

Understanding the Potential of U-FAST Sintering Process – An Overview

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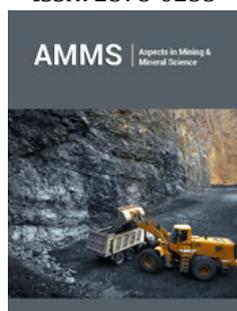
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Abstract

As Tungsten carbide has a number of desired mechanical properties, including temperature resistance, high strength and toughness, which are suitable for industrial cutting tools therefore it is receiving great interest in cutting industry. One of the effective methods for the synthesis of cemented carbide is powder metallurgy. Sintering, a critical part of P/M is occurred by pressing the powder into die and then heat is provided for a period of time until the sintering temperature reaches. Numerous conventional sintering methods were used to prepare tungsten carbide such as vacuum sintering and microwave sintering. The conventional methods are time consuming and require high temperature and high pressure. Some of these issues are resolved by current sintering methods such as spark plasma sintering by reducing the overall time of process and use of commercial grain growth inhibitors. Still, there are challenges that need more research in terms of sintering method that can produce binderless WC. In this study, along with the overview of several conventional and modern sintering processes, upgraded field assisted sintering technology (U-FAST) has been discussed from the perspective of manufacturing carbides. Technical details and microstructural features of U-FAST sintered WC are discussed.

Keywords: Sintering; U-FAST; Tungsten carbide; Microstructure; Conventional sintering

Abbreviations: U-FAST: Upgraded Field Assisted Sintering Technology; SEM: Scanning Electron Microscope; SPS: Spark Plasma Sintering; PPS: Pulse Plasma Sintering; ANFF: Australian National Fabrication Facility

Introduction

Powder metallurgy has been around for more than a century and has been expanding mainly due to its economical processing, unique properties and captive processes [1]. Powder metallurgy has been proven to be an effective method for producing hard materials, including carbides [2]. Metal alloys with any composition can be produced by using powders with the desired purity and fineness [3]. Due to the modern alloy constitution relying mainly on X-ray investigation, the alloys being in powder form is beneficial. Powder metallurgy allows the alloys to be examined, determining the exact phases present, before they undergo sintering [3]. In conventional sintering methods, the heat is transferred to the material using conduction, radiation and convection [4]. The material is heated from the outside causing the surface and the cross section to be heated at different rates, which creates a non-uniform heating [4]. The time to heat a material up to the desired temperature using the conventional sintering methods is long, into the hundreds of minutes [4]. Although the conventional methods have been used for over a century, other methods are needed to increase the efficiency and properties of the sintered material.

Modern sintering methods such as vacuum, microwave and plasma sintering are more efficient than the conventional methods due to their increased and more uniform heating rates [5]. Although these methods are better than conventional sintering they still yield undesirable grain growth. In carbides, if the grain growth can be controlled during heating and cooling cycle of sintering, mechanical properties can be enhanced [6]. WC has much

greater qualities when it is sintered using nanoscale powder. WC powder can be reduced from micron/submicron size to nanosized by mechanical ball milling. This can achieve up to 10nm sized powders over a large period of time [6,7]. Nanocrystalline WC-Co powders lose their nanoscale characteristics upon sintering due to extremely rapid grain growth during sintering; therefore, grain growth inhibitors are used [5]

Upgraded Field Assisted Sintering Technology (U-FAST) is a new sintering technique developed in Poland from a company known as GeniCore. The process is a step up to the SPS technology dedicated to powder materials' sintering. This technology uses an impulse power supply similar to that of SPS, however, it allows for a much higher voltage to be reached [8]. The U-FAST technology allows for full densification in a much shorter period of time and lower temperature when compared to the other sintering methods. In the fabrication of pure WC samples, no grain growth inhibitors or binding materials were present when sintering using U-FAST [8]. This technology is able to benefit from short current impulses

and eliminates the capacitor battery circuit and spark gap of low stability in the energy supply circuit [8]. The materials produced using the U-FAST technology have very minimal grain growth even when using nanocrystalline and submicron powders [8]. This paper presents an overview of conventional and modern sintering processes along with their advances in P/M and adds some excellent achievements of the U-FAST process in manufacturing WC samples without binders or any commercial grain growth inhibitors.

Experimental Procedure

The raw materials used in this study were of three different sizes of pure WC particles. The particles were commercially purchased from Taegu Tec in South Korea and may have been milled as part of production. Details of the initial particles are given in Table 1. Sintered samples were fabricated using the initial particles without any commercial grain growth inhibitors. No binders were used as part of the composition. The three different sizes of WC powders were then sintered using U-FAST.

Table 1: Details of initial WC particles used in U-FAST process.

Sample	Particle size	Composition	Particle Shape	Milling Condition
1	100nm	WC	Spherical	Unmilled after purchase
2	500nm	WC	Spherical	Unmilled after purchase
3	1-3 μ m	WC	Spherical	Unmilled after purchase

Table 2: The list of parameters used in U-FAST process.

Internal dia. Of die	19.5mm
External dia. Of die	40mm
Temperature	2000 °C
Heating Rate	100 °C
Cooling Rate	100 °C
Holding Time	5 min
Load	15KN
Vacuum Pressure	5 x 10 ^{e-5} mbar
Pre-sintering Pressure	1 Tonn

Before sintering, the WC powders were manually pressed with 1 Tonn to generate the green samples. The process started by pouring powder into a graphite mould. Once the green samples were obtained. The die along with the green samples was placed in the U-FAST chamber. Then chamber was closed and heat source - a pulsing electric current was turned on. Once enough energy and heat was applied, the powder was compressed with 15 KN load. The temperature of 2000 °C was used and the processing pressure in vacuum chamber was 5x10^{e-5}mbar. The parameters used for the process are mentioned in the (Table 2) below: Three discs were prepared with 19.5mm diameter and 2mm thickness. All the fabricated discs were manually cut into quarters by using diamond blade saw to mount cross-section surface along with the top surface. Then polishing was done on the mounted sample using a Tegramin 25 polishing machine (Struers). The samples were then etched to remove the deformed layer deposited during

grinding and polishing also to reveal the microstructural detail of the polished mount or the crystalline structure of the sample. The samples were etched using Murakami reagent (10gm Potassium ferricyanide, 10gm NaOH and 100ml of water) for 80-120 seconds. The observation of microstructure of the samples was done with the help of an FEI F50 Scanning Electron Microscope (SEM) having an EDS analyser.

Advances in Sintering Techniques in Manufacturing Materials

Microwave sintering versus conventional sintering

The work by Breval et al. [9] compares the microwave and conventional sintering for WC-Co samples. In the conventional sintering the heating starts from surface, which thermally conducts from heating element to holding vessel and from outside to inside of the sample, thus it takes a considerable amount of time. However it was found in the study that in microwave sintering the heating is caused by oscillations of free electrons in microwave oven at high frequency microwave (~2.45GHz) in cobalt, free carbon and of ions in WC [10,11]. Microwave sintering needs only minutes to complete the sintering because the thermal equilibrium reaches fast, while conventional sintering takes several hours due to thermal gradient from the surface to the centre of the sample. Hence the grain growth is less in microwave compared to conventional sintering.

The same initial powder was sintered in both the conventional furnace and in the microwave furnace at 2.45GHz. WC-6 wt% Co was used as raw material. Conventional sintering was performed at 1450 °C for approximately 1h with up- and down-ramps in the

order of 6-20h and microwave sintering was performed at 1300-1450 °C for approx. 10min with up- and down-ramps in the order of 60-90min. The micrographs (Figure 1) had shown that the grain size is smaller in microwave-sintered samples as compared to conventional sintered. It was also pointed out that by using microwave sintering fine grain growth can be achieved without

using any growth inhibitors that usually degrade the mechanical properties of material. Also, the microwave-sintered samples achieved 1-5GPa better hardness than conventional method. The advantage of microwave sintering over conventional is its better grain growth even in shorter time and lower temperature.

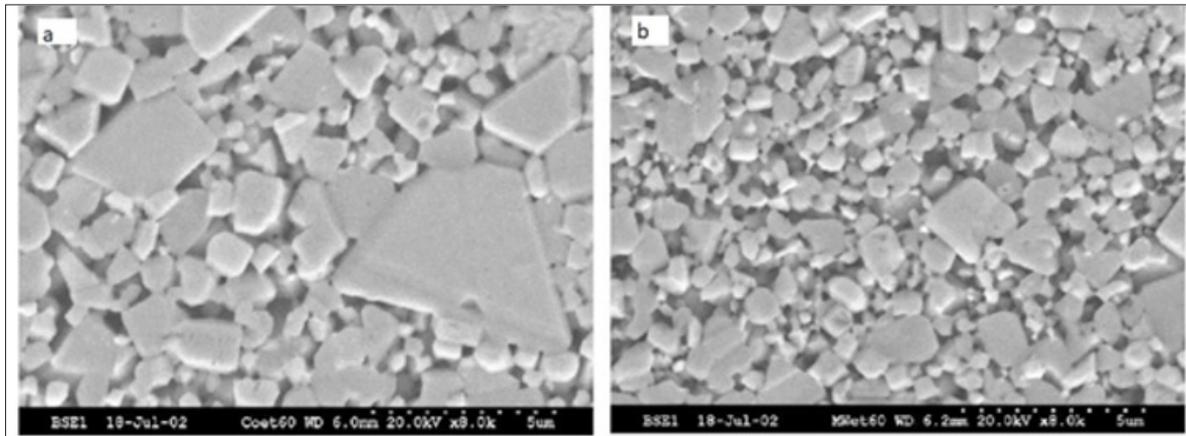


Figure 1: SEM images of WC/Co: a) conventionally sintered and b) microwave sintered [9].

Vacuum sintering

Liang et al. [12] synthesized WC-Co cemented carbide by vacuum sintering. The composition included WC (99.5wt. %), Co (99.9wt. %), VC (99wt. %) and Cr_3C_2 (99wt. %). The disk shaped green sample was prepared with 100 MPa of pressure by pressing the dried powder. Also, the required amount of 5 wt. % solution of PVA was added as a binder. Then sintering was done at 1280 °C~1400 °C in vacuum sintering furnace with pressure applied under 1 Pa. XRD analysis has shown that only WC and Co_3W_3C (γ -

phase) were present with a relative density of over 95%. (Figure 2) shows the microstructure of the WC-Co cemented carbide with different wt. % of Co. The microstructure was dense and well distributed including some bulky grains. It was found that in this method the hardness was increased with increasing temperature because the relative density increases with temperature. However, the grain size also increased with the highly increased temperature. The issues associated with this method are that it is a slow process and comes with the additional step of preparing green sample before sintering.

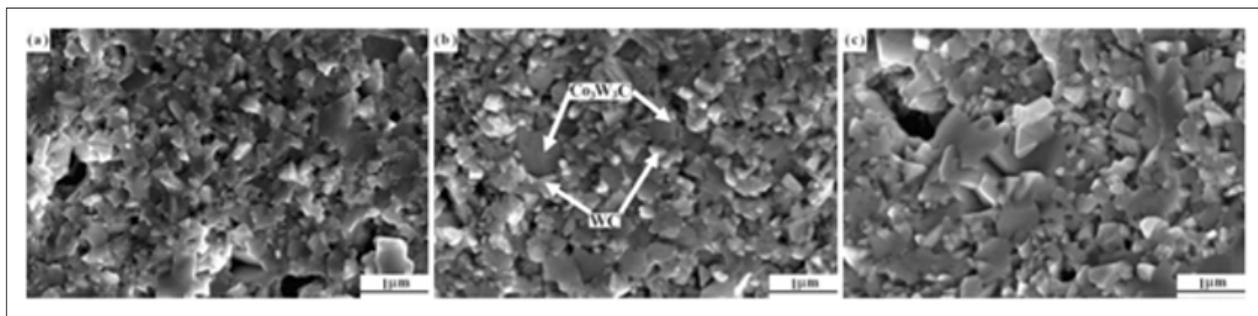


Figure 2: SEM images of fractured surface of (a) WC-6Co, (b) WC-8Co, (c) WC-10Co cemented carbide prepared at 1400°C in 1hr [12].

Spark plasma sintering

In the Spark Plasma Sintering (SPS) method the sintering is performed at lower temperatures and lasts for shorter periods than in conventional methods. The powder is heated through a pulse current, which causes spark discharges to be ignited in the pores and these discharges remove the absorbed gases and oxides from the surfaces of the particle [5]. The sintering process occurs under the influence of a current and pressure simultaneously [13]. (Figure 3) shows the relative density with respect to the crystallite size and pressure when using the SPS method to produce zirconium

oxide. It is now believed that through faster and better diffusion mechanism, plasma-based sintering is able to reach theoretical density in materials [2].

Pulse plasma sintering

The work by Michalski et al. shows that pulse plasma sintering (PPS) process can be carried out at relatively low temperatures when compared to that of SPS [5]. The spark charges that are present in PPS remove the gaseous content, which enables an easier path for the bonding of the particles or grains [5]. The grain growth

of WC in a WC-10 wt% Co alloy obtained by sintering nanosized (5-20nm) WC-Co powder drastically increased; first observed at 200nm after 30 seconds of sintering at 1400 °C, and finally 2m after an additional 30 seconds of sintering [6]. The source of energy for the Pulse Plasma Sintering (PPS) is a 200F capacitor battery; this heats up the powder that is held in a graphite die through periodically repeated current pulses that last several hundreds of microseconds. Due to the electrical pulses having a short duration (500µs) compared to the interval between the pulses (0.5s–5s) the average temperature of the powder being sintered is lower than the temperature reached during exposure to the current pulse [5]. The PPS process is much more intensive due to the high energy that is delivered in a pulse which is greatly larger than in the other

methods' [5]. The sintering process promotes growth of tungsten carbide grain size through the presence of the liquid Co phase, this is undesirable as the mechanical properties deteriorate as the grain size increases [5]. The grain growth comes from the high-rate diffusion of WC through the liquid Co phase. Smaller grain size in WC is highly desirable as it causes an increased hardness and improved mechanical properties, whereas an increase in Co binding phase equates to an increased fracture toughness but decreased hardness [5]. The fracture toughness and hardness of the WC-12 wt.% Co carbides sintered using the pulse plasma method are shown in Figure 4. Both the fracture toughness and the hardness are observed to increase up to 1100 °C and then decrease when the temperature is increased to 1200 °C.

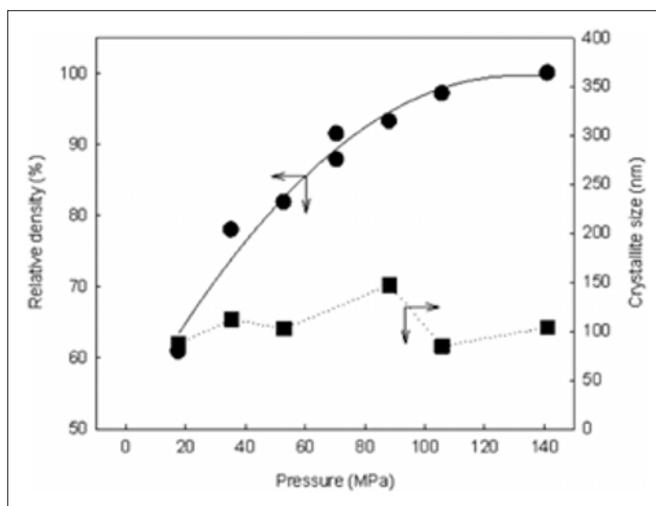


Figure 3: Effect of applied pressure on density and grain size of cubic Zirconia. Constant temperature of 1200°C, hold time 5min, heating rate 200°C/min [13].

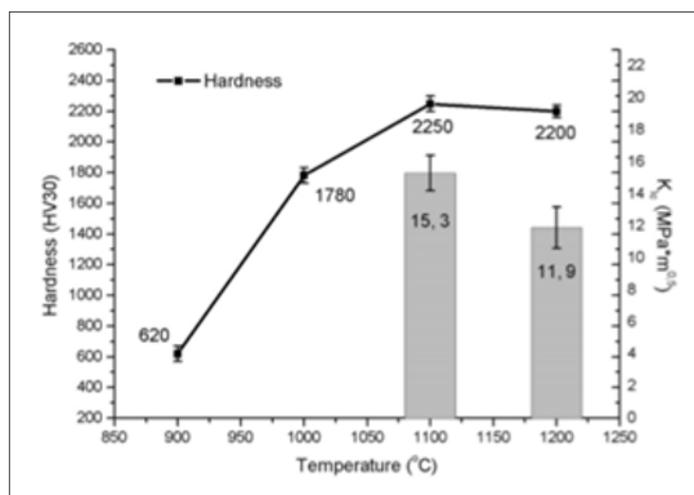


Figure 4: Variation of the hardness and fracture toughness with the sintering temperature [5].

Upgraded field assisted sintering technology (U-FAST)

U-FAST is a low voltage technology, which is the updated version of modern methods such as pulse plasma compaction. Heating up of powders in U-FAST is realized by very narrow electrical pulses. Very short current pulses below of 1 millisecond are used to heat the material, which allows producing a material with good

properties such as pure WC. The U-FAST technology allows for full densification (Figure 5) in a much shorter period of time and lower temperature when compared to the other sintering methods. The shorter time and less energy consumption make this method ideal to fabricate nanocrystalline materials and submicron without unfavorable grain growth during the sintering process. The current

impulse is shorter and the interval between impulses is shorter than that of the SPS methods, this is what facilitates the faster densification and diffusion in U-FAST. The materials produced by

U-FAST are Al_2O_3 at 1100 °C, dense Al_2O_3 - ZrO_2 ceramics at 1150 °C, dense W-SiO_2 composites at 1600 °C, amorphous bulk metallic glasses ZrCuAlNb with full density without crystallization at 400 °C.



Figure 5: Image of the U-fast machine.

The advantages of new technology over the others are that its short period of time reduce the unfavourable grain growth during sintering. The sintering is performed at uniaxial pressure. One of the main benefit is its low processing temperature and low pressure without compromising the mechanical properties of the material. It can be observed from the SEM images that WC particles can bind together without any binder Figure 6. Some of the grains are closely

packed which are showing complete diffusion of the interfaces of particles. The grain growth is minimal even when full densification is achieved. The high hardness was achieved in the range of 2400-2500 HV and the fracture toughness was 15-18 $\text{MPa}\sqrt{\text{m}}$. The UFAST technology makes it possible to synthesize pure WC without any binder and growth inhibitor, which can deteriorate the mechanical properties of material.

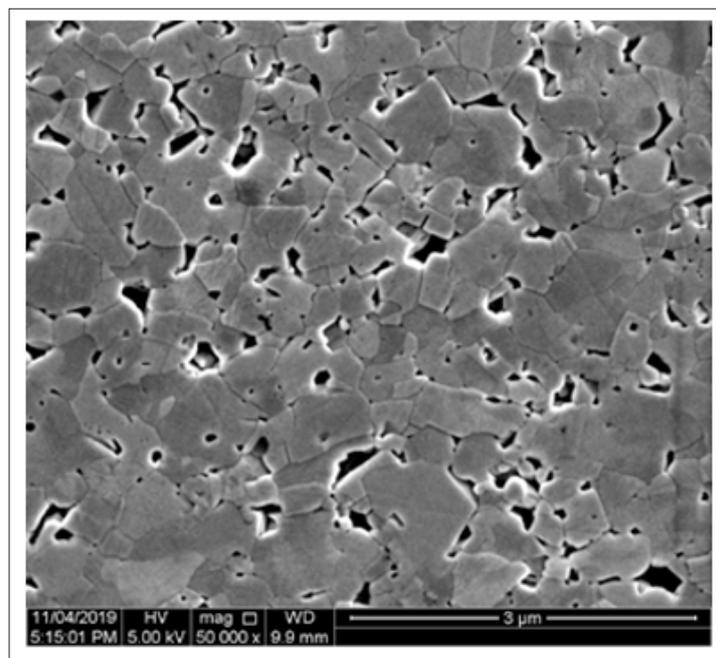


Figure 6: SEM micrograph (using SEs) of pure WC (initial particle size of 100nm) synthesized by U-FAST technology at 2000°C without using any binder and grain growth inhibitor.

Conclusion

Powder metallurgy is an important part of the modern-day sintering techniques as a large variety of materials are produced from powders. The U-FAST method is highly beneficial to sintering technologies as it yields better mechanical properties of cemented carbides with more efficiency. The results achieved show very positive trends with the U-FAST sintered tungsten carbide samples yielding higher hardness values than pulse plasma compacted tungsten carbide. The tungsten carbide particles were seen to be able to bind successfully when there is no binder or grain growth inhibitor when U-FAST was used. If the use of the U-FAST method could be amplified, it would have a very positive impact on the mechanical properties of cemented carbides produced through sintering.

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