Comparative Study of Conventional and Hybrid Microwave Sintering of Aluminum and Magnesium based Nanocomposites

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ABSTRACT
Sintering is one of the important steps in powder metallurgy methodology and is usually realized through conventional resistance furnaces. Process evaluation revealed that microwave assisted sintering can lead to a reduction of 86% in sintering time and energy savings of 96% when compared to conventional sintering. Most importantly, the study established the viability of microwave sintering approach used in place of conventional sintering for magnesium based formulations.

In the present paper comparative study has been made on the basis of the facts and findings available from the various literatures available in the field of powder metallurgical engineering.

KEYWORDS: Microwave sintering, Magnesium, Aluminum, Nanocomposites, Mechanical properties

INTRODUCTION:
Based on electromagnetic theory, metals are known to reflect microwaves and have limited penetration depth of a few microns. Moreover, special precautions have to be taken when placing metals in a microwave cavity due to the possibility of electrical discharge in the form of arcing which may damage the magnetron. As a result, research on harnessing microwave energy for heating is focused mainly on the processing of food, ceramics and polymers, with very limited work carried out so far on microwave heating of metals.

Currently, microwave heating is mainly used for the processing of food and in selected applications such as rubber vulcanization, drying of ceramics, chemical and pharmaceutical synthesis, cancer treatment, minerals processing, waste remediation etc [Stein, 1994; Clark and Folz, 2005; Wong & Gupta, 2007 (a) & (b)]. Cost savings can also be achieved with the elimination of inert atmosphere during microwave sintering.

Microwave heating is an emerging technology that can be used for the rapid and efficient heating of a wide range of different materials [Stein, 1994; Clark and Folz, 2005]. Some of the advantages of microwave heating include reduction in processing time, volumetric and uniform heating, selective and controlled heating, improved properties, environmental friendliness and potential in the synthesis and processing of novel and/or nanostructured materials.

Next, microwave sintering of hardmetals was reported by groups of researchers from the United States and Germany. In 1999, investigators from Penn State University demonstrated that metals in the form of powders can be heated rapidly using microwaves producing materials with better properties than conventional heating [Roy et al., 1999]. This opens up a great opportunity for the application of microwaves to process metal based materials to harness the many advantages of microwave heating. Recently, microwaves have been applied for the sintering of various metals and composites, melting of metals and metal ores, joining or brazing of metals and heat treatment of metals [Gupta and Wong, 2007]. Literature review of existing work performed on microwave sintering of metals revealed limited research conducted on the microwave sintering of metals and metal matrix composites (MMCs) [Gupta and Wong, 2007]. Most of the researches carried out so far involved the sintering of mainly ferrous and copper alloys.

Based on the following characterizations the entire focus of the paper is concentrated for the comparative studies:

Microstructural Characterization and Mechanical characterization

Microstructural Characterization
Finer microstructure can be observed in microwave sintered pure magnesium when compared to its conventionally sintered counterpart. This can be attributed to the shorter processing time for microwave sintering which leads to minimal microstructure coarsening. Microstructure characterization conducted on microwave sintered and extruded composite samples revealed the presence of a network of nanosized particulates decorating the particle boundaries of the matrix similar to that observed by other researchers.
working on aluminium and magnesium based composites reinforced with nanoparticles [Ferkel and Mordike, 2001; Kang and Chan, 2004]. Similar fabrication technique involving blending of powders, compaction, sintering and extrusion were employed in these studies, the main difference lies in the method of sintering (conventional resistance heating versus microwave heating). The micrographs revealed the presence of minimal porosity which is also supported by the results of experimental density measurements.

**Mechanical Characterization**

An improvement in microhardness was observed in microwave sintered pure aluminum and magnesium when compared to conventionally sintered counterparts. This can be attributed to the finer microstructure of the microwave sintered samples which is consistent with the findings of other investigators where it has been shown that microwave sintered metal compacts have higher hardness than their conventionally sintered counterparts [Anklekar et al., 2005; Roy et al., 1999]. The increase in hardness of magnesium matrix with the addition of nano-size reinforcements can be attributed primarily to the: (i) presence of harder nanopowder reinforcements in the matrix and (ii) higher constraint to the localized matrix deformation due to the presence of harder phases. Results of tensile testing revealed an overall improvement in the mechanical properties of microwave sintered samples. For pure magnesium, an improvement of 15% in 0.2%YS, 17% in UTS, 8% in failure strain and 61% in WOF was observed in microwave sintered specimens when compared to conventionally sintered specimens. For microwave sintered aluminum, an improvement of 14% in 0.2%YS, 18% in UTS and 17% in WOF was observed. Failure strain was reduced marginally by 0.7%. A simultaneous improvement in the average values of 0.2% yield strength, ultimate tensile strength and work of fracture for aluminum and magnesium over the conventionally sintered ones clearly indicates an improved microstructural integrity and refined microstructural features of the microwave sintered materials. The results of tensile testing revealed an improvement in 0.2%YS and UTS for all the composite formulations investigated. The largest improvement in strength was shown by Mg1.0Cu composite formulation with an increase of ~60% in 0.2%YS and ~26% in UTS. The increase in 0.2%YS and UTS can be attributed to:

(i) Work hardening due to the strain misfit between the reinforcing particulates and the matrix,
(ii) The formation of internal thermal stresses due to different thermal expansion behavior between the nano reinforcements and the matrix,
(iii) Reduction in grain size and
(iv) Effective load transfer between matrix and reinforcements.

Increase in failure strain with the addition of nano-size SiC and Al attributed to the activation of non-basal slip [Agnew and Duygulu, 2005]. Increase in ductility has also been observed in the past when Ti [Hassan and Gupta 2002], Mo [Wong and Gupta, 2005], CNT [Goh et al, 2006] and nano-A12O3 [Hassan and Gupta, 2004] was added to Mg. The reduction in failure strain for Mg1.0Cu can be attributed to the coupled presence of harder copper reinforcement and brittle Mg 3Cu intermetallic phase in the matrix which leads to plastic incompatibility and serve as potential crack initiation sites under tensile loading.

**RESULTS**

The different formulations synthesized in this study and the sintering conditions. Conventionally, the sintering temperature is maintained between 0.7 to 0.85 of the absolute melting point [German, 1996; Schaffer et al., 1999]. For all the materials investigated in this study, microwave sintering time was adjusted based on temperature calibration of the experimental setup so as to closely reach the melting point of the material. The results of macrostructural characterization on the sintered billets revealed absence of sintering defects such as circumferential or radial cracks and warpage in spite of the rapid heating during microwave sintering. Prior to extrusion, a small section of the billet was carefully removed and polished for macrohardness measurements at selected locations across the diameter of the billets. The hardness values obtained for pure aluminum and magnesium. The results revealed that near dense materials can be obtained using the fabrication methodology adopted in this study. The highest porosity was observed in Mg/1.0A12O composite samples and was limited to 0.83%.

Microstructural characterization studies revealed minimal porosity in the samples congruent with the results of density measurements. For composite samples, a continuous network distribution of nanoparticulates along
the grain boundaries was observed. A finer microstructure can be observed in microwave sintered magnesium when compared to its conventionally sintered counterpart.

Results of X-ray diffraction of materials were analyzed for Mg reinforced with SiC and Al. The results of hardness measurements revealed marginally superior micro hardness in hybrid microwave sintered monolithic samples when compared to conventionally sintered monolithic samples. The microhardness of composite samples was also observed to be superior to the monolithic samples. In magnesium and aluminum monolithic samples sintered using hybrid microwave assisted rapid sintering, a simultaneous improvement is observed in 0.2% yield strength (YS), ultimate tensile strength (UTS) when compared to conventional sintered samples. Similarly, magnesium nanocomposites displayed improved 0.2% YS and UTS with the addition of reinforcements.

DISCUSSION

The use of microwave sintering leads to a significant reduction in the processing time without compromising the end properties of the material. For conventional sintering of pure magnesium, the total sintering time required is 169 minutes (based on a heating rate of 10ºC/min from room temperature to 512ºC with no intermittent isothermal holding time and a 2 hours soaking duration at 512ºC.

For microwave sintering, the total sintering time was only 25 minutes (heating from room temperature to 640ºC and no soaking required). Assuming the cooling rate to be almost identical for both conventional and microwave sintering, an 85% reduction in processing time was achieved with the use of microwave sintering coupled with external susceptors in place of conventional sintering. Similarly for sintering of aluminum, a reduction of 82% in processing time is achieved. During conventional sintering, the heating rate is limited by the capability of the machine to achieve fast heating rates (due to slow resistive heating of the heating elements and heat transfer via thermal radiation to the material) and also to prevent large thermal variation within the compacts to avoid cracking or warpage. The slower heating rate, the need for holding at intermittent temperature to reduce thermal variation and long soaking time for sintering increases the total processing time of the compacts. For microwave heating coupled with external susceptors, rapid heating rates in excess of 20ºC/min can be easily achieved since the powder compact can absorb microwave energy directly and be heated rapidly from within. External susceptors provide radiant heating to the samples externally thereby reducing the thermal variation in the compacts. This is supported by macro hardness measurements conducted on the as-sintered compacts which revealed a lesser degree of variation in hardness across the diameter of the compact for microwave sintered magnesium compared to conventionally sintered magnesium.

Another advantage of microwave sintering is the rapid sintering of the metallic materials. It was indicated that a rapid heating rate will better activate the bulk transport and thereby improve the sintered properties [German, 1996]. Rapid heating minimizes grain growth and enhances the mechanical properties of materials. The micro structural and mechanical properties results obtained in this study supported the feasibility of the simple and inexpensive set-up used in the present study to sinter pure aluminum and magnesium.

CONCLUSIONS

Following are the main conclusions that can be drawn from this study:

(1) On comparing hybrid microwave sintered samples with conventional one, it has been found that the hardness, 0.2%YS, and UTS of monolithic magnesium and aluminum samples improved. Compared to conventional sintering, hybrid microwave assisted rapid sintering can lead to lowering in processing time up to 85% and energy savings of 96%. Micro structural characterization revealed that the microstructure for microwave sintered magnesium is smaller when compared to conventionally sintered magnesium. Properties of aluminum and magnesium can be enhanced using hybrid microwave sintering conducted close to melting temperature unlike conventional one. Nano-size reinforcements formed a continuous network along the grain boundaries of the matrix in magnesium composite formulations. Mechanical characterization revealed an increase in hardness, 0.2%YS and UTS of magnesium with the addition of nano-size reinforcements. Failure strain was improved with the addition of SiC and Al2O3 ceramic reinforcements but displayed the opposite trend with the addition of metallic copper as reinforcement. Significant reduction in processing time has been found in case of microwave sintering in contrast to conventional sintering. Not only has this but it also...
allowed the sintering of reactive magnesium metal without the aid of a protective inert atmosphere unlike conventional one. Finally it can be assured that the microwave technology has tremendous potential in saving energy and minimizing the production cost and duration without compromising the quality of the sintered product. Cost savings can also be achieved with the elimination of inert atmosphere during microwave sintering.

REFERENCES