The Aluminum Based Composite Produced by Self Propagating High Temperature Synthesis

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Self-propagating high-temperature synthesis method can be used for producing aluminum and boron carbide based composites. The experimental composites were fabricated using cobalt and carbon as catalysts. The microstructure of the material was studied using Scanning Electron Microscopy and the mechanical properties were determined using micro-hardness testing. Al/B_4C based composites with improved properties were obtained and the role of Co/C catalysts was studied.

Keywords: aluminum, boron carbide, composite, SHS process, catalysts.

1. INTRODUCTION

At present time many compounds have been produced by Self-propagating High-temperature Synthesis (SHS) process [1]. Boron Carbide (B_4C) is a hard material (9.5 + in Mohs scale) and low specific gravity (2.52 g/cm^3). By using additives (e.g., graphite, cobalt) B_4C sintered at high temperatures (> 2000 °C) can produce microstructures with a high density (~ 98.2 % of theoretical density) [2]. Full density is usually achieved through hot pressing technique however, even in a fully dense form, the sensitivity of boron carbide to brittle fracture remains a major limitation. SHS method has a very wide range of applications [3]. It has been used to produce a wide variety of materials including carbides, catalysts and carriers characterised by high activity and selectivity with high physical and mechanical properties [1, 2].

SHS is a relatively novel and simple method for making certain advanced ceramics, composites and intermetallic compounds. This method has received considerable attention as an alternative to the conventional furnace technology. SHS process is based on a system that involves an exothermic combustion. High purity of the product obtained, the possibility to obtain a metastable phase, and the possibility of simultaneous synthesis and densification. Higher purity of the product is a consequence of the high temperatures associated with combustion and the volatile impurities removed as the wave propagates through the sample. Based on the heat released from the reaction of metal powder (fuel) with oxygen (oxidizer) in the presence of other metal oxides hence, after the initial ignition by an external heat source, the reaction is able to regenerate itself while the resulting high temperature sufficient for the synthesis of the desired ceramic product [3, 4]. Al, with Boron carbide and the use of a catalyst is to improve the properties of composite materials. This study compared the use of carbon and cobalt as catalysts on Al composites reinforced by boron carbides. Hardness testing and tracking microstructure by SEM is used to obtain the value of the comparison of the SHS process. In our study the improvement of B_4C-Al composite properties was received introducing of Cobalt and metal to initial powder before milling as catalyst.

2. EXPERIMENTAL DETAILS

Aluminum powder with average particle size 100 μm was received from Aluminum Powder Company Ltd, AlPoCo. Boron carbide had particle size in the range of 17.2 to 74.5 μm and with a mean of 17.2 μm. Al and B_4C powder mixture was mechanically activated by attrition mill. The grain size after attrition milling was The SHS process decreased to ~ 400 nm. The volume fractions used in the composite without catalyst was 50:50 % and volume fraction for composite with catalyst was 48% Al, 45% B_4C, 5% Co and 2% C.

Powder composite was milling by attritor for 5 hours. The powder mixtures were densified by vibration and compacted in the steel container. The SHS-process ignition temperature was around 800 ± 20 °C and then increased during the SHS process up to 1100 ± 20 °C for the used powders content [5]. The heating rate at SHS was higher than attrition mechanical processes. After process the capsules were immediately pressed the heating, then under normal pressure under stress of 150 MPa as shown in Fig. 1.

After SHS, the green samples were removed from the steel container. HV_{10} hardness is measured. Tabletop Scanning Electron Microscope TM-100 Hitachi was used to obtain the phase distribution. Image-J software was used for quantitative microstructure. Density measurements were performed by Archimedes method using Mettler Toledo ME 204.

3. RESULTS AND DISCUSSION

SHS is process that begins at a temperature of 800 to 1200 °C and resulted to composite Al/B_4C forming. SHS

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with temperatures below 1000 °C cannot be achieved by sintering technique without pressure, because the contact angle between Al and B_4C is not obtained in a short time. Densification hampered caused by chemical reactions occur faster than the reaction of capillarity between Al with B_4C. The pressing at 150 MPa which is react form result the powder of rearrangement.

However Halverson [1] obtained a low contact angle in a short amount of time because the vapor pressure of aluminum also increases with increasing of temperature. Then the dynamic nature of the contact angle of aluminum on B_4C will result in mass transfer across the solid-liquid phase interface. Because the system is moving toward a state of chemical equilibrium, B_4C and Al react to form composite (see Eq. 1).

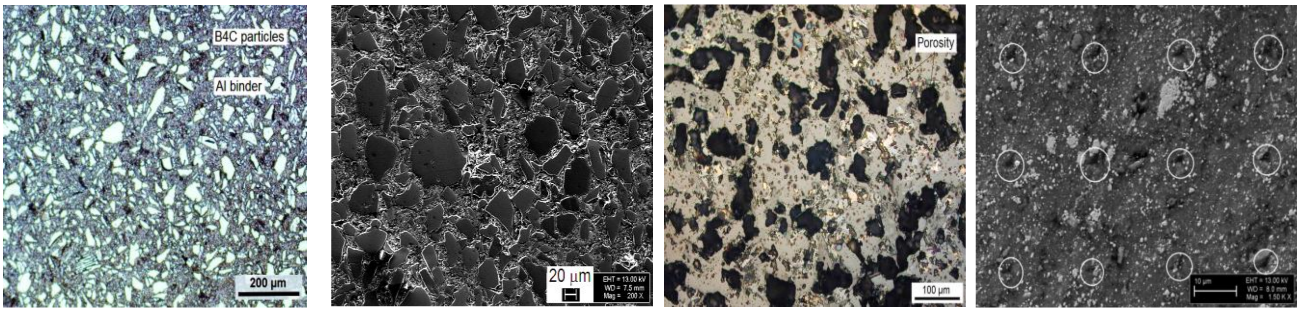
$$3B_4C + 5\,Al \rightarrow AlB_{12} + Al_4C_3. \tag{1}$$

Fig. 1. Stage of SHS Process: preparation of materials, attritor milling and hydraulic pres

Xanthopoulou [2] used Co to increase the activity of catalysts such as the improvent of the properties of composite materials.

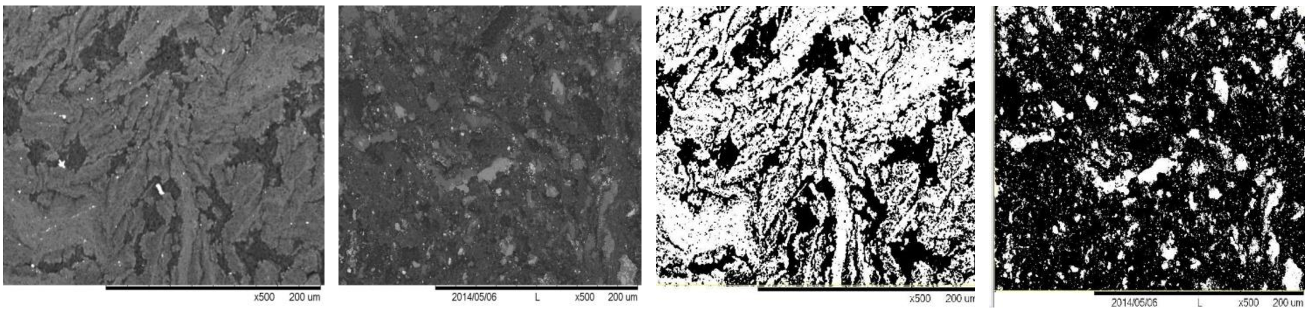
Table 1. Mechanical Properties of Composite base Aluminum (Hardness, Density and Porosity measurement)

Material	Vf (%)	Milling time (h)	Hardness (HV_{10})		Density (g/cm^2)	
					Measurement	Average
Al/B_4C	50:50	5	89.2 88.5 86.3 87.0 86.1	87.4 (σ=1.3)	2.125 2.128 2.146	2.13 (84%)
Al/B_4C Catalysts Co-C	48:45:5:2	5	104.8 102.6 164.7 187.5 179.2	147.7 (σ=41.0)	2.388 *2*.428 2.431	2.42 (82%)



a b c d

Fig. 2. The results of optical and scanning electron microscopy of aluminum-boron carbide based composite: a – SHS processed composite of Al/B_4C without catalyst; b – fine – grained composite Al/B_4C with the Co-C catalyst; c – pores in Al/B_4C composite as result of new phases forming at presence of C, Co-catalysts; d – ultrafine grained composite of Al/B_4C with the Co-C catalyst. The micromechanical properties measurements test points are indicated



a b c d

Fig. 3. The results of Scanning Electron Microscopy of Al based composite: a – composite Al/B_4C without catalyst; b – composite Al/B_4C with the Co-C catalyst; c – the results of measurements of grain extracts Image-J software of the composite Al/B_4C without catalyst; d – the results of measurements of grain extracts Image-J software of the composite Al/B_4C with the Co-C catalyst

When Co is increased even further in the starting material, the excess Co oxide appears in SHS products that alter the overall function of the dehydrogenation catalyst with the high temperatures [6 – 8]. Co and C are used as a catalyst to improve the mechanical properties and the results of the reaction.Cobalt and carbon used as a catalyst to improve the mechanical properties and the results of the reaction shown as in Eq. 2:

$$Al + B_4C + Co + C \rightarrow AlB_4C + 2Co. \quad (2)$$

The use of Co-C as the catalyst is able to improve significantly the mechanical properties such as hardness (see Table 1). Without using Co-C catalyst, the hardness of Al based composite is 87.4HV_{10} but by using Co-C catalyst the hardness improve significantly to 147.7HV_{10}. The improvement of hardness caused by the improvement of fine grains. The size of the grains become more finer due to the reaction of Al and B_4C with the catalysts. If the temperature above 1000 °C is able to decrease the grain size, the mechanical properties will improve. However, the improvement of mechanical properties will decrease the density of Al – based composite as a result of increasing the porosity (Fig. 2). The results of composites mechanical properties are listed in Table 1.

The microstructure evolution depending on attrition milling time and processing stage are presented in Fig. 2. By using particle analysis tools in Image J software, the grain size diameter can be obtained, assumptions the grain area *(A)* is in the spherical form as shown in equation (3) so the particle diameter *(d)* can be obtain.

$$d = \sqrt{\frac{4A}{\pi}}\,. \quad (3)$$

Based on the image processing area, average particle calculated by using the analysis particles on image feature, so the image processing can measure the average diameter calculated using equation (2) and the results are shown in Table 2.

Table 2. The results of the average particle size analysis of the samples with 500x magnification. SEM image J software

Material	Area of particle, μm^2	Particle diameter, μm	Average area of particle, μm^2	Average particle diameter, μm
Al/B4C	47.64 44.27	11.68 7.50	45.95	9.59
Al/B4C Co-C	4.38 3.37	2.36 2.07	3.87	2.21

The grain size of Al-based composite without catalyst measured by Image J sofware is 9.59 μm while using catalyst the grain size becomes finer i.e. 2.21 μm. Catalyst effected the mechanical properties caused the changing of grain morphology from flake to be a round. Morover, it produces more uniform grain. The catalyst is reacted at temperature from 1000 – 1200 °C, so the density reduced to 82 % and it is resulted the increasing of porosity about 16.6 %.

4. CONCLUSIONS

The use of Co-C catalysts in Al-based composite significantly increases the hardness. Initially, the hardness of Al based composite with B_4C as reinforced is about 87.4 Hv_{10} and after the addition of Co-C catalysts the hardness become 147.7 Hv_{10}, furthermore, the grain size become finer. Without using Co-C catalyst the grain size of Al-based composite with B_4C is about 9.59 μm but after adding Co-C catalyst, the grain size become finer i.e. 2.21 μm.

Morphology of grain size aluminium based composites B_4C without catalysts is flake and with catalysts Co-C the morphology is round. Changes in grain structure in high temperature (Co-C at 1000 – 1200 °C) reactions lead to a decrease in density so the porosity increases.

Acknowledgments

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