 1(b).





Fig. 1. Optical image of 18% B₄C mixed with Al6061 at different magnifications (a) X10, (b) X50.

Figs. 2 shows SEM images of 18% B₄C mixed with Al6061. Point EDS analysis was conducted to determine the composition at various positions on the specimen. The result of the qualitative point EDS analysis of the specimen shown in Fig.2(b) indicate that point 1 and 2 in the figure represent the aluminum and boron carbide with a chemical composition of 83.62 atomic percent (at.%) B and 16.38 at.% C. Point 3 contained 3.74 at.% Si and 22.67 at.% Ti, which correspond to the TiAl₃ [5]. At point 4, the Fe and Si concentrations were 13.85 and 11.40 a.t.%, respectively, which correspond to the Al_{7.4}Fe₂Si [6].





Fig. 2. SEM images of 18% B₄C mixed with Al6061 (a) X250, (b) X1000.

Table II: EDS analysis results (at.%) of 18% B₄C/6061Al.

Point No	. Al	Si	Ti	Fe	В	С
1	100					
2					83.62	16.38
3	73.59	3.74	22.67			
4	74.75	11.40		13.85		

3.1 Electrochemical corrosion test

From the previous research, the electrochemical behavior of Al-B₄C composites undergoes significant changes with increasing B₄C ratios [9]. According to the polarization curves presented in Fig. 3, it's observed that while the corrosion potential remains unaffected by the increased B₄C ratio, both the corrosion current density (icorr) and corrosion rate (mm/year) experience a notable increase, as indicated in Table 3. This finding contradicts the initial expectation that incorporating less corrosive B₄C particles would enhance corrosion resistance under standard conditions [8]. Instead, the test results (Table 2) demonstrate the opposite effect. This unexpected behavior is attributed to the fact that the introduction of hard particles, such as B₄C, disrupts the continuity of the protective films formed on the surface of aluminum [9]. These protective films typically serve to inhibit corrosion by acting as barriers between the metal and the corrosive environment. However, the presence of hard particles can disturb this protective layer, leading to increased exposure of the aluminum substrate to corrosion agents and consequently higher corrosion rates.



Fig. 3. Polarization curves of the prepared Al–B₄C composites [7].

Table 3: Polarization results of composite pellets [7].

Sample	E _{corr} (V)	i _{corr} (µA cm ⁻²)	Corrosion rate (mm/year)
%10 B ₄ C	-1.125	52.25	0.573
%20 B4C	-1.121	111.34	1.230
$\%30 B_4C$	-1.105	197.76	2.201
%40 B4C	-1.135	375.36	4.121

4. Conclusion

The microstructure of two kinds of Al/B₄C MMCs was analyzed by OM and SEM. The corrosion properties and corresponding mechanisms of Al/B₄C MMCs were investigated. The outstanding obtained results were summarized as follows:

• From EDS analysis, there are Al_{7.4}Fe₂Si. and TiAl₃ precipitates as well as B₄C particles in Al-B₄C MMC.

• The polarization curves from previous research indicate that as the B₄C ratio increased in the Metal Matrix Composites (MMCs), there was a noticeable rise in both the corrosion current density and corrosion rate. Nonetheless, the corrosion potential remained unaffected despite the increase in the B4C ratio [7]. Al1100+30% B₄C is expected to exhibit higher corrosion current density and corrosion rate than Al6061+18% B₄C.

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