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Effect of Dry and Wet Ball Milling Process on Critical Powder Loading and Mixture Properties of Fine WC-10Co-0.8VC Powder

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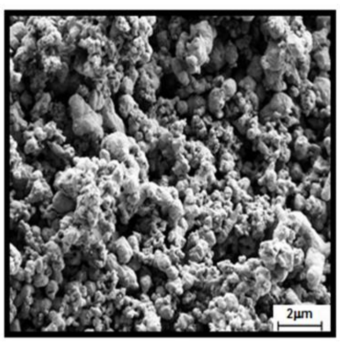
Graphical abstract



Abstract

Micro powder injection molding (µPIM) has great potential for the production of micro cemented carbide parts that require high hardness and toughness. The main stages of the µPIM process include mixing the powder and organic binder, injecting, debinding, and sintering. High critical solid loading of submicron tungsten carbide (WC) powder is one of the requirements in the micro powder injection molding process, which is not obtained easily. This paper investigates the effects of ball milling on critical solid loading of submicron WC. Dry and wet ball milling processes were used to prepare a powder mixture with composition of WC-10Co-0.8VC (wt-%). Critical powder volume concentration (CPVC) was determined using the torque variation method, and the powder characteristics were assessed using scanning electron microscopy and energy dispersive X-ray spectroscopy. CPVC was at 42% and 50% for the dry and wet ball milling processes, respectively. Apparent and tap densities of the powder mixture were achieved at 2.4 g/cm^3 and 2.96 g/cm^3 after dry milling and at 2.54 g/cm^3 and 3.39 g/cm^3 after wet milling, respectively. Wet ball milling causes fine particles to de-agglomerate and improves the critical solid loading, which is advantageous for submicron cemented tungsten carbide injection molding. The homogeneity of the powder mixture can improve under longer time of wet milling process and it can be expected that reduce microstructure defects in sintered components.

Keywords: Ball milling; micro powder injection molding (µPIM); cemented tungsten carbide (WC-Co); mixture physical properties

Abstrak

Proses pengacuanan suntikan serbuk mikro (µPIM) merupakan proses berpotensi tinggi dan mempunyai kelebihan dari segi kos bagi komponen karbida terekat mikro yang berciri kekerasan tinggi. Peringkat penting dalam µPIM merangkumi pencampuran serbuk dan pengikat, pengacuanan suntikan, penyahikatan dan pensinteran. Akan tetapi, muatan serbuk genting yang tinggi bagi serbuk submikron tungsten karbida merupakan kriteria yang sukar. Maka, kesan pengisaran bebola terhadap muatan serbuk genting WC submikron turut dikaji. Pengisaran bebola secara kering dan basah digunakan untuk menyediakan campuran WC-10Co-0.8VC (wt%). Kepekatan isipadu serbuk kritikal (*critical powder volume concentration-CPVC*) ditentukan dengan kaedah kilasan. Ciri serbuk yang diperoleh dinilai dengan mikroskop imbasan elektron (*Scanning Electron Microscopy*, *SEM*) and spektoskopi tenaga penyebaran (*Energy Dispersive Spectroscopy-EDS*). CPVC diperoleh iaitu 42 % dan 50 % bagi pengisaran bebola kering dan basah masing-masing. Ketumpatan ketara dan tap bagi serbuk setelah pengisaran bebola secara kering adalah 2.4 g/cm^3 dan 2.96 g/cm^3 manakala ketumpatan ketara dan tap setelah pengisaran bebola secara basah turut mencapai 2.54 g/cm^3 dan 3.39 g/cm^3. Kecekapan pengisaran bebola secara basah turut mengurangkan penggumpalan dan meningkatkan muatan serbuk genting, ini memberikan kelebihan bagi pengacuanan suntikan tungsten karbida submikron. Di samping itu, pengisaran bebola secara basah dalam tempoh yang panjang turut meningkatkan kehomogenan campuran serbuk dan mengurangkan kecacatan struktur sinter.

Kata kunci: Pengisaran bebola; pengacuanan suntikan serbuk mikro (µPIM); tungsten karbida terekat (WC-Co); ciri fizikal campuran

1.0 INTRODUCTION

Micro powder injection molding (μPIM) is an effective process for the large-scale and low-cost manufacturing of micro parts and microstructured parts with complex shapes [1]. μPIM is a net-shaped production process that reduces or eliminates any finishing process required for parts with high wear resistance. Thus, μPIM presents economic advantages for the production of hard metals cemented carbide (WC-Co) [2, 3]. μPIM is adapted from powder injection molding, and the process has the following four main steps: Mixing: compounding the fine powder and organic binder into feedstock; Injecting: shaping the parts from feedstock as in plastic injection molding; Debinding: removing the binder in molded parts; and Sintering: thermal process to achieve final high-density components [2, 4]. For the fabrication of micro components in μPIM, powder particle size must be fine enough to obtain a smooth surface and acceptable optimal accuracy [5]. Submicron WC is suitable for manufacturing micro parts via μPIM [1, 6].

In the first stage of μPIM, feedstock is prepared using a mixing powder and binder. The ratio of powder to total powder and binder content is called solid loading [2, 6]. The optimum powder-to-binder ratio is referred to as critical powder volume concentration (CPVC) [7]. Determination of the point where the powder is perfectly packed and all vacancies between particles are filled with binder is important in μPIM [2]. Different methods are used for predicting critical solid loading. Several studies measured CPVC using the evolution of torque variation during the mixing of the powder and the binder [8, 9]. Mutsuddy and Ford [10] offered a method by modifying the ASTM standard No. D-281-31, and they determined CPVC by recording torque variation during the progressive addition of oil to the powder. Reddy *et al*. [7] also used mixing torque to measure the CPVC of different ceramics and metal powders by adding oil. Critical solid loading of WC-Co was determined by measuring density versus composition [11]. Some researchers also predicted the CPVC of ceramics through viscosity measurements [12].

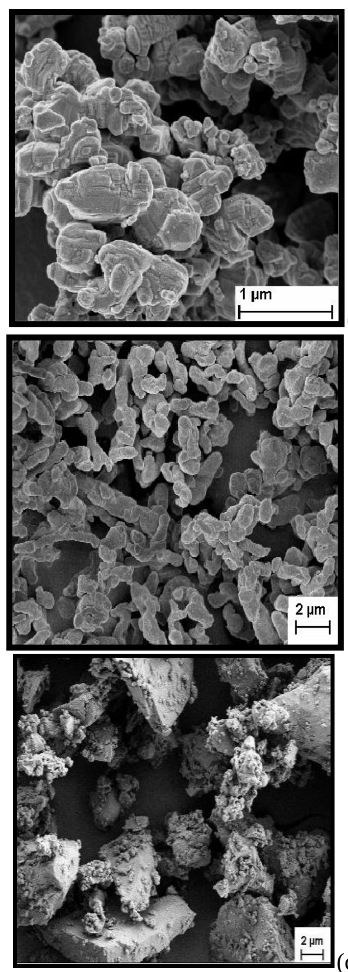
Finer particles usually used in μPIM have high surface area and lower wettability with binders, causing low critical solid loading [13]. Powder and binder were separated in high-pressure injection molding [14] and they had high shrinkage in final parts at low solid loading in μPIM [15]. Good rheological properties of feedstock, acceptable dimension tolerance, and higher mechanical properties are obtained via accurate solid loading [16].

Yang and German [17] found that the critical solid loading of nanophase cemented carbide can be increased via effective milling. A number of studies also reported the improved solid loading of some materials, such as superfine pure molybdenum powder [18] and agglomerated 97W-2.1Ni-0.9Fe heavy alloy powder [19], using the milling process. Increasing solid loading to a reasonable point for fine powder particle size is one of the main challenges in PIM. However, far too little attention has been paid to investigate on influence of milling on critical solid loading and properties of fine WC-CO powder mixture. In this regard, the current study mainly aims to evaluate the effect of ball milling-mixing submicron WC with Co and VC on critical powder loading. Moreover, the effects of dry and wet ball milling processes on mixture properties are investigated as well.

2.0 EXPERIMENTAL

In the current study, the raw materials used include WC (99.5% pure), Co (99.8% pure), and VC (99% pure) powders with mean particle sizes of less than 1, 1.6, and 2.5 μm, respectively. Milling was performed under two conditions of dry and wet ball milling processes using Fritsch Pulverisette-6 to prepare a powder mixture with theoretical composition of WC-10Co-0.8VC (wt-%). Hard metal vial and ball were used to prevent contamination, and ethanol was used as milling medium. The powder mixtures were milled for 1, 3, and 6 h in wet and 6 h in dry condition respectively at a rotation speed of 100 rpm. The weight ratio of the ball-to-powder was 4-to-1. After wet milling, the powder was dried in a vacuum oven at 100 °C. Morphology and homogeneity of the powder mixture were investigated using scanning electron microscopy (SEM, Zeiss Supra 55-VP FESEM). The elemental composition of the powder mixture was evaluated using energy dispersive X-ray spectroscopy (EDS) in three areas with side length of 10 μm. Figure 1 shows the microstructures of the raw materials. WC had an irregular morphology and high degree of agglomeration, whereas Co and VC had angular and irregular shapes, respectively. The apparent density of powders was measured using Hall flowmeter. The mechanical tapping is applying to determine tap density. The height of 100 g of powder taken in a 100 ml cylinder and tapped around 300 times is recorded. The mass of powder divided by its volume after the test gives its tap density.

CPVC was measured using the oil absorption technique [10]. Brabender measuring mixture (W50 EHT), roller blades, and Oleic acid oil were selected for mixing. Oleic acid oil was added to the mixture stepwise, and torque was recorded as a function of time. CPVC was determined based on the oil content, which caused the maximum torque.



(a) (b) (c)

Figure 1 SEM images of raw material powder (a) WC, (b) Co, and (c) VC

3.0 RESULTS AND DISCUSSION

The apparent and tap densities of the mixture are shown in Table 1. Apparent density was lower than theoretical density (14.3 g/cm^3) because of the powder characteristics. Fine powders have high specific surface area and high friction between powder particles, Therefore powder particle cannot flow easily and consequently causing low apparent density. The tap density after 6h wet milling was greater than dry milling, which is ideal for PIM [2]. The results in Table 1 show that effective wet ball milling can improve tap density to 3.39 g/cm^3 after 6 h of milling and produces better powder characteristics for PIM processing. The apparent and tap densities of the powder mixture after 6 h of wet milling were similar with mixed WC-5TiC-10Co, which is reported for PIM [11].

Table 1 Apparent and tap densities of milled WC-10Co-0.8VC

Ball mill condition	Apparent density(g/cm^3)	Tap density(g/cm^3)	% increasing
Dry milling	2.40	2.96	23
Wet milling (1h)	2.32	2.89	25
Wet milling (3h)	2.40	2.95	23
Wet milling (6h)	2.54	3.39	33

Figure 2 shows the CPVC of the mixture at different milling conditions. Critical powder loading after dry milling is 42%, while results indicate that wet milling improved critical solid loading to 50% after 6 h of milling. Li *et al*. [18] report milling case to reduction in particle size and the powder of sponge-like agglomerates was broken down to smaller pieces. These researchers also improved powder loading through milling. Figures 3(a) and 3(b) show the comparison of the microstructure of the mixture after dry and wet milling processes, respectively. The SEM image in Figure 3(b) shows that wet milling can reduce agglomeration. It seems possible that these results are due to the original grains were broken into individual compact pieces. However, the SEM image in Figure 3(b) reveals that the powder mixture achieved a certain degree of agglomeration after 6 h of wet milling. It still a somewhat agglomerated and a portion of aggregated particle. Therefore, longer milling is required to achieve regularly small particle. High specific surface area increases the van der Waals force between particles, causing the agglomeration of the powder [20]. High agglomeration tendency causes low critical solid loading. Dihoru *et al*. [21] used a combination of torque rheometry and capillary and developed a neural network model to predict the maximum powder loading without powder-binder segregation. They found that irregular powder morphology leads to smaller maximum solids loadings. The WC powder was also irregularly shaped and fine in this work. It can be expected lead to lower than maximum solid loading. De-agglomeration of powder during the effective wet milling process can cause higher than critical powder loading for fine WC as shown in Figure 2. The present results are significant in the possibility of selecting suitable powder loading and feedstock viscosity in subsequent stages of PIM. These results of the current study are consistent with those of Yang and German [17] who found milling is required to de-agglomerate and reduce particle size of pre-alloy nanophase carbide powder. These researchers found that the critical solid loading of nanophase carbide powder can improved after effective milling.

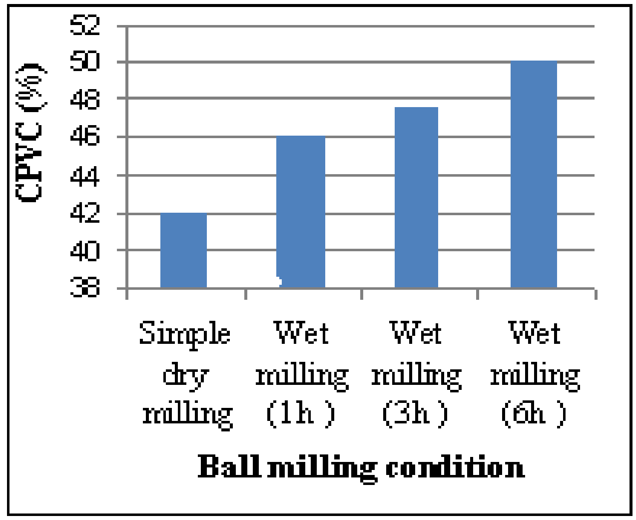
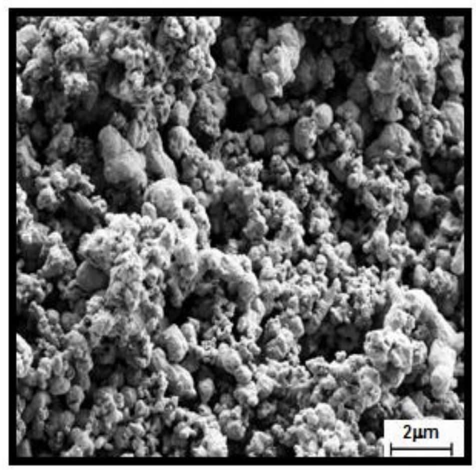
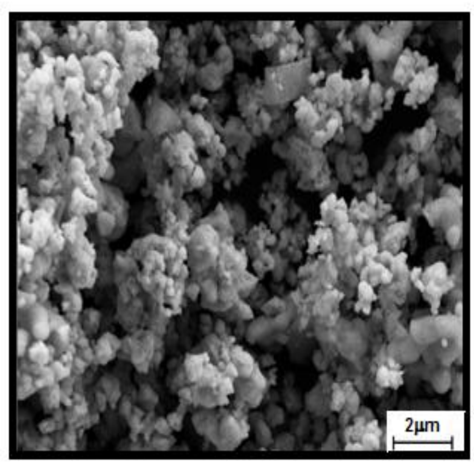


Figure 2 CPVC percentage in different ball-milling conditions



(a)



(b)

Figure 3 SEM images of powder mixture via (a) dry milling (6h) and (b) wet milling (6 h)

To obtain advanced WC-Co mixture with minimal porosity and defects require appropriate milling. Any inhomogeneity from raw materials by milling cannot perfectly be eliminated by subsequent stages of manufacturing [22]. The mixing homogeneity powder mixture can be evaluated and determined based on the element composition detected by EDS [23]. The distribution uniformity of element composition in WC-Co powder mixture was investigated by EDS in this work. The average EDS element compositions of the mixed powders for at least three measurements are given in Table 2. High uniformity was achieved in the mixed powder at a longer time of wet ball milling with the elemental compositions of the powder mixture closer to the theoretical composition of WC-10Co-0.8VC. Uniform distribution of Co and VC in the powder mixture reduced grain growth of WC during sintering and it produced less Cobalt lake in the final microstructure [22]. Heterogeneity in milled-mixed powder cannot eliminate the next stage of μPIM. Thus, higher than homogenous distribution of Co and VC in raw material causes better mechanical properties and fewer defects in sintered components [19].

Table 2 EDS element compositions of powder mixture WC-10Co-0.8VC

Ball mill condition	W (wt-%)	Co (wt-%)	V (wt-%)
Simple dry milling	85.38	14.34	0.29
Wet milling (1h)	78.91	19.95	1.38
Wet milling (3h)	84.18	14.77	1.05
Wet milling (6h)	85.86	13.2	0.87

■4.0 CONCLUSIONS

In this investigation, the aim was to assess influence of milling condition on powder properties and critical powder loading of fine WC-10Co-0.8VC powder mixture. The results from this study show that the wet milling helped to improve the CPVC. The agglomerated submicron WC powder was broken down into smaller pieces via wet ball milling, and the de-agglomeration of powder was effective in improving critical solid loading. The current study demonstrated that wet milling not only increased critical powder loading but also caused a homogeneous mixture of WC-10Co-0.8VC powder, which is helpful for the subsequent stages of μPIM. The tap density of powder also increase under longer time of wet milling that is ideal for PIM.

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