Solid lubricant effect on the microstructure and hardness of the

Functionally graded cemented tungsten carbide

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**Abstract**

Cemented tungsten Carbide (WC-Co) is preferred cutting tool material, having tungsten carbide (WC) reinforcement embedded in Cobalt (Co) matrix. Higher hardness, fracture toughness and wear resistance are the essential characteristics for cutting tool materials and are inherited by cemented tungsten carbide. The controlled distribution of Co composition in the form of gradient makes Functionally Graded Cemented Tungsten Carbide (FGCC) and results in customized material properties but only difficulty is Co migration. Additionally, wear resistance of FGCC is further improved by including solid lubricant in the form of gradient. The desired gradient is developed by Powder Metallurgy route using Spark Plasma Sintering (SPS), which eliminates the migration of Co. The present work deals with development of FGCC with and without solid lubricant and comparisons of their microstructure and hardness. The obtained results confirm the variations of microstructure and hardness in both FGCC samples (with and without solid lubricant). The presence of solid lubricant decreases the hardness, so FGCC without solid lubricant is having higher hardness.

***Keywords*:** Functionally graded cemented tungsten carbide; Spark Plasma Sintering; solid lubricant.

1. **Introduction**

Tungsten carbide (WC) is most suitable cutting tool material due to its high hardness, but due to its brittle nature it requires some binder phase. Usually Cobalt (Co) is preferred as a binder, addition of WC in Co matrix provide toughness to cemented carbide [1]. In cutting tool application the Co percentage varies from 5-15%.The increasing Co content improves toughness and reduces hardness. Hence it is very difficult to get balance of toughness and hardness. Functionally Graded Material (FGM) is new concept where composition can be varied according to the requirement, consequently balance between toughness and hardness can be achieved [2]. Hence cemented carbide is prepared in such a manner that surface and core of material have high WC and Co content respectively. In this way promising combination of hardness and fracture toughness can be achieved. Cemented carbide prepared with this kind of composition gradient is known as FGCC [3]. Although this concept of gradient is having enormous advantages, still it is a challenge to prepare effective gradient. Most commonly adopted method for preparation of cemented carbide is Powder Metallurgy (PM) route through liquid phase sintering. The gradient development using liquid phase sintering is difficult due to migrating nature of Co. After reaching liquid phase temperature initially deposited Co gradient homogenized. This problem was effectively encountered by Spark Plasma Sintering (SPS) method [1]. In this method it is possible to perform compaction and sintering together at solid state temperature with high heating rate. This solid state sintering at short span of time suppressed Co migration and initially deposited gradient is consolidated. FGCC is having hard surface but its wear resistance decreased at high temperature due to low hot hardness while high-speed machining. This problem is encountered by proper cooling technique at the time of machining but due to the strict environmental policies, it would be better to adopt dry machining process [4]. There are several methods are available for dry machining but the new area of research is the application of solid lubricant [4]. This solid lubricant will be included in the Co matrix as reinforcement.

The previous studies revealed that the presence of solid lubricant in composite matrix makes it self-lubricating but at the same time mechanical properties degraded [4-6]. Researchers have done extensive work to improve the mechanical properties and found that, preparation of solid lubricant gradient is the only solution to optimize the composition for performance enhancement. Solid lubricant is required only at surface so it would be better to provide composition gradient according to application [1]. In addition to this volume fraction and composition gradient can be optimized in such a way that there is development of compressive and tensile residual stresses at the surface and core respectively due to the material processing. The compressive residual stress will hinder the crack propagation and improve the life and properties of developed functionally graded material [7]. A. Xing et al. proposed design model for symmetrical functionally gradient ceramic tool materials and developed Al2O3-TiC FGM in such a way that compressive residual stresses formed on the surface layers so that the stresses created from external loadings can be neutralized [8]. J. Zhao et al. developed Al2O3-(W, Ti)C and Al2O3-TiC symmetrical functionally graded ceramic tool materials and achieved improved thermal shock resistance by composition variation [9]. Xu et al. designed and developed self-lubricating functionally graded Al2O3-(W,Ti)C cutting tool material by incorporating solid lubricant CaF2 [10]. C. Xu et al. showed that the wear resistance and antifriction property of cutting tools can be improved simultaneously by gradient self-lubricating ceramic material, with surface and middle layers having compressive and tensile stresses respectively [7, 9]. A. Muthuraja et al. has prepared monolithic cemented carbide (WC-Co) with solid lubricant (CaF2) [4]. The developed WC-Co-CaF2 tested for abrasive as well as adhesive wear resistance and found the improvement in wear resistance [5-6]. The present authors have also developed WC-Co-CaF2 composite With Co and CaF2 gradient using Spark Plasma Sintering process [1].

The present work deals with the development of Functionally Graded Cemented Tungsten Carbide with and without addition of solid lubricant. The possibility of high heating rate in Spark Plasma Sintering process makes the choice for preparation of FGCC, results in smaller grain size and suppression of Co migration. The prepared samples are characterized by microstructure and hardness to show the effect of solid lubricant on the FGCC.

1. **Experimental procedure**

WC-Co is basic cutting tool material and CaF2 is adopted as a solid lubricant because of its capability to provide lubrication at high temperature. WC-Co-CaF2 is adopted as a cutting tool material. WC is work as reinforcement in a Co binder (for cutting tool application 5-15 wt.% Co), additionally CaF2 (4-5 wt.%) is also included. These materials are mixed in a Planetary ball mill at rotational speed of 250 rev/min with the powder to ball weight ratio as 1:5 for 40 h milling time using WC balls and vial. The ball-milled material is consolidated by SPS using Graphite die and punch. The ball-milled materials are deposited in the form of layer inside the die. In present development 5 layers are selected due to die size constraint. The layer composition is decided by “Power law” and optimized in such a way that compressive and tensile residual stresses are generated at outer and middle layer respectively. Fig.1 is showing the arrangement of layers composition in the designed FGCC. The selected parameters are: 1100°C sintering temperature, 100°C/min heating rate, 100MPa consolidation pressure and 10 min holding time. In SPS Joule heating is done by simultaneous application of pulsed electric current and pressure. The consolidation process was monitored by displacement of the punch while processing. The punch also worked as electrode for generation of pulse current in deposited powder. This consolidation is performed by two mechanism solid state sintering and liquid phase sintering. The consolidated sample had cylindrical shape (20mm diameter and 5mm thickness) and characterized for microstructure and hardness.

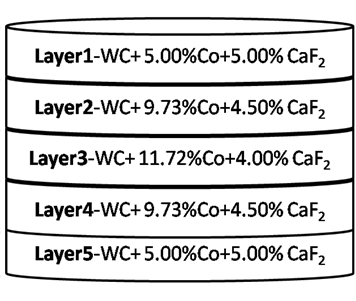


Fig. 1. Composition distribution in FGCC

1. **Result and discussion**
   1. Microstructural characterization of Spark Plasma Sintered sample

The microstructural examination of consolidated samples is done with the help of Scanning Electron Microscope (SEM). This investigation is performed on cold mounted polished samples. The polishing is performed using the different grades of paper and velvet cloth with diamond paste. These polished samples are etched by Murakami’s reagent (K3FeCN6+KOH+Water) to reveal the grains and grain boundaries. The SEM micrographs were taken at particular layers along the thickness direction so the variation of microstructure with composition can be observed. Fig.2 shows the SEM micrograph of FGCC without solid lubricant, each micrograph present a different layer. These layers are deposited symmetrically along the thickness, so outer layers (1 and 5) as well as layer 2 and 4 exhibits similar microstructure features. The sintering is performed at 1100°C by solid state sintering and results in Co neck formation between WC particles followed by Co melting, which leads to densification. The outer layers (1 and 5) are having more porosity than middle layer. Two kinds of phases are visible in the micrograph, dark and light grey phases (light grey phase represent WC and light grey phase Co). In middle layer amount of dark grey phase is comparatively high.

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Fig. 2. Micrograph of FGCC without Solid lubricant (CaF2)

FGCC with solid lubricant is also prepared at 1100°C sintering temperature. Fig. 3 is showing SEM micrograph FGCC with solid lubricant. It is also having similar features as in Fig.2, the light grey phase represent WC and dark grey phase Co. Layer 1, 5 and layer 2, 4 are similar to each other due to same composition. The outer layers are having higher porosity than middle layer. The outer layer is having less Co content as compare to middle and having less porosity due to more densification. The current microstructure observations are in line with the available literature [1, 11-12].

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Fig. 3. Micrograph of FGCC with Solid lubricant (CaF2)

* 1. Hardness of spark plasma sintered samples

The change in Co volume fraction is responsible for the microstructure variations. Similarly this Co percentage causes variation in the hardness along its thickness direction. Higher Co percentage results in lower hardness and vice versa. The hardness measurement is performed using Vickers hardness tester along the thickness direction of the polished samples. The indenter is diamond shape, dwell time is 10 sec and load is 9.8N. Hardness of FGCC without solid lubricant varied from 1341 to 1741HV at 9.8N load. The hardness value of FGCC with solid lubricant is varied from 1171-1357HV. Higher hardness is obtained at the outer layer and lowest at the middle in both samples due to Co composition gradient. The obtained hardness values are in accordance with the available literature [1, 11]. The variation in Co volume fraction in the prepared samples is confirmed by the EDS analysis and the variation is given in the Fig. 4(a).

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| E:\Desktop\New folder\Paper7\Cobalt.jpg | E:\Desktop\New folder\Paper7\Hardness.jpg |
| (a) | (b) |

Fig. 4. (a) Variation of Cobalt percentage along the thickness at different layers (b) Variation of hardness along the thickness at different layers

1. **Conclusion**

The FGCC with and without solid lubricant is successfully developed by SPS process. The variation in SEM micrograph, hardness value and EDS mapping confirms the presence of gradient. The FGCC with solid lubricant is posses more porosity and less harder than other sample due to presence of CaF2 phase. The presence CaF2 phase is responsible for the reduction in hardness of FGCC. The Co volume fraction gradient is also responsible for hardness variation; at outer layer higher hardness is observed as compare to middle layer due to less Co volume fraction. Additionally the outer layer exhibits higher porosity than middle layer due to less Co volume fraction.

**References**

[1] R. S. Parihar, S. G. Setti and R. K. Sahu, Journal of Composite Materials 52(10), 1363-1377 (2017).

[2] R. S. Parihar, , S. G. Setti and R. K. Sahu, Science and Engineering of Composite Materials, 25(2), 309-336 (2016).

[3] X. Wang, K. S. Hwang, M. Koopman, Int J Refract Met Hard Mater, 36, 46-51 (2013).

[4] A. Muthuraja and S. Senthilvelan, Int J Refract Metals Hard Mater. 48, 89-96 (2015).

[5] A. Muthuraja and S. Senthilvelan, Int J Refract Metals Hard Mater. 52, 235–244 (2015).

[6] A. Muthuraja and S. Senthilvelan, Int J Refract Metals Hard Mater. 51, 91–101 (2015).

[7] C. Xu, G. Xiao, Y. Zhang and B. Fang, Ceramics International 40, 10971–10983 (2014).

[8] A. Xing, Z. Jun, H. Chuanzhen and Z. Jianhua, Materials Science and Engineering A248, 125–131 (1998).

[9] J. Zhao, X. Ai, J. Deng and J. Wang, Journal of the European Ceramic Society 24, 847–854 (2004).

[10] C. H. Xu, G. Y. Wu, G. C. Xiao and B. Fang, Int J Refract Metals Hard Mater. 45, 125–129 (2014).

[11] S. I. Cha, S. H. Hong, and B. K. Kim, [Materials Science and Engineering: A](https://www.sciencedirect.com/science/journal/09215093) 351, 31–38 (2003).

[12] M. Eriksson, M. Radwan and Z. Shen, Int J Refract Metal Hard Mater. 36, 31-37 (2013).