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Enhancing Mechanical And Structural Properties Of TiC Inserts Using Microwave Energy

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Abstract:

Two different compositions of Titanium Carbide (TiC) inserts consisting of 97%TiC3%Ni and 93%TiC7%Ni were synthesized using conventional sintering (CS), microwave sintering (MS) and Hot Isostatic Pressing (HIP). The influence of sintering time and sintering temperature on the mechanical and structural properties of this TiC inserts were examined. The compaction load of 103 MPa was used to produce the green samples. Different time-temperature combinations were used for each type of heat treatment. 97TiC3Ni composition produced inserts with higher density, hardness and strength compared to the 93TiC7Ni composition for all three different sintering methods. Meanwhile, hybrid microwave sintering produced TiC inserts with the highest density, hardness and strength compared to conventional sintering and HIP. Improved mechanical and structural properties were observed in samples sintered using microwave energy for just 10 minutes compared to 10 minutes using HIP and 30 minutes of conventional sintering.

1. Introduction

Synthesis of various materials ranging from metal alloys, ceramics and composites has proven to be successful using microwave energy [1-7]. Microwave sintering is a method of heating that involves energy conversion which is different from the conventional sintering that concerns energy transfer. In microwave sintering, the heat is generated internally within the material instead of originating from external sources. In the process of microwave heating, the materials absorb microwave energy themselves and then transform it into heat within the sample volume [8]. The energy is directly transferred to the material through the interaction of electromagnetic waves with atoms and molecules leading to heating [9]. Microwave sintering has the potential for rapid processing with refinements in microstructure and enhanced properties. Hybrid microwave sintering is much more uniform and effective compared to conventional sintering. This results in a reduction of processing time and energy consumption.

The microwave processing of ceramics can be used as an alternative method to conventional sintering of ceramics because of the potential advantages that exist, such as rapid heating, more uniform microstructures, penetrating radiation, and higher densities [10-12]. Several studies have been directed towards a comparison of the dielectric behaviour of microwave and conventionally sintered ceramics [13-14]. All of these studies showed that the microstructure and superconducting properties of microwave sintered material is close to those observed for conventionally sintered ceramic material. Initially, successes in microwave heating and sintering were confined to mainly oxide and some non-oxide ceramics. Today, this has been extended to cemented carbides for cutting and drilling applications with improved performance, and has been successfully commercialized [7].

Titanium carbide (TiC) has been widely used in the fields of wear resistance tools and aerospace materials. Titanium carbide based composites with nickel alloys and iron alloys are currently used in high performance applications where wear and corrosion are the main sources of material failure. Due to its high melting point, the production of bulk materials containing the TiC component has been made possible by TiC powder consolidation [15].

This research aims to compare the effects of hybrid microwave sintering, conventional sintering and HIP on the physical behavior of TiC powder compacts with respect to strength, hardness, density and microstructure. In this project two different powder compositions; 97TiC3Ni and 93TiC7Ni have been used to compare the mechanical and structural properties of the TiC inserts. These powders have been compacted using cold pressing and then sintered using HIP (1400ºC, 10 minutes), conventional furnace (1245ºC, 30 minutes) and microwave furnace (273ºC, 10 minutes).

2. Experimental Method

2.1 Sample preparation

Titanium Carbide powder of 99.5% purity with a size of 20µm was used as the matrix material, while Nickel (Ni) powder of 99.5% purity with a size of 10µm was used as the binder for both compositions (97TiC3Ni and 93TiC7Ni).

Titanium carbide powder and Nickel were weighed and mixed in a ball milling machine (Fritsch) for 10 hours with a speed of 150 rpm. Spherical steel balls with 10 mm diameter and 1.47 g/ball were used. A total of 34 balls were used for the milling each time. Samples were hydraulically pressed (4350.L Carver Inc.) at 103 MPa and then cold isostatically pressed (AIP-CP360) at 103 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) (30µm) 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 [4]. 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 [16].

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Table 1: Experimental conditions [16]

Composition	Compaction Method	Compaction Load	Sintering Equipment	Sintering Temperature	Total Sintering Time
97TiC3Ni	Hydraulic Press	103.42 Mpa	Conventional Furnace	1250ºC	30 minutes
93TiC7Ni					
97TiC3Ni	Hydaulic Press and CIP	103.42 Mpa and 339.75 Mpa	Microwave Furnace	273ºC	10 minutes
93TiC7Ni					
97TiC3Ni	Hydaulic Press	103.42 Mpa	Hot Isotatic Pressing	1400 ºC at 339.75 Mpa	10 minutes
93TiC7Ni					

2.2 Mechanical Testing

The sintered samples were tested for their density, hardness, strength 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.

Microhardness of the specimen was determined using Shimadzu microhardness tester. Microhardness measurement was carried out using Vickers hardness with a pyramidal diamond indenter (face angle of 136º, 1.961N indenting load) for a load dwell time of 20 seconds.

Strength for the circular inserts was measured using the compression tester (INSTRON 4485). The solid cylindrical insert (disk) was compressed between two flat dies. Tensile stresses are developed perpendicular to the vertical centerline along the disk during compression. Fracture begins and the disk splits into half vertically. The tensile strength of the material from the compression test can be calculated from Equation 1.

$$\text{Tensile strength, } \sigma = \frac{2P}{\pi d t} \qquad (1)$$

where P is the load at fracture, d is the diameter of the disk and t is the thickness.

2.3 Microstructural Analysis

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. Samples were etched using 20% Hydrogen Peroxide and 80% distilled water.

3. Results and Discussion

3.1 Physical Appearance

Green compacts appeared black in colour. Both conventionally and microwave sintered specimens appeared in black and grey colour while for the HIP samples, it turned out to be silvery lustrous grey colour as shown in Figure 1 [16]. This physical appearance was observed for both compositions (97TiC3Ni and 93TiC7Ni). For the microwave sintered samples, there was no swelling or shrinkage noticed. Its shape and size remained the same as after the compaction.

Meanwhile, the conventionally sintered samples swelled to about 0.01 mm from the surface after sintering. This is due to the heating mechanism and temperature of the furnace compared to microwave. In conventional heating, heat was transferred from the heating element to the sample's surface, and then conducted to the interior. Hence, all reactant powders experienced the same temperature before ignition took place. In hybrid microwave sintering, heat is generated within the sample itself by interaction of microwaves with the molecules of the material. Thus, leading it to a volumetric heating process.

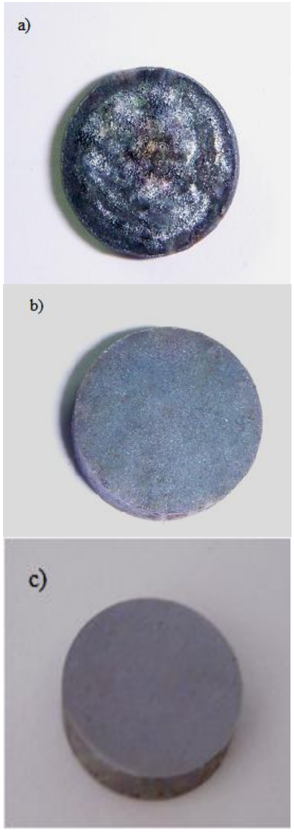

Figure 1: Appearance of the specimens after sintering, (a) CS (b) MS (c) HIP [16]

3.2 Density

Table 2 shows the density of the samples increased after the HIP, microwave and conventional sintering. Microwave sintering produced samples with highest density increment followed by HIP and conventional sintering as shown in Figure 2.

Table 2: Summary of density results [16]

Sintering methods	Composition (%wt)	Time (min)	Temperature (°C)	Green Density (g/cm^3)	Sintered Density (g/cm^3)
CS	97TiC3Ni	30	1250	2.601	5.212
CS	93TiC7Ni	30	1250	2.601	5.201
MS	97TiC3Ni	10	271	5.274	5.767
MS	93TiC7Ni	10	240	5.399	5.692
HIP	97TiC3Ni	10	1400	5.483	5.635
HIP	93TiC7Ni	10	1400	5.483	5.573

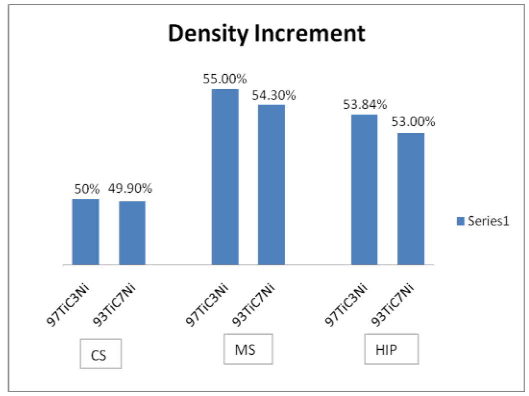

Figure 2: Density increment after the sintering process [16]

3.3 Hardness

Hybrid microwave sintered samples are generally harder than HIP and conventionally sintered samples. 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.

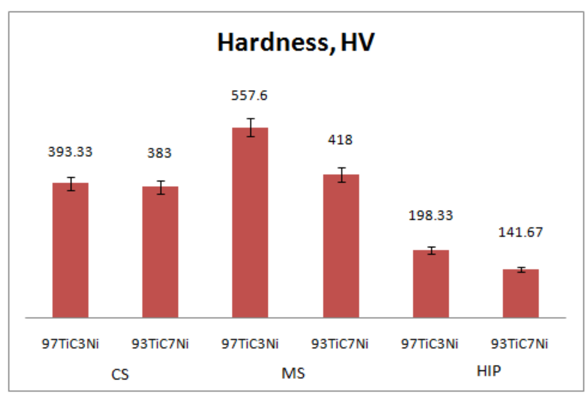

Figure 3: Microhardness results [16]

3.4 Strength

Hybrid microwave sintering has shown to produce higher strength in TiC inserts compared with the conventionally sintered ones. Higher strength is observed in the 97TiC3Ni composition for the conventional and microwave sintering methods. Hybrid microwave sintering have produced samples with higher tensile strengths; 23.8% and 23.5% higher than conventional sintering for 93TiC7Ni and 97TiC3Ni respectively. As for the HIP samples, both compositions have shown not to fracture during the compression test. This shows that the HIP samples were actually not brittle at all. Both HIP compositions have resulted in increased toughness rather than being brittle. Microwave sintering is capable of producing improved mechanical properties compared with the conventionally sintered samples at a lower temperature. Table 3 summarizes the results obtained for the tensile strengths using compression test for all three types of sintering methods.

Table 3: Summary of tensile strength

Sintering Method	Composition	Strength (Mpa)
CS	93TiC7Ni	47.88
CS	97TiC3Ni	51.97
MW	93TiC7Ni	62.99
MW	97TiC3Ni	68.50
HIP	93TiC7Ni	Did not break
HIP	97TiC3Ni	Did not break

3.5 Microstructural Analysis

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 [16]. 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 [4].

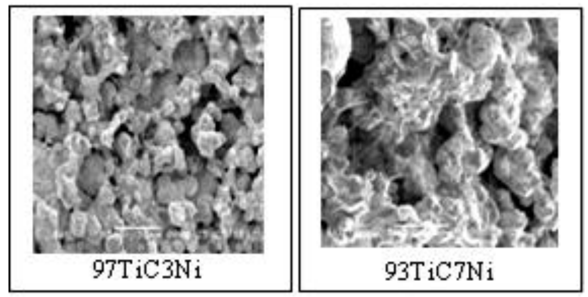

Figure 4: Green Microstructures [16]

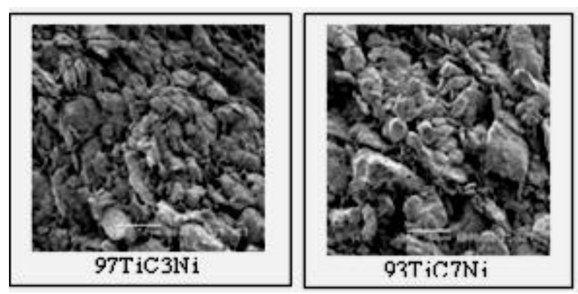

Figure 5: Microwave Sintered Samples [16]

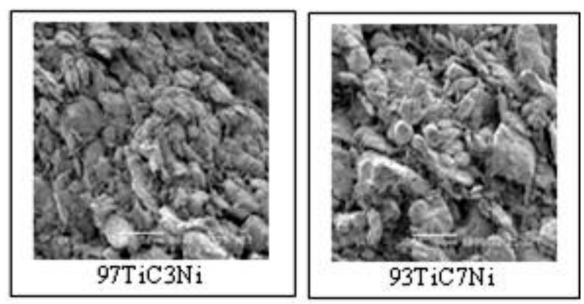

Figure 6: Conventionally Sintered Samples [16]

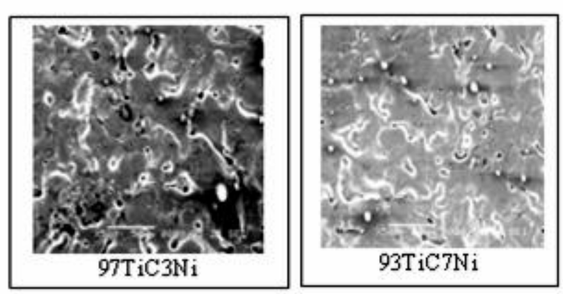

Figure 7: Hot Isostatically Pressed Samples [16]

4. Summary

TiC inserts can be successfully sintered using microwave energy. 97TiC3Ni composition apparently has shown to have better mechanical and structural properties compared to 93TiC7Ni composition. Microwave sintered samples have the highest increment in density; 55% and 53.4% for 97TiC3Ni and 93TiC7Ni respectively followed by HIP and conventionally sintered samples. Microwave sintered samples have higher tensile strengths; 23.8% and 23.5% higher than conventional sintering for 93TiC7Ni and 97TiC3Ni respectively. Microwave sintering produced samples with enhanced mechanical properties in terms of density, hardness and strength. This is a result of the less grain growth found during microwave sintering. Furthermore, microwave sintering offers reduction in total processing time (93% and 50%) compared to conventional sintering and HIP respectively due to the uniform heating at a rapid rate. Microwave sintered samples resulted in a more uniform and finer microstructure due to the rapid heating effect which does not allow sufficient time for grain growth. Small, rounded and uniformly distributed pores can be seen in microwave sintered samples compared to large, angular and non-uniform pores observed in conventionally sintered and HIP samples.

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