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Sintering of TiB_2-Al composites using HP-HT method

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ABSTRACT

Purpose: The subject of the work was to study the effect of the sintering temperature on the properties and structure of TiB_2-Al composites.

Design/methodology/approach: TiB_2-Al composites reinforced with 70 vol.% ceramic particles were prepared by powder metallurgy method. The high pressure - high temperature (HP-HT) method was employed to consolidate the sinters. Composites were sintered at pressure of 7.0 ± 0.2 GPa and temperatures of 520°C and 600°C. The duration of sintering was 60 seconds. In order to investigate the structure changes, the scanning electron microscope was applied. Young modulus measurements were carried out using ultrasonic method. Mechanical properties were determined by Vickers hardness tester.

Findings: Two variants of TiB_2-Al with respect to the sintering temperature (520°C and 600°C) were obtained by the HP-HT method. The application of the higher temperature of 600°C and pressure of 7.0 ± 0.2 GPa and time of 60 seconds permits to obtain the higher properties of this composites in comparison with the sinters which were obtained at 520°C.

Practical implications: From a practical position it is important to optimize the sintering densification of TiB_2-Al composites by high pressure -high temperature (HP-HT) method.

Originality/value: The results from this work can be useful in determining conditions for sintering the materials with the high amount of titanium diboride.

Keywords: Manufacturing; Aluminium; TiB_2 ceramic; Composites; Sintering; HP–HT technique

MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Among various ceramic particulates, titanium diboride (TiB_2) is expected to be one of the best reinforcements for composites due to its high hardness (3400 HV), high melting temperature (3225 °C), low density (4.451 g/cm^3), high electrical conductivity, good chemical stability, good corrosion resistance and outstanding tribological properties [1,2]. Literature [3] showed that sintering densification of TiB_2 is very difficult. In the pressureless sintering processing, the sintering temperature of pure TiB_2 is higher than 2200°C and the density of sintered materials is not more than 95% of the theoretical density. The hot pressing sintering processing has been considered as an effective candidate sintering processing for TiB_2 ceramics. The main features of hot pressing sintering include lower sintering temperature, high sintering speed, and a uniform microstructure of sintered materials. Studies showed that hot pressing increased the density of TiB_2 ceramics significantly [3-6]. Sulima *et al.* [7] produced the titanium diboride ceramics by the high pressure - high temperature (HP-HT) technique. In case of the HP-HT method, simultaneous action of pressure and temperature influences the short duration (only a few minutes) in comparison with the free sintering which progress for even a dozen hours. It is worth emphasizing, that the TiB_2 ceramics were sintered without the use of sintering agents. The application of the temperature of 1500ºC±50ºC and pressure of 7.2 ± 0.2 GPa and time of 60 seconds permits to obtain the TiB_2 ceramics without cracks. The obtained sinters were characterized by very high density and isotropy of properties.

Titanium diboride characterized by many advantages in comparison with traditional ceramic particulate reinforcements such as silicon carbide (SiC) or alumina (Al_2O_3). For example, SiC particles react with liquid aluminium to form a reaction layer at the reinforcement-matrix interface. The brittle reaction product (Al_4C_3) reduces the mechanical properties of the composite [8]. However, TiB_2 particles are thermodynamically stable in liquid aluminium [9]. Thus, the addition of TiB_2 to a metal matrix can greatly improve strength, hardness, and wear resistance in comparison with other ceramic reinforcements [10,11]. Several studies were reported on the use of the various techniques for the fabrication of aluminium-TiB_2 composites such as the conventional powder metallurgy [12]], the hot isostatic pressing (HIP) [13] and in situ processes [14,15]. This paper reports on the preparation, properties and structure of TiB_2-Al composite produced by high pressure -high temperature (HP-HT) method of the sintering [16].

2. Experimental procedure

In the present study, TiB_2-Al composites were fabricated with the titanium diboride powders (Atlantic Equipment Engineers, below 10 μm average grain size, purity 99.9%) and Al powders (Benda-Lutz, about 100 μm average grain size, purity 99.7%). Initial phase composition of mixtures for the samples preparation were as follow 70vol.% TiB_2 + 30vol.% Al.

The powder mixtures were formed into discs (15 mm in diameter, 5 mm high) by pressing in a steel matrix under pressure of 200 MPa. Samples were heated using a ceramics gasket provided with an internal graphite heater. For the densification of composites of the powder the high pressure-high temperature (HP-HT) Bridgman type apparatus was used. Compacts were obtained at pressure of 7.0 ± 0.2 GPa and at temperatures of 520 °C and 600°C. The samples were HP-HT sintered for 60 seconds.

Density was measured by hydrostatic method. Uncertainty of measurements was no more than 0.02 g/cm^3 which gave us a relative value of error below 0.5 %.

Young's moduli of the samples obtained by the HP-HT sintering were measured basing on the velocity of the ultrasonic waves transition through the sample using ultrasonic flaw detector Panametrics Epoch III. The accuracy of calculated Young's modulus was estimated at 2 %.

The samples for Vickers hardness measurements and microstructure analysis were prepared through lapping on a cast iron plate with diamond paste. The Vickers microhardness studies were carried out using FM-7 microhardness tester. The applied load was 0.98 N.

The chemical characterisation of TiB_2-Al composites was carried out by X-ray diffraction using Cu K_α radiation and by energy dispersive X-ray microanalyser (EDS). The microstructures were observed using JEOL ISM-6460 LV scanning electron microscopy (SEM with an accelerating voltage of 30 kV).

3. Results and discussion

The results of the HP-HT sintering process were presented for TiB_2-Al compacts for the temperature of 520°C and 600°C, respectively. The selected physical and mechanical properties of the TiB_2-Al composites are given in Table 1 and Table 2.

Table 1.
Selected physical and mechanical properties of the TiB_2–Al composites (70vol.%TiB_2 + 30vol.%Al) obtained by the HP-HT method

compacts	T [°C]	Density (R_o) [g/cm^3]	R_o/R_{Teor}	Poisson's ratio
TiB_2-Al (1)	520	3.84	97	0.20
TiB_2-Al (2)	600	3.94	100	0.19

Table 2.
Selected physical and mechanical properties of the TiB_2–Al composites (70vol.%TiB_2 + 30vol.%Al) obtained by the HP-HT method

compacts	T [°C]	Young's modulus E [GPa]	$\frac{E}{E_0}$ [%]	HV1
TiB_2-Al (1)	520	171	42	200
TiB_2-Al (2)	600	194	48	300

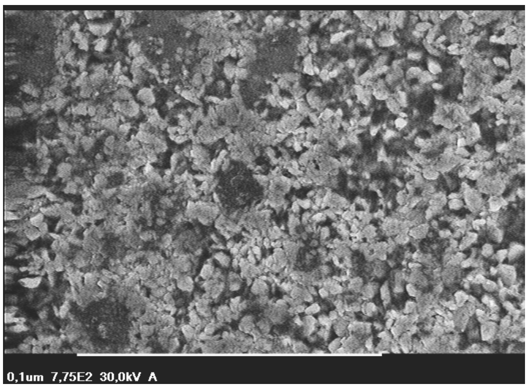
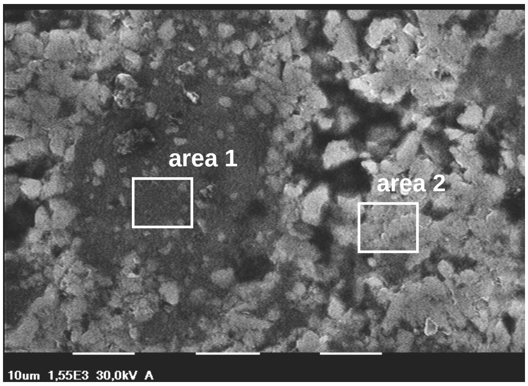


Fig. 1. Microstructure of the Al-TiB_2 composite (sample 1) after the sintering at temperature of 520°C

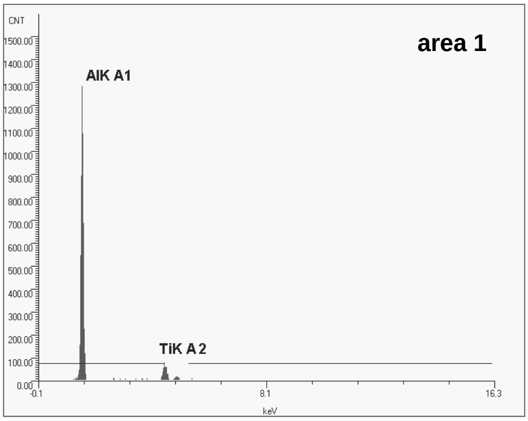
The TiB_2-Al specimens which were sintered at the temperature of 520°C reached density of 3.84 g/cm^3 corresponding to 97% of theoretical density (3.94 g/cm^3). However, the Young's modulus, Poisson's ratio and Vickers hardness for these ceramics were 171 GPa and 0.20 and about 200 HV1, respectively. Figures 1 and 2 illustrate the microstructure of this composite. The XRD analysis indicated the presence only of TiB_2 and aluminium in sintered TiB_2-

Al composites (Fig. 3). The results of the examinations indicated that the microstructure was characterized by non-homogenous distribution of the aluminium in the composite (dark areas on Fig. 1 and 2a.,b). Additionally, small TiB_2 grains were observed in the aluminium area. However, in the grey areas the grains (Fig. 2a,c) composed mainly of TiB_2 phase were located.

a)



b)



c)

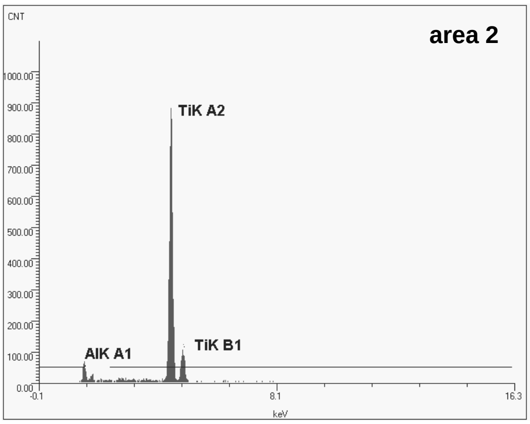


Fig. 2. a) SEM microstructure of the TiB_2–Al composite (sample 1) after sintering at temperature of 520°C and EDS analysis corresponding: b) area scan 1, c) area scans 2

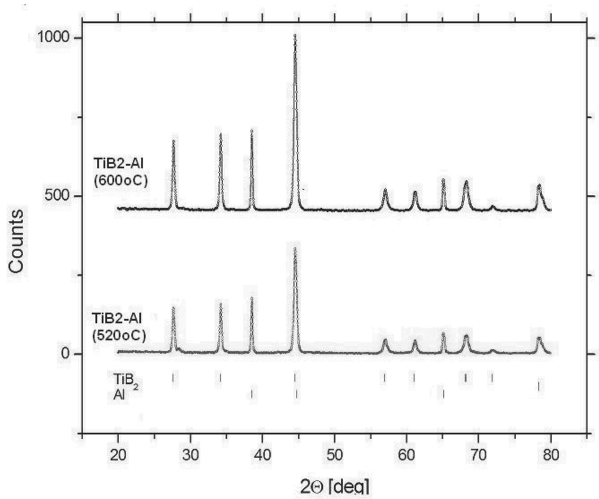


Fig. 3. X – ray diffraction pattern of TiB_2-Al composites obtained by the HP-HT method

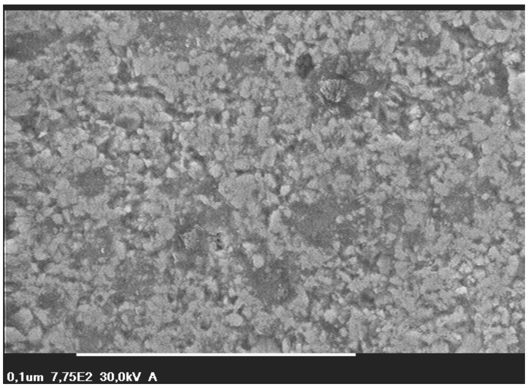


Fig. 4. Microstructure of the TiB_2-Al composite (sample 2) after sintering at temperature of 600°C

In the case of the TiB_2-Al composite which was sintered at the higher temperature of 600°C, the density received value of 3,94 g/cm^3 (Tab.1). This value corresponds to 100% of the theoretical density (3.94 g/cm^3). Moreover, the Young's modulus is 194 GPa, Poisson's ratio is 0.19. Average Vickers hardness is about 300 HV1 for this composite. Generally, the higher properties were obtained at the higher temperature. Figures 4 and 5 show the typical microstructure of TiB_2-Al composite after sintering at the temperature of 600°C. In principle, this microstructure is similar to the microstructure of the composite obtained at the temperature of 520°C. However, microstructural investigations indicated that the higher temperature causes more homogenous distribution of the aluminium in the composite (dark areas on Figs. 4 and 5a,b) in comparison with the compacts which were obtained at 520°C (dark areas on Fig. 2). Also, in aluminium area some small TiB_2 grains were observed. Moreover, SEM and XRD studies allowed to identify TiB_2 phase as grey phase areas grains in Figures 4 and 5a,b.

a)

b)

c)

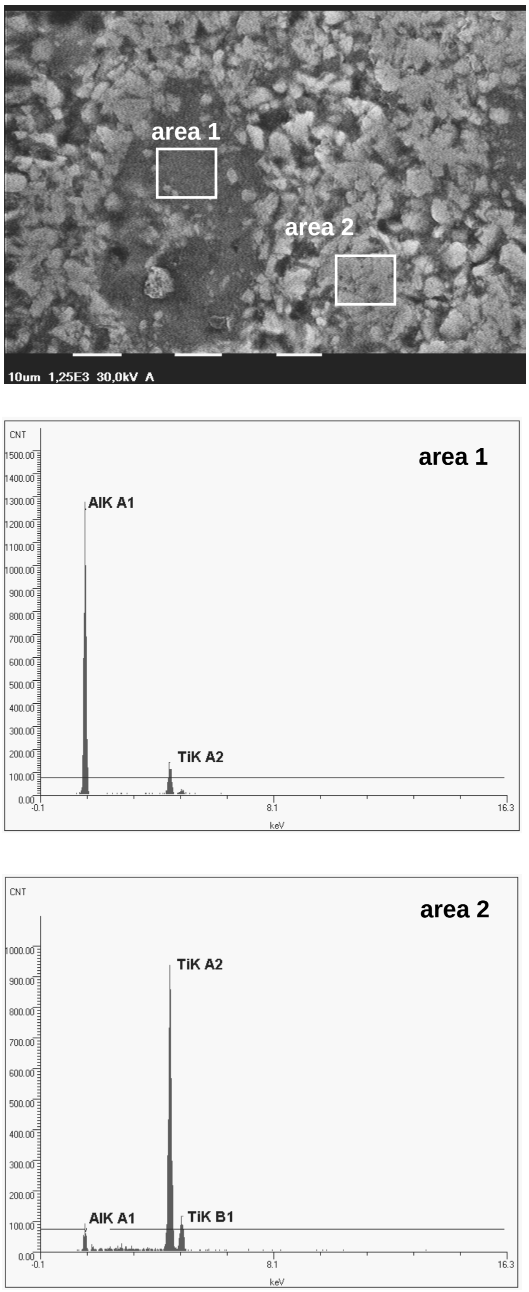


Fig. 5. a) SEM microstructure of the TiB_2-Al composite (sample 2) after sintering at temperature of 600°C and EDS analysis corresponding: b) area scan 1, c) area scans 2

4. Conclusions

The TiB_2-Al composites with initial content of TiB_2 phase equal to 70vol.% were obtained by the HP-HT method. The examinations indicated that the properties and microstructure of TiB_2Al composites depends on temperature of sintering process. Application of the higher temperature of 600°C resulted in the higher properties and more homogeneous microstructure of the TiB_2-Al composites. Materials are characterised high level of consolidation, R_o/R_{toer} for materials sintered at 600°C is 100%.

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