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Fabrication of Homogenous Dispersion TiB_2-Al_2O_3 Composites

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Abstract: In order to get a homogenous mixture and compact of TiB_2-Al_2O_3, hybridization as a surface modification method was used to prepare nano-scale Al_2O_3 coated TiB_2 particles. PE-wax particles were first coated onto TiB_2 particles by hybridization, and then the nano-scale Al_2O_3 particles were coated onto the surface of TiB_2 coated by PE-wax particles again. SEM, TEM and EDS were used to characterize the microstructure of as-received core/shell particles and its compacts. The experimental results show that a particle-scale homogenous dispersion of TiB_2 and Al_2O_3 can be formed not only in mixed powder but also in dewaxed compacts. The compacts then were sintered by gas-pressing sintering (GPS). Finial products show improved mechanic properties comparing with reference samples fabricated by normal ways.

Key words: TiB_2/Al_2O_3; hybridization; dispersion

1 Introduction

Titanium diboride (TiB_2) is a ceramic material with excellent potential properties: high melting point, high hardness and good electrical conductivities. Such unique properties give TiB_2 materials wide applications, such as high temperature electrodes, wear components, mechanical seals, aerospace parts, cutting tools and Hall-Heroult cell cathodes[1,2]. However, its applicability as a structural material is still restricted for its brittleness, poor sinterability and anisotropic thermal expansion. Compositing is a possible way of avoiding some of these problems[3,4]. It has been reported that adding metals (such as Fe[5], Ni[6] *etc*) and second ceramics phase[7] could improve the poor sinterability and brittleness of TiB_2. Though so many works have been done on it, it is still far away from TiB_2 and its composites' application as main structural parts. There are many reasons for it. One important reason is discreteness of mechanical properties of TiB_2 and its composites. The discreteness comes form two way, ceramics nature and heterogeneous microstructure. The later one is also one critical problem that many researchers try and tried to overcome. Their results show that an effective mixing way can help to solve this problem.

Hybridization system developed by Koishi *et al* [8] has been concerned in many fields such as drugs, cosmetics and toners, for it is a method to prepare complexes of organic and inorganic materials in a solvent-free system by a mechanical shock process involving dry blending and dry impact blending. Honda and Koishi have reported that adhesion and embedding of fine particles onto other large particle surfaces were often observed in the dry blending of binary powder mixtures with two different sizes, and that the adhesion among particles was due to Van der Waals interaction. Our previous works[9] has proved that nano-scale Al_2O_3 particles could be coated on the several-micrometer TiB_2 particle. If every TiB_2 particle can be enwrapped by an Al_2O_3 layer, whose thickness is linear to the TiB_2 particle size, it is obvious that the TiB_2 particle-scale homogenous dispersion is achieved.

Based on the above assumption, hybridization, a surface modification method, was used here to mix several-micrometer TiB_2 and nano-scale Al_2O_3. The microstructure characters of as-received core/shell particles and its stability during post powder metallurgy process were studied in detail.

2 Experimental

Titanium diboride was synthesized by self-propagating high temperature synthesis (SHS) method was our lab[10]. Alumina (Sumitomo Chemical) powder was commercial one. The properties of the as-received powders are shown in Table 1. The SEM images of TiB_2 and Al_2O_3 powders used are shown in Fig.1 (a) and (b) respectively. Transition layer used was spherical polyethylene wax $–[CH_2–CH_2]_n–$ (PE-wax), and 1 μm in mean particle size. Its softening temperature is 385K. Hybridization was carried out in air and the process details are same with the Ref.[9].

Considering the average particle size of alumina is about 100 nm, a 50 nm-thickness PE-wax and 100

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Table 1 Powder characteristics of the titanium diboride and alumina

Powder characters	Titanium diboride (TiB_2)	Alumina (Al_2O_3)
Manufacturer type	SHS by ourselves	Sumitomo AKP-G008
Particle diameter (d_{50})	5.8 μm	80 nm

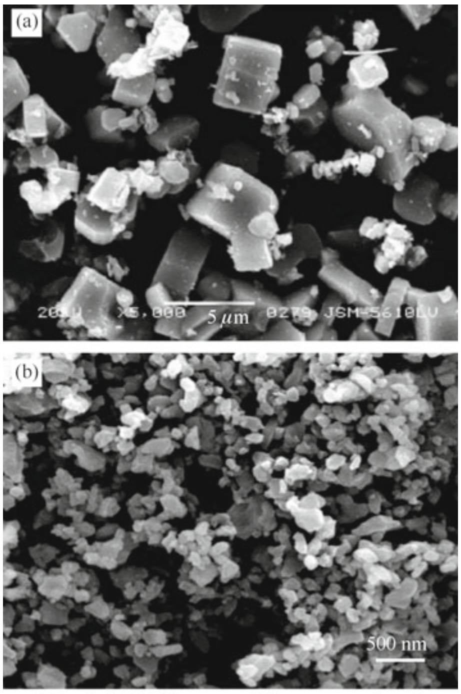

Fig.1 SEM images of the as-received powders TiB_2; (b) Al_2O_3

nm-thichness alumina are expected. Of course there will be some loss of the initial components and the initial shape of TiB_2 also will have some effects, the volume ratio of TiB_2: PE-wax: Al_2O_3 is set as 100:5:15.

A normal scan electron microscope (SEM) of JSM-5610LV and A field emission scanning electron microscope (FESEM) of FEI-Siron 200 were used to observe the surface morphology and microstructure of the mixed powder. The energy dispersive X-ray spectrum (EDS) and X-ray diffraction (XRD) were used to confirm the surface information of the core/shell particles. The particle size distribution was determined by a laser particle size analyzer.

3 Results and Discussion

It is known that the impact force and shearing force, which depends on speed of rotor rotation, are the main drivers of PE-wax coating on TiB_2 [9]. A higher rotation speed of 15,000 r/min and 10 min duration were used here to make PE-wax have enough mobility and form proper film with small volume content of PE-wax and alumina.

Fig.2a shows the representative FESEM images of as-received powder. Almost no plate-like TiB_2 particles had been found in the as-received powders. Main particles in the mixture are nearly sphere and

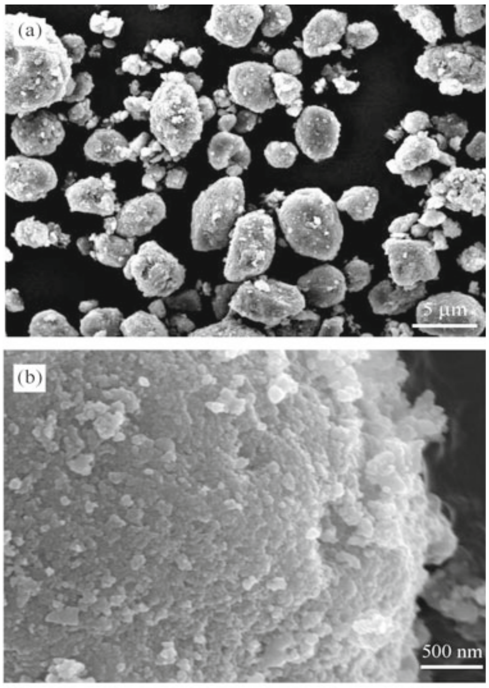

Fig.2 FESEM images of (a) as-received powder and (b) surface of single core/shell particle

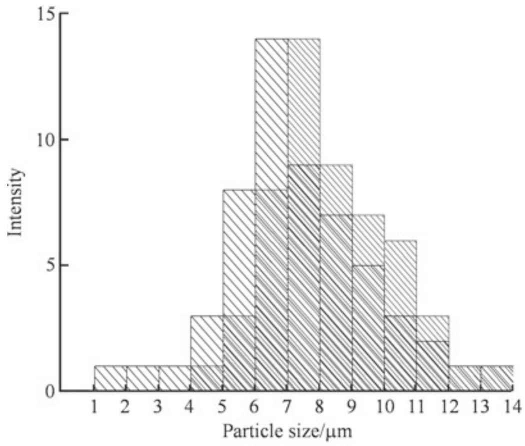

Fig.3 Particle size distribution of as-received and initial TiB_2

have smooth curved surface (shown as Fig.2b). Results also show that a longer duration can make the surface more smooth and spherical. Comparing the particles size in the surface with that of initial components, the surface must be PE-wax and Al_2O_3. EDS results also support it.

The as-received particle size distribution was measured by a laser particle size analyzer. Because the PE-wax can not dissolve in water at room temperature, distilled water was used as the liquid media. Results are shown in Fig.3. The red bar is that of as-received powder and the blue bar is that of initial TiB_2 powder. If there are some very small particles exiting, such as nano Al_2O_3, their content must be very small and almost can not be found in the SEM images and measured particle size distribution. The average particle size (6.3 μm) of as-received powder is a little bigger than that of initial TiB_2 particle (5.8 μm). One of the reasons is the distribution of as-received powder becomes narrow. The bigger-particle part keeps no change and the small-particle part shift to the bigger one. It may means there are some small TiB_2 particles have been bonded together, but for bigger TiB_2 particles there must be only one in the mixed particle. The other reason is the coated BE-wax and Al_2O_3 layer on TiB_2 particle. The average height of the coated layer about 100-200 nm can be deduced from the change.

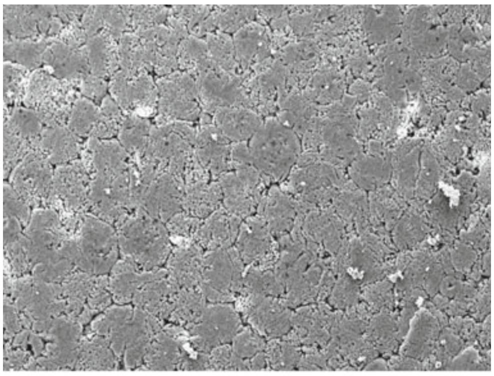

Fig.4 SEM image of surface of pre-pressed and dewaxed specimen

The stability of as-received particle was also investigated here. The as-received powder was putted into a steel die and pressured with a pressure of 50 MPa. Discs with a diameter of 50 mm and a height of 20 mm were gained. Then these discs were placed in a vacuum furnace and the dewax time and temperature were 12 h and 800 ℃ respectively. Fig.4 shows the SEM image of dewaxed specimen. The shape of deformed as-received particle is still clear. That is to say the Al_2O_3/PE-wax/TiB_2 core/shell particles have enough stability before sintering. Fig.5 shows microstructure of the sintered discs. The dark area is TiB_2 and the grey one is Al_2O_3. Because the sintering temperature is 1 550 ℃, far away from the TiB_2 minimum sintering temperature (1 750 ℃)[11], the TiB_2 does not grow while Al_2O_3 grows obviously. These sintering behaviors will destroy the Al_2O_3 enwrapped TiB_2 structure. However the dispersion of two components still keeps well.

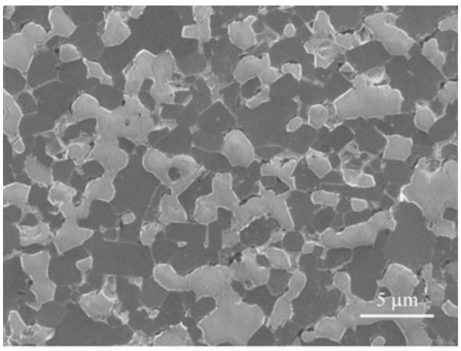

Fig.5 SEM image of microstructure of sintered disc

The bending strength and Rockwell hardness of sintered specimens were also measured. Every specimen was cut to six standard bars and three specimens were used to measure the bending strength and hardness are measure on every bars for 5 times. Statistical bending strength and Rockwell hardness are 523±41 MPa and 92.2±0.1 respectively. Comparing 463±87 MPa and 91.4±1.4 of specimens fabricated by normal way, not only the properties has a slight improvement, the discreteness of the value decreases obviously.

4 Conclusion

Hybridization, a surface modification method, was used to make dispersion well several micrometers TiB_2 / nano-scale Al_2O_3 mixture. PE-wax particles were first coated onto TiB_2 particles to work as a transition layer and binder. Results show that a 100-200 nm Al_2O_3 layer can be coated onto the surface of TiB_2 and form a core/shell mixture powder. The core/shell particles have a good stability and can be kept in post metallurgy processes and help to achieve dispersion well TiB_2/Al_2O_3 composites. The bending strength and Rockwell hardness of sintered specimens with the special powder are 523±41 MPa and 92.2±0.1 respectively. Comparing 463±87 MPa and 91.4±1.4 of specimens fabricated by normal way, not only the properties has a slight improvement, the discreteness of the value decreases obviously.

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