School of Mechanical Engineering

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EFFECT OF PARTICLE SIZE AND SINTERING PROCESSES ON MECHANICAL PROPERTIES OF CEMENTED CARBIDES

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PhD Thesis

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ABSTRACT

Cemented carbide has been widely used as hard materials in industrial machining as cutting tools and as moulds in metal shaping. Prior research suggests that many processing techniques have been applied to improve its manufacturing conditions, microstructural characteristics and mechanical properties, namely hardness, fracture toughness and wear resistance. Although it is considered to be one of the most stable composites in terms of industrial requirements, challenges have been faced in the areas and routes of manufacturing, and consolidation with a desired set of mechanical properties via different processing techniques.

In the field of cemented carbides, the understanding of the processes such as powder refinement and consolidation or sintering, contributing to the mechanical properties is critical. The aim of this project was to examine three different groups of powder size of Tungsten Carbide-Cobalt (WC-Co), which is the dominant compositional elements among all cemented carbides, and four consolidation techniques including plasma, microwave and conventional sintering, in order to understand the relationship between these processes and mechanical properties. This study started with analysing three different particle size for both Tungsten Carbide (WC) and Cobalt (Co), which then resulted into three different types of mixed powder samples based on their size. Once the compositions were made through either high or low energy ball milling, experiments proceeded to compaction and sintering stage. The chosen composition for the study was WC – 7.5 wt. % Co, which was kept constant, while particle size, sintering process, sintering temperature, pressure and other parameters were varied. The final and overall objective was to establish a three way relationship among particle size, processing routes and mechanical properties of WC – 7.5 wt. % Co.

It was found that Pulse Plasma Sintering (PPS) method is the most successful in achieving excellent physical and mechanical properties including density (fully dense), hardness (2000 HV) and fracture toughness (15.3 MPa√m), and displays significantly improved microstructural behaviour in cemented carbides sintered at lower than conventional temperature, ensuring time and energy efficiency. Spark Plasma and Microwave Sintering were found to be the most efficient and effective after PPS in terms of mechanical

properties, considering the entire particle size range and the other variable parameters.

This thesis first outlines the basic understanding of cemented carbides, their fields of application, types, and processes that are involved during their manufacture. It then presents an overview article which is a chapter as well that presents an understanding of what has been done specifically in the areas of processing techniques, through powder refinement and consolidation highlighting the areas where challenges are faced. The mechanical properties along with certain microstructural aspects like grain growth and phases are also elaborated as part of this paper. The second paper discusses the processing of particles before sintering processes are applied and how a homogeneous and optimized mixture can be achieved. Four more chapters come from four papers that are either published or under review focusing on the different consolidation processes and their effects on mechanical properties and microstructural behaviour of WC- 7.5 wt. % Co. The thesis finally brings together a conclusion from all the separate segments of the project, highlighting the key findings and comparisons and provides information on future work.

Future work will involve further investigation on sintering mechanisms and to resolve one of the most critical question, whether the presence of plasma can be confirmed during the use of some of the modern sintering methods.

THESIS DECLARATION

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Md Raihanuzzaman Rumman, 11th May 2016

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Dedication

Mom, your words of inspiration and prayers resonated greatly with me recounting all the curiosities you entertained as I learned to categorize the world around me in a way I would like to believe, conscientious. You were, are, and always will be priceless.

List of Publication

[1] **Raihanuzzaman RM**, Xie Z, Hong SJ, Ghomashchi R. Powder Refinement, Consolidation and Mechanical Properties of Cemented Carbides—An Overview. Powder Technology. 2014;261:1-13.

[2] **Raihanuzzaman RM**, Jeong TS, Ghomashchi R, Xie Z, Hong S-J. Characterization of Short-Duration High-Energy Ball Milled WC–Co Powders and Subsequent Consolidations. Journal of Alloys and Compounds. 2014;615:S564-S8.

[3] **Raihanuzzaman RM**, Han S-T, Ghomashchi R, Kim H-S, Hong S-J. Conventional Sintering of WC with Nano-sized Co Binder: Characterization and Mechanical Behavior. International Journal of Refractory Metals and Hard Materials. 2015.

[4] **Raihanuzzaman RM**, Xie Z, Hong S-J, Ghomashchi R. Effect of Spark Plasma Sintering Pressure on Mechanical Properties of WC–7.5 wt% Nano Co. Materials & Design. 2015;68:221-7.

[5] **Raihanuzzaman RM**, Lee Chang Chuan, Zonghan Xie, Reza Ghomashchi Mechanical Properties and Microstructural Behaviour of Microwave Sintered WC – Co (Under Review).

[6] **Raihanuzzaman RM**, Marcin Rosinski, Zonghan Xie, Reza Ghomashchi Mechanical Properties and Microstructural Behaviour of Pulse Plasma Sintered WC – Co (Under Review).

LIST OF ADDITIONAL PUBLICATIONS

Not included in this thesis

[1] **Raihanuzzaman RM**, Chuan LC, Xie Z, Ghomashchi R. Microwave Sintering and Its Application on Cemented Carbides. World Academy of Science, Engineering and Technology, International Journal of Chemical, Molecular, Nuclear, Materials and Metallurgical Engineering. 2015;9:930-3.

[2] Song J-W, **Raihanuzzaman RM**, Lee I, Hong S-J, Myung Koo J. Refinement Behavior of Scrap Silicon by Mechanical Milling. Current Nanoscience. 2014;10:441-4.

[3] **Raihanuzzaman RM**, Park H, Ghomashchi R, Kwon T, Son H-T, Hong S. Effect Of Ti Powder Addition On The Fabrication Of TiO_2 Nanopowders. Archives of Metallurgy and Materials. 2015;60:1473-7.

[4] Song J-W, **Raihanuzzaman RM**, Hong S-J. Consolidation of WC–Co Alloys by Magnetic Pulsed Compaction and Evaluation of their Mechanical Properties. Powder Technology. 2013;235:723-7.

[5] Song J-W, **Raihanuzzaman RM**, Hong S-J. Mechanical Properties of WC-Co Alloys with Various Mixing and Milling Conditions using High Energy Ball Miller. Current Nanoscience. 2014;10:62-5.

Summary of Each Paper

Paper 1 (Published)

Cemented carbides, owing to their excellent mechanical properties, have been of immense interest in the field of hard materials for the past few decades. Although a number of processing techniques have been developed to obtain nanostructured cemented carbide powders with grain size of less than 100 nm, they are expensive, as such, the challenge remains for producing nano-sized powders economically. The lack of understanding and control of grain growth and its effect on deformation and fracture of the resulting carbide materials is another area that requires proper attention. In addition, the effect of binder materials and their content on the mechanical properties of cemented carbides is not clearly understood yet. This review aims to address some of the key issues and challenges faced in the research and development of cemented carbides, especially on powder refinement and consolidation, and their effect on mechanical properties.

Paper 2 (Published)

The aim of this study was to observe the effect of short-duration, high-energy ball milling on the mechanical properties of sintered WC–Co. Mixed powders of WC–7.5 wt. % nano Co were ball milled at three different time using WC vial and balls, while other parameters were kept constant. The powders were then consolidated using spark plasma sintering. Density and hardness of the sintered samples were measured as a function of milling time, used to mix the powders prior to consolidation. It was found that both density and hardness increased with milling time, with hardness reaching a maximum of 14.95 GPa for the sample milled for 10 min. Microstructures of the sintered samples suggested a slight decline in grain size and increase in Co distribution with increasing milling time. It was also evident that milling of WC–Co powders resulted in the sintered samples having overall irregular shaped grains.

Paper 3 (Published)

In this study, WC particles of 1–3 μm were blended with two different sizes of Co particles and sintered conventionally at two different temperatures. Compaction of initial powders was carried out using a relatively new dynamic compaction method called Magnetic Pulsed compaction (MPC). The maximum Vickers hardness for the samples found was around 1353 HV (13.27 GPa), while the maximum fracture toughness was observed at 4.6 $MPa \cdot m^{1/2}$. The marked changes in density, hardness, fracture toughness and crack behaviour observed in the samples indicate strong correlations among particle size, sintering process and mechanical properties of cemented carbides. In addition, the nature of crack initiation and propagation, which is indicative of the trend in fracture toughness of materials, was observed and analysed.

Paper 4 (Published)

In this study, Spark Plasma Sintering (SPS), a relatively fast consolidation method assisted with simultaneous application of heat and pressure, was used to produce bulk carbides from mixed WC–7.5 wt. % Nano Co powders. Different pressures ranging from 30 to 80 MPa were applied during sintering to explore its effect on the mechanical properties of resulting carbides. The maximum hardness was found for the samples pressed at 80 MPa, which is ~1925 HV (18.88 GPa), higher than that of the commercially available cemented carbide tools. The marked changes in porosity, hardness, and crack propagation observed in the samples show that the sintering pressure have considerable impact on the mechanical properties of cemented carbides. In particular, the nature of crack initiation and propagation, which is indicative of the fracture toughness of materials, was studied and clarified.

Paper 5 (Under Review)

Tungsten or in general cemented carbides have been of great interest within industrially manufacturable hard materials for their excellent mechanical properties, especially in the last few decades, with a number of new and revamped processing techniques emerging in order to develop high performance carbide tools. Microwave sintering or heating is known for its application on a range of materials, which includes ceramics as well. Although it has been widely used, its effect however on grain growth of materials still requires clear understanding. Furthermore, the effect of sintering temperature and initial particle size and how they influence microwave behaviour for such range of materials is another area that requires clear understanding. This article focuses on microwave sintering being used on cemented carbides using a range of final sintering temperatures and initial particle sizes, which sheds light on the entire process and its effectiveness on ceramic processing. The microstructural features associated to the sintering process have also been focused as part of the study, addressing some of the key issues and challenges faced in the research.

Paper 6 (Under Review)

Tungsten carbides-based inserts have been considered as one of the dominant hard materials in the cutting industry, receiving great interest for their excellent combination of mechanical properties. Pulse plasma sintering (PPS) process has been applied to a series of WC-Co samples with varying sintering temperature, initial particle size and sintering pressure in order to study the mechanical and microstructural behaviour. The quality of the products as well as the mechanical properties and microstructural features this process yields are commendable and worth looking into. A high hardness of more than 2000 HV has been achieved while a maximum fracture by toughness of 15 MPa. $m^{1/2}$ was found in samples that were sintered at 1100 ^{o}C and 100 MPa. Microstructural features like grain growth and other properties are discussed with respect to the varying parameters. While grain size shows an incremental pattern with increasing temperature, it was still possible to limit them to a great extent ensuring high mechanical properties. The effect of sintering pressure in the range of 60-100 MPa, while keeping sintering temperature constant was found to be almost negligible.