UNIVERSITI TEKNIKAL MALAYSIA MELAKA

IMPROVING THE DENSIFICATION OF TUNGSTEN CARBIDE BY LIQUID PHASE SINTERING

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor's Degree in Manufacturing Engineering Technology (Process & Technology) (Hons.)

by

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APPROVAL

This report is submitted to the Faculty of Engineering Technology of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering Technology (Process & Technology) (Hons.). The member of the supervisory is as follow:

………………………………

(Project Supervisor)

ABSTRAK

Dalam projek ini, tungsten karbida, WC seramik dihasilkan dengan menggunakan kaedah pensinteran fasa cecair, LPS untuk meningkatkan penumpatan. Umumnya, WC memerlukan suhu yang tinggi bagi meningkatkan penumpatannya. Oleh itu, agen pensinteran telah ditambah untuk mengurangkan suhu pensinteran bagi meningkatkan penumpatannya. Bagi menangani masalah ini, WC-Co telah dikaji dengan profil pensinteran dan parameter yang berbeza. Projek ini mengkaji kesan komposisi kandungan Co, suhu pensinteran dan masa rendaman dalam menghasilkan penumpatan yang lebih baik. Dua komposisi kandungan Co iaitu 3 dan 12 wt.% Co dipilih. Sampel WC-Co telah dibakar pada suhu pensinteran yang berbeza dalam julat 1200-1400°C. Dalam usaha untuk mendapatkan sampel yang tumpat, suhu pembakaran optimum 1350^{o}C telah dipilih dengan masa rendaman yang berbeza. Pencirian dan pengukuran WC-Co dikaji dari segi pembentukan fasa, analisis unsur, saiz partikel, ketumpatan dan kekerasan. Berdasarkan kajian ini, WC-12 wt.% Co dengan masa rendaman selama 7 jam menunjukkan prestasi yang baik dari segi kekerasan yang tinggi (10.9430x10^{-6} GPa), ketumpatan pukal tinggi (11.3103 g/cm^{3}) dan mikrostruktur seragam.

ABSTRACT

In this work, the fabrication of tungsten carbide, WC ceramic by using liquid phase sintering (LPS) was investigated to improve the densification. In general, WC requires high temperature to improve its densification. Thus, sintering aid was added to reduce sintering temperature in order to improve densification. In order to tackle this problem, WC-Co was studied with different sintering profiles and parameters. This work studied the effect of composition of Co content, sintering temperature and soaking time which in turn to improve densification. Two composition of Co contents; 3 and 12 wt.% Co are selected. The WC-Co samples were sintered at different sintering temperature in the range of 1200-1400°C. In order to obtain densified sample, the optimum sintering temperature of 1350^{o}C was selected with different soaking times. The characterization and measurement of WC-Co was studied in term of phase formation, elemental analysis, particle size, density and hardness of WC. Based on this work, WC-12 wt.% Co with soaking time of 7 hours showed a good performance in term of high hardness (10.9430x10^{-6} GPa), high bulk density(11.3103 g/cm^3) with uniform microstructure.

DEDICATION

To my beloved parents

ACKNOWLEDGEMENT

Bismillahhirahmannirahhim, with the name of Allah S.W.T, we felt very thankful for those who are given support and guide in order to finish this work. Peace and blessings is upon Muhammad S.A.W, the true Messenger of Allahfor this bountiful bond of loves to his ummah.

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LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

WC	-	Tungsten Carbide
Co	-	Cobalt
Ni	-	Nickel
ASTM	-	American Society for Testing and Materials
XRD	-	X-Ray diffraction
FESEM	-	Field emission scanning electron microscopy
GPa	-	Gigapascal
%	-	Percent sign
^{o}C	-	Degree celcius
θ	-	Theta

CHAPTER 1
INTRODUCTION

This chapter describes research background, problem statement, objectives, and scope of project as a general view about this project. The structure of this research is briefly explained as well to ensure a better visualization of the sequence of the entire study.

1.1 Research background

Tungsten carbide with molecular formula; WC is an inorganic chemical compound from a group of transition metal IV-VI. It can be referred as carbide, whereby it consists of two different elements namely tungsten and carbon with equivalent ratio. It is also known that dense WC exhibits outstanding results in mechanical properties i.e. high hardness (18-22 GPa), high tensile modulus (700 GPa), high melting point (2870^{o}C) and good thermal expansion coefficient (5.5×10^{-6} K^{-1}) in comparison to other transition metal carbides. It was reported that the mechanical properties of WC is dependent on particle size, whereby its hardness, wear resistance and transverse rapture strength are relatively high with decrease in particle size (Kurlov and Gusev, 2013).

WC based cemented carbides have been served as important cutting tools and wear resistant parts due to their excellent mechanical properties. However, the use of metallic binders limits its corrosion resistance ability as well as high temperature performance. Considering this, binderless WC cemented carbides have been attracted much attention in recent years. In order to overcome the sintering difficulty of this covalent material, which is attributed to their rigid covalent bonds and low diffusion mobility, fine WC powder with the enhanced sinterability is desired.

In general, WC is difficult to achieve high densification by using Solid Phase Sintering (SPS) method. This is because the full sintered density of WC can be achieved when temperatures as high as 2700^{o}C is employed (Yuttanant, 2009). As related, the sintering aid is use to help WC achieve fully densification. According to Wei (2014), WC-Co hardmetals consists of hard phase WC and ductile phase Co, are preparing by liquid phase sintering (LPS). Yuttanant (2009) said that addition of Group VII transitions metals including Ni, Pd, Pt, Co, and Fe can beneficially enhance the sintering kinetic of W powder, thus reduce its sintering temperature.

1.2 Problem statement

In the earlier section, WC has a great performance in mechanical properties. Nevertheless, there is obvious drawback during densification at elevated temperature. In order to tackle this issue, added of sintering aid will be studied in term of weight content, sintering temperature and soaking time towards densification of WC.

1.3 Research objectives

Several objectives that are significant to be studied are listed as follows:

i. To fabricate WC with Co added samples by Liquid Phase Sintering (LPS).
ii. To improve densification of WC-Co samples.

1.4 Scope of research

In general, this work is more focused on the fabrication and characterization of WC samples. As stated earlier, LPS is mainly used for densification whereby three parameters will be studied in terms of sintering aid content, sintering temperature and soaking time. The optimized will be selected for further characterization i.e. hardness, density, phase formation and microstructure.

Generally, this work can be divided into three main parts. Figure 1.1 shows flowchart the scope work involved in this study.

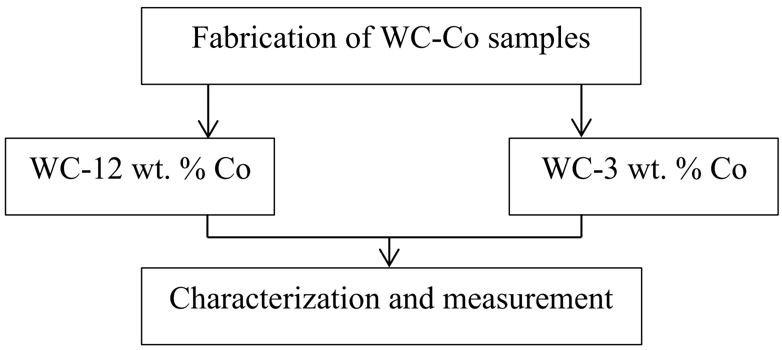

Figure 1.1: Flowchart of the study

CHAPTER 2
LITERATURE REVIEW

In this chapter covered previous study of tungsten carbide, WC in term of background, properties and application in industry. Besides that, this chapter describes mechanical and physical properties of tungsten based material in respond to have the best selection of high strength material for military components.

2.1 Tungsten carbide, WC

WC is combination of two groups of materials that are metals and ceramics. Agnieszka et.al, (2014) pointed out that polycrystalline WC belongs to the group of materials that called sintered carbides that have defined in the following ways. Based on definition WC from Agnieszka et al.,(2014), WC is a group of material that consist of mixture of one or more of finely divided tungsten, titanium, and tantalum and vanadium carbides in a matrix of cobalt or nickel. Most of previous study stated about mechanical properties of WC in term of application in industry. Firstly, Shi et al.,(2012) state that the WC have been served as necessary cutting tools and wear resistant part due to the their superiority mechanical properties. Secondly, Aravinth et al, (2012) claimed that transition metal carbides have many excellent properties such as good thermal, structural and superior electronic characteristics. Besides that, WC is a valuable tool and dies material caused by high melting point and high hardness, with good thermo-mechanical and thermo-chemical properties (Aravinth et al., 2012).

2.1.1 History of WC

On 1893, WC was study by H. Moissan. Next, fused tungsten metal has been produced through electro thermal way by Moissan on 1897. According to Karlov and Gusev, 2013, the initial tungsten metal was in powder form. The industrial production of hardmetals based on WC is activated 20-25 years after finding WC and goes on at present. The determination of the active fundamental investigations of WC crystal and electronic structure and physicochemical properties of both WC as such as related compounds and system that contain it was the practical interest in WC. Besides that, nanocrystalline WC was the most studied were associated with the synthesis and application since 20 years ago. The studies of WC and WC based hardmetals began at the Institute of Sold State Chemistry of the Ural Division of the RAS (ISSC UrD RAS) after 1992 on the initiative and with the immediate participation of Prof. A. A Rempel(Karlov and Gusev, 2013).

2.1.2 Properties

In general, the properties of WC such as relative density, grain microstructure and mechanical properties (hardness) are highly dependent on many parameter studies. There can be simplified the processing method, grain orientation and densification. The factors are discussed briefly as follows:

2.1.2.1 Mechanical properties

The mechanical properties show the performance of the material. Mechanical properties in term hardness are objective of this research. Hardness is measure of the resistance of a metal to plastic deformation. The hardness of WC samples in different temperature is dissimilar. Shi et al., (2012) come out with table that shows the mechanical properties of WC at different temperature.

Table 2.1: Mechanical properties of WC at different sintering temperature

Sample/ condition	Relative density (%)	Vickers' hardness (HV_{10}) (GPa)	Fracture toughness, K_{1C} (MPa $m^{1/2}$)
WC 1600/1h	90.3	12.8±0.1	4.0±0.3
WC 1700/1h	96.6	20.6±0.5	3.9±0.1

As a conclusion, the optimum sintering temperature, relative density and mechanical properties are interconnection to produce good criteria of WC. However, sintered sample at 1800°C and above prove the abnormal grain growth.

2.1.2.2 Grain microstructure

Grain microstructure is arrangement of small grains of material that can be identified by using microscopy. The expected result in this research is to obtain uniform microstructure and single phase formation of WC. As theoretical, the uniform microstructure affected the material's performance. According to Shi et al., (2012), the high sintering temperatures of WC influenced the grain growth.

Based on the previous study, the grain microstructure was growth in uniform order (0.68±0.27µm) at 1600°C and some grains is unchanged. Nevertheless, a fine microstructure was observed after hot-pressed at 1700°C (2.14±0.57µm). In contrast, the increasing temperature to 1800 and 1900°C give results of abnormal grain microstructure (Shi et al,.2012). Cha and Hong (2003) find out that the abnormal grain microstructure of WC during sintering temperature. As conclusion, the uniform grain microstructure of WC is depended to optimum sintering temperature. The illustration grain microstructures of WC in different temperature are shown in Figure 2.1.

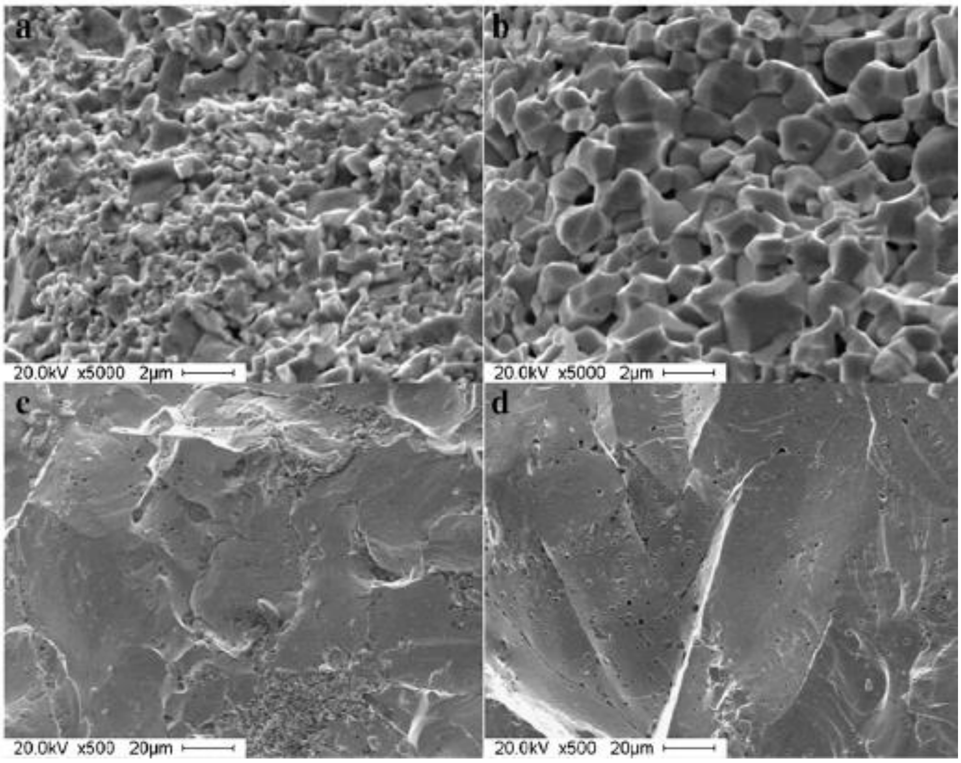

Figure 2.1: SEM microstructure of fracture surfaces of the samples sintered at different temperature: (a) 1600°C, (b) 1700°C, (c) 1800°C, and (d) 1900°C.

2.1.2.3 Relative density

Relative density of WC samples by optimum temperature produce hardness properties of WC. Shi et al., (2012) stated that the punch displacement began slowly at 1200°C. However, the speed up increasing rate of punch displacement was observed at temperature range from 1500°C to 1700°C because of the intensive interparticle sintering. According to Shi et al., (2102), a diffusion mechanism is related in the active densification and pore shrinkage. Based on previous experiment, density of WC at sintering temperature 1600°C possessed a low density of 90.3%, whereas densities of WC sample sintered at 1700 to 1900 °C were much higher and varied in a narrow range of 96.4-96.9% (Shi et al., 2012). Therefore, the high sintering temperature produces dense WC. Nevertheless the high sintering temperature causes abnormal grain microstructure. The optimum sintering temperature is preferred to obtain good performance of WC.

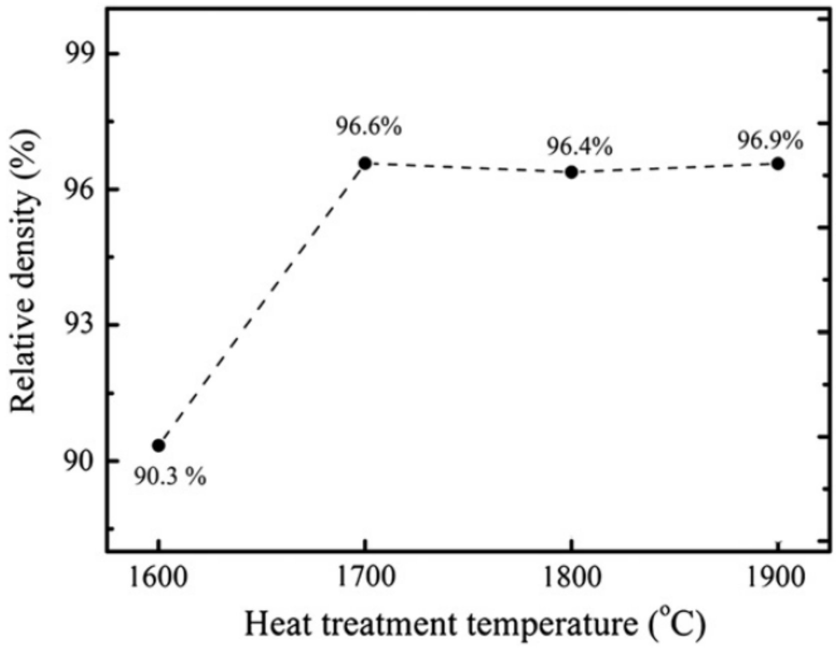

Figure 2.2 : The relationship between sintering temprature with relative density of WC.

2.2 Tungsten based material

This subtopic describes several tungsten based material that familiar in industry application. The listed tungsten based material is compared in term mechanical properties, relative density, phase formation and microstructure. Finally, all of this material is summarized to have characteristic of high strength material and its application.

2.2.1 Pure tungsten

W is a high-temperature structural material. According to Qiang and Pengwan, (2014), W have highest melting point (3410°C) compared to another metal elements. Besides that, W also has high strength at elevated temperature, excellent compatibility with liquid metals, low sputtering yield, good thermal conductivity and low coefficient of thermal expansion (Qiang, 2014). Table 2 shows the relative