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The Effect of Cobalt, Vanadium Carbide and PKS Activated Carbon Addition on WC-Co Composite: A Study using Taguchi Method

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ABSTRACT

WC-Co, which is also known as cemented carbide, is widely used in metal cutting industry and wear related applications. Numerous studies have been made to improve the properties of the composite. Thus, this research paper is focusing on selection of WC-Co formulation using Taguchi method based on design factors such as percentage of cobalt, vanadium carbide and PKS activated carbon. The WC-Co composite is produced using powder metallurgy technique and sintered under nitrogen-based atmosphere. Based on the results obtained using Taguchi orthogonal array, it is found that WC-8Co-0.3C-0.4VC and WC-6Co-0.4VC are the optimized combination of levels for all the three control factor for hardness and flexural strength respectively.

Keywords: *WC-Co; cemented carbide; powder metallurgy; vanadium carbide; PKS activated carbon*

Introduction

Cemented tungsten carbide, WC-Co is widely used in various machining-related applications due to its superior properties. WC-Co exhibit high and stable hardness over wide range of temperature, high elastic modulus, high

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thermal conductivity and low thermal expansion. These properties make the composite versatile and cost-effective tool and materials for wide range of applications. Although WC-Co has high physical and mechanical properties, researchers continue to seek improvement of the composite to meet industries demand. Due to the needs of increasing production and reducing manufacturing cost, machining operation is always targeted to solve these matters. Therefore, the properties improvement of the composite is inevitable with study on nanocrystalline WC grains have become hot issue over the last decade.

The synthesis and sintering of ultrafine powders to produce nanocrystalline WC grain structure has potential to increase its hardness while maintaining the toughness of the composite. It is reported that uniformly distributed grains with size of 300 nm improves the mechanical properties and cutting performance of WC-Co composite [1]. Beside WC, using finer particle size of powder for cobalt also offers several advantages such as increasing fracture toughness and wear resistance. Furthermore, it is also known to be effective in reducing residual porosity and cobalt pooling [2]. The content of cobalt significantly affects the properties of WC-Co with the increment of its composition reduce hardness, strength and wear resistance but increase the toughness [3, 4].

Despite enhancing the physical and mechanical properties, fine powder particles are extremely sensitive to processing condition due to its high reactivity, which often leads to WC grain growth [5-7]. Grain growth occurs during sintering process through dissolution of small WC grain and reprecipitates on larger grains in liquid Co, in which rapid grain growth during heat-up and normal grain growth during isothermal holding [8, 9]. However, addition of hard refractory particles (also known as grain growth inhibitor) could produce pinning effect and inhibit grain growth [2, 8, 10]. This is because the solubility of WC in the liquid phase saturated with inhibitor carbide is decrease, hence reducing its coarsening rate [5]. It is reported that vanadium carbide, VC is the best grain growth inhibitor and very effective in limiting normal and abnormal grain growth [5, 7, 9, 11-12]. VC acts as grain growth inhibitor by forming precipitates along WC grain boundaries, reducing the affinity and migration of Co onto WC particles [7, 11].

Addition of small amount of free carbon also could act as grain growth inhibitor [5]. Other than that, free carbon also could enhance densification process of WC-Co composite [14]. Furthermore, it can prevent decarburization of WC during sintering process which results to formation of brittle complex carbide due to deficiency of carbon content [11, 15, 16]. However, few works have been done on addition of free carbon using activated carbon from palm kernel shell, PKS. Local carbon from agricultural waste is expected to become competitive due to price hike of coal and petroleum. Increasing of environmental awareness also plays major role in utilization of local carbon since Malaysia is one of the largest oil palm producers in the world, contributing to high amount of waste which is unfavourable to environment [17, 18].

Although research on PKS carbon in WC-Co is lacking, but various studies on the materials in hard and friction applications have been made such as automobile disc brake and brake pad [19, 20]. This work may serve as preliminary studies on applicability of PKS carbon as an addition in cemented carbide. This work also investigates the effect of cobalt, vanadium carbide and PKS carbon composition on physical and mechanical properties of WC-Co composite.

Methodology

Design of Experiment

Taguchi method consists of L_{16} orthogonal array was selected using Minitab statistical software. Design of experiment was done to optimize WC-Co formulation to obtain high microhardness and flexural strength which are important properties of WC-Co. Three factors and four levels were chosen to cover the experimental region such as shown in Table 1 and Table 2, with total of 16 samples to be produced. The responses in this study are hardness and flexural strength, where larger value is better.

Consolidation of WC-Co

Before WC-Co was consolidate, PKS carbon was crushed to fine powders for 3 hours as preparation for mixing process. The particle size of PKS carbon after crushing at D50 using particle size analyser is 5.41 µm meanwhile particle size for WC, Co and VC are 0.8 µm, 3.51 µm and 5.06 µm respectively. WC-Co composite was consolidated using wet mixing process using turbula mixer based on the formulation generated by Taguchi orthogonal array.

Table 1. Design parameters for WC-Co composition.

Symbol	Factors	Level, wt%			
		1	2	3	4
A	Cobalt	5.0	6.0	7.0	8.0
B	PKS Carbon	0	0.1	0.2	0.3
C	Vanadium Carbide	0.4	0.5	0.6	0.7

Table 2. Design of Taguchi orthogonal array L_{16}.

Sample Identification	Parameters and Levels		
	A	B	C
S1	1	1	1
S2	1	2	2
S3	1	3	3
S4	1	4	4
S5	2	1	2
S6	2	2	1
S7	2	3	4
S8	2	4	3
S9	3	1	3
S10	3	2	4
S11	3	3	1
S12	3	4	2
S13	4	1	4
S14	4	2	3
S15	4	3	2
S16	4	4	1

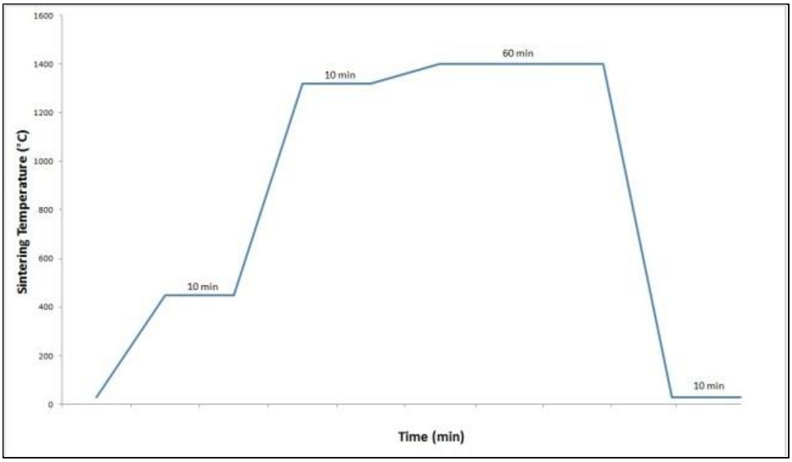


Figure 1. Sintering schedule for WC-Co composite.

The green body was formed using uniaxial pressing application at 625 MPa. Cold-isostatic pressing is introduced in-between compaction and sintering process to obtain a denser and uniform green density distribution. In this work, the green body was subjected to a pressure of 200 MPa. The composites were sintered in tube furnace under nitrogen-based atmosphere (95% N_2 – 5% H_2). The sintering schedule is such as shown in Figure 1.

Results and Discussions

Holding steps are introduced during sintering of WC-Co composites since it is proven to be more effective compared to direct heating and offers several advantages. This is because holding step offers several advantages (at different temperature) such as eliminates residual gases, promote melting and homogeneous distribution of cobalt [2, 21, 22]. Therefore, introduction of holding steps during sintering process is crucial to enhance densification and development of rigid WC skeletal structure.

Table 3 shows the density, hardness and flexural strength of WC-Co sintered samples. Although density is not include as one of the response in Taguchi orthogonal array, its results are still considered as important since both hardness and flexural strength can be correlate with density. Based on the result obtained, it is found that the density of WC-Co is generally lower when the composition contains high VC content, which is 0.6-0.7%. This finding is similar to several literatures which reported that addition of VC slightly reduces WC-Co sintered density [8, 13]. However, cobalt and PKS carbon content does not have any significant effect on the density.

Table 3. Physical and mechanical properties of WC-Co.

Sample Identification	Density, g/cm^3	Hardness, HV	Flexural Strength, MPa
S1	13.42	188.4	220.9
S2	13.23	798.7	203.7
S3	12.96	619.9	204.8
S4	12.73	908.8	198.6
S5	13.86	465.2	245.6
S6	13.58	739.5	306.8
S7	13.14	701.8	247.9
S8	12.77	648.5	198.8
S9	13.03	953.1	234.6
S10	13.88	410.9	185.4
S11	12.90	897.7	142.3
S12	13.10	719.1	161.8
S13	12.75	1254.8	187.7
S14	12.84	914.9	191.3
S15	13.03	749.1	169.1

S16	13.34	1513.9	203.2

High hardness value were observed in sample S13 (WC-8Co-0.7VC) and S16 (WC-8Co-0.3C-0.4VC). It is strongly suggested that the increase in hardness is because of VC and PKS act as grain growth inhibitor and control the grain size of WC particles [8, 10, 13]. Hence, prevent grain growth from occurring which can lead to reduction of WC-Co properties. It is also believe that high percentage of cobalt content also contributes to the increment due to the increase amount of liquid phase formation in the composite [21]. The flexural strength for all the samples is comparable to each other, but lower compared to commercial sample. This is probably due to the addition of VC that could leads to formation of porous structure, thus explains the lower density obtained for several samples [2, 11, 13].

Table 4. Percentage of contribution for S/N ratio and means value for hardness.

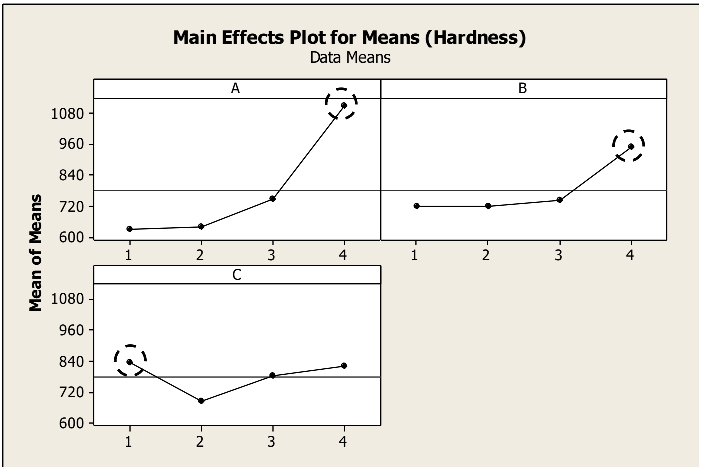
Factors	DOF	SS S/N	SS Mean	S/N Contribution (%)	Mean Contribution (%)
Cobalt	3	77.5	606709	29.79	40.89
PKS Carbon	3	31.7	151152	12.18	10.19
Vanadium Carbide	3	5.9	558.35	2.27	3.76

Table 5. Percentage of contribution for S/N ratio and means value for flexural strength.

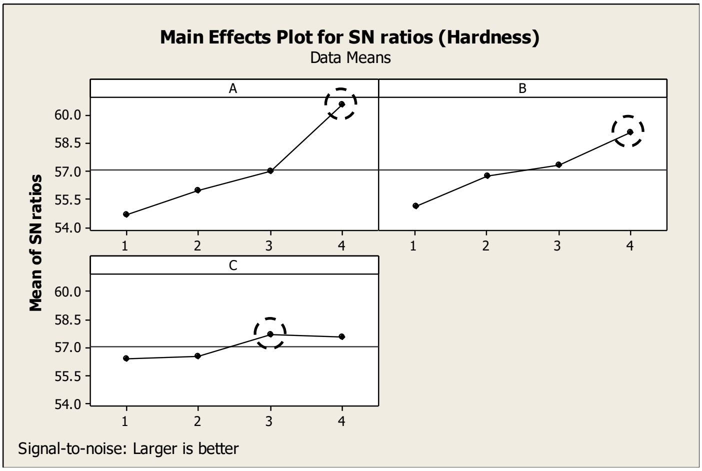
Factors	DOF	SS S/N	SS Mean	S/N Contribution (%)	Mean Contribution (%)
Cobalt	3	18.85	11492	49.44	49.93
PKS Carbon	3	6.95	3896	18.24	16.93
Vanadium Carbide	3	1.37	1097	3.59	4.77

Hardness and flexural strength results were analysed using combination of signal-to-noise (S/N) ratio, means value and analysis of variance (ANOVA) in order to identify the optimum design parameters. Table 4 and Table 5 show the percentage of contribution, obtained from

ANOVA for S/N ratio and means for each factors and responses. A higher contribution is required to determine either S/N ratio or means value to be used as optimum design parameters.



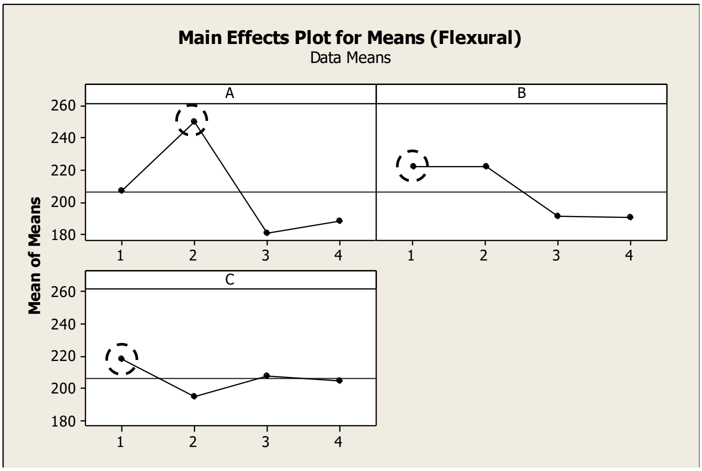
(a)



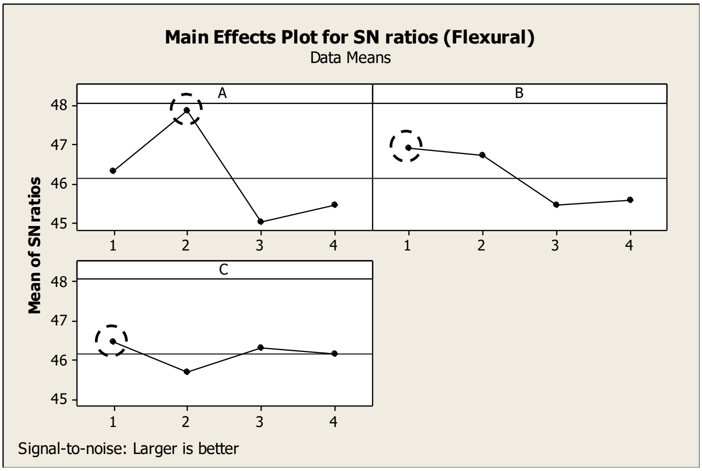
(b)

Figure 2. Optimum design parameters for hardness using (a) means value and (b) S/N ratio.

From Figure 2 and 3, a higher S/N ratio or means value corresponds to better mechanical properties where higher hardness and flexural strength can be obtained. For highest hardness value, the optimized combination of levels is WC-8Co-0.3C-0.4VC meanwhile for highest flexural strength, the combination of levels is WC-6Co-0.4VC.



(a)



(b)

Figure 3. Optimum design parameters for flexural strength using (a) means value and (b) S/N ratio.

Although there is not much difference for the cobalt content, but the formulation differs greatly in term of existence of PKS activated carbon in the composite between the optimized formulations obtained using the L16 Taguchi orthogonal array. This is because the addition of PKS activated carbon is expected to increase the hardness of the composite since it will act as free carbon agent that will enhance densification as well as inhibiting grain growth [5, 14]. However, addition of carbon will increase the brittleness of the composite, hence reduce its flexural strength.

Conclusion

L_{16} Taguchi orthogal array was generated to study on the optimization of WC-Co formulation to achieve high hardness and flexural strength. Three optimized formulations have been obtained which are WC-8Co-0.3C-0.4VC for high hardness value meanwhile WC-6Co-0.4VC for high flexural strength.

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References

[1] S. M. Asri, A. A. Mahaidin, S. A. Manaf and T. R. Jaafar, "Microstructure, mechanical properties and cutting performance of sintered sub-micron WC-6wt%Co hardmetal powder," *Proceedings of 19th Scientific Conference of Electron Microscopy Society of Malaysia* (2010).

[2] G. –H. Lee and S. Kang, "Sintering of nano-sized WC-Co powders produced by a gas reduction-carburization process," *Journal of Alloy Compounds* 419 (1-2), 281-289 (2006).

[3] S. Kalpakjian and S. R. Schmid, Manufacturing: Engineering and Technology (Pearson, Singapore, 2014), pp. 600-616.

[4] Y. V. Milman, S. Luyckx, V. A. Goncharuck and J. T. Northrop, "Results from bending tests on submicron and micron WC-Co grades at elevated temperature," *International Journal of Refractory Metals and Hard Materials* 20 (1), 71-79 (2002).

[5] Z. Yao, J. J. Stiglich and T. S. Sudarshan, "Nanosized WC-Co holds promise for the future," *Metal Powder Report* 53 (3), 20-33 (1998).

[6] J. M. Olson and M. M. Makhlouf, "Grain growth at the free surface of WC-Co materials," *Journal of Materials Science* 36, 3027-3033 (2001).

[7] R. Gonzalez, A. Ordonez and J. M. Sanchez, "HIP after sintering of ultrafine WC-Co," *International Journal of Refractory Metals and Hard Materials* 23 (3), 193-198 (2005).

[8] D. –H. Xiao, Y. –H. He, W. –H. Luo and S. Min, "Effect of VC and NbC additions on microstructure and properties of ultrafine WC-10Co cemented carbides," *Transactions of Nonferrous Metal Society of China* 19, 1250-1255 (2009).

[9] X. Wang, Z. Z. Fang and H. Y. Sohn, "Grain growth during the early stage of sintering of nanosized WC-Co powder," *International Journal of Refractory Metals and Hard Materials* 26, 236-241(2008).

[10] W. Acchar, C. Zolfrank and P. Greil, "Microstructure and mechanical properties of WC-Co reinforced with NbC," *Materials Research* 7 (3), 445-450 (2004).

[11] F. A. Costa Oliveira, A. C. Lopes, J. C. Fernandez, J. Sacramento and M. A. Valente, "Fracture behaviour of a new submicron grained cemented carbide*," Fracture Behaviours of Hardmetals* 20 (1-2) (2008).

[12] S. G. Huang, L. Li, K. Vanmeensel, O. Van der Biest and J. Vleugels, "VC, Cr_3C_2 and NbC doped WC-Co cemented carbide prepared by pulse electric current sintering," *International Journal of Refractory Metals and Hard Materials* 25, 417-422 (2007).

[13] L. Sun, C. Jia, R. Cao and C. Lin, "Effect of Cr_3C_2 additions on the densification, grain growth and properties of ultrafine WC-11Co composites by spark plasma sintering," *International Journal of Refractory Metals and Hard Materials* 26 (4), 357-361 (2007).

[14] S. I. Cha and S. H. Hang, "Microstructure of binderless tungsten carbides sintered by spark plasma sintering process," *Materials Science and Engineering* A356, 381-389 (2003).

[15] R. P. Singh, "Synthesis and characterization of tungsten carbide nanoparticles," Masters thesis, Thapar University, Patiala (2008).

[16] C. –J. Liu, A. Ohmori and Y. Harada, "Effect of powder structure on the structure of thermally sprayed WC-Co," *Journal of Materials Science* 31, 785-794 (1996).

[17] I. M. Dagwa, P. F. Builder and J. Achebo, " Characterization of palm kernel shell powder for use in polymer matrix composites," *International Journal of Mechanical and Mechatronics Engineering* 12 (4), 88-93 (2012).

[18] Allwar, A. M. Noor, and M. A. M. Nawi, "Textural characteristics of activated carbon prepared from oil palm shells activated with $ZnCl_2$ and pyrolisis under nitrogen and carbon dioxide," *Journal of Physical Science* 19 (2), 93-104 (2008).

[19] O. A. Koya and T. R. Fono, "Palm kernel shell in the manufacture of automotive brake pad," *Technoexpo*, (2009).

[20] D. R. Reddy, S. K. Singh and B. N. Banoth, "Development of a new bio-degradable friction material for brake pads from kernel shell," *International Journal of Mechanical Engineering and Materials Science* 4 (1), 1-6 (2011).
[21] N. I. Izura, M. A. Selamat, N. M. Diah and T. R. Jaafar, "The evaluation of microstructure and mechanical properties of sintered sub-micron WC-Co powders," *Proceeding of National Metallurgical Conference* (2007).
[22] M. A. Selamat, S. A. Manaf, N. M. Diah and T. R. Jaafar, "Powder metallurgy processing of hardmetal powder," *Solid State Science and Technology* 18 (1), 194-201 (2010).