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STUDY ON THE DENSIFICATION OF ALN-TIB$_2$ COMPOSITES BY HOT PRESSED SINTERING

Abstract. *The AlN-TiB$_2$ composites were prepared by hot pressing sintering process and the effects of TiB$_2$ content and sintering process on the densification of AlN-TiB$_2$ composites were studied. The results showed that at 1900℃ for 1h, the relative density reached more than 98.6% and the TiB$_2$ content had no effect on densification of the composites. The phase composition and microstructure of the composites were also studied. Two-phase BN and TiN are newly formed in the sintering process of AlN-TiB2 multiphase material, so the multiphase material is composed of four phases, the primary crystal phase is AlN and TiB2, and the secondary crystal phase is BN and TiN. With the increase of sintering temperature and the extension of holding time, the density of AlN-TiB2 multiphase materials gradually increases. The optimum sintering temperature and holding time were 1900℃ and 1h respectively. The addition of TiB2 does not affect the sintering of multiphase materials.The multiphase materials have achieved high density.*

Keywords: aluminum nitride; titanium boride; hot pressed sintering; densification

Introduction

Aluminum nitride has unique physical and electrical properties. It can be compounded with a variety of matrix materials to produce composite materials with excellent electrical, thermal and mechanical properties, and has a broad application prospect in the field of microelectronics technology [1; 2]. Titanium boride has the characteristics of high melting point and electric conductivity [3]. Therefore, it is of great significance for developing new electronic materials of AlN-TiB$_2$ system to study the relationship between the process, structure and properties of AlN-TiB$_2$ system multiphase materials by adopting the design principle of multiphase materials and combining the material properties of aluminum nitride and titanium boride [12].

Microwave attenuation properties of AlN-TiB$_2$ system multiphase materials have been studied [4], but few studies on densification have been reported publicly. Therefore, in this paper, AlN and TiB$_2$ were used as raw materials, mixed by ball milling, and hot pressing sintering process was adopted to study the influence of TiB$_2$ content, sintering temperature and holding time on the densification of AlN-TiB$_2$ composite materials.

Experimental Part

Preparation of AlN-TiB$_2$ composites materials

Using AlN and TiB2 as raw materials, Y_2O_3 as sintering agent, zirconia ball as grinding ball, anhydrous ethanol as grinding medium, in the high-energy planetary mill ball grinding for 4h, and then the material drying, into the graphite mold, placed in the hot pressing furnace, AlN-TiB$_2$ multiphase materials were prepared in argon (purity ≥ 99%) atmosphere, 30 MPa pressure, different sintering temperature and holding time [11].

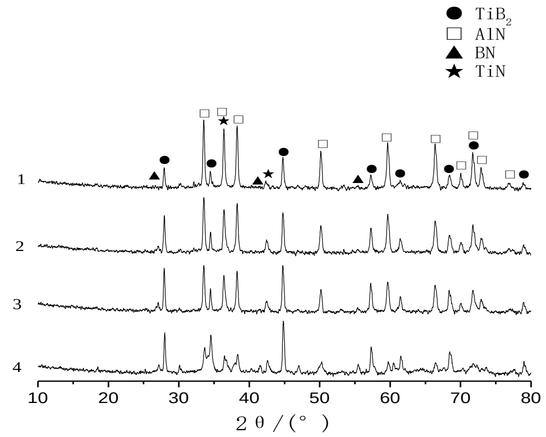
Analysis Methods

The sample density was measured by Archimede method, and the relative density was calculated according to the theoretical density. Swiss ARL X 'TRA X-ray diffractometer was used to analyze the phase of the samples. The microstructure of sample section was observed by JSM-5900 scanning electron microscope of JEOL, Japan.

Results and Discussion

Phase composition analysis

Studies have shown that the phase composition of materials has a crucial influence on the properties of materials [5 – 10]. Therefore, the analysis of phase composition of AlN-TiB$_2$ composite is one of the key factors affecting the densification of the composite. Figure 1 shows the X-ray diffraction of multiphase materials with different TiB$_2$ contents. As can be seen from the figure, the multiphase material contains not only AlN and TiB$_2$ phases, but also a small amount of BN and TiN phases, which increase with the increase of TiB$_2$ content. This is because a small amount of Oxidized B_2O_3 and TiO_2 in TiB$_2$ raw material react with AlN to form BN and TiN. Therefore, the AlN-TiB$_2$ multiphase material is composed of four phases, with AlN and TiB$_2$ as the primary crystal phase and BN and TiN as the secondary crystal phase.



1-10wt% TiB_2 2-25wt% TiB_2 3-35wt% TiB_2 4-50wt% TiB_2

Figure – 1 XRD patterns of composites with different contents of TiB_2

Densification analysis

Influence of sintering temperature

Fig. 2 shows the relationship between sintering temperature and the relative density of AlN-10wt%TiB_2 composite. As can be seen from the figure, with the increase of sintering temperature, the relative density of the multiphase material increases gradually. This indicates that the densification of AlN-TiB_2 multiphase materials increases with the increase of temperature, and the sintering temperature has an important influence on the densification performance of the multiphase materials. When the temperature reaches 1900℃, the densification properties of the composite materials will not change significantly when the temperature increases. This indicates that increasing the sintering temperature below 1900℃ is beneficial to the densification properties of materials, but when the sintering temperature exceeds 1900℃, increasing the sintering temperature does not have an obvious promoting effect on the densification properties.

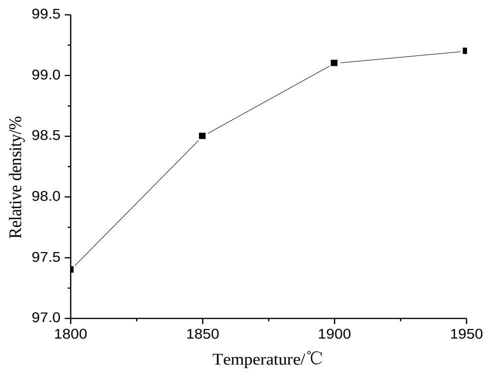


Figure 2 – The curve of relative density of AlN-10wt%TiB_2 composite with temperature

Influence of holding time

Fig. 3 is the curve of relation between holding time and relative density of AlN-10wt%TiB_2 composite. It can be seen from the figure that with the increase of holding time, the relative density of the multiphase material increases gradually. When holding time reaches 1h, the multiphase material reaches a good density, and the sintering performance of the material does not change significantly with the increase of time. This indicates that a certain holding time is necessary for sintering of AlN-TiB_2 multiphase materials, but when holding time exceeds 1h, the increase of holding time has no obvious effect on sintering properties.

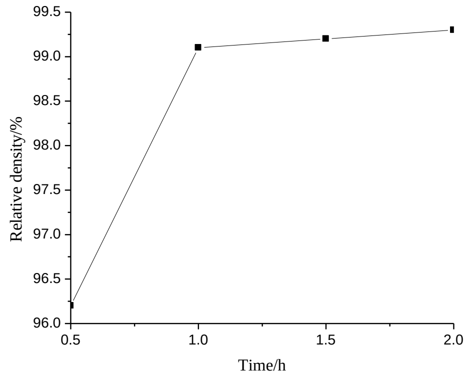


Figure 3 – The curve of relative density of AlN-10wt%TiB2 composite with time

Influence of TiB_2 content

Fig. 4 is the curve of relation between TiB_2 content and relative density of AlN-TiB_2 composite materials. As can be seen from Figure 4, with the increase of TiB_2 content, the relative density of the multiphase material is between 99.0% and 99.1%. This indicates that the addition of TiB_2 does not affect the sintering of AlN-TiB_2 multiphase materials, and the multiphase materials have reached a high density.

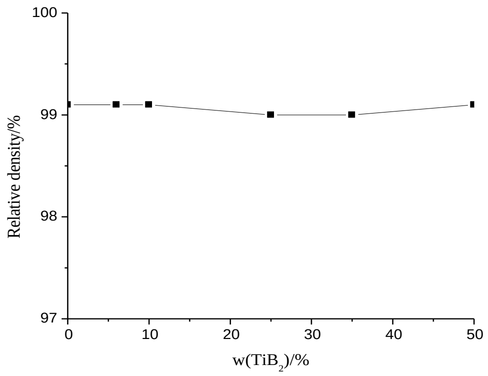
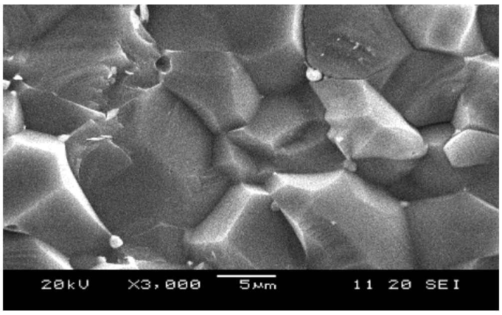
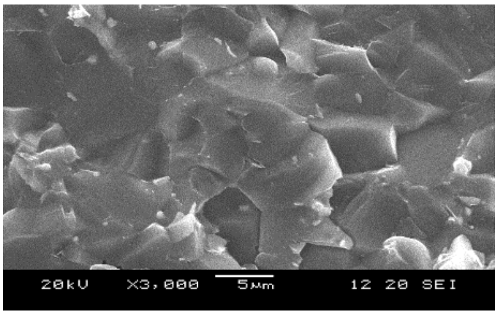


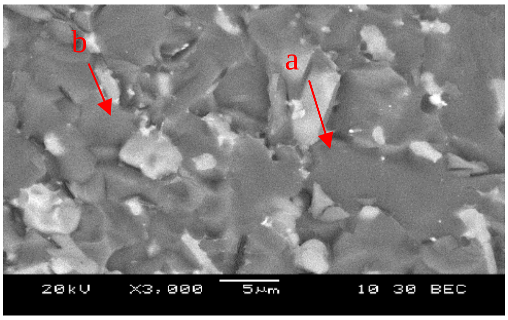
Figure 4 – The curve of relative density of AlN-TiB_2 composite with contents of TiB_2



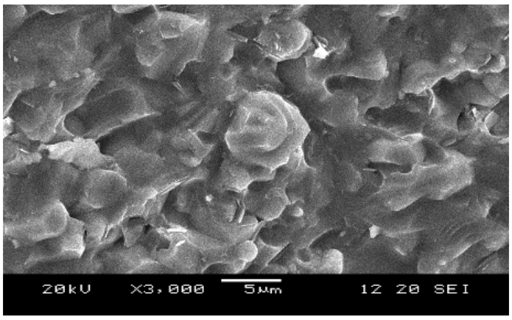
(1) 0wt%TiB_2



(2) 10wt%TiB_2



(3) 25wt%TiB_2



(4)50wt% TiB_2

Figure 5 – SEM of composites with different contents of TiB_2

Fig. 5 is the SEM diagram of sections of multiphase materials with different TiB_2 contents. As can be seen from the figure, the compatibility between AlN and TiB_2 is good, the grains are closely bonded, and only a few pores exist. This indicates that the presence of TiB_2 does not hinder the sintering of the multiphase materials, and the multiphase materials have achieved good densification, which is consistent with the results measured in FIG. 4. EDS analysis of A and B in Fig. 5 (3) was performed (see Fig. 6 and Fig. 7). The results showed that the bright white area was TiB_2, a small amount of Au was introduced when spraying Au, and the dark area was AlN.

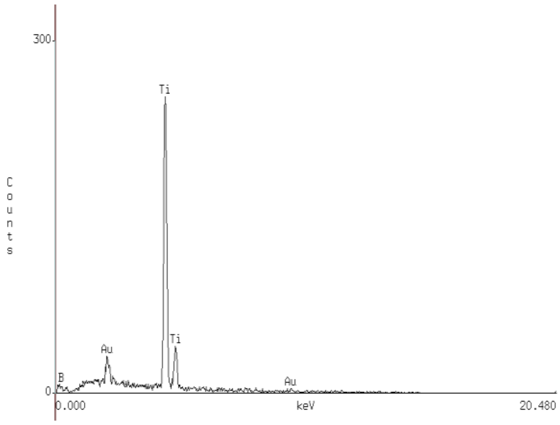


Figure 7 – EDS analysis in Fig. 5 (3) b

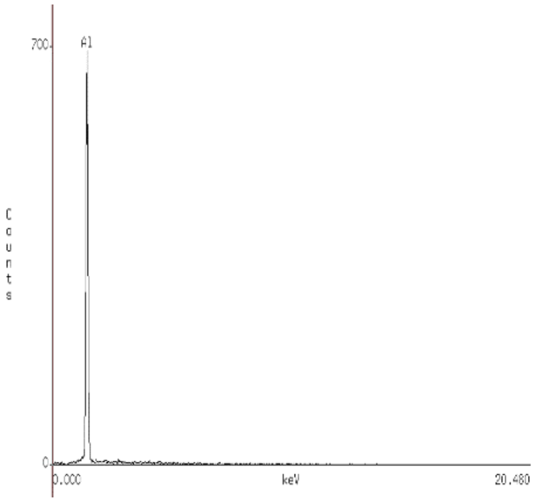


Figure 6 – EDS analysis in Fig. 5 (3) a

Conclusions

1) Two-phase BN and TiN are newly formed in the sintering process of AlN-TiB2 multiphase material, so the multiphase material is composed of four phases, the primary crystal phase is AlN and TiB2, and the secondary crystal phase is BN and TiN.

2) With the increase of sintering temperature and the extension of holding time, the density of AlN-TiB2 multiphase materials gradually increases. The optimum sintering temperature and holding time were 1900℃ and 1h respectively.

3) The addition of TiB2 does not affect the sintering of multiphase materials.The multiphase materials have achieved high density.

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ДОСЛІДЖЕННЯ УЩІЛЬНЕННЯ КОМПОЗИТІВ ALN-TIB2 МЕТОДОМ ГАРЯЧОГО СПІКАННЯ

Анотація. *Композити AlN-TiB2 були отримані методом спікання гарячим пресуванням, досліджено вплив вмісту TiB2 та процесу спікання на ущільнення композитів AlN-TiB2. Результати показали, що при 1900℃ протягом 1 год відносна густина досягала понад 98,6% і вміст TiB2 не впливав на процес ущільнення композитів. Також досліджено фазовий склад і мікроструктуру композитів. Двофазні BN і TiN новоутворюються в процесі спікання багатофазного матеріалу AlN-TiB2, тому багатофазний матеріал складається з чотирьох фаз, первинна кристалічна фаза - AlN і TiB2, а вторинна кристалічна фаза – BN і TiN. З підвищенням температури спікання і подовженням часу витримки щільність багатофазних матеріалів AlN-TiB2 поступово зростає. Оптимальна температура спікання та час витримки становили 1900 ℃ та 1 год відповідно. Додавання TiB2 не впливає на спікання багатофазних матеріалів. Багатофазні матеріали досягли і набули високої щільності.*

Ключові слова: нітрид алюмінію; борид титану; гаряче пресування спікання; ущільнення

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