Effects of Vanadium Carbide on Sintered WC-10%Co Produced by Micro-powder Injection Molding
(Kesan Vanadium Karbida ke atas WC-10%Co Bersinter dihasilkan melalui Pengacuan Suntikan Serbuk Mikro)

WONG YEE NING, NORHAMIDI MUHAMAD, ABU BAKAR SULONG*, ABDOLALI FAYYAZ & MUHAMMAD RAFI RAZA

ABSTRACT

Ultrafine, cemented tungsten carbide (WC) possesses exceptional hardness, wear resistance and high strength in various applications. In this study, WC was produced through micro powder injection molding (μPIM), which is also applicable for metals and ceramics in producing complex parts with high-dimensional accuracy. Different inhibitors, such as VC, Cr_2C_3, NbC, or TaC, were added to improve the mechanical properties of WC and control its grain growth. The effects of a grain growth inhibitor were investigated by adding VC in WC–10%Co–nVC, where n = 0 to 1.2 wt. %. The mechanical properties of the sintered part, such as hardness and flexural strength, were determined. The morphology and elemental distribution of the samples were studied by field emission scanning electron microscopy and energy-dispersive X-ray spectroscopy. X-ray diffraction was employed to study the phases of the obtained samples. The results showed that the sample with 0.4 wt. % VC (optimal amount) sintered at 1410°C exhibited the highest theoretical density, hardness and flexural strength of 95.2%, 1973±31 HV and 2586±172 MPa, respectively. The average grain size measured was 519±27 nm. VC acted as a grain growth inhibitor during sintering, thereby improving the mechanical properties.

Keywords: Flexure strength; grain growth inhibitor; microstructure; micro powder injection moulding; XRD

ABSTRAK

Ultra halus, tungsten karbida terikat (WC) memiliki ciri kekerasan yang tinggi, rintangan haus dan kekuatan yang tinggi dalam pelbagai aplikasi. Dalam kajian ini, WC dihasilkan melalui pengacuan suntikan serbuk mikro (μPIM) yang turut digunakan dalam bidang logam dan seramik untuk menghasilkan produk kompleks dengan ketepatan dimensi yang tinggi. Perencat lain seperti VC, Cr_2C_3, NbC atau TaC telah ditambah untuk meningkatkan sifat mekanik WC dan mengawal pertumbuhan butirnya. Kesan perencat pertumbuhan butir telah dikaji dengan menambah VC ke dalam WC-10%Co-nVC dengan n=0 - 1.2 wt. %. Sifat mekanik pada bahagian bersinter seperti kekerasan dan kekuatan lenturan diukur. Taburan morfologi dan unsur sampel dikaji dengan pancaran medan mikroskop imbasan elektron (FESEM) dan spektroskopi tenaga penyebar sinar-x. Pembelauan sinar-X digunakan untuk mengkaji fasa sampel yang diperoleh. Keputusan menunjukkan sampel dengan 0.4 wt. % VC (jumlah optimum) yang disinter pada suhu 1410ºC mencatatkan teori ketumpatan tertinggi, kekerasan dan kekuatan lenturan masing-masing pada 95.2%, 1973±31 HV dan 2586±172 MPa. Purata saiz butir yang dikira ialah 519±27 nm. VC berperanan sebagai perencat pertumbuhan butir semasa proses pensinteran, justeru memperbaiki sifat mekaniknya.

Kata kunci: Kekuatan lenturan; mikrostruktur; pengacuan suntikan serbuk mikro; perencat pertumbuhan butir; XRD

INTRODUCTION

Cemented tungsten carbide (WC) is known for its exceptional hardness, wear resistance and high strength. However, WC is difficult to machine because of its hardness, especially in fabricating products with small sizes and complex shapes. In this study, micropower injection molding (μPIM) was used to enhance the capability of WC for the low-cost mass-production of components in metal, ceramics and carbides with micro-geometrical features and complex shapes at low cost (Piotter et al. 2008).

In hard metals industry, sintered materials with ultrafine grain size exhibit extremely high flexural strength. The dependence of the mechanical behavior of cemented carbides on their grain size is due to the rapid grain growth that occurs during the early sintering stage.

WC grains tended to aggregate via coalescence of grains (Fang et al. 2009). Thus, various methods have been developed to inhibit grain growth, such as applying small percentage of a grain growth inhibitor or by using advance sintering technologies e.g. spark plasma sintering (SPS), microwave sintering and rapid hot-pressing sintering. These advanced sintering methods are restricted to the laboratory and may be impractical for mass production (Xiong et al. 2008). Small amounts of grain growth inhibitors such as VC, Cr_3C_2, NbC, TaC, Mo_2C and other carbides, were added to control the grain growth of WC (Xiao et al. 2009; Xiong et al. 2008). Less than <1.0% mass fraction of grain growth inhibitors are added to avoid formation of precipitation in a WC-Co interface that can cause embrittlement.

Vanadium carbide (VC) is considered the most effective grain growth inhibitor because of its high solubility and diffusivity in liquid cobalt phase at relatively low temperature (Fang et al. 2005). The effect of VC as a grain growth inhibitor has been explained in the presence of a liquid metal binder by slowing the mechanism of solution re-precipitation of W and C in a liquid binder phase (Huang et al. 2007; Morton et al. 2005; Sun et al. 2008). By limiting the dissolution of W and C grains in the liquid phase, grain growth is consequently slowed down. Sun et al. (2008) investigated the amount of inhibitors (Cr_3C_2) by comparing them to an inhibitor-free sample that was consolidated using pressure-assisted SPS technique. The results showed that the amount of Cr_3C_2 should be controlled within a certain range to achieve desired hardness and toughness. The experimental results showed that optimum hardness, toughness and bending strength can be obtained when 0.9 wt. % NbC is added with WC (Huang et al. 2008). Higher NbC content decreases the mechanical properties because of the formation of large (Nb, W) C grains.

In previous studies, cemented carbide has been fabricated mainly by pressing method; limited studies have μPIM. The aim of this study was to investigate the effects of VC as a grain growth inhibitor to control the grain size and microstructure of WC produced via μPIM. This study focused on the addition of an inhibitor by minimizing grain growth during liquid phase sintering. The optimal VC content that did not cause formation of pores, which cause brittleness, was determined. Densification, mechanical properties, microstructural behaviour and phase relations were investigated and compared with inhibitor-free samples.

MATERIALS AND METHODS

WC (99.5% purity) and Co (99.8% purity) were manufactured and supplied by Alfa Aesar, USA, where as VC (99% purity) was manufactured by Changsha Asian Light, China. Composite powders (WC-10%Co-nVC) with various compositions of VC (n = 0, 0.4, 0.8, 1.2 wt. %) were prepared via planetary ball milling (Pulverisette 6, FRITSCH). The powder mixtures were dispersed and ball-milled in ethanol at 100 rpm for 6 h. The slurry mixtures were dried at 100°C in an oven for 24 h and then crushed. The dried composite granules were mixed in brabender W50E machine using multiple-component binder system, which consisted of paraffin wax, polyethylene and stearic acid (Ani et al. 2013; Zakaria et al. 2014). This binder system played an important role during the injection molding process, acting as vehicle that packed particles into desired shapes and also providing shape retention until the sintering process. The feedstock was prepared by mixing (30 rpm) binder components with powder mixtures at 140°C to reduce the feedstock viscosity. The feedstock was then injected into a micro dumb-bell-shaped mold. The mold temperature was 105°C and the barrel temperature was kept at 140°C (Heng et al. 2014, 2013). Debinding

was performed via two-step debinding techniques. The compacts were first immersed in n-heptane at 35°C for 3 h to extract the paraffin wax via solvent extraction. The compacts were then debound in a split debinding furnace at 500°C to remove the remaining binder (PE), which served as the backbone polymer to provide structural strength and handling support. Lastly, sintering was conducted in a vacuum furnace at temperatures varying from 1370 to 1450°C at a heating and cooling rate of 10°C/min, with a dwell time 3 min; this process was performed when the Co phase is in liquid state, in accordance with previous studies by the researchers (Petersson & Ågren 2005; Tsai 2011).

The sintered compacts were analyzed to determine the effect of VC on densification, mechanical properties and microstructure of the ultrafine WC. The density of the samples was measured using Archimedes' displacement method according to metal powder industries federation (MPIF) standard 42. All of the measurements were obtained using a model BSA224S-CW electronic balance with ethanol as the medium. Tranverse rupture strength (TRS) was measured using Instron 5567 at a speed rate of 1 mm/min with 1kN load cell. Hardness of the sintered components were measured according to MPIF standard 43 by using Mitaka Vicker's indentation hardness test, under a dwell load of 29.42 N and 15 s dwell time. The specimens were cold mounted and polished to 'mirror-appearance' followed by etching with Murakami reagent. The metallographic surface microstructures were observed using an ultrahigh resolution field emission-scanning electron microscopy (FE-SEM; Zeiss Supra 55-VP). The average grain was measured using a linear intercept method. The measurements were made on polished and etched cross- sections of metallographic specimens by calculating the length of a random intercept. The distribution of elements was determined by energy dispersive X-ray (EDX) analysis in combination with FE-SEM. The phases of the starting powder and sintered hard metals were identified by X-ray diffraction (XRD, Siemens D5000). All of the measurements were conducted using a standard procedure at 2θ from 20° to 90° at 0.5°/10 s.

RESULTS AND DISCUSSION

MICROSTRUCTURAL EVOLUTION IN THE MPIM PROCESS

FE-SEM Supra 55 was used to examine the different stages of the μPIM process. As shown in Figure 1(a), the microstructure of the powder mixture (WC–10%Co–0.8wt.%VC) that was produced by planetary ball milling showed the homogeneous dispersion. The WC powder exhibited slight agglomeration with near spherical and irregular shape, which was ideal for injection molding process (German & Bose 1997). Figure 1(b) shows the component structure of a green part formed when heated feedstock (powder-binder mixture) is forced into a cavity where it cools and retain the shape of cavity. The green parts were then undergone binder removal in heptane

(solvent debinding) followed by a thermal debinding step at 500°C to form brown parts. Figure 1(b) shows the structure of a compacted part where the binder covered the metal particle. The green parts were solvent debound in heptane followed by a thermal debinding step at 500°C. Figure 1(c) shows the fractured surface of a specimen after the solvent debinding process. Polyethylene remained as the backbone molecule and was subjected in chain scission by providing structural strength and handling support until subsequent thermal degradation. Large surface pores formed during the thermal debinding process (Figure 1(d)) as heat softened the binders, forming capillary stress that induced the rearrangement of the particles, in which regions with low packing density were pulled apart from the regions with high packing density leaving a fragile structure (Chua et al. 2013).

Defect free samples of WC were produced via μPIM (Figure 2). No geometrical changes were observed between the green and brown powder injection-molded structures (Jamaludin et al. 2009). The surface and edge of the green micro-component in Figure 2(a) is smooth and well filled. In Figure 2(d), the sintered part shows 22% shrinkage compared with the green part. A linear shrinkage varied from 15 to 25%, depending on the feedstock composition during the production of the micro parts using μPIM (Heng et al. 2014, 2013; Piotter et al. 2008).

DENSIFICATION BEHAVIOUR OF WC COMPOSITE

In μPIM materials, the compact density was increased after sintering because of pore elimination during the heating process. Figure 3 shows the relative density of the sintered samples with various inhibitor contents at different sintering temperatures. Densifications occurred with increasing sintering temperature at the same amounts of VC. Both sintering temperature and inhibitor content affected density. Higher sintering temperature increased the Co melt fluidity, leading to improve capillary pressure and porosity filling. Therefore, the compact is denser at a higher temperature (Mahmoodan et al. 2011). Highest densification was achieved at 1450°C (highest sintering temperature) for the inhibitor-free sample. A slight decrease in density was observed when VC was added gradually. During densification at 1410°C, the density decreased from 95.8% for pure WC–10Co to 95.2, 93.3 and 91.5% for the cemented WC with 0.4, 0.8, and 1.2 wt. % VC, respectively.
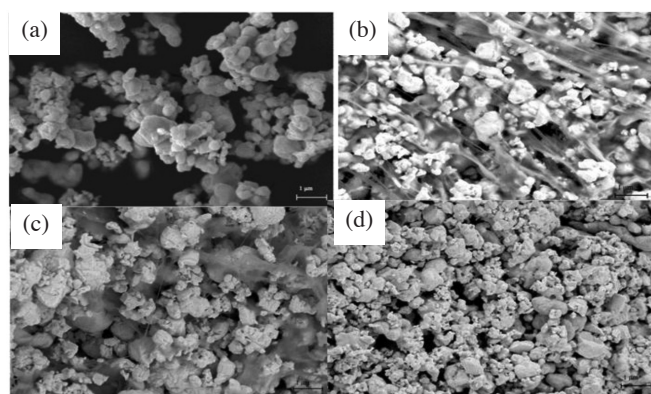

FIGURE 1. FE-SEM micrographs of (a) composite powder of feedstock, (b) green part, (c) fracture surface of green part after solvent debinding and (d) fracture surface of the brown part (10000× magnification)
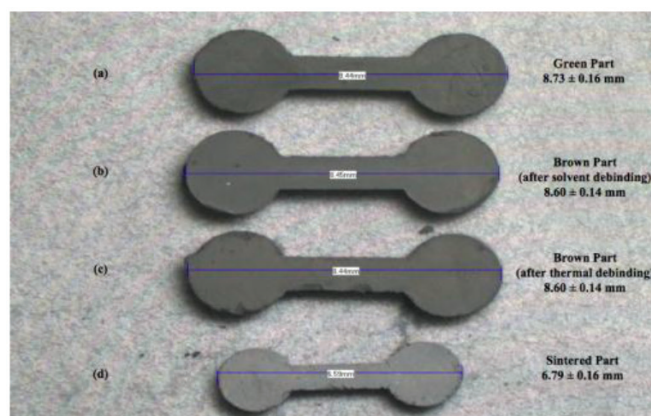

FIGURE 2. Geometry changes: (a) green part after injection, (b) brown part after solvent debinding, (c) brown part after thermal debinding and (d) sintered part after sintering

With the addition of VC, the solubility of WC in the Co phase was decreased, thereby hindering alloy densification in the liquid-phase sintering-solution re-precipitation stage (Sun et al. 2011). This phenomenon may be caused by the high viscosity of the liquid phase containing the VC and the low capability to fill the porosities. Thus, the VC-containing sample did not achieve the maximum density even at higher sintering temperatures (Mahmoodan et al. 2011).

MECHANICAL PROPERTIES

The Vickers hardness of WC–Co cemented carbides with different compositions of inhibitor and sintering temperatures are shown in Figure 4. The hardness value obtained from the inhibitor-free WC–10%Co was 1498±43 HV, which was close to the Ultra Carbide Inc. and ISO 3878 standard values. The hardness increased with the increase in the amounts of VC until a maximum hardness value of 1973±31 HV was obtained with the addition of 0.4 wt. % VC.

The effect of VC content on TRS is shown in Figure 5. The overall value obtained was lower than the theoretical value from the cast parts. Li et al. (2013) reported that WC–10%Co with an average grain size of 0.84 μm exhibited flexural strength of 3376±227 MPa. According to German and Bose (1997), a typical WC–10%Co composition that was fabricated by PIM exhibited a fracture strength ranging from 1700 to 3000 MPa, if sintering was performed under defect-free conditions. The main reason for the low TRS is the sensitivity of the TRS to porosity level; a high porosity causes poor and inconsistent TRS values (Fang 2005). The TRS value increased to 2586±172 MPa at 0.4 wt. % VC, which dropped with further addition of VC. TRS values after sintering increased appreciably until 1410°C due to the strong bond formed between WC grains. During liquid phase sintering process, formation of eutectic phase helped densification process by infiltrating porosity. However, densification process was incomplete at low sintering temperature causing the component to have higher porosity level. However, it can be observed that TRS decreased when temperature increase more than 1410°C. The data and analysis from previous work stated that TRS improved with increasing Co content in cemented tungsten carbide component (Gille et al. 2002). When temperature is too high (1450°C), cobalt phase tends to evaporate through sintering process and caused to low Co content in matrix. Hence, the effectiveness of VC is very depending on sintering temperature.

The EDX results showed that only 6.87% of Co remained in the sintered parts. The flexural strength obtained in this study was close to the value obtained by Li et al. (2013) in which 2645±203 MPa was achieved from WC–6%C. Janisch et al. (2010) suggested that a minimum cooling rate ranging from 25 to 50°C/min is required to avoid Co spreading. Fang (2005) also stated that TRS value is highly dependent on the Co content and grain size.
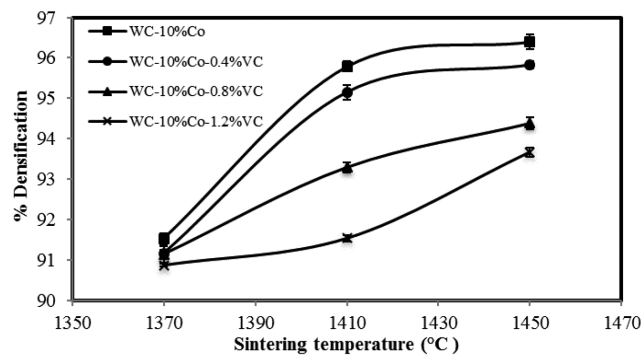
FIGURE 3. Sintered density of WC–10%Co with different VC contents at 1370 to 1450°C
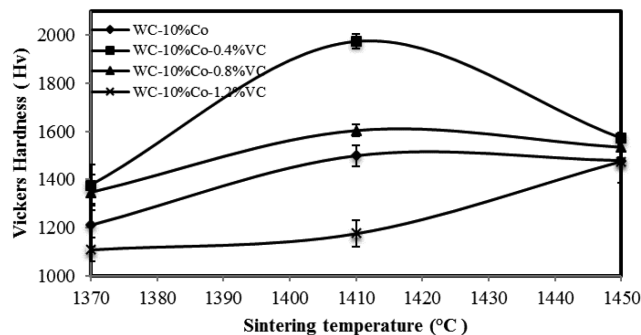
FIGURE 4. Effects of grain growth on hardness value of WC–10%Co with and without addition of VC

The results indicated that the mechanical properties were improved by the addition of VC. However, the amount of inhibitor should be controlled at a certain range to achieve optimal values of hardness and flexural strength.

MICROSTRUCTURAL BEHAVIOUR OF THE SINTERED PARTS

Determining microstructural changes are important to understand property evolution. The morphology and sintering behavior of WC were studied using FE-SEM Supra 55. The microstructures of the sintered compacts via liquid-phase (Co) sintering are shown in Figures 6(a) and 6(b). According to Lei and Wu (2009), the bright and grey regions appeared to be WC and Co phase, respectively.

During liquid phase sintering, the process of grain growth for WC-Co alloy can be described as Oswalt ripening process where solid particles tends to reduce surface energy by forming larger grains. The presence of grain growth inhibitor VC reduced growth rate. The decreased in growth is attributed to a lowering of difference in interfacial free energies due to segregation of inhibitor to interface of WC grains. The segregation is accompany face specific absorption, face-orientated deposition change edge energy associated with a 2-dimensional nucleation process (Morton et al. 2005). Hence, WC grain is inhibited due to incorporate of VC as impurities atom in blocking WC nucleation.

The mean grain size was obtained by analyzing the microstructures using average grain intercept method. The addition of VC aided in reducing the grain size. The average grain size of 0.4 wt. % VC was reduced from 748 to 519 nm, which is 31% lower than the sintered inhibitor-free sample. The mean grain size with added VC was finer than the VC-free sample. In addition, the samples with VC showed uniform grain distribution and effective restraint of abnormal grain growth.

The growth rate was reduced with the addition of a grain growth inhibitor. The addition of VC at the WC–Co interface prevented phase transformation; thus, the dissolution of WC in the binder phase was suppressed, thereby prolonging the formation of normal grain growth and minimizing abnormal grain growth (Fang et al. 2009; Sun et al. 2007). Morton et al. (2005) mentioned

that the decrease in growth is attributed to a lowering in the difference of interfacial free energies because of the segregation of the inhibitor with the interface of the WC grains. As a result, the inhibitor-free WC formed large, fully developed, prism-shaped grains, whereas the grains with 0.4 wt. % VC showed a multi-faced, layered structure.

The hardness and TRS of the alloy (as determined by grain size) can be explained using the Hall-Petch relationship, which states that the refinement of the WC grains leads to superior mechanical properties. The addition of VC inhibited the WC grain growth, resulting to an increase in hardness and TRS (Ouyang et al. 2012; Sun et al. 2008; Xiao et al. 2009).

PHASE RELATION OF THE VC SUBSTITUTED CEMENTED CARBIDE ALLOYS

The phase composition of the raw powder and alloys sintered at 1410°C were analyzed by XRD (Figure 7). The XRD patterns indicated that only WC and Co phases occurred in the nanocrystalline composite powders. The eta (Co_3W_3C) phase was observed in the cemented carbide without VC addition. The active powder tended to absorb oxygen during milling, which caused the formation of undesirable Co_3W_3C phase. The occurrence of the Co_3W_3C phase affects the mechanical properties of the cemented carbide (Xiao et al. 2009). Thus, the addition of VC, which is a grain growth inhibitor, reduced the oxygen concentration and decreased the volume fraction of the Co_3W_3C phase.

CONCLUSION

WC–Co–VC hard metal is produced by μPIM process. The addition of an inhibitor, 0.4 wt. % VC, improves the mechanical properties of WC–10%Co – 0.4 wt. % VC by inducing the production of finer grains at sintering temperature of 1410°C. Excessive addition of VC may affect liquid cobalt saturation concentration and the densification process, which can reduce the overall properties. The mechanical properties of the hard metal can be improved by adding VC, but excessive addition may cause reversible effect. The measurement results,
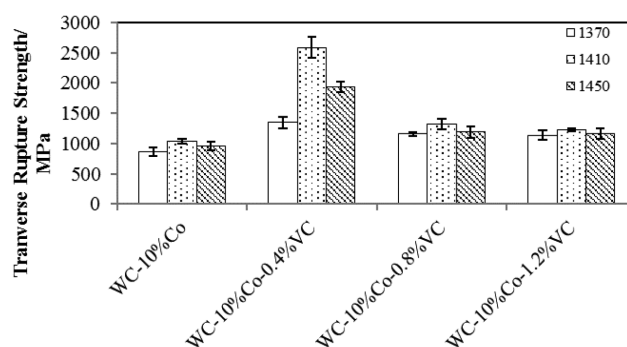

FIGURE 5. Transverse rupture strength of samples versus sintering temperature
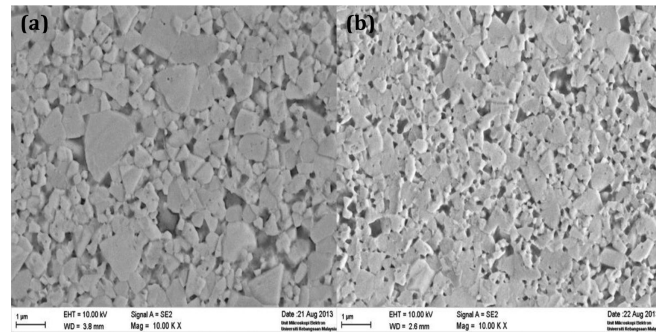
FIGURE 6. Microstructure of a polished section of (a) WC–10%Co and (b)
WC–10%Co–0.4% VC that were sintered at 1410°C (10000× magnification)
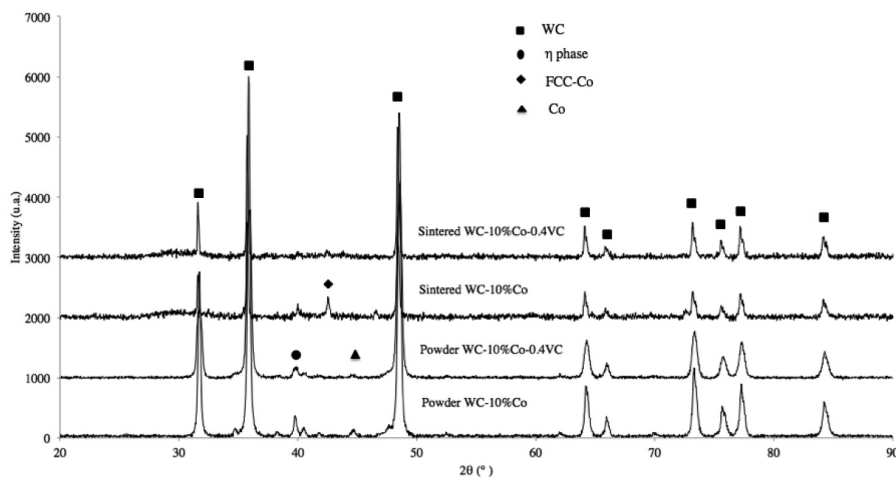

FIGURE 7. XRD patterns of powders and sintered alloys

such as hardness, transverse rupture strength and average grain size, support this enhancing effect. Compared with that of the inhibitor-free sample, the optimal composition WC–10Co – 0.4 wt. % VC exhibits a reduced average grain size from 748 to 519 nm, improved hardness from 1498±73 to 1973±53 HV and increased flexural strength from 1036 to 2586 MPa. Therefore, the addition of grain growth inhibitor effectively prevented the formation of large WC grains. However, the amount of VC should be controlled within a certain range to achieve the optimal mechanical properties of the cemented carbides.

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Department of Mechanical & Materials Engineering
Faculty of Engineering & Built Environment
Universiti Kebangsaan Malaysia
43600 Bangi, Selangor D.E.
Malaysia

*Corresponding author; email: abubakar@ukm.edu.my