Study of TiC Cutting Tool Insert Using Microwave Synthesis

Shahdura Hammad Thauri^a, Tasnim Firdaus Ariff^b

and A. N. Mustafizul Karim^c

International Islamic University Malaysia

Engineering Faculty

Manufacturing and Materials Engineering Department

Jalan Gombak, 53100 Kuala Lumpur, Malaysia

^ashahdura_ht@hotmail.com, ^btasnim@iium.edu.my, ^cmustafizul@iium.edu.my

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Abstract. Microwave processing ceramics is emerging fast as a new field of ceramic processing and material synthesis. The past year has witnessed significant progress in the aspects of commercialization and application of the technology to new areas. Further research states that Titanium Carbide (TiC) is the best cutting tool due to its high melting point and by that reason this project aims to show the difference between conventional sintering, microwave sintering and Hot Isostatic Pressing (HIP). Two different compositions consisting of 97TiC3Ni and 93TiC7Ni were sintered using conventional furnace, microwave furnace and HIP. Density, hardness and microstructure analysis were carried out on these TiC inserts. 97TiC3Ni produced higher density and hardness values compared to 93TiC7Ni for all three different methods. Microwave sintering and HIP. Microwave produced samples with improved density and hardness in a shorter processing time which is 93% faster than conventional sintering and 50% faster than HIP.

1. Introduction

Powder metallurgy (PM) is a process whereby a material powder is compacted as a green body and sintered to a net shape at elevated temperatures. There are challenging demands from the PM industry for new and improved sintering process with finer microstructures and enhanced physical and mechanical properties. This is where the microwave technology is found to be advantageous.

New development and innovative ideas in the area of materials processing have often led to discovery of new materials processing, with interesting and useful properties and new technologies which are faster, better, cheaper and greener. Striking examples of such innovations are recent developments in the area of microwave processing of ceramics [1-3]. Although many potential advantages of utilizing microwaves to process ceramics have long been recognized, it is only now that this field has finally been shown to be take-off stage. Microwave heating was conceived over 50 years ago, its use in ceramic processing is relatively new.

Microwave heating is fundamentally different from the conventional one in which thermal energy is delivered to the surface of the material by radiant and or convection heating that is transferred to the bulk of the material via conduction. In contrast, microwave energy is delivered directly to the material through molecular interaction with the electromagnetic field. Microwave heating is the transfer of electromagnetic energy to thermal energy and is energy conversion rather that heat transfer. Since microwaves can penetrate the material and supply energy, heat can be generated throughout the volume of the material resulting in volumetric heating [4].

The search for new techniques is driven by a need to improve sintering productivity, either by faster sintering or more uniform heat transport. A wide variety of techniques are investigated including: microwave, induction, plasma, electrical discharge and laser heating [5]. Interest and activity in microwave processing has been continuously increasing, and the use of the microwave

technology in industrial application is also growing with new developments in both engineering and design of microwave systems. This project is undertaken with the aim in understanding the effect of microwave synthesis and sintering process on the physical behavior of powder ceramic part with respect to hardness, density and microstructure analysis.

2. Experimental Procedure A. Sample preparation

In this study, Titanium Carbide powder of 99.5% purity with a size of $20\mu m$ was used as the matrix material. Nickel powder of 99.5% purity with a size of $10\mu m$ was used as a binder for the composition of 97TiC3Ni and 93TiC7Ni.

Titanium carbide powder and Nickel were weighed and mix in a ball milling machine (Fritsch) for 10 hours with a speed of 150 rpm. Samples were hydraulically pressed (4350.L Carver Inc.) at 103.42MPa and then cold isostatically pressed (AIP-CP360) at 103.42 MPa into cylinder pallets with a diameter of 10.5 mm and thickness of 4 mm. The sample for each composition (97TiC7Ni and 93TiC7Ni) was sintered using three different methods; conventional sintering, microwave sintering and Hot Isotatic Pressing (HIP).

Two samples (97TiC3Ni and 93TiC7Ni) were sintered using conventional furnace (Nabertherm GmbH) with a temperature of 1250°C for 30 minutes. Another two samples were sintered using microwave hybrid sintering technique by employing SiC (100g) (30μ m) as susceptor in a crucible setup to separate between the specimen and SiC powder. The sample was heated using a modified domestic microwave oven (Panasonic, model ST 557M) with an output power of 1100 kW and magnetron operating frequency of 2.45 GHz.

After sintering for 10 minutes, the specimen was taken out from the microwave furnace and the K type thermocouple was used to measure the temperature at the specimen's surface. There are some limitations with this temperature measurement method in which it was unable to display the temperature profile change along the heating process and invariably there will be some heat loss during temperature measurement. However previous research by Gupta and Wong has proven the reliability of temperature measuring method [6]. In another set of experiment, two samples were also sintered using HIP (AIP- HP630) at 1400°C for 10 minutes. Sintering time and compaction load for each composition is summarized in Table 1.

Composition	Compaction Method	Compaction Load	Sintering Equipment	Sintering Temperature	Total Sintering Time
97TiC3Ni	Hydraulic Press	103.42 Mpa	Conventional Furnace	1250°C	150m inutes
93TiC7Ni		105.42 Мра			
97TiC3Ni	Hydaulic Press and CIP		Microwave Furnace	273°C	10 minutes
93TiC7Ni		Mpa			
97TiC3Ni	Hydaulic Press	102 (22)	Hot Isotatic	1400 °C with	10
93TiC7Ni		103.42 Mpa	Pressing	339.75 Mpa	10 minutes

Table 1: Summary of sintering time and compaction load for each experimental condition

B. Characterization

The sintered samples were tested for their density and hardness values and followed by microstructural analysis. The density of each specimen was measured before and after sintering using Archimedes' principle. A Precisca XT220A electronic balance with accuracy 0.0001g was used for recording the weights and densitometer. Specimens were weighed in the air and immersed in distilled water. The green density was calculated on the basis of the mass and volume of the samples.



Mechanical behavior of the specimens was assessed in terms of microhardness. Microhardness of the specimen was determined using Shimadzu microhardness tester. Specimen microhardness measurement was carried out using Vickers hardness with a pyramidal diamond indenter (face angle of 136°, 1.961N indenting load) for a load dwell time of 20 seconds. The Vickers hardness (HV) is determined from the formula shown in Eq.1:

$$HV = \frac{1.854F}{D^2}.$$

F= applied load (kgf) D= diagonal of the impression made by the indenter (m)

Samples for microstructural analysis using Scanning Electron Microscopy (SEM) (JEOL-JSM 5600) were polished (Metapol-2 polisher) with alumina solution till most of the surface scratches were removed.

3. Result and Analysis

A. Physical Appearance

After compaction the green compact appeared black in colour. After sintering, both conventional and microwave sintered specimen appeared in black and grey colour while for HIP it is silvery lustrous grey colour as shown in Figure 1. Both compositions (97TiC3Ni and 93TiC7Ni) have the same physical appearance after the sintering. During microwave hybrid sintering, it was observed that the sample had the same shape as after the compaction while for conventional sintering it was observed that sample swelled for about 0.01mm from the surface after the sintering.

This is due to the heating mechanism and temperature of the furnace compared to microwave. In conventional heating, heat was transferred from heating element to sample surface, and then conducted to the interior, therefore all reactant powders experienced the same temperature before ignition took place. In microwave heating, heat is generated within the sample itself by interaction of microwave with material thus it will have a volumetric heating.

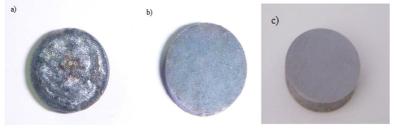


Figure 1: Appearance of the specimens after sintering, (a) Conventional sintering (b) Microwave sintering, (c) Hot isotatic pressing

B. Density

Table 2 shows the density of the samples increased after the HIP, microwave and conventional sintering. Figure 2 proves that microwave sintering produced a sample with highest density increase followed by HIP and conventional sintering.

Conventional Sintering (CS), 30 minutes

Composition	Temperature	Green Density g/cm3	Density After g/cm3
97TiC3Ni	1250°C	2.601	5.212
93TiC7Ni	1250°C	2.601	5.201

Microwave Sintering (MS), 10 minutes

Composition	Temperature °C	Græn Density g/cm3	Density g/cm3
97TiC3Ni	271	2.601	5.767
93TiC7Ni	273	2.601	5.692

Hot Isotatic Pressing	(HIP), 10 minutes

Composition	Temperature °C	Græn Density g/cm3	Density g/cm3
97TiC3Ni	1400	2.601	5.635
93TiC7Ni	1400	2.601	5.573

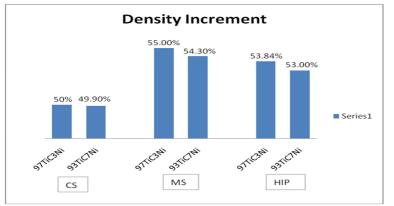


Figure 2: Density increase after the sintering

B. Hardness

Microwave hybrid sintered specimen is generally harder than HIP and conventional sintered sample. Figure 3 shows that sintering using microwave energy results in highest hardness, (557.6 and 418HV) compared to HIP and conventional sintering. Similarly, 97TiC3Ni gives the highest hardness compared to 93TiC7Ni for each of sintering method. Although the temperature for conventional sintering and hot isotatic pressing were high compared to microwave sintering, the samples for microwave sintering produced harder specimens compared to all three processing methods.

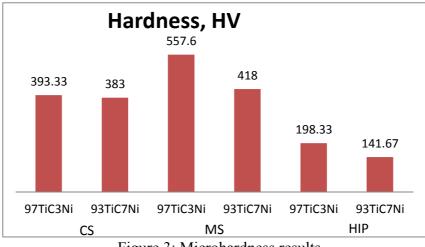


Figure 3: Microhardness results



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C. SEM

Figure 4 shows the green microstructure of both compositions. The grain size of the sintered samples have increased for all of the sintering methods since existence of grain growth can be seen. However, the grain growth for microwave sintered samples are relatively low due to rapid heating. Figures 5, 6 and 7 show the differences in the microstructure from the samples which were sintered using conventional, microwave and HIP respectively. The heating of microwaves is very rapid as the material is heated by energy conversion rather than by energy transfer which occurs in conventional techniques [7].

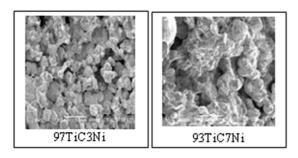


Figure 4: Green Microstructures

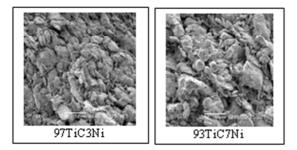


Figure 6: Conventionally Sintered Samples

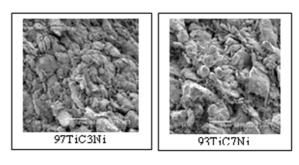


Figure 5: Microwave Sintered Samples

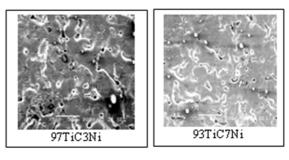


Figure 7: Hot Isostatically Pressed Samples

4. Conclusion

Based on the experimental results, the proposed microwave hybrid sintering experimental set up can be successfully utilized to sinter TiC with Nickel as the binder. Microwave sintered sample has the highest increment in density (55% and 53.4%) for 97TiC3Ni and 93TiC7Ni respectively compared to all the three methods. Microwave sintered samples give the highest value of hardness (557.6 and 418 HV) for 97TiC3Ni and 93TiC7Ni respectively compared to conventional sintering (393 ad 383 HV) and hot isotatic pressing (198.33 and 141.67 HV).

Microwave sintering offers reduction heating time (93% and 50%) compared to conventional sintering and HIP respectively due to the uniform heating at a rapid rate. Due to the rapid heating, microwave sintered sample resulted in a finer microstructure, thus enhancing the mechanical properties. It has been demonstrated that a finer grained structure TiC and Ni has been observed in microwave sintered samples. Small, rounded and uniformly distributed pores can be seen in microwave sintered samples compared to large, angular and nonuniform pores observed in conventionally sintered and hot isotatically pressed samples. This is due to the hybrid heating using microwave and susceptor which heating is from inside to the outside and susceptor provides radiant heat from outside to inside.



2121

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