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THE EFFECT OF Ti ADDITION ON THE MICROSTRUCTURE AND FRACTURE TOUGHNESS OF BN-Al COMPOSITE MATERIALS SYNTHESIZED BY VACUUM INFILTRATION

WPŁYW DODATKU Ti NA MIKROSTRUKTURĘ I ODPORNOŚĆ NA PĘKANIE KOMPOZYTÓW BN-Al WYTWORZONYCH PRZEZ INFILTRACJĘ W PRÓŻNI

In this paper, we studied the effect of Ti addition on the microstructure and fracture toughness of Boron nitride- Aluminum (BN-Al) composite materials that were synthesized by vacuum infiltration. The BN-Al composite materials were fabricated by preheating the [Ti+BN] preforms at 1700° for 1 hour before Al alloys were infiltrated into the preforms in a vacuum atmosphere at 1100° for 2 hours. X-ray diffraction (XRD) patterns showed that the diffraction peaks of titanium diboride (TiB_2) appeared when the [Ti+BN] preforms were preheated. It is thought that metal Al protected are visible and this could be achieved by the generation of TiB_2 when Al infiltrated into the preform from fractography. The matching fracture toughness of the [Ti+BN] preforms gradually improve when Ti content was increased.

Keywords: BN-Al; Ti; Ceramic composites; Microstructure; Fracture toughness

W niniejszej pracy badano wpływ dodatku Ti na mikrostrukturę i odporność na pękanie kompozytów aluminium-azotek boru (BN-Al), które zostały zsyntetyzowane przez infiltrację w próżni. Kompozyty BN-Al zostały wykonane przez podgrzewanie preform [Ti+BN] w 1700°C przez 1 godzinę, po czym stopy aluminium infiltrowano do preform w atmosferze próżni w temperaturze 1100°C przez 2 godziny. Dyfrakcja rentgenowska (XRD) wykazała, że piki dyfrakcyjne diborku tytanu (TiB_2) pojawiły się gdy preformy [Ti+BN] zostały podgrzane. Uważa się, że aluminium jest chronione co widoczne jest na przełomach i może to być osiągnięte przez wytwarzanie TiB_2 gdy Al przeniknęło do preformy. Odporność na pękanie preform [Ti+BN] stopniowo poprawia się, gdy zawartość Ti została zwiększona.

1. Introduction

Metal matrix composites (MMCs) reinforced with a ceramic phase are of great interest because of the combined effects of metallic and ceramic materials relative to the corresponding monolithic alloy [1]. Various fabrication methods have been developed, such as powder metallurgy [2], stir casting [3, 4], squeeze casting [4-6] and the metal infiltration method [7-10].

Metal infiltration processing is a promising method for preparing high volume fraction ceramic/metal composite materials because it 1) has the potential to realize the net shape of these materials at low cost and 2) can overcome the obstacle that ceramic/metal composite materials are difficult to machine into complex shapes due to their hardness and brittleness [11].

Boron nitride (BN) is an interesting ceramic material because of its unique combination of properties, such as its low-density, high melting point, high thermal conductivity and high electrical resistivity [12, 13]. There are many reports that the BN/Al system shows much better wettability than several other ceramic/metal systems [10, 14-16], but the interface reaction is so strenuous that the BN is almost completely consumed, and the interface reaction product is a disadvantage to the mechanical properties of the resulting material. Therefore, the element Ti was introduced to solve the interface problem. The vacuum infiltration method was selected to reduce the disturbance caused by oxygen.

The aim of this work was to study the effects of Ti addition on the microstructure and the fracture toughness of BN-Al composite materials synthesized using the vacuum infiltration method.

2. Experimental

The starting materials used in this work were Ti powder (purity>99.8%, average particle size 38 μm), h-BN powder (purity>99.9% BN, average particle size 5 μm), and aluminum alloy (trademark 5083).

The Ti and BN powders were blended using a rolling ball mill with polyurethane balls for 24 hours. Then, the Ti-BN powder was mixed with PF resins-acetone solution as an adhesive. The resulting powders were granulated using a 60 mesh

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sieve for further use, and the powder mixtures were uniaxially pressed into preforms of 30 mm diameter in steel die and held under 150 MPa pressure for 1 minute. The composition of the resulting preforms is shown in Table 1.

Preforms were manufactured by being preheated in a vacuum carbon tube furnace to 1700° for 1 hour (10°/min) in a vacuum; graphite crucibles were prepared to perform the experiments. Al ingot (5083) was kept on top of the [Ti+BN] preform compact. The crucible containing the Al ingot and the [Ti+BN] preform were introduced into the furnace and heated to 1100° under vacuum at a rate of 10°/min. The temperature of the ingot and preform was held for 2 hours before being cooled inside the furnace.

The phases of the composites were examined by X-ray diffraction (XRD) with a Philips diffractometer using Cu K_a, radiation and a scan rate of 10 deg/min. The detailed structure of the fresh surfaces was investigated using a scanning electron microscope (SEM). In addition, EDS was used for composition analysis.

The porosity of the composites was investigated using the Archimedes method. The fracture toughness was evaluated using a WE-10A hydraulic universal testing machine. The fracture toughness was measured by the single-edge notched beam method (specimen size = 28 mm×4 mm×3 mm, notch width = 0.2 mm, notch depth = 2 mm, bend span = 20 mm and load speed = 0.05 mm/min).

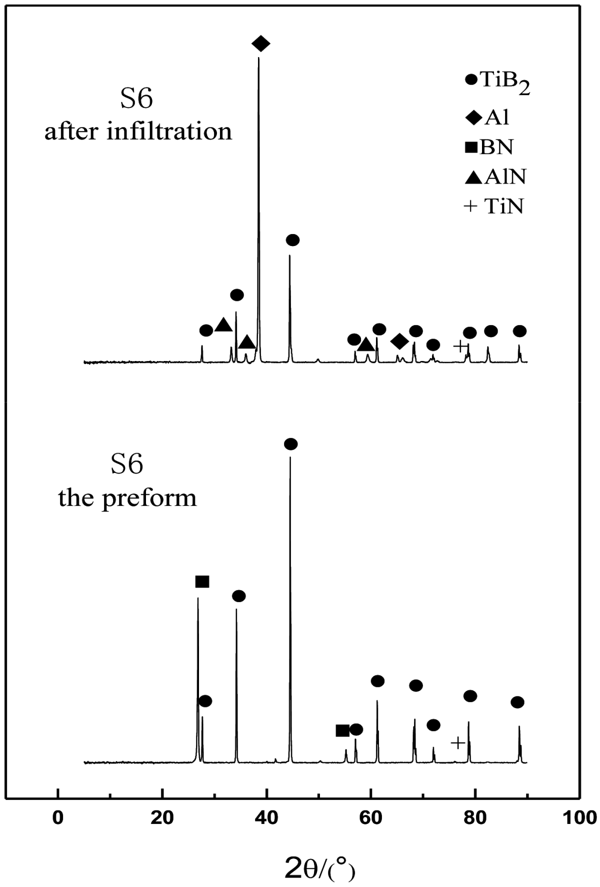

Fig. 1. XRD patterns of the S6 [Ti+BN] preform and the S6 composite material

3. Results and discussion

3.1. The Phase of the Composites

XRD analysis was performed to determine the nature of the chemical compounds in the preforms and the composite materials. Figure 1 shows the XRD pattern of the S6 [Ti+BN] preform and the S6 composite material synthesized by vacuum infiltration. The XRD data showed that strong TiB_2 peaks exist in the preform with no signs of the Ti peaks. This confirms that the Ti has been completely transformed into TiB_2. According to the XRD results, the chemical reaction had occurred for the [Ti+BN] preform.

$$Ti + BN \rightarrow TiB_2 + TiN \qquad (1)$$

Because of the Ti added in the starting materials, a large amount of TiB_2 was produced following the chemical reaction (1). The resulting preform contained a large amount of TiB_2, which is regarded as a phase that can provide positive wetting properties to aluminum infiltration.

For XRD analysis of S6 after infiltration, several reactions had occurred between Al, TiB_2 and BN. The results show that composites synthesized by BN+Al give rise mainly to an AlN phase. This confirms that the addition of the [Ti+BN] preform simultaneously into molten Al leads to reaction (2).[17]

$$Al(l) + BN(s) \rightarrow \alpha AlB_{12}(s) + B(inAl) + AlN(s) at T > 985° \qquad (2)$$

3.2. Micrographs of Metal Infiltration

Figure 2 shows the microstructures of the S1 preform and the S1 composites. Figure 2(A) shows the SEM image analysis of the S1 preform samples. When the microstructure of the composites in Figure 2(A) was examined, large pores and pore walls were observed where TiB_2 and BN particles accumulated. Figure 2(A) confirms that a honeycomb of the preform is formed by preheating. Figure 2(B) shows the polished S1 composite sample after infiltration. Figure 2(C) shows the EDS mapping of aluminium. As shown in Figure 2(B) and Figure 2(C), the infiltration of the porous [Ti+BN] preform was successfully achieved. The porosity test results show that approximately 100% of the total pores in the preforms were impregnated by the liquid aluminum. This supports the hypothesis that vacuum infiltration is a sufficient method to overcome the surface tension between BN and TiB_2. This microstructure photograph indicated that BN-Al composite materials were successfully prepared.

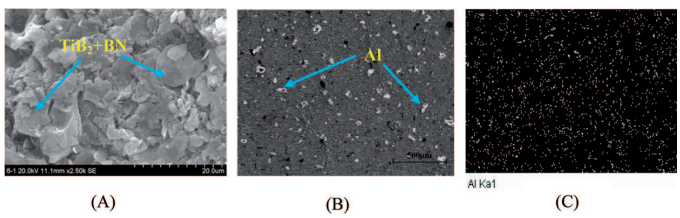

Fig. 2. Microstructure photographs (A) S1 [Ti+BN] preform; (B) S1 composite material after infiltration; (C) The SEM-EDS analysis of the element Al in the S1 composite material

3.3. The Fracture Toughness of the Composites

Table 1 shows the fracture toughness of BN-Al composite materials with varying Ti content. The fracture toughness increased from 3.5 MPa·m$^{1/2}$ to 9.8 MPa·m$^{1/2}$ when the Ti content was increased in the preform. Ti addition significantly contributed to the fracture toughness of the composite materials because the titanium diboride (TiB_2) which was formed in the preform (confirmed by XRD) during the preheating process may restrict the interface reaction between the liquid metal aluminium and BN. The interface reaction has a notable effect on the mechanical property of the composite materials because the ceramic phase can react completely with the infiltrated aluminium, making the ductile metal aluminium change into other reactants, which reduced the fracture toughness of the composite materials.

TABLE 1

The pressure of the preform and the fracture toughness of the composites with varying Ti content

Sample name	Composition	Pressure	Fracture toughness
S1	5% Ti, 95% BN	150 MPa	3.5 MPa·m$^{1/2}$
S2	10% Ti, 90% BN	150 MPa	3.9 MPa·m$^{1/2}$
S3	15% Ti, 85% BN	150 MPa	4.8 MPa·m$^{1/2}$
S4	20% Ti, 80% BN	150 MPa	5.4 MPa·m$^{1/2}$
S5	30% Ti, 70% BN	150 MPa	7.9 MPa·m$^{1/2}$
S6	40% Ti, 60% BN	150 MPa	9.8 MPa·m$^{1/2}$

3.4. Fractography of the Microstructures of the Composites

Fig. 3 shows the fractography of the S4 composites. It was observed via fractography that the composites have almost no porosity and the micrograph analysis allows us to conclude that the infiltration was successful. Dimples are scattered all around an enlarged roughness section. EDS test results show the following: the center of the roughness is the metal Al, the brim of the metal is titanium diboride (TiB_2), the farthest areas from the metal Al are comprised of AlN and BN. Obviously, Al in proximity to TiB_2 has no reactivity with BN; by contrast, Al can react with BN if TiB_2 is not in the immediate area. The increasing fracture toughness may be attributed to the existence of the dimple. When the Ti content is increased, the appearance of the dimple also increases. This sequence of increasing dimple size will eventually result in an increase in fracture toughness.

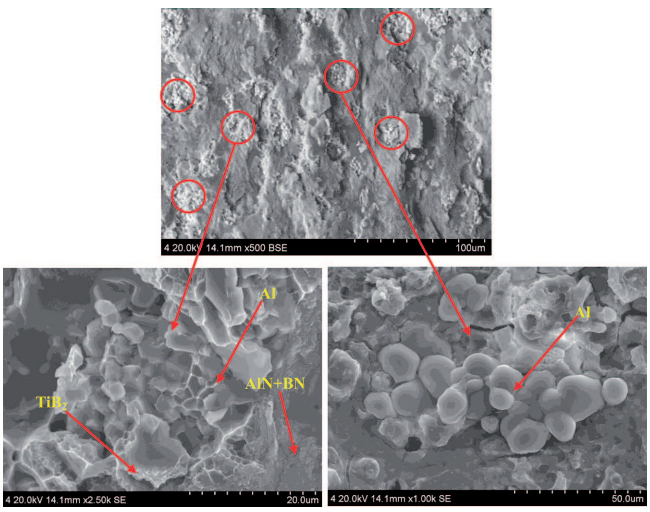

Fig. 3. Fractography of the sample S4

4. Conclusions

1. In this paper we studied the effect of Ti addition on the microstructure and fracture toughness of BN-Al composite materials. The composite materials were prepared from Al with different Ti:BN content ratios by vacuum infiltration after preheating the [Ti+BN] preforms.
2. Research and observation revealed that because of Ti addition, the microstructure of the composites was changed, and dimples were observed in fractography.
3. The fracture toughness increased from 3.5 MPa·m$^{1/2}$ to 9.8 MPa·m$^{1/2}$ when the Ti content was increased from 5% to 40% and was obtained over the entire range of Ti percentage composition by restricting the interface reaction.

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