EFFECT OF ALN CONTENT ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF BN-BASED COMPOSITES

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Keywords: BN-AlN composites, microstructure, bending strength, fracture toughness

Abstract

In this study, AlN was introduced into hot pressed BN-based composites as reinforcement. A new phase of Si_3AlON_7 appeared when AlN was added. When AlN content was higher than 10 vol%, amorphous silica disappeared. At the same time, the relative amount of Si_3AlON_7 increased with raised AlN. Bending strength and fracture toughness of BN-based ceramics was improved by adding AlN. As the AlN content increased, the bending strength and fracture toughness of BN -AlN ceramics firstly increased and then decreased, the elastic modulus slightly increased. The composite with 5vol.% AlN exhibited the highest bending strength (247.0 MPa) and fracture toughness (4.02 $MPa\cdot m^{1/2}$), which was 57.5% and 40.6% higher than that of BN-based ceramics, while the elastic modulus was 10.2% higher than the latter.

1 Introduction

Hexagonal boron nitride has excellent combination of high melting point, high thermal conductivity, good thermal shock resistance and chemical inertness. H-BN can be easily machinable using conventional metal cutting techniques because of the low hardness [1,2]. But the lower strength has limited the broader application of boron nitride.

AlN ceramics have high thermal conductivity, high strength, high hardness and low thermal expansion which close to that of silicon [3,4]. To improve the machinability, h-BN was introduced into AlN ceramics as the weak-boundary phase [5~8].It has been known that the addition of hexagonal boron nitride (h-BN) improves the machinability of AlN. AlN–BN based ceramics can be machined easily since cleavage plane of h-BN can facilitate crack propagation during machining, thereby decreasing the cutting resistance.

In this paper，to overcome the low strength problem of BN ceramics, AlN was introduced into BN-based ceramics to prepare the BN–AlN ceramics. The mechanical properties, phase analysis and surface observation were systemically investigated by three point bending, XRD and SEM.

2 Experimental procedure

The initial powders used in this study were: h-BN powder(Dandong Chemical Engineering institute Co., Ltd, Liaoning, China), average particle size 0.8μm (> 99%); AlN (Nitrogen compounds Co., Ltd. Liaoning, China), average particle size 3 μm (> 99%); Amorphous(Lianyungang Guangyu Quartz Co., Ltd, Jiangsu, China), average particle size 2 μm (> 99%). The powder mixtures h-BN plus AlN and Amorphous were ball-milled for 24 h in a polyethylene bottle using ZrO_2 balls and ethanol as the grinding media. Then the slurry was dried in a drying baker and screened. The resulting powder mixtures were hot pressing at 1700 °C for 30 min under a uniaxial load of 30 MPa in N_2 atmosphere. The microstructures features of the composite were observed by a FEI Quanta 200FEG scanning electron microscope (SEM). SEM samples were sputter-coated with gold. Flexural strength was tested in three point bending on 3×4×36 mm bars, using a 30 mm spanned a crosshead speed of 0.5 $mm{\cdot}min^{-1}$. Fracture toughness (K_{IC}) was evaluated by a single-edge notched beam test with a 16 mm span and a crosshead speed of 0.05 $mm{\cdot}min^{-1}$ using 2×4×20 mm test bars. The mechinal properties were record on a American Instron 5569 Electron-Mechanical Universal Material Testing Machine. The XRD spectra of the powder sample were recorded on a *Japan* Rigaku D/max-γB X-ray diffractometer with Cu-Kα (λ=1.5405 Å) radiation which operated at 60 kV and 80mA.

3 Results and discussion

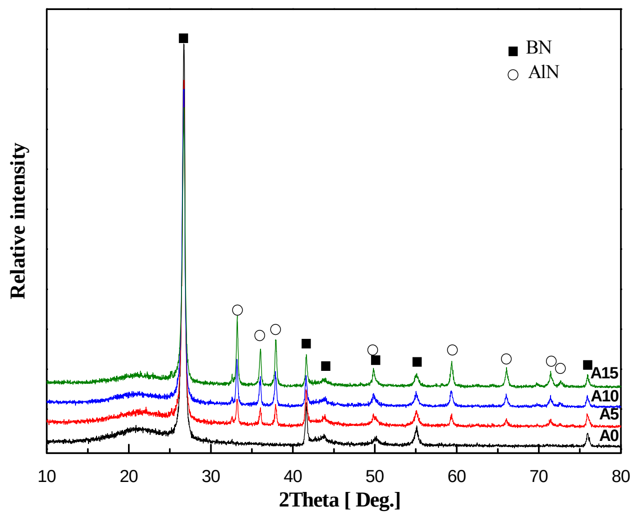

Figure 1.X-ray diffraction patterns of original powder

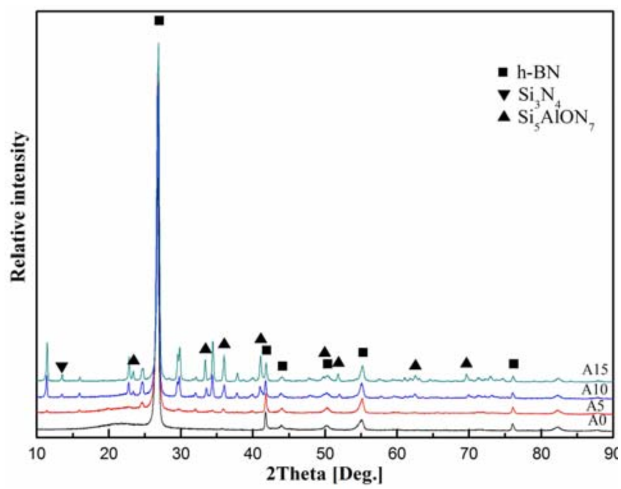

Figure 2. X-ray diffraction patterns for the BN -AlN composites

Fig 1.shows X-ray diffraction patterns of original powder. before sintering The powders were consisted by h-BN、AlN and fused silica. The sample of X-ray diffraction patterns for the BN- AlN composites materials consolidated by hot-pressing are presented in Fig. 2. After

sintering the primary crystalline phases of h-BN were detected in every case. And with the accession of AlN, SiAlON phase were present. In Fig. 2 it can be found that when AlN content was lower than 5 vol%, amorphous silica still can be observed. While AlN content was higher than 10 vol%, amorphous silica disappeared.
Since AlN and amorphous silica in a chemical reaction under high temperature conditions, so with the adding of AlN, it will consume part of the amorphous silica. When AlN content was 5 vol%, as the amount of silica consumed less so it can still found weak peaks of amorphous silica. When increased content of AlN, it will consume more amorphous silica which make the peaks of amorphous silica disappear.

	AlN [vol%]	**Density [g/cm^3]**	**Flexural Strength [MPa]**	**Young's Modulus [GPa]**	**Fracture Toughness [MPa·m$^{1/2}$]**
0	0	2.06	156.8±5.1	65.0±1.6	2.86±0.17
A5	5	2.10	247.0±23.0	73.1±2.5	4.02±0.39
A10	10	2.16	234.5±22.1	74.8±2.9	3.96±0.34
A15	15	2.23	223.3±15.8	73.4±3.2	3.29±0.19

Table 1. Mechanical properties of composite materials

Table 1 shows the effect of AlN addition on the density flexural strength, modulus and fracture toughness of h-BN matrix composites. Flexural strength and fracture toughness increased with an increase in the level of the additive. The flexural strength from156.8MPa for h-BN-matrix composites to 247MPa for containing 5 vol% AlN, increased by about 57.5%. The corresponding the fracture toughness from 2.86 MP·am$^{1/2}$ to 4.02MP·am$^{1/2}$, increased by about 40.6%. As the density of AN is higher than BN, so the density of composites increased with the increasing of AlN.
In order to analysis the cause for the increased in strength and fracture toughness resulting from the addition of AlN, the Young's modulus was measured as a function of AlN content. Table1 shows the results. The modulus increased by about 12.5% with the addition of 5 vol% AlN, from 65.0GPa for h-BN- matrix composites to 73.1GPa for samples with 5 vol% AlN added. Thus, the addition of AlN increased in the modulus as well as a decrease in the size of fracture source; both contribute to the observed increased in flexural strength and fracture toughness.
Comparision of the BN- matrix composites and the BN-AlN composites; it can be found that flexural strength and fracture toughness significantly improved, compared with the modulus. It is known that the thermal shock parameter (*R*)[9] is affected by flexural strength and modulus. In this experiment with the addition of AlN, the ratio of the flexural strength and modulus varying degrees of increase compared with BN matrix composites composites. It is helpfull to improve the material's critical temperature difference (ΔT_c), thereby improving the thermal shock resistance of materials.

$$R = \Delta T_c = \frac{\sigma_f (1-\nu)}{E\alpha_1} \quad (1)$$

Fig. 3 shows SEM pictures of Vickers indentation crack paths on the surfaces of BN-AlN materials which perpendicular to the direction of the applied pressure in hot-pressing. Due to the lower strength of base material, making the crack in the process of expansion is similar to a straight line extended(Fig. 3(a)) when the crack tips encountered the reinforcement, as the higher strength of reinforcement makes the crack cannot pass through, which on its surface deflection (Fig. 3(b)). As the crack in the process of deflection will increase its expansion path and thus consume more energy, thereby improving the fracture toughness of the BN- AlN composites.

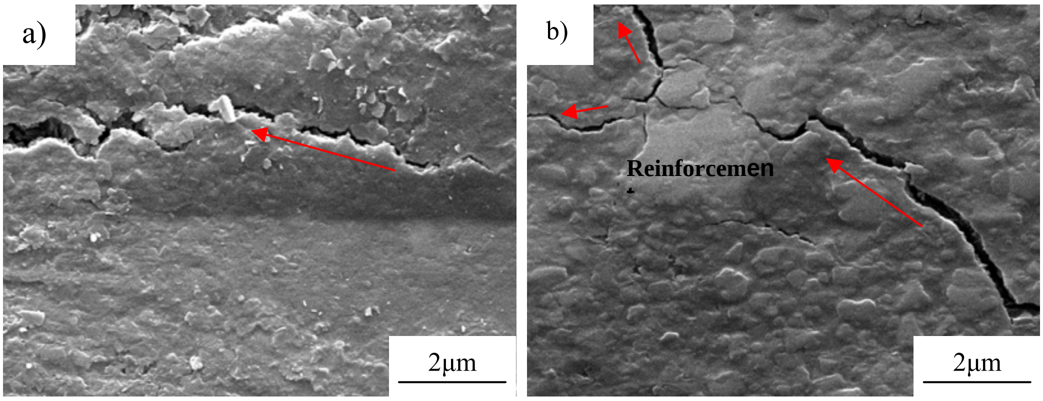

Figure 3. Crack propagation in BN-SiO2-AlN composites

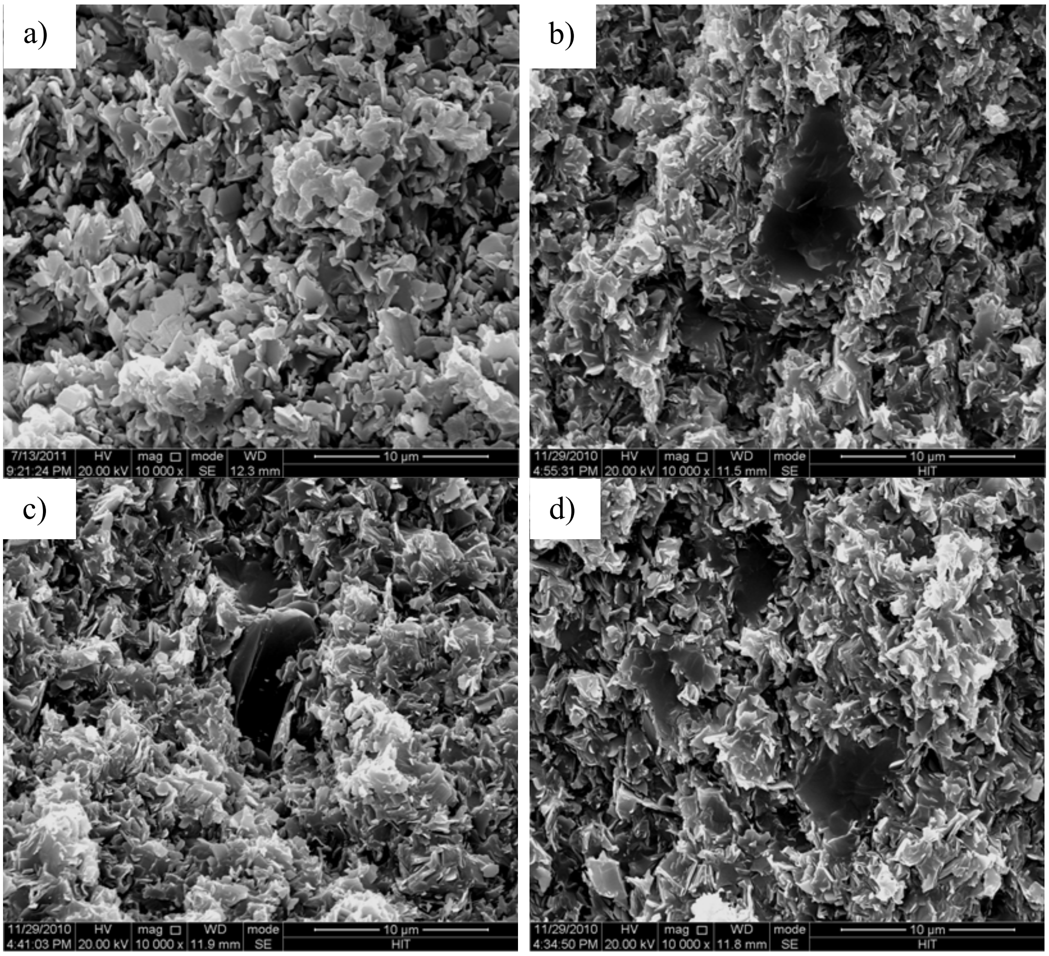

Figure 4. The fracture surfaces of AlN/h-BN composites after three-point bending test:(a) 0 vol% AN, (b) 5 vol% AN, (c) 10 vol% AN and (d) 15 vol% AN.

Fig. 4 shows SEM micrographs of fracture surfaces after bending tests. The fracture surface of BN-SiO_2 matrix shows intergranular fracture patterns. But with the addition of AlN, the materials performance of complex fracture patterns, as intergranular fracture and transgranular fracture coexisted.

Summary

(1) The BN-AlN composites were consolidated by hot-pressing at 1700℃for 30min under 30MPa, and with the accession of AlN, after sintering new phase SiAlON were present.

(2) Flexural strength, fracture toughness and Young's modulus increased with adding of AlN. When the AlN content was 5vol.% the flexural strength and fracture toughness reached a maximum, respectively 247.0±23.0MPa and 4.02±0.39 MPa·m$^{1/2}$. When further increasing the content of aluminum nitride, the material flexural strength and fracture toughness showed a downward trend.

Acknowledgements

This work was supported by National Natural Science Foundation of China --Innovative Research Groups (51021002)

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