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Investigating the effect of sintering temperature on the microstructure and hardness of cemented tungsten carbide/steel bilayer

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Abstract. In this article, the effect of sintering temperature was studied on the microstructure and hardness of cemented tungsten carbide and steel bilayer fabricated via the powder metallurgy route. Cemented tungsten carbide was reinforced with Fe and also used as the base material for the other layer to ensure compatibility between layers. Carbon addition was varied in the steel part composition to avoid carbon-deficient bilayer samples. Optical images revealed cracks at the interface which opened up more in bilayer samples sintered at 1295°C due to high mismatch in strain rate and excessive diffusion rate. Increase in sintering temperature turned grain particles coarser and lead to a decline in hardness values while increase in carbon addition enhanced densification and progressively increased hardness. Hardness values measured far away from the interface were observed higher than those close to the interface due to the formation of weak bond at the interface.

1. Introduction

Multilayered components are preferred over single components as they incorporate unique properties from selected materials within a component to meet specific demands. Cemented tungsten carbide is commonly used as cutting and drilling tools by virtue of its superior hardness and wear resistance properties [1,2]. Meanwhile, Fe–W–C steel on the other hand is majorly known for its excellent toughness property [3] which makes the combination of both components a suitable requirement in machine industries. Among the numerous processing techniques used in fabricating multifunctional components for industrial applications, powder metallurgy (PM) has gained a wider interest from researchers owing to its economic importance, flexibility and near-net production of components [4,5]. Manufacturing of tailored components via PM employ limited processing steps of powder blending, co-compaction which ensures handling of pressed compacts with green strength and co-sintering which is the major step of this route as it consolidates different layers through inter-particle reaction, diffusion and consequently bonding. Co-sintering temperature which is important in the PM route is often obtained from thermochemical calculations, phase diagrams and experimental analysis of individual materials involved [6]. Therefore, the co-sintering temperature must be carefully selected and monitored to ensure acceptable properties and good quality of sintered bilayer compacts.

The adhesive strength and integrity of co-sintered multilayered components is influenced by several factors arising from powder compositions, sintering parameters (sintering temperature, time, rate, environment) during the sintering cycle which when not closely studied and controlled could result in shrinkage mismatch, interfacial separation/fracture, presence of detrimental phases. While trying to adjust powder composition to ensure compatibility of different layers in a bilayer/multilayer, detrimental phases may be formed around the interface [7] and increasing temperature to accelerate interlayer diffusion and enhance densification might also result in shrinkage mismatch [8] and consequently delamination. Temperature increase in processing multilayer components enhance densification and also comes with the challenge of dissimilar shrinkage of layers deteriorating the mechanical properties. However, with numerous researchers [9–11] having worked on developing multifunctional components, the challenges mentioned above have been a major set-back and understood to compromise the resultant properties and quality of these components. To develop a multilayer component with good bonding, layers must possess similar densification rate during sintering [12,13] and compatibility between layers should be ensured for interlayer diffusion.

This study aims at explicitly investigating the microstructure and hardness property after co-sintering of cemented tungsten carbide (WC–Fe–C) and steel (Fe–W–C) bilayer at different sintering temperatures. The hardness property was evaluated at both the interface region and away from the interface to gain a good understanding of the relevance of interfacial reaction to mechanical properties.

2. Experimental Procedure

In order to ensure compatibility between layers, cemented tungsten carbide was reinforced with low carbon steel as binder (WC–6wt. %Fe–0.2wt. %C). The percentage of carbon was varied in steel layer (Fe–6wt. %W–xC) layer (x=0.2, 0.4, 0.6, 0.8wt. %). High purity graphite C_{gr} (4μm), 99% pure tungsten (4–6μm) and 99.95% pure tungsten carbide (3–6μm) were all supplied by Accumet Materials, Co, Ossining, NY USA while iron by Sumitomo, Sdn. Bhd, Malaysia. 2wt. % Polyethylene Glycol (PEG 2000) dissolved in acetone was added to the powder formulations to avoid cracks later on in green compacts and mixed in a Fritsch Planetary Monomill for 3hrs at 800 rpm then dried in Hahn rotary evaporator at 56°C at 50 rpm to evaporate acetone. 7.5g and 7.0g of WC base and Fe base respectively were loaded simultaneously into a 20mm diameter cylindrical die and compacted by applying a force of 30kN using an Instron Universal Testing Machine to produce green bilayer compacts with different carbon addition (C_{gr}) shown in (Figure 1). Green bilayer compacts were then sintered using Vecstar tube furnace in two stages of (a) pre-sintering at 360°C for 1hr to ensure complete removal of PEG and (b) sintering at 1290°C and 1295°C for 1hr at heating rate of 5°C/min. The cooling rate after sintering was 20°C/min. Microstructural analysis of sintered bilayer compacts was carried out using Olympus optical microscope with the stream essentials software while hardness of sintered compacts was evaluated using Wilson Wolpert Micro-Vickers hardness tester. It is worth noting that bilayer samples will be later referred to as *MC* and variation of carbon addition (0.2, 0.4, 0.6 and 0.8wt. %) as earlier stated will be attached for clarification.
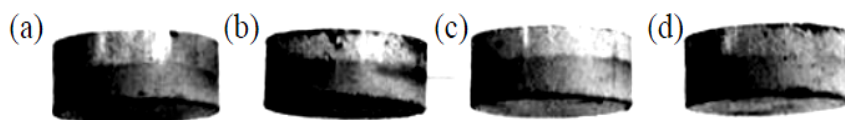

Figure 1. Green bilayer compacts with different levels of carbon content, C_{gr} (a) 0.2 (b) 0.4 (c) 0.6 (d) 0.8 wt. %.

3. Results and Discussion

3.1 Microstructure of bilayer compacts

The microstructure of bilayer compacts sintered at 1290°C and 1295°C for 1h are presented in Figure 2 and Figure 3 respectively. These optical images further confirm the reaction between layers. For

bonding to take place in bilayer through co-sintering, chemical reactions by inter-particle diffusion must take place between layers and the liquid required to initiate this reaction starts forming around 1180°C [7]. In this study, weak interface caused by high mismatch in strain rate were observed in bilayer compacts which opened up more in samples sintered at 1295°C resulting into partial contacts between layers (Figure 7). Pores (dark holes/spots) due to carbon vacancies were observed more in samples sintered at 1295°C especially in the WC base layer and gradually disappeared with increase in carbon addition as observed in (MC–0.8) Figure 3. The increase in carbon addition was shown to gradually widen cracks in both samples sintered at 1290°C and 1295°C. This is in relation to the report of [14,15] that the addition of carbon can only lead to rapid diffusion and enhance densification. It is worth mentioning that embedded in-between the layer's interface is resin used during polishing.
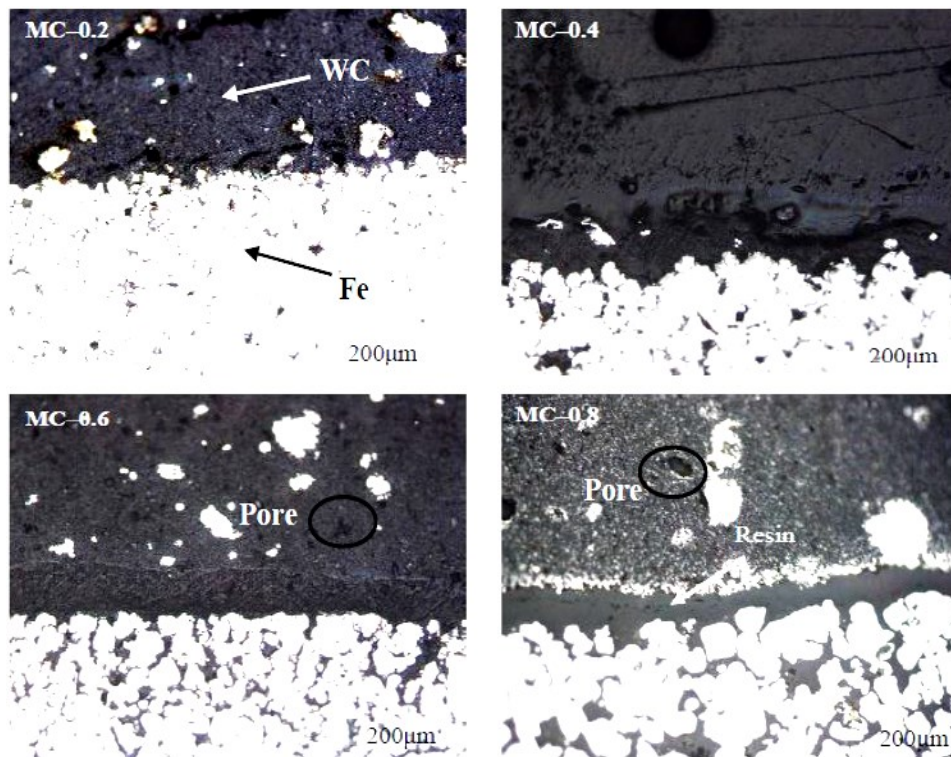

Figure 2. Optical images of microstructures at the interface of bilayer compacts with different levels of C_{gr} sintered at 1290°C.
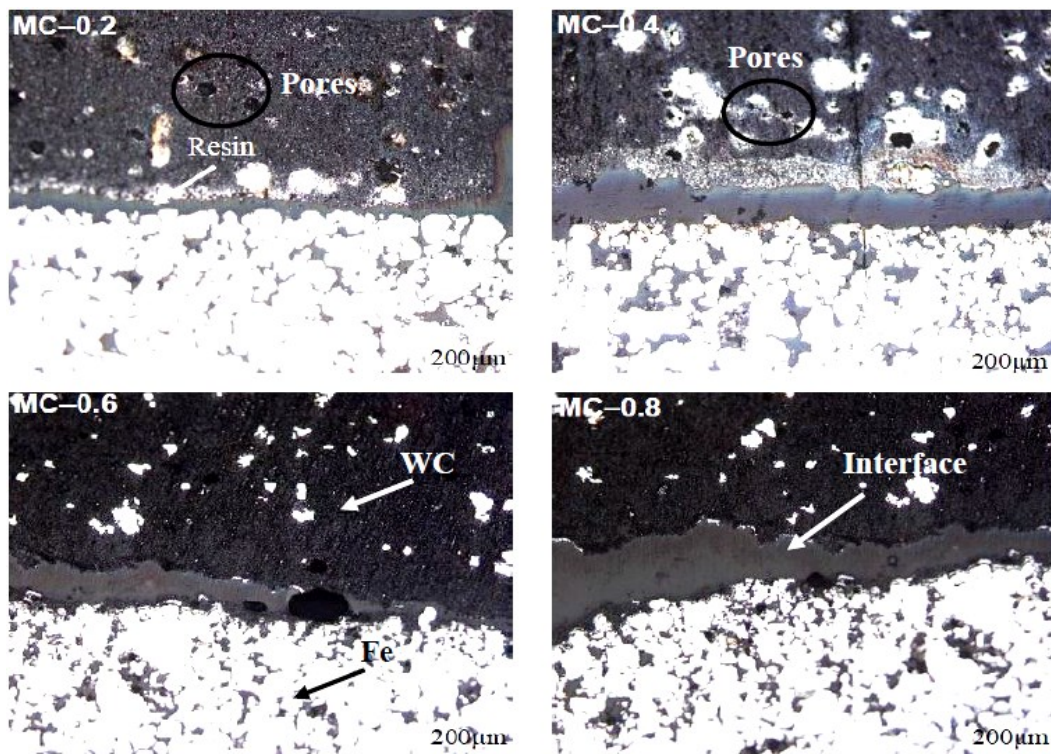
Figure 3. Optical images of microstructures at the interface of bilayer compacts with different levels of C_{gr} sintered at 1295ºC.

3.2 Hardness of bilayer compacts

A material under loading is able to resist permanent deformation due to a considerable hardness property. Therefore, in the process of trying to enhance densification of cemented carbide/steel bilayer under elevated sintering temperature, it is necessary to understand its effect on the hardness property. Hardness measurement was evaluated by automatically pressing the tip of Vickers diamond indenter on polished surfaces of already sectioned sintered bilayer compacts (Figure 4) at a load of 1000gf under a dwell time of 10s.
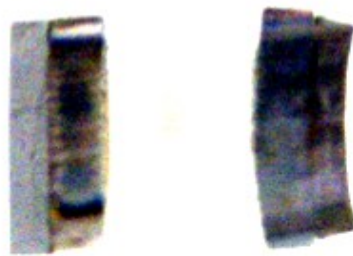

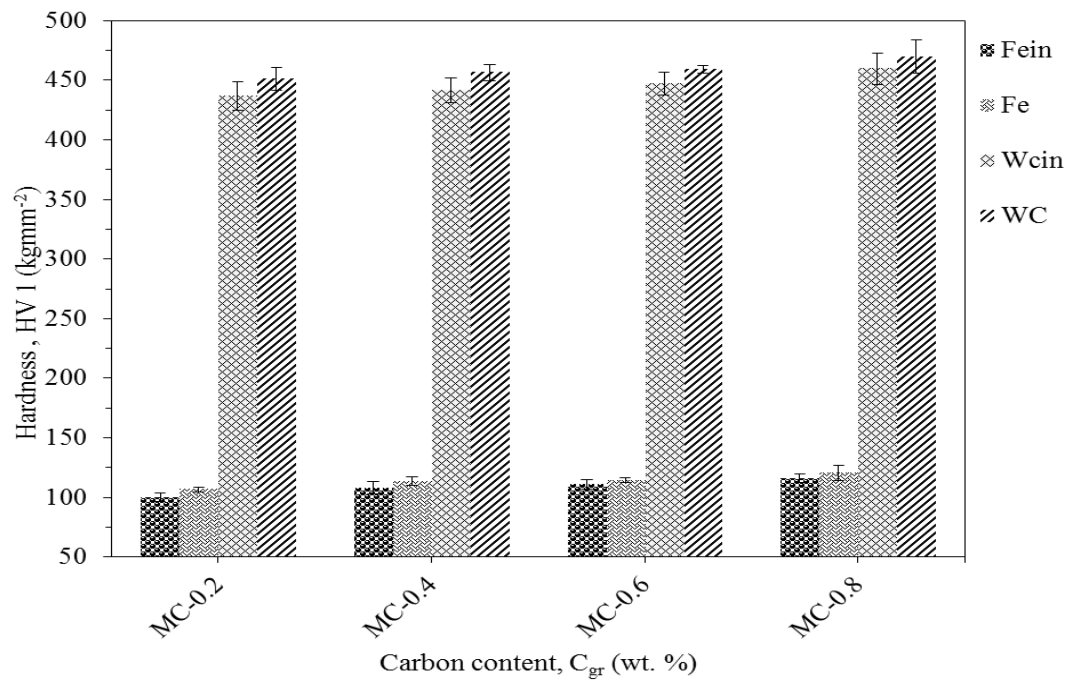**Figure 4.** Front-view of sectioned bilayer

Figure 5. Hardness results of bilayer compacts sintered at 1290°C for 1h.

As presented in Figure 5, it can be concluded that hardness values of bilayer compacts sintered at 1290°C recorded close to the interface of both steel (Fe_{in}) and cemented tungsten carbide (WC_{in}) are seen to be lower than those far away from the interface (Fe & WC). The reason can be partly attributed to the formation of weak bonding at the interface owing to excessive diffusion rate and high mismatch in strain rate which results into appearance of cracks between layers (Figure 7). The bilayer with highest carbon content (MC–0.8) sintered at 1290°C was seen to possess the highest hardness value of (120.77 and 469.80 kgmm^{-2} for Fe base and WC base respectively as compared to that of MC–0.2, 106.67 and 451.13 kgmm^{-2}). It is worth noting that addition of carbon compensates for carbon losses during sintering [3] thereby enhancing densification.
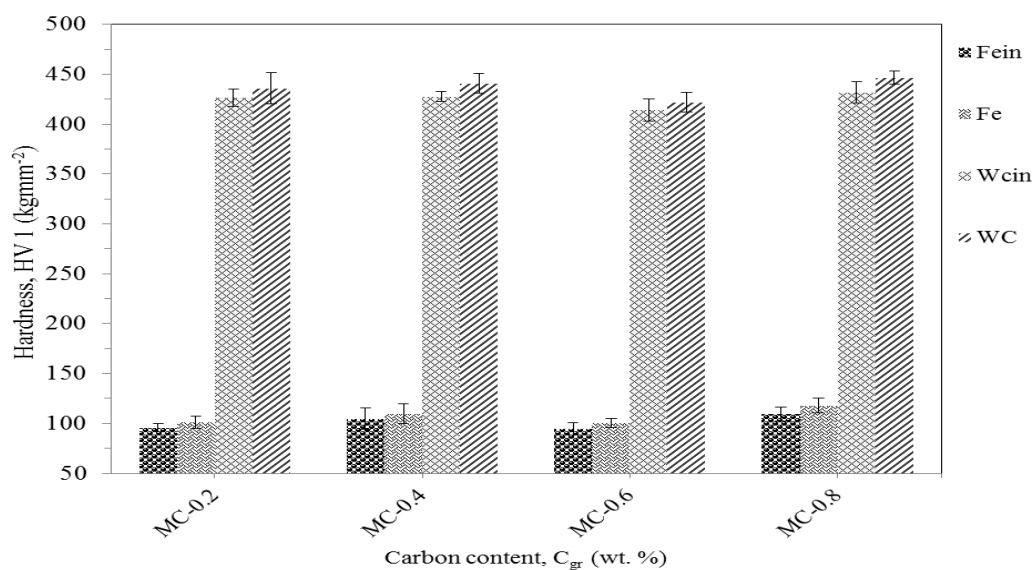

Figure 6. Hardness results of bilayer compacts sintered at 1290°C for 1h.

Hardness results of bilayer compacts sintered at 1295°C presented in Figure 6 is seen to be lower than those recorded at 1290°C. This is in accordance with the findings of [16,17] where hardness was reported to decrease with increase sintering temperature. The microstructure of the particle grain structure turns coarser with increasing temperature which may cause a decline in hardness. The finer the grain sizes of particles, the higher the hardness property [18].

Figure 7 shows crack opening at the interface of sintered bilayer compacts sintered at (a) 1290°C and (b) 1295°C. Crack opening in the latter was seen to be wider which results in weaker interface as higher temperature at a long sintering time only resulted in delamination. The coefficient of thermal expansion (CTE) play a significant role in the compatibility of layers as it determines the expansion rate during sintering and cooling rate after sintering. The CTE for tungsten carbide is known to be between $0.4 - 0.7 \times 10^{-5}$ °C^{-1} and $1.2 - 1.5 \times 10^{-5}$ °C^{-1} for austenitic stainless steel [19]. With this difference in CTE, a reduced holding time would in turn minimize interfacial defect in sintered WC/Fe bilayer.
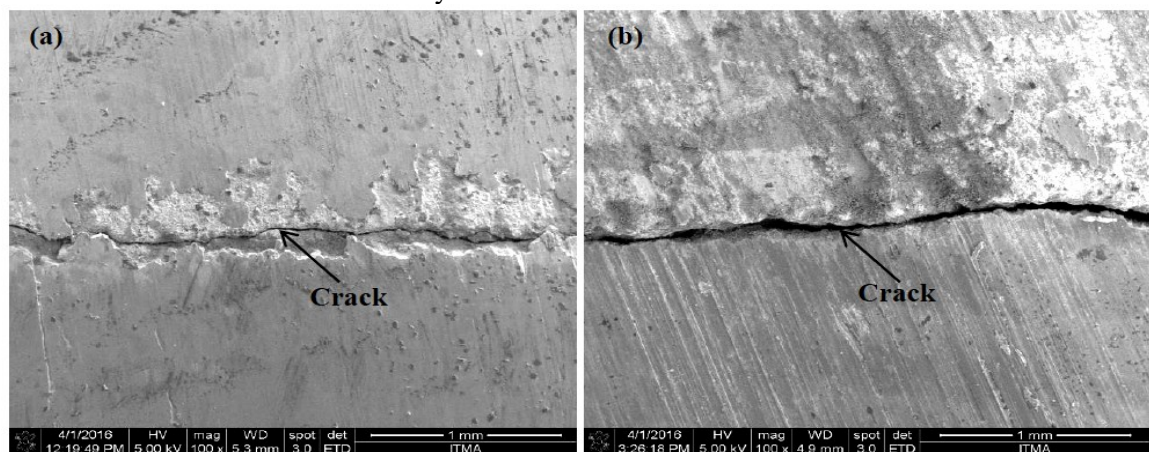

Figure 7. Crack propagation in bilayer sintered at (a) 1290°C and (b) 1295°C.

4. Conclusion

Cemented tungsten carbide and steel bilayer has been fabricated through powder metallurgy with various carbon contents in the steel part composition. Majorly, the effect of sintering temperature on the microstructure and hardness property was studied. Cracks were observed at the interface of bilayer compacts sintered at 1290°C which opened up more in samples sintered at 1295°C owing to excessive inter-particle diffusion and high mismatch in strain rate during sintering. Increase in carbon addition enhanced densification which in turn slowly widens the cracks at the interface. Hardness was observed to be on a decline as grain particles turned coarser with sintering temperature increase and values measured far away from the interface were seen higher than those close to interface which was attributed to the formation of weak bond between layers. It is suggested that the compatibility of WC/Fe bilayer at this sintering temperature can further be improved if the holding time (60mins) is reduced.

References

[1] Li T, Li Q, Fuh J Y H, Yu P C and Wu C C 2006 Effects of lower cobalt binder concentrations in sintering of tungsten carbide *Mater. Sci. Eng. A.* 430 113–119

[2] Yao Z, Stiglich J J, Sudarshan T S 1999 Nano-grained tungsten carbide–cobalt (WC/Co) *Mater. Modif.* 1–27

[3] Thomazic A, Pascal C and Chaix J M 2011 Interface reactivity and induced phenomena during sintering in a model steel/cemented carbide bimaterial. *Adv. Eng. Mater.* 13 594–598

[4] Largiller G, Bouvard D, Carry C P, Gabriel A, Müller J and Staab T 2012 Deformation and cracking during sintering of bimaterial components processed from ceramic and metal powder mixes. Part I: Experimental investigation *Mech. Mater.* 53 123–131

[5] Mosbah A Y 2001 Sintering, microstructure and properties of WC-FeAl composite materials Doctor of Philosophy Thesis, Department of Materials Engineering, University of Wollongong *http://ro.uow.edu.au/theses/1519*

[6] Pascal C, Thomazic A, Antoni-Zdziobek A and Chaix J M, 2012 Co-sintering and microstructural characterization of steel/cobalt base alloy bimaterials *J. Mater. Sci.* 47(4) 1875–1886

[7] Pascal C, Chaix J M, Doré F and Allibert C H 2009 Design of multimaterial processed by powder metallurgy: Processing of a (steel/cemented carbides) bilayer material *J. Mater. Process. Technol.* 209(3) 1254–1261

[8] Biswas K and Upadhyaya G S 1998 Co-sintering of tungsten alloy slurry coated alumina composites and their properties *Mater. Des.* 19(5) 231–240.

[9] Cai P, Green D and Messing D 1997 Constrained Densification of Alumina / Zirconia Hybrid Laminates, I: Experimental Observations of Processing Defects *J. Am. Ceram. Soc.* 80 1929–1939.

[10] Dourandish M and Simchi A 2009 Study the sintering behavior of nanocrystalline 3Y-TZP/430L stainless-steel composite layers for co-powder injection molding *J. Mater. Sci.* 44 1264–1274.

[11] German R, Heaney D and Johnson J 2005 Bi-material components using powder injection molding: densification, shape complexity, and performance attribute *Adv. Powd. Metall. Particul. Mater.* 4 41–52.

[12] Simchi A, Rota A and Imgrund P 2013 An investigation on the sintering behavior of 316L and 17-4PH stainless steel powders for graded composites *Mater. Sci. Eng. A.* 424 282–289

[13] Szewczyk-Nykiel A and Bogucki R 2018 Sinter-bonding of AISI 316L and 17-4 PH stainless steels *J. Mater. Eng. Perform.*

[14] Gonzalez R, Echeberria J, Sanchez J M and Castro F 1995 WC-(Fe, Ni, C) hardmetals with improved toughness through isothermal heat treatments *J. Mater. Sci.* 30 3435–3439

[15] Ojo-kupoluyi O J, Tahir S M, Azmah Hanim M A, Baharudin B T H T, Matori K A, Anuar M S 2017 Role of carbon addition on the microstructure and mechanical properties of cemented tungsten carbide and steel bilayer *Int. J. Adv. Manuf. Tech.* 92 3363–3371

[16] Zhao Z L, Jianwei T, Huaguo M and Xianfeng Z W 2015 Investigation on the mechanical properties of WC–Fe–Cu hard alloys *J. Alloys Compd.* 632 729-734

[17] Guo B, Zhang L, Cao L, Zhang T, Jiang F and Yan L 2018 The correction of temperature-dependent Vickers hardness of cemented carbide base on the developed high-temperature hardness tester *J. Mater. Process Technol.* 255 426-433

[18] Fang Z Z 2005 Correlation of transverse rupture strength of WC-Co with hardness *Int. J. Refract. Met. Hard Mater.* 23 119–127

[19] Brandes E A and Smithells C J 1983 Metals reference handbook *Butterworths, London.*