Improvements in the Development of Silicon Nitride Inserts using Hybrid Microwave Energy for Machining Inconel 718

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Abstract—Hybrid Microwave (HMW) sintering is becoming popular for heating ceramics, composites, metals and alloys. It is becoming a trendy research due to its capabilities in saving time, energy and money, increasing efficiency and productivity, improving mechanical and structural properties. Silicon Nitride (Si3N4) is widely used as a cutting insert for machining cast iron, hard steel and nickel based alloys. It is mass produced by traditional powder technology. This research aims to increase the tool life of the Silicon Nitride inserts by enhancing the mechanical and structural properties by using Hot Isostatic Pressing (HIP) at 1800°C and followed by hybrid microwave (HMW) post-sintering at 200°C for 10 minutes with the aid of Silicon Carbide powder as susceptor. Three different compositions (88%, 90% and 92% Si3N4) were synthesized in order to find the optimum composition using these techniques. Mechanical and structural properties were analyzed. The combination of HIP+HMW post sintering for just 10 minutes significantly enhanced the density (93-97%TD), hardness (18-22%), compressive strength (2-4%) and produced finer uniform grains particularly for 88% and 90% Si3N4 when compared with HIP samples without any post sintering effect. Hence, the 88% and 90% Si3N4 compositions produced by the combination of HIP+HMW post sintering are potentially suitable candidates for machining hard materials such as Inconel 718.

Keywords—Silicon carbide; hybrid microwave post sintering; hot isostatic pressing; enhanced densification; improved mechanical properties; Inconel 718

I. INTRODUCTION

Ceramic inserts are widely used in the field of high speed machining of hard materials. Silicon nitride (Si3N4) is an example of an insert that has a unique set of outstanding properties such as high hot hardness, high wear resistance, high fracture toughness, improved oxidation and chemical resistance [1]. Ceramic inserts are generally produced by traditional particulate processing techniques.

Hybrid microwave energy has gained popularity in the world of particulate processing of ceramic materials. Charmond et al. investigated on the densification and microstructure changes of 2 mol% yttria-stabilized zirconia nano powder and concluded that hybrid heating produced homogeneous microstructures whereas direct microwave heating led to rather heterogeneous microstructures due to thermal gradients [2]. Thauri et al. analyzed on the mechanical properties of titanium carbide (TiC) in hybrid microwave sintering; and found out that samples with improved density and hardness were produced by hybrid microwave sintering in a shorter processing time which is 93% faster than conventional sintering and 50% faster than Hot Isostatic Pressing [3]. ITO ceramics generally are difficult to achieve full densities using conventional heating because of the volatilization property of both indium oxide (In2O3) and tin oxide (SnO2) at high temperatures. However, Chen et al. managed to achieve 99% of theoretical density by doping it with Zinc Oxide and sintering it using hybrid microwave energy in just 25 minutes [4].

Hybrid microwave heating is a function of the material providing rapid heating to the sample [6](Oghbaei and Mirzae, 2010). The use of susceptor with microwave energy results in uniform volumetric heating; from inside to outside as well as from outside to inside. Furthermore, it also contributes to the
rapid heating process. In addition, the rapid initial heating via susceptors becomes the key factor to execute the energy efficient microwave processing for the poorly microwave absorbing materials [7].

Microwave processing for sintering or post-sintering ceramics has advantages such as increased heating rates, uniform heating, reduced cost and improved mechanical and structural properties compared to conventional methods [8-11] (Huang et al., 2009; Ariff et al., 2014; Oakley, 2014; Xu, 2017). Most of the researches pertaining to microwave sintering of Si₃N₄ in general focus on the comparison of microwave with conventional heating in terms of its mechanical and structural properties. The actual performance is rarely seen in action.

Machining extremely hard materials such as tungsten and nickel alloys contribute to a higher wear rate and shortened tool life which contributes to an increased tooling cost. Full HMW sintering using an industrial microwave furnace has proven to reap outstanding properties at lower temperatures and faster processing times when compared with conventional sintering [12]. However, high investments may be required in purchasing an industrial microwave furnace (with higher heating capability) which does not make the process so appealing to industries. Therefore, this research is intended to cater for cheaper options by simply using domestic microwave oven and apply post sintering methods which can eventually enhance properties of the inserts and increase tool life. Hence, this research aims in determining a suitable composition of Si₃N₄ inserts that can be produced by Hot Isostatic Pressing (HIP) and followed by post sintering at 200 °C for only 10 minutes using Hybrid Microwave (HMW) energy. The outcome of this research is intended to produce inserts that can be used for machining hard materials; particularly Inconel 718.

II. EXPERIMENTAL PROCEDURE
A. Preparation of samples
Three different compositions of Silicon Nitride powders were used in this experiment as shown in Table I to produce round cutting tool inserts. The powder consists of Silicon Nitride (Si₃N₄), Yttrium Oxide (Y₂O₃), Magnesium Oxide (MgO), Aluminium Oxide (Al₂O₃) and Silicon Dioxide (SiO₂). These powders (Alfa Aesar) with the size of 0.5 µm were weighed using the digital weighing scale (Sartorius MECAPOL P230). Etching was done using phosphoric acid (H₃PO₄) for 100-120 seconds at room temperature. A Sputter Coater (SC7620) was used to coat the samples in order to make it conductive. Micro structural analysis was performed using Universal Testing Machine (401MVA), followed by compression test to determine the compressive strength and its corresponding tensile strength of the Si₃N₄ inserts using Universal Testing Machine (INSTRON 3367) with a tonnage of 100 kN. The results were compared with the commercial tool (Sandvik Coromant RNGN 6060) which can be used to machine hard materials, such as Inconel 718.

| Sample | Si₃N₄ (%) | Y₂O₃ (%) | MgO (%) | Al₂O₃ (%) | SiO₂ (%) |
|--------|-----------|-----------|---------|------------|---------|
| 1      | 88        | 5.5       | 2.5     | 2      | 2       |
| 2      | 90        | 4         | 2.5     | 2      | 1.5     |
| 3      | 92        | 2.5       | 2.5     | 2      | 1       |

The powders were compacted using cold press; manual pellet pressing machine (MP-15T) with a load of 150 kN and a holding time of 5 minutes for each sample. 18 samples with average diameter of 13.64 mm and average thickness of 5.33 mm were produced for each composition.

B. Sintering Process
The green samples were placed into the HIP furnace (AIP6-30H) at 1800°C at a heating rate of 5°C/min with 1 hour holding time. Argon gas was used in this HIP process. Then, 9 samples from the HIP were taken for further post-sintering using HMW. The Si₃N₄ samples were placed vertically inside a small alumina crucible with a diameter of 30 mm at the opening and covered with a lid. This is to ensure that the heating is uniform all around the insert. The small crucible was then placed inside a larger crucible with a diameter of 65 mm at the opening and submerged inside 50 cm³ (~ 300 mesh) of Silicon Carbide (SiC) powder (Alfa Aesar) which functions as a susceptor to aid in rapid hybrid microwave heating (Fig. 1). The larger crucible is covered with a lid as well. The crucibles were later placed inside the domestic microwave oven (Panasonic NN-C999TS) with a magnetron operating frequency of 2.45 GHz for 10 minutes at 200°C.

![Fig. 1: Setup of the Si₃N₄ sample inside the alumina crucible for HMW heating](image)

C. Mechanical Testing
The dimensions of the Si₃N₄ samples were recorded; the physical appearance and shrinkage were analyzed. The densities of the Si₃N₄ samples from each composition were measured using Densimeter (OK-300). Then, hardness test was performed using Vickers Micro-hardness Tester (401MVA), followed by compression test to determine the compressive strength and its corresponding tensile strength of the Si₃N₄ inserts using Universal Testing Machine (INSTRON 3367) with a tonnage of 100 kN. The results were compared with the commercial tool (Sandvik Coromant RNGN 6060) which can be used to machine hard materials, such as Inconel 718.

D. Scanning Electron Microscope
The Si₃N₄ samples were polished with the aid of Micro Polish Alumina (0.3 µm) on a polishing machine (PRESI MECAPOL P230). Etching was done using phosphoric acid (H₃PO₄) for 100-120 seconds at room temperature. A Sputter Coater (SC7620) was used to coat the samples in order to make it conductive. Micro structural analysis was performed by using the Scanning Electron Microscope (JEOL JSM-5600).
III. RESULTS AND DISCUSSION

A. Physical Appearance

The Si₃N₄ samples before and after sintering can be seen in Fig. 2. It is clearly obvious that the samples have undergone shrinkage. The three compositions: 88 Si₃N₄, 90 Si₃N₄ and 92 Si₃N₄ have very similar shrinkage values for both HIP (-8%) and HIP+HMW (-10%). In addition, change in color from the green condition (light grey) to after HIP (dark grey) and after HMW (grey) was observed.

![Image: Physical appearance of the Si₃N₄ samples](image)

B. Mechanical Properties

The densities for the three Si₃N₄ compositions are shown in Table II. Results show that HIP alone produced samples with 93%, 92% and 90% of theoretical density (TD) for 88, 90 and 92 wt% Si₃N₄ respectively. However, after post sintering with HMW for only 10 minutes at 200°C, the densities significantly increased to 97%TD, 96.6%TD and 93%TD. Certain amounts of additives such as Y₂O₃, MgO and Al₂O₃ were added to produce a glassy intergranular phase during sintering in order to achieve proper densification of the Si₃N₄ samples which can enhance metal cutting performance.

| TABLE II. DENSITY AND HARDNESS OF Si₃N₄ SAMPLES |
|------------------------------------------------|
| Sample | Density (g/cm³) | Hardness (HV) |
|--------|----------------|---------------|
|       | HIP            | HIP+HMW       | HIP            | HIP+HMW       |
| 88Si₃N₄ | 3.031          | 3.158         | 1124           | 1438          |
| 90Si₃N₄ | 2.997          | 3.136         | 1099           | 1396          |
| 92Si₃N₄ | 2.911          | 3.023         | 1002           | 1219          |
| RGN6060| 3.219          |               | 1454           |               |

Meanwhile, the hardness value for 88Si₃N₄ is the highest (1438 HV), followed by 90Si₃N₄ (1396 HV) and 92Si₃N₄ (1219 HV). This conforms to the findings with the density values of the samples where hardness increases as density increases. The density and hardness of HIP+HMW for both 88Si₃N₄ and 90Si₃N₄ are quite close to one another because of the weight percentage of MgO (2.5%) and Al₂O₃ (2%) that remained unchanged in both compositions. The 88Si₃N₄ and 90Si₃N₄ (HIP+HMW) samples have the density and hardness values which are quite similar to the commercial insert (RGN6 60).

The compressive strength values for these three compositions of Si₃N₄ samples are shown in Fig. 3. The compressive strength for each sample was calculated using (1) which is commonly used for ceramics and brittle materials in disk shape (Kalpakjian and Schmid, 2014),

\[ \sigma = \frac{2P}{\pi dt} \]

where \( \sigma \) is the tensile strength (MPa), \( P \) is the load applied along the centerline of the disk (N), \( d \) is the diameter of the sample (mm) and \( t \) is the thickness (mm).

![Image: Mechanical properties of Si₃N₄ samples](image)

Tensile strengths are generally low for ceramics due to its natural characteristics. All three compositions possessed almost similar tensile strength values in the range of 404 - 452 MPa; the highest value was found in 88Si₃N₄ (HIP+HMW) while the lowest value in 92Si₃N₄ (HIP). Ceramics are stronger in compression; which justifies the significantly larger compressive strength values for all three compositions of the Si₃N₄ samples. Larger compressive strengths were found in HIP+HMW samples when compared with HIP alone. 88Si₃N₄ produced the largest compressive strength (1578 MPa), followed by 90Si₃N₄ (1469 MPa) and 92Si₃N₄ (1260 MPa). Post sintering for 10 minutes using HMW resulted in an increase in compressive strength by 4.2%, 3.9%, and 2.1% for the 88Si₃N₄, 90Si₃N₄ and 92Si₃N₄ respectively.

Hybrid microwave post sintering provides efficient internal volumetric heating. The electromagnetic energy
which is converted into heat, is penetrated directly into the Si₃N₄ samples. The SiC powders which act as a susceptor with good microwave absorption characteristics, help improve heating rate. Heat is generated uniformly throughout the Si₃N₄ sample as a result from the energy transfer.

C. Micro Structural Analysis

The SEM images for the Si₃N₄ samples are shown in Fig.4. 88Si₃N₄ appeared to be very dense with uniform fine grained microstructure for both HIP and HIP+HMW. Meanwhile, for the 90Si₃N₄ (HIP), uniformly distributed small sized pores were visible and the HIP+HMW sample produced uniform grain size. Larger sized pores were noticeable in the 92Si₃N₄ (HIP) and larger grain size in 92Si₃N₄ (HIP+HMW). Better densification and improved microstructure are observed in the 88Si₃N₄ and 90Si₃N₄.

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