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Sintering of BN/cemented carbide composites under an electric field for improved mechanical performances

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Abstract: In the present study, attempts were made to fabricate cemented carbide (WC/Co) composites toughened with cubic boron nitride (cBN) for structural applications using a field assisted sintering technique. The effects of the content of cBN and the soaking time during the sintering process on the consolidation and mechanical performance of the materials were studied. It was found that with the increase of the cBN content from 0 to 30vol.%, the densification of the composites was hindered and relative density decreased from 99.5% to 92.18%. Phase transformation of the cBN to the hexagonal structure was related to the content of cBN due to the joule heat generated in the interface and tended to occur when a cBN content higher than 5vol% was incorporated and sintered for more than 8 minutes. Both the hardness and toughness showed considerable improvements with an introduction of 5 vol.% cBN and continuous decrease with its further addition. A maximum enhancement of around 12%

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and 20% were achieved for the hardness and toughness respectively. A long period sintering degraded the mechanical performances of the cBN-WC/Co composites by forming the pores or holes and promoting the phase transformation of the cBN. The results obtained are expected to lay a foundation for the fast fabrication of high performance structural materials.

Keywords: sintering; composites; consolidation; phase transformation; properties

1. Introduction

The past decades have witnessed a booming consuming of cemented carbides (WC/Co) in such various applications as drilling and mining due to their excellent mechanical properties [1-10]. The existence of WC grains equips cemented carbides with high hardness, strength and good wear resistance, whereas binder phases contribute the toughness and ductility to the alloys. Despite their wide application in industry, attempts have been continuously made to produce new types of densified cemented carbides with enhanced mechanical performance by incorporating new phases. Cubic boron nitride (cBN) possesses extraordinarily high hardness which is only inferior to that of diamond and is featured by higher thermal stability and chemical stability than diamond[11, 12]. Due to these attractive properties, BN are extensively utilized as a strengthening phase to improve the mechanical performance of the cemented carbides[13, 14]. Nonetheless, the transformation from a high hardness cBN to hexagonal BN (hBN) with a graphite structure and inferior mechanical properties tends to occur in a regular liquid sintering when the temperature is higher than the eutectic point of Co and WC since the thermal stability of cBN can be highly reduced by the presence of liquid and the formation of the hBN is accelerated by the dissolution-precipitation process during the liquid

sintering. As a result, the mechanical properties of the composites will be severely degraded[13]. To well preserve the cBN phase many advanced sintering techniques, such as capacitor electrical discharge consolidation (CEDC) and electrical resistance sintering (ERS) have been put forward to achieve sintering processes in a shorter processing time and at a lower temperature. In CEDC high-voltage discharge obtained by a capacitor bank and released with an extremely short pulse creates a significant current density passing through a powder compact [15] while the ERS process directly introduces electric current to sinter a powder compact under the effects of Joule heat [16]. In particular, coupled multi-physical-fields activated sintering (CMPFAS) and spark plasma sintering (SPS) are among the widely used ERS [10, 17, 18]. The major differences between the CMPFAS and SPS are twofold. First, In the CMPFAS, the formation of spark is hardly noted while the electric current can produce spark discharge in gaps between particles in the SPS. Second, a heating rate as high as 10^5 °C s^{-1} and an electric current as high as 10^5 A can be achieved in CMPFAS, which are greatly higher than those the SPS can introduce. Using CMPFAS, Huang et al. successfully prepared cemented carbides with a relative density of up to 97% at a very low temperature of 850 °C [17]. Zhou et al. investigated the correlation between the sintering temperature and the consolidation of the WC/Co alloys and found a relative density of up to 98.76% could be produced at a temperature of 1200°C [10].

In this study, investigation was carried out to evaluate a new type of structural material of cBN-WC/Co composites using the CMPFAS. The effects of the content of cBN and the soaking time on the consolidation process and mechanical performance of the materials were studied.

2. Materials and methods

WC powder (99.85%, 0.6 μ m, Mingwu Ltd, Chengdu, China), Co powder (98.5%, 2 μ m, Mingwu Ltd, Chengdu, China) and cBN powder (98.5%, 10-14 μ m, Fulaike, Zhengzhou, China) were used in this study. Scanning electron microscope (SEM) images and x-ray diffraction(XRD) patterns of WC, Co and cBN powder are presented in Figure 1. Powder mixtures with volume ratio of 90vol.%WC-10vol.%Co: cBN=95:5, 90:10, 80:20, 70:30 were produced respectively by planetary ball-milling the powder for 2 hours at a ball-to powder ratio of 2. Figure 1g-h shows the SEM image of the powder mixtures and large cBN particles are well dispersed in the powder mixtures and covered with WC and Co powder of smaller sizes. Sintering was performed in a Gleeble-1500D thermal-mechanical (Figure 2a). A cylinder graphite die with a size of Φ 10mm $\times$ 10mm was filled with 0.7-0.8g powder and placed between two electrodes before the sintering. A thermocouple was introduced on the die by soldering to monitor the actual temperature and a temperature of 1200 $^{\circ}$ C was adopted in the sintering experiments. The sintering process started by heating the powder to 200 $^{\circ}$ C for 2 minutes in order to drive out the residual gas within the powder and then further to the sintering temperature for different periods of time between 8 and 16 minutes. A pressure of 75 Mpa and a heating rate of 30 $^{\circ}$ C/s were used throughout the whole heating process. The densities of the specimens were calculated through the Archimedes' principle and microstructures and fracture surfaces were examined using SEM. Phase compositions were obtained using a Bruker Advance D8 XRD diffractometer with Cu K α 1 radiation (λ = 0.154056 nm), source power of 40 kV and 40 mA at room temperature in θ -2 θ Bragg-Brentano configuration geometry. Vickers hardness was determined by introducing force of HV30 on the polished surfaces with a hardness

tester. Cracks from the indents in the hardness tests were employed to estimate the fracture toughness according to the following equation previously used [19].

$$K_{IC}=0.0752P/c^{3/2}$$

where K_{IC} is the fracture toughness, P the load and c the crack length.

Raman spectra were used to evaluate the phase transformation of the cBN in the sintered samples with a laser excitation wavelength of 532 nm and 2400 lines grating.

3. Results

Typical shrinkage and temperature profiles for the cemented carbides and the WC/Co composite reinforced with BN during the sintering process are given in Figure 2b-2c. It is evidently noted that dramatic shrinkage displacements take place at round 1050°C for both materials and the shrinkage displacement for the cBN-WC/Co composite is significantly smaller than that for the matrix alloy, indicating the addition of cBN reduces the densification process of the alloys. A further observation of the curves suggests the sintering process can be separated into four stages: (I) pre-heating stage, (II) heating stage, (III) soaking stage and (IV) cooling stage. Initially, powder is compressed within the die under the action of the pressure and subjected to the electrical heating. At a low temperature, significant change hardly happens to the powder compact and a slight shrinkage approximately 10% of the total is produced in the pre-heating period. It is worthy of note that the holding at 200°C is intended to drive out the residue gas within the powder compact so as to facilitate the densification process by reducing the distance between particles and to improve the electrical performance of the powder compact by enhancing the passing of electrons through the samples (stage I). With the further heating process, the powder compact is heated swiftly to the sintering

temperature and the porosity within the powder compact is decreased considerably, resulting in the enhanced densification and significant shrinkage (stage II). Due to the high contact resistance of the interfaces among powder, it is expected that the actual temperature in some local area might be higher than the preset temperature and therefore liquid phase probably from cobalt or eutectic phase of WC and Co might be formed. Once the liquid phase is produced, it would swiftly fill into the pores and reduce the porosity of the sample due to the capillary force. In the soaking period (stage III), shrinkage displacement increases slightly due to the thermal expansion and indicates densification of the sample proceeds slowly while in the cooling period a moderate shrinkage is noticed due to the thermal contraction caused by a sharp decrease in the temperature (stage IV).

Mechanical properties of the sintered samples with varied contents of cBN were given in Table 1. It can be noted that all of the specimens reach a high density and the densities decrease gradually from 99.50 to 92.18% with the increasing content of the cBN from 0% to 30%. The hardness exhibits a significant increase when a small percentage of 5vol.%cBN is added and presents a continuous decrease with the increasing content of the cBN. An approximately 12% increase in hardness is obtained by adding cBN. However, it is unanticipated that an introduction of a higher content of cBN results in a lower hardness given that cBN is much harder than the cemented carbide matrix. As later discussed, the possible explanation for this fact is the cubic structure for BN transforms to the hexagonal structure, leading to the formation of the hBN phase with a lower hardness. Fracture toughness derived from the hardness presents a similar pattern. It initially increases from 7.8 to 9.39 with the addition of 5vol.%cBN and decreases significantly to 6.79 with the incorporation of 30vol%cBN. A

maximum increase of approximately 20% in fracture toughness is achieved.

Comparisons of the results between the current work and previous ones are also shown in Table 1. Evidently, the composite with 5vol.%cBN obtained in this work presents a higher hardness than those produced by using the spark plasma sintering (SPS) or hot isotatic pressing(HIP) and shows a comparable value in toughness as those SPS-processed at 1250 °C and 1300°C. Although the fracture toughness of the composites fabricated in the presented work exhibit significant lower values than the HIPed sample, the HIP process takes a much longer time.

A further observation of the indents (Figure 3a-c) indicates that crack formed during the loading can propagate along the grain boundaries between the cBN and the matrix, helping release the stress and improve the toughness. In addition, propagation through the cBN is also observed as well, implying the strengthened interface between the cBN and the matrix. The BSE images of the sintered samples with varied contents of cBN are given in Figure 3d-3i. As can be noted, cBN particiles are homogeneously embedded in the WC/Co matrix, indicating a well dispersion of cBN in the powder mixtures was achieved during the ball milling process. A further observation implies that small particles arising from the wear debris of cBN particles are dispersed within the matrix and are expected to provide additional strengthening effect. Typical XRD patterns for the cemented carbides and the composite with 30vol.% cBN sintered at 1200°C for 12 minutes are shown in Figure 4a-4b and indicate the presence of phases of WC and cBN in the sintered samples and hBN is hardly detected. However, when it comes to the BN, XRD might not be sufficiently sensitive to examine its phase transformation, as suggested that identification and quantification by XRD of hBN and cBN in the presence of WC are very difficult because of the very strong mass-absorption

coefficients of WC/Co compared with both phases of BN, which prevents the detection of cBN converted into hBN at the interfaces[13, 20]. This is also implied by the unexpected lower hardness of the materials loaded with a higher content of BN in Table 1. Raman characterization was therefore used to determine the potential structural transformation of the BN within the composites and the typical results are given in Figure 4c-4e. It can be observed that pure cBN powder exhibits two distinct peaks at the wavenumbers of around 1050 and 1300 respectively. cBN in the sintered 5vol.%cBN-WC/Co composites shows the similar signatures with a higher background and implies that cBN remains after the sintering. The higher background can be attributed to the strong interaction between cBN and the matrix. Nonetheless, when a high content of 30vol.% cBN was added, a signature of a big lump is noted and in sharp contrast with what is observed in the cemented carbides and the composite with a content of 5vol.%cBN. This implies that a higher content of cBN added tends to promote the phase transformation of BN from cubic to hexagonal structure, leading to the degradation of mechanical properties of the composites[21]. The content related phase transformation of cubic BN is expected to be closely associated with the mechanism of the field assisted sintering used in the presented study. Since the resistance of the interfaces between the cubic BN and the cemented carbides matrix is significantly large, when the current passes through the sample a great deal of joule heat will be generated, leading to a higher temperature than the preset one at some local area. When a higher content of cubic BN is introduced, more interfaces will be created and more heat energy will be produced. A higher temperature around local area and even potentially a higher one for the bulk sample will likely be achieved, enhancing the phase transformation of cubic to hexagonal boron nitride.

The above results indicate that a small addition of cBN will appreciably improve both the hardness and the fracture toughness. However, it is certainly desired that the composites with improved properties can be produced by adding a relatively large amount of cBN and controlling the phase transformation. Therefore, effects of the soaking time on the composite with a high content of 20vol.% cBN were investigated to shade more light on the microstructural evolution and mechanical performance of the composites.

BSE and SEM observations of the 20%cBN-WC/Co composite are given in Figure 5a-5e. It is noted that large cBN particles are well dispersed and securely embedded in the fine WC/Co matrix. Pores are scarcely observed on the polished and fractured surfaces, indicating the composites were well consolidated. As expected, the Raman spectra (Figure 5f-5h) of the BN in the 20%cBN-WC/Co composite lose the distinctive features of cBN and there are no noticeable peaks observed at the wavenumbers of around 1050 and 1300. In addition, there is a peak around 1500 wavenumber for BN sintered for 16 min and implies the formation of hBN [22]. It is therefore expected that a long period processing will promote the transformation of the BN from the cubic to hexagonal structure and a sintering time shorter than 8 min will be preferred to produce cBN reinforced WC/Co composites with desired performance. WC/Co alloys were also examined for a deep analysis of the matrix. It can be observed from Figure 6a-6c that grain size of the WC in the cemented carbides remains nearly the same with the soaking time increasing from 8 to 16 min. However, a big hole is found after a long period sintering process and might arise from the evaporation of the liquid phase potentially from the melting of Co and eutectic reaction between WC and Co. XRD result in Figure

6d indicates only WC and Co phase exist in the final sintered sample and no traceable decarbonisation phases as W_2C are detected after the sintering process.

The mechanical properties of the composites reinforced with 20vol.% cBN are presented in Table 2. It is observed that WC/Co composites in general were successfully densified while significant decreases in hardness and toughness are obtained after a long holding time. The results are consistent with the observation of microstructures and expected to be attributed to the evaporation of liquid phase within the sintered samples. With the addition of 20%cBN, the density of the composites initially exhibit a slight increase from 95.57% to 96.96% with the increase of the holding time and a significant decrease to 92.68% with the further sintering while the values of both hardness and toughness show descending trends. The lower density and inferior mechanical performance of the composites in a long sintering time might be caused by the phase transformation from the cubic to hexagonal structure and the evaporation of the liquid phase.

4. Discussion

Overall, the mechanical performance of the cemented carbides composites toughened with BN during the field assisted sintering is largely related to the content of cBN added and the phase transformation of BN occurred during the sintering process, which is determined by the joule heat, the heating rate and the sintering time based upon the following equation[18].

$$\frac{I}{A} = \sqrt{\frac{\rho C}{\sigma}} \cdot \sqrt{\frac{dT}{dt}} \quad (1)$$

$$Q = I^2 R t \quad (2)$$

Where I is current, A the cross-sectioned area, C specific heat, α the coefficient of the electrical resistance, ρ the density, T the temperature, t the time, Q heat, R the electrical resistance.

From the above equations it can be seen that the current density is proportional to the heating rate. At the same heating rate, a higher electrical resistance and a longer sintering time will produce a larger amount of joule heat. In the case of cBN-WC/Co composites, heterogeneous BN/matrix interfaces are present in the materials and expected to possess a higher contact resistance than the matrix/matrix interfaces. A high addition of cBN therefore tends to increase the resistance of the compact and lead to the generation of a great deal of joule heat when a current density is introduced. This explains why the more cBN is added, the more likely it would transform from cubic to hexagonal structure, resulting in a low hardness in the composite with a high content of cBN, as indicated by the Raman signature and the hardness of the 30vol.%cBN-WC/Co composite shown in Figure 4e and Table 1. It should be noted that the Raman result indicates that when a low addition of 5 vol.%cBN is incorporated, phase transformation hardly occurs during the sintering process, implying temperature rise contributed by the joule heat caused by a low addition of cBN is insignificant and actual temperature during the sintering process is below the phase transformation point. When a loading of cBN higher than 5vol.% is introduced, additional large resistance arising from the heterogenous interfaces would be produced. As a result, the corresponding joule heat generated would give rise to an increase in the temperature of the sample, which might be higher than the phase transformation point, leading to the occurrence of the transformation from cubic to hexagonal structure.

On the other hand, it is noted from the Table 2 that the hardness of all of the 20vol.%cBN-WC/Co composites exhibit a lower value than those of the WC/Co composites and an increase in the sintering time causes a decrease in the hardness of the 20vol.%cBN-WC/Co composites. This fact suggests that a reduction in sintering time from 16 min to 8min fails to prevent the phase transformation from happening in the composite with a 20vol% BN, as indicated by the Raman result shown in the Figure 5f-5h and further corroborates besides the sintering time, the cBN content which to a large extent determines the joule heat produced in the composites during the field assisted sintering process is another important factor influencing the phase structure and the mechanical performance of the cBN-WC/Co composites.

4. Conclusions

In the current work, cBN-WC/Co composites were successfully produced by a field assisted sintering process. Mechanical properties of the composites were examined and correlated with the microstructure. The joule heat generated during the field assisted sintering can favour the formation of liquid phase, enhancing the densification process. A small addition of 5 vol.% cBN significantly improves the toughness and hardness of the matrix and a maximum enhancement of approximately 12% and 20% were achieved for the hardness and toughness respectively. A further addition of cBN higher than 5 vol.% fails to impart better mechanical performance to the composites. A long period of sintering process causes the evaporation of liquid phase and promotes the transformation from the cubic to hexagonal structure for BN, leading to the significant degradation of the mechanical properties of the cBN-WC/Co composites. The research results obtained may pave the way for the production of a new type of composites reinforced with cBN to suit engineering applications.

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Figure and Table captions

Figure 1. SEM images and XRD patterns of the WC (a, d), Co (b, d) and cBN (c, f) powder and SEM images of the powder mixtures (g-h).

Figure 2. Schematic of the sintering machine (a) and sintering profiles of the WC/Co alloy (b) and the 20vol%cBN-WC/Co composite (c) soaked for 12 min.

Figure 3. Optical images of the indents formed in the hardness test (a-c) and BSE images of the cBN-WC/Co composites with varied contents of cBN. (a, d) 0 vol.%, (e) 5vol.%, (f) 10 vol.% (e, g) 20 vol.% (c, h) 30vol.% (i) a higher magnification image (30vol.%) .

Figure 4. Typical XRD patterns of the sintered cBN-WC/Co composites ((a) 0 vol.%, (b) 30 vol.%) and Raman spectra of cBN powder (c) and cBN in the composites((d) 5vol.% (e) 30vol.%)

Figure 5. BSE (a-c) and SEM (d-e) images and Raman spectra (f-h) of the 20vol.%cBN-WC/Co composites processed for varied time. (a, f) 8min, (b, g) 12min, (c, h) 16min.

Figure 6. BSE images and XRD patterns of the WC/Co alloys processed for varied time. (a) 8min, (b) 12min and (c) 16min.

Table 1. Mechanical properties of the cBN-WC/Co composites as a function of the cBN content

Table 2. Mechanical properties of the 0 vol. % and 20 vol. %cBN-WC/Co composite as a function of soaking time

Table 1. Mechanical properties of the cBN-WC/Co composites.

Sintering temperature (°C)	Soaking time (min)	cBN content (Vol.%)	Relative density (%)	Vickers hardness (HV)	Fracture toughness (MPa m ^{1/2})
1200	12	0	99.50	2136.36±54	7.80±0.28
1200	12	5	99.30	2399.25±45	9.39±0.38
1200	12	10	99.09	2023.68±51	8.10±0.30
1200	12	20	96.96	1570.90±70	7.02±0.27
1200	12	30	92.18	1507.36±110	6.79±0.70
1200(SPS)	5	0	99.86	1869±20	6.68±0.50 ^[14]
1250(SPS)	5	25	99.40	1752±60	10.90±0.80 ^[14]
1300(SPS)	5	25	99.39	1847±80	8.43±0.90 ^[14]
1300(SPS)	7.5	25	99.52	2123±40	10.97±0.50 ^[14]
1100(HIP)	150	30	99.80	2150±12	15.40±0.80 ^[13]

Table 2. Mechanical properties of the 20vol.%cBN-WC/Co composite

Sintering temperature (°C)	Soaking time (min)	cBN content (Vol.%)	Relative density (%)	Vickers hardness (HV)	Fracture toughness (MPa m ^{1/2})
1200	8	0	99.61	2194.14 ± 38	7.90 ± 0.29
1200	12	0	99.50	2136.36 ± 54	7.80 ± 0.28
1200	16	0	98.11	1818.24 ± 75	7.46 ± 0.48
1200	8	20	95.57	1782.00 ± 66	7.42 ± 0.65
1200	12	20	96.96	1570.90 ± 70	7.02 ± 0.27
1200	16	20	92.68	1344.66 ± 118	6.83 ± 0.25