ABSTRACT

Hybrid Microwave sintering has become a growing interest for heating and synthesizing ceramic materials due to its capabilities in successfully enhancing densification and improving mechanical and structural properties. Silicon Nitride (Si$_3$N$_4$) based cutting inserts have outstanding properties for machining hard materials such as cast iron, hard steel and nickel based super alloys. This research aims to analyze the effect of different susceptors on the tool life of various Si$_3$N$_4$ inserts [90Si$_3$N$_4$ 4Y$_2$O$_3$ 2.5MgO 2Al$_2$O$_3$ 1.5SiO] that have been synthesized in machining Inconel 718. The Si$_3$N$_4$ inserts have been synthesized by means of 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 three different susceptor; Silicon Carbide (SiC), Graphite (G) and mixture of (SiC + G) powders. Density, hardness, micro structural properties and tool wear were analyzed. HMW post sintering for only 10 minutes using SiC, G and SiC+G susceptors enhanced the density (97-98%TD) and hardness (27-58%) significantly. Finer uniform grains and less porosities were produced particularly for Si$_3$N$_4$ inserts produced by HMW (SiC+G) when compared with HMW (SiC) and HMW (G). Tool life for the Si$_3$N$_4$ inserts were improved by 10-17% HMW(SiC), 20-53 % HMW(G) and 32-88% HMW(SiC+G) for the cutting speeds of 100, 125 and 160 m/min. Hence, the mixture of SiC +G powders as susceptors produced the best outcome for Si$_3$N$_4$ inserts with enhanced densification, hardness, wear resistance, and longer tool life (88% increment at 100 m/min) when compared with the commercial tool (RNGN 6060) in machining Inconel 718.

Keywords - silicon carbide; hybrid microwave post sintering; hot isostatic pressing; enhanced densification; improved hardness; improved wear resistance; longer tool life; Inconel 718
industries should readily be confronted with materials that are more difficult and challenging to machine than already existing materials.

Silicon Nitride (Si$_3$N$_4$) based cutting tool materials are found to have superior fracture toughness compared to alumina-based cutting tools. It is chemically inert and extremely resistant to heat and are usually desirable in high speed applications. The addition of yttrium oxide (Y$_2$O$_3$) and magnesium oxide (MgO) in the composition of Si$_3$N$_4$ enhances densification and increases the toughness of the inserts in metal machining operation (Burdun 1987). Altin et al. 2007 noted that the ceramic cutting tools are suitable for high speed machining super alloys, and the cutting speed increased up to three to five times than the carbide tools. Si$_3$N$_4$ cutting tools are believed to have concentrated on improving the high temperature, properties of strength, hardness and oxidation resistance. It is also outstanding with the wear resistance, both impingement and frictional modes. Due to this kind of superior toughness that the Si$_3$N$_4$ has, it would give a very positive result in the machining process.

The microwave energy is a process where it transfers the energy directly into materials by ionic induction, dipole relaxation and photon-photon interaction. From this, each crystal lattice in the molecules of the composites will raise at constant amplitude of vibration. This promotes the uniform heat distribution for the whole ceramic body. The densification of ceramic inserts is enhanced and significant reduction in porosities in the ceramic is observed (Cheng et al. 2000, Upadhyaya et al. 2001). Hybrid microwave heating is an improvement process from direct microwave heating. Hybrid microwave heating provides dual directional heating in which one is from the microwave energy and another one is from the heat of susceptor. The material selection for susceptor is plays an important role in hybrid microwave heating as it contributes to the rapid heating process. Silicon carbide (SiC) and graphite are possible materials as a susceptor since they have good absorbing microwave characteristics. Both SiC and graphite share the same properties; good wear, corrosion and thermal shock resistance. They can sustain high temperature and provide rapid heating to the sample in the microwave (Oghbaei and Mirzaee 2010).

Increased wear rates result in shorter tool life can lead to frequent changes in tools and increase in tool cost as well. Tool life has a direct influence in tool cost. By increasing tool life, the frequency of tool changing, tool cost and wear rates can be reduced. Several researches have been conducted in machining Inconel 718 with the aim to improve machining performance. These include hybrid machining, cryogenic machining and pre-heating and cooling-assisted technologies which successfully managed to extend tool life and improve surface integrity (Wang et al. 2003, Kaynak 2014, Zhuang et al. 2015). Tool wear and breakage are often the main problems that will reduce the performance of the product and the quality itself particularly in machining hard materials and superalloys. If the machining performance is not good, it will decrease the quality of product and increase the cost of production. Therefore, in order to increase quality in product and reduce the cost of production, the performance of inserts in the machining process need to be improved. Nevertheless, this justifies the importance of enhancing the mechanical properties of the cutting tool insert. The density, hardness, wear resistance, and tool life of the inserts must be enhanced. 90Si$_3$N$_4$ 4Y$_2$O$_3$, 2.5MgO 2Al$_2$O$_3$, 1.5SiO was selected for this research based on previous study by the authors (Ariff et al. 2018). Thus, this study is focused on improving the performance of Silicon Nitride (Si$_3$N$_4$) based inserts produced by Hot Isostatic Pressing (HIP) and followed by post sintering using hybrid microwave (HMW) energy with three different susceptors: silicon carbide, graphite and mixture of silicon carbide and graphite. The tool wear and tool life of the developed Si$_3$N$_4$ based inserts in machining Inconel 718 superalloy are analyzed.

II. EXPERIMENTAL PROCEDURE

2.1 Silicon Nitride (Si$_3$N$_4$) Insert Preparation

Five different powders (Alfa Aesar) with the size of 0.5 µm were used in this experiment, consisting of Silicon Nitride (Si$_3$N$_4$), Yttrium Oxide (Y$_2$O$_3$), Magnesium Oxide (MgO), Aluminium Oxide (Al$_2$O$_3$) and Silicon Dioxide (SiO$_2$). 90wt% Si$_3$N$_4$, 4wt% Y$_2$O$_3$, 2.5wt% MgO, 2wt% Al$_2$O$_3$, and 1.5wt% SiO were used to produce the Silicon Nitride inserts. These powders were weighed using the digital weighing scale (Sartorius CP224S) and then mixed in a planetary ball mill (FRITSCH 5) for 6 hours at 150 rpm with the aid of the steel balls (Ø 8.85 mm) in the ratio of number of balls to powder weight 1:5. 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. 6 samples with average diameter of 13 mm and average thickness of 5 mm were produced.

2.2 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, the 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 of susceptor powder. The susceptor powders (Alfa Aesar) with the size of ~ 300 mesh were used in HMW post sintering to aid in rapid hybrid microwave heating; 50 cm³ Silicon Carbide (SiC), 50 cm³ Graphite (G) and mixture of 25 cm³ (SiC) + 25 cm³ (G). The larger crucible was covered with a lid as well. The crucibles were later placed inside the domestic microwave oven (Panasonic NN-CD997S) with a magnetron operating frequency of 2.45 GHz for 10 minutes at 200°C. 2 samples from each susceptor were prepared.

2.3 Density and Hardness

The dimensions of the Si₃N₄ samples were recorded. The densities of the Si₃N₄ samples from each susceptor were measured using Densimeter (OK-300). Then, hardness test was performed using Vickers Micro-hardness Tester (401MVA). The results were compared with the commercial tool (Sandvik Coromant RNGN 6060) which can be used to machine hard materials, such as Inconel 718.

2.4 Scanning Electron Microscope

The four Si₃N₄ samples; HMW (SiC), HMW (G), HMW (SiC + G) and the commercial tool (RNGN 6060) were observed under the Scanning Electron Microscope (SEM) (JEOL JSM- 5600). These inserts were polished with Micro Polish Alumina (0.3 µm) on a polishing machine (PRESI MECAPOL P230). Phosphoric acid (H₃PO₄) was used for etching the inserts for about 100-120 seconds at room temperature. A Sputter Coater (SC7620) was used to coat the samples in order to make it conductive.

2.5 Machining

A cylindrical rod (Inconel 718) with a diameter of 80 mm and length 500 mm was used in this wet machining experiment to determine the tool life for the Si₃N₄ inserts that were produced from HIP and followed by post sintering using HMW (SiC), HMW (G) and HMW (SiC + G). The rod was divided into 5 segments of equal lengths (80 mm) and machined using a turning operation on a lathe machine (HARRISON M600). Three suitable cutting speeds (V) were used; 100, 125 and 160 m/min. A feed of 0.2 mm/rev and 0.2 mm depth of cut were used. The machining time taken to machine each segment was recorded. The flank wear was recorded after machining each segment using a microscope (Meiji Techno FU 1010). The results were tabulated into graphs to determine wear rates as well as tool life for each Si₃N₄ insert produced. The results were compared with the commercial tool insert (RNGN 6060).

III. RESULTS AND DISCUSSION

3.1. Physical Appearance

The Si₃N₄ samples after the HMW post sintering can be seen in Fig. 1. The 90 Si₃N₄ samples have undergone similar shrinkage values (-10%) after HMW post sintering with SiC, G and mixture of SiC and G. Color changes were observed and noticed that the samples changed from light grey (green condition) to slightly darker grey after HMW post sintering. Color changes in the samples appeared to be very similar from using all three different type of susceptors.

![Physical appearance of the Si₃N₄ samples from post sintering using HMW with different susceptors](image)

3.2 Density and Hardness

The densities for the three Si₃N₄ samples which were HMW post sintered are shown in Table 1. Results show that HIP alone produced samples with 92% of theoretical density (TD). Nevertheless, after post sintering with HMW for only 10 minutes at 200°C, the densities significantly increased to 96.6%TD, 97.3%TD and 98.3%TD from using SiC, G and SiC + G respectively. Meanwhile, the hardness value for HMW (SiC + G) is the highest (1741 HV), followed by HMW (G) (1556 HV) and HMW (SiC) (1396 HV). This correlates with the density values of the samples where hardness increases as density increases. The HMW (G) has increased by 7% while HMW (SiC + G) is 20% higher in hardness when compared with the commercial tool insert (RNGN 6060). The mixture of graphite with SiC (another microwave absorber material) can enhance the performance of the sample in an extensive way.
The density and hardness of HMW (SiC) is the lowest among all the samples and is lower by 2.5% in density and 4% in hardness when compared with the commercial tool insert (RNGN 6060). The density and hardness of HMW (SiC) is the lowest among all the samples and is lower by 2.5% in density and 4% in hardness when compared with the commercial tool insert (RNGN 6060). SiC has a lower thermal conductivity when compared with graphite which resulted in heating that is not as efficient as using graphite or mixture of SiC and graphite (Chandrsekaran et al. 2013). However, skin depth value of heating increases with decreasing conductivity (Mondal 2010). Nevertheless, for pure graphite which has higher thermal conductivity than pure SiC results in decreased skin depth penetration of microwave but improved rapid heating enhances densification and hardness more effectively when compared with SiC.

The improvement in the hardness of Si₃N₄ insert (HMW) (SiC + G) is because of larger amount of heat produced from microplasma (spark effect) of graphite which is transferred to SiC particles effectively. This contributes to enhanced rapid heating since SiC absorbs the microwave energy and heat from graphite. So, the heating rate of the mixture is greater than the pure G and SiC susceptor alone. It is believed that when using both SiC and G as the susceptors, there is a stronger interfacial bonding between all the compositions of Si₃N₄ at elevated temperature due to the penetration of heat throughout the skin depth increased with decreasing conductivity (Chandrasekaran et al. 2013, Menendez et al. 2010).

3.3 Micro Structural Analysis

The SEM images for the Si₃N₄ samples are shown in Fig. 2. Si₃N₄ sample produced from post sintering HMW (SiC + G) appeared to be very dense with uniform fine grained microstructure. Meanwhile, for the HMW (G), larger grain size with uniformly distributed small sized pores was visible. Larger sized pores with larger grain size were noticeable in the HMW (SiC). Better densification and improved microstructure are observed in the Si₃N₄ sample from HMW (SiC + G) when compared with using Graphite and SiC alone as susceptors. The mixture of SiC and G as susceptors results in improved rapid heating and prevents further grain growth which results in enhanced densification.

3.4 Tool Life Analysis

Flank wear measurements from machining Inconel 718 were obtained only for the three Si₃N₄ inserts that were synthesized by HMW; HMW(SiC), HMW(G) and HMW(SiC+G). The insert prepared by HIP alone had insufficient strength and could not be used for machining Inconel 718 as it broke the moment machining started. Results of the flank wear measurements can be seen in Figs. 3-5 for 100 m/min, 125 m/min and 160 m/min accordingly. The flank wear measurements were compared directly with the ready-made insert (RNGN 6060). Data were extrapolated to the maximum flank wear for the Si₃N₄ inserts which was taken as 0.7 mm. Results show that all the three HMW post sintered Si₃N₄ inserts performed significantly better in terms of wear rates when compared with the RNGN 6060 for all three cutting speeds. From these graphs, the tool life was obtained. The summary of the tool life for all the Si₃N₄ inserts is shown in Fig. 6. HMW (SiC+G) has shown to have the longest tool life with the largest increment (32-88 %), followed by HMW (G) (20-53 %) and HMW (SiC) (10-17%) when compared with RNGN 6060 in all the three cutting speeds (100 m/min, 125 m/min and 160 m/min).
Taylor’s Tool Life equation (Eq.1) was used to determine the respective tool life equations for all the Si$_3$N$_4$ inserts used in this research,

$$VT^n = C$$ (1)

where $V$ is the cutting speed (m/min), $T$ is the tool life (min), $n$ is the exponential value and $C$ is the constant value. The tool life graph was plotted (Fig. 7) to obtain the corresponding values of $n$ (from the slope) and $C$ (the intercept). The summary of the tool life equations obtained is listed in Table 2. It is observed that the value of $n$ decreases with increasing hardness of the tool insert. The tool life is longer when the exponential value $n$ is smaller.

The performance of Si$_3$N$_4$ insert from HMW (SiC + G) in machining Inconel 718 has been justified through the enhancement of tool life which does correlate with the improved densification, hardness and microstructure. The ability of HMW (SiC + G) prolonging tool life up to 88% at 100 m/min is considered a great breakthrough in terms of saving cost, and reducing tool changing frequency.

IV. CONCLUSION

Post sintering of Si$_3$N$_4$ inserts through HMW energy for only 10 minutes at 200°C with SiC, graphite and mixture of SiC and graphite powders as susceptor has significantly improved densification, hardness and wear resistance. Hence, tool life of the synthesized Si$_3$N$_4$ inserts (HMW (SiC+G)) in machining Inconel 718 is enhanced significantly up to 88% in machining Inconel 718 at 100 m/min. Mixture of SiC and graphite powder has shown to exhibit itself as the best form of susceptor because of their ability to complement each through their characteristics; i.e. SiC is not as conductive as graphite, nevertheless, it is able to couple with microwave better than graphite. The outcome from using SiC alone and graphite alone are not as good as when they are used in combination together. HMW post sintering can be an economical method to enhance mechanical and structural properties of Si$_3$N$_4$ inserts which in addition prolongs tool life and eventually can reduce tool changing cost and the frequency of changing the tool insert.
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