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Microwave Sintering and Its Application on Cemented Carbides

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***Abstract*—**Cemented carbides, owing to their excellent mechanical properties, have been of immense interest in the field of hard materials for the past few decades. A number of processing techniques have been developed to obtain high quality carbide tools, with a wide range of grain size depending on the application and requirements. Microwave sintering is one of the heating processes, which has been used to prepare a wide range of materials including ceramics. A deep understanding of microwave sintering and its contribution towards control of grain growth and on deformation of the resulting carbide materials requires further studies and attention. In addition, the effect of binder materials and their behavior during microwave sintering is another area that requires clear understanding. This review aims to focus on microwave sintering, providing information of how the process works and what type of materials it is best suited for. In addition, a closer look at some microwave sintered Tungsten Carbide-Cobalt samples will be taken and discussed, highlighting some of the key issues and challenges faced in this research area.

***Keywords*—**Cemented carbides, consolidation, microwave sintering, mechanical properties.

I. INTRODUCTION

FOR industrial applications, materials can be processed in a number of ways, and powder metallurgy is one that has been of great interest in recent times. The process includes several stages, one of which is the compaction of powders into green samples, which would then be sintered or further consolidated at a higher temperature and/or pressure in order to be able to impart the green samples desired shape and mechanical properties. During the sintering process which is a critical part of the manufacturing chain, the particles, initially compacted into green bodies, go through a high degree of growth in terms of grain size; a phenomenon that is undesirable, and needs to be controlled to an extent that is experimentally feasible. A number of techniques have been used in an attempt to reduce the grain size, including some innovative approaches taken in response to this emerging challenge faced in PM industry [1].

Microwave sintering is one of the processes that essentially became quite influential in ensuring unique microstructural features and mechanical properties, as well as low consumption and manufacturing cost for a wide range of materials including metals, ceramics and composites [2]. The way microwave sintering works is inherently different from how conventional sintering functions. When it comes to conventional sintering which is still being used in most manufacturing industries for production of materials, thermal energy is directed to the surface of the material through either radiation or convection process, which is then carried to the rest of the material via a relatively slow conduction method. In microwave sintering, the energy that is delivered however penetrates through the material through molecular interactions, which capitalizing on the electromagnetic field renders the energy transfer faster than the conventional process. The transformation within microwave sintering from electromagnetic to thermal energy is considered to be a conversion of energy rather than just heat transfer. The use of microwave sintering ensures that the generated heat is generated through the entire volume of the material, which will eventually lead to the material experiencing rapid heating. In addition, unlike conventional sintering, which is known to over-cure the surface than the interior, leaving traces of temperature gradients, it is easier to prepare samples without having thermal gradient present using microwave sintering.

This paper briefly covers the comparative advantages of microwave sintering over other sintering options, and presents some discussions on areas of applications with a special focus on cemented carbides.

II. MICROWAVE SINTERING: ADVANTAGES AND APPLICATIONS

The heating mechanism in microwave sintering is the main factor differentiating factor from other conventional heating methods when it comes to sintering materials. A list of tangible advantages of using microwave heating includes, but not limited to the following [3]:

- Faster and enhanced diffusion processes,
- Reduced energy consumption,
- Faster heating and cooling rates,
- Reduced processing times,
- Reduced sintering temperatures,
- Enhanced mechanical properties,
- Improved physical properties,
- Simplicity of operation,
- Reduced processing costs,
- Improved production quality,
- Production of innovative and advanced materials,
- Reduced health and environmental hazards

The understanding and control of microwave sintering are critical for many industrially manufacturable materials and products. While the feature ensures proper penetration of heat

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through materials, eventually leading to rapid heating, processes used on samples prior to microwave heating is of importance as well. For example, the nature of the green sample also affects the level of precision and quality to be expected from microwave sintering. It used to be thought that all metals are prone to radiating microwave or could generate plasma formation, which is why they are not safe to be considered for microwave heating [4]. It was later clarified that this idea was only applicable to sintered metals or those in bulk condition, at ambient temperature, and would not affect particles being heated at an elevated temperature range. This clarification then opened windows for microwave heating as a possible and rapidly growing tool for materials sintering.

A. Low Temperature Microwave Applications

The applications of microwave sintering are quite varied, as it has been used by a number of industries over the last few decades. However, based on temperature, applications of microwave sintering can be classified into two main groups, low and high temperature [4]. Low temperature (less than 500^{o}C) microwave applications cover the following areas:

- Communication (Technology used in cellular devices for establishing long distance communicative route)
- Food processing
- Rubber industry (Mainly focusing on preheating and vulcanization)
- Drying of wood based materials/products
- Ceramic drying
- Pharmaceutical products (Manufacture and preservation)
- Polymer
- Printing material
- Biomedical work

B. High Temperature Microwave Applications

Involvement of microwave heating at elevated temperatures exceeding 500^{o}C is relatively new, and has been observed only in the past couple of decades. High temperature applications of microwave sintering have primarily been focused on the following areas:

- Ceramics
- Composites
- Metallic materials
- Aerospace defence

Microwave sintering furnaces can be of different size, shape and capacity and depend primarily on the manufacturing requirement of the products. A sample list of specifications for a typical microwave sintering furnace is given in Table I [5]

As seen in Fig. 1, the microwave is produced by a generator, sometimes called magnetrons. The actual chamber where the heating takes place is insulated to avoid temperature gradient radiating inside and outside of the heating zone. The resistance heating tuner (R-H Tuner) helps with the heating while preventing rapid fluctuations in temperatures.

Ro et al. [8] carried out some extensive work to determine the difference in time between microwave sintering and conventional heating for a number of hard materials that were initially compacted and then sintered.

TABLE I
SAMPLE FEATURES OF A MICROWAVE FURNACE [5]

Microwave frequency	2.45 GHz
Output power	0 – 1.95 kW continuously variable
Maximum operating temperature	1700^{o}C
Maximum heating rate	50 ^{o}C/min
Temperature measurement	Infra-Red pyrometer
Temperature Control	PID controller with ramp and soak
Maximum load	1 Kg
Chamber size	390 mm (W) x 450 mm (D) x 480 mm (H)
Atmosphere	Air, Argon, Hydrogen, Nitrogen
Data acquisition	PC Interface System to record the process values as a function of time.

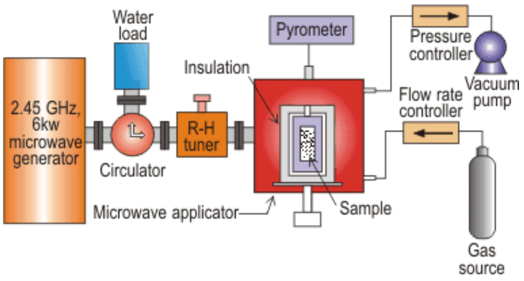

Fig. 1 Schematic diagram of a microwave furnace assembly [6], [7]

It was found that microwave heating was able to shorten the sintering cycle, which excludes the cooling phase, by a factor of 3 in comparison with conventional sintering [8]. The significant difference in heating time between conventional and microwave sintering evidently led to the understanding that microwave heating doesn’t only allow a faster path for sintering of materials, but also consumes much less electricity or energy due to a shorter processing cycle as opposed to conventional heating. By taking advantage of the features of microwave sintering, it is possible to sinter hard metals as well as other materials with presence of metallic binders at temperatures that are at least 50-100 K lower than the temperatures required for conventional sintering [8]. This, in effect, changes the entire dynamics of the heating pattern, as materials will be able to reach their desired sintering state well below the melting temperatures. Moreover, due to the fact that a lower temperature achieves the desired sintering state, grain growth within particles will be limited to a great extent. Grain growth inhibition is one of the critical challenges during sintering steps, since it is almost directly proportional to the temperature used in the process, meaning that a higher temperature would lead to a larger average grain size, as opposed to low temperature results. Fine grain size is a strong indicator of how well the product performs in terms of mechanical properties, and is expected to not deviate much from its original size, be it in grain or particle form. While this deviation is more in conventional sintering than microwave heating, it is easier for the latter to provide better balance in terms of mechanical properties, operational requirements and cost. Even in terms of densification, the reduction in final temperature and duration of the process while achieving enhanced mechanical properties, suggest that densification starts and reaches its final state faster than any other processes; a behavior that can be attributed to the simultaneous reduction of both open and closed porosities during microwave sintering.

III. Recent Developments

A. Metal Matrix Composites

As opposed to previous popular belief, materials in the form of particles with size less than 100µm can be microwave sintered at 2.45 GHz, even at elevated temperatures [1]. It is also now possible to understand that there might be a correlation among electrical conductivity of a material, final temperature, frequency and the successful operation of microwave sintering. With these ideas coming together and blending in, a number of metallic powders with various alloy compositions including but not limited to Fe, Co, Ni, Al, Sn, W, Mo have been successfully sintered using microwave processing [4]. The results were exceptional in terms of reaching a desired set of mechanical properties, microstructural features and time boundaries.

The work [9] focused on using both microwave and conventional furnaces for sintering of 92.5W–6.4Ni–1.1Fe compacts. In both cases, the powders were first compacted into green bodies, and then sintered via one of the sintering furnaces. It was found that W-Ni-Fe alloys that were consolidated using microwave furnace had a 75% reduction in sintering time as opposed to conventional heating. Added to that was the final sintering temperature that was lower for microwave heating. It was observed that the non-stoichiometric binder composition in the study did not result in formation of any brittle intermetallic phases after microwave sintering, which could explain the higher mechanical properties illustrated for the samples. Significantly low grain growth was observed in the final sintered samples that went through microwave processing (Fig. 2). The mechanical properties of the samples were also tested as part of their study, which concluded that microwave sintering not only allowed room for improvements in terms of microstructural features, but also yielded higher mechanical properties, including hardness of the sintered samples compared to their conventional counterparts.

In an attempt to differentiate the characteristics between conventional sintering involving a liquid phase, and microwave sintering, Breval et al. [10] used both sintering methods on several compositions of WC-Co. This study also concluded that microwave sintering played a crucial role in limiting grain growth of WC, as opposed to conventional sintering, and also ensured greater uniformity of Co or other binder phases in the matrix. Between microwave and conventionally sintered samples, the difference in WC grain size was quite large, which eventually resulted in a better and finer distribution of cobalt in the microwave-sintered sample. Unlike the conventional process, according to the principles, the major source of the heating in the microwave oven is oscillations of the free electrons at high frequency microwave in cobalt and in free carbon, and of ions in WC [11]. It is worth noting that the contribution of heat from oscillations of magnetic domains in Co is very small because of its small hysteresis [12].

TABLE II
CONVENTIONAL AND MICROWAVE SINTERING OF 92.5W–6.4NI–1.1FE ALLOY: COMPARISON OF STEREOLOGICAL PARAMETERS AND TUNGSTEN'S (W) SOLUBILITY IN THE MATRIX [9]

Attributes	Heating mode	
	Conventional	Microwave
W (vol.%)	78.0 ± 1.8	84.5 ± 4.7
Avg. grain size of W (µm)	17.3 ± 0.8	9.4 ± 0.5
Connectivity	1.8 ± 0.8	1.9 ± 0.7
Contiguity, Cg	0.32 ± 0.10	0.42 ± 0.09
SV (cm^{-1})		
W–W	0.04 ± 0.01	0.17 ± 0.04
W–M	0.15 ± 0.02	0.13 ± 0.03
Avg. dihedral angle (deg)	53 ± 7	63 ± 8
W concentration in Ni–Fe matrix (wt.%)	30.3 ± 2.5	21.7 ± 3.6

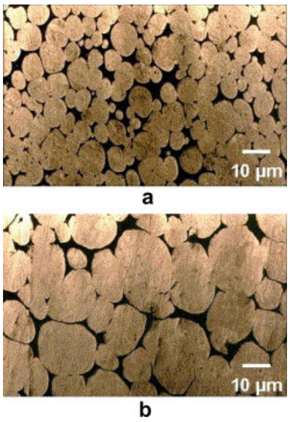

Fig. 2 Scanning electron micrographs of (a) microwave and (b) conventionally sintered W–Ni–Fe alloy [9]

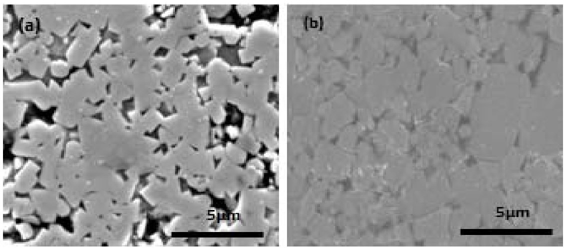

Fig. 3 (a) SEM micrograph of WC-7.5Co, microwave sintered at 1250°C. The initial particle size of both WC and Co were 1-3 µm. The final grain size observed was close to the original size of the particles, showing almost no grain growth in most areas. (b) SEM micrograph of WC-7.5 Co, conventionally sintered at around 1400 °C, with nanoparticles being included in the composition before sintering. A large proportion of grains went through significant growth and coarsening during the process [13].

For our study related to microwave sintering, a number of WC-7.5 wt% Co powder samples were first prepared, with three different size range; micron, submicron and nano. It was of interest to us to observe the differences in mechanical properties and microstructural features of these samples once sintered using microwave sintering. Since temperature plays an important role in sintering behavior of materials [14], the final sintering temperature was varied as well. It was found in our studies that at low temperature (1000°C), microwave sintering causes the particles to lightly join without any traces of liquid phase generated during the process. While that essentially succeeds in keeping particle or grain sizes in check, and not far from their original size, the mechanical behavior observed for those samples are rather towards the lower end. In contrast, employing slightly higher temperature, 1250°C or above causes the particles to bond in a better manner (Fig. 3 (a)), while also ensuring enhanced mechanical properties. Fig. 3 (b) on the other hand shows a micrograph of a conventionally sintered WC- 7.5% Co where nanoparticles were used as part of the composition before sintering was conducted [13]. It was found that a large proportion of grains went through considerable amount of growth before reaching the final sintering temperature for conventional sintering. Grain growth in microwave sintering is another critical factor that was observed in our work. It was concluded that grain growth inhibition is something that is present irrespective of the initial particle size. However, observation on several microwave sintered samples suggests that not every single grain behaves in one specific way. In most cases, the microstructure is a combination of grains that barely demonstrated any growth or coarsening, and grains that did to an extent. The mechanical properties, namely hardness, seemed to indicate very promising results, by outperforming their conventionally sintered counterparts.

IV. CONCLUSION

Microwave sintering has the potential to reduce processing time and enhance the quality of a product as microwave is capable of transferring energy throughout an entire volume of a material. The fact that the transfer of energy during this process takes place at an atomic level brings many advantages over other processing techniques. It is now clear that microwave sintering is applicable for a wide range of materials including alloys. The advantages of using this process are many, including but not limited to grain growth inhibition, faster heating, low temperature sintering, reduction in energy consumption and less precipitation of selective phases. For many metal matrix composites and ceramics, the use of microwave sintering has brought about exceptional results, with improved mechanical properties and distinguishable microstructural features.

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