Microwave sintering and mechanical properties of PM copper steel

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Microwave processing has gained worldwide acceptance as a novel method for heating and sintering a variety of materials from food to rubber to specialty ceramics, as it offers specific advantages in terms of speed, energy efficiency, process simplicity, novel and improved properties, finer microstructures, and lower environmental hazards. In the present paper, microwave sintering of modulus of rupture (MOR) bar samples of PM copper steel (MPIF FC-0208 composition) and the comparative evaluation of the mechanical properties using both microwave and conventional sintering techniques has been reported.

The starting powder characteristics and the processing details of copper steel bar samples sintered in a conventional furnace and in an in house modified commercial microwave oven has been covered at length. In this study, the sintering temperature used typically ranged between 1100 and 1300°C, soaking time ranged from 5 to 20 min, and the atmosphere was controlled using flowing forming gas (mixture of 95% $N_2 + 5\%$ H₂). Microwave sintering resulted in higher sintered density, higher Rockwell hardness (HRB), and higher flexural strength as compared with conventional sintering. The improved mechanical properties of microwave sintered samples can be mainly attributed to the evolution of distinct porosity distribution, primarily consisting of small, rounded, and uniformly distributed pores as against large, angular and non-uniformly distributed pores observed in the case of conventional sintering. PM/0916

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INTRODUCTION

Microwaves are electromagnetic radiation with wavelength ranging from about 1 mm to 1 m in free space, and frequency ranging from 0.3 GHz to 300 GHz. However, only narrow frequency bands centred at 915 MHz and 2.45 GHz are permitted for research purposes. Microwave heating of materials is fundamentally different from conventional heating in that the heat is generated internally within the material instead of originating from an external heating and subsequent radiative transfer. Microwave heating is a very sensitive function of the material being processed and depends upon such factors as size, geometry, and mass of the sample. In actual practice, the sample in fact becomes the source of heat during microwave processing.

Microwave processing has gained a lot of significance in recent times for materials synthesis and sintering mainly

because of its intrinsic advantages such as rapid heating rates, reduced processing times, substantial energy savings, novel and improved properties, finer microstructures, and being environmentally more clean. Recent reviews on micro-wave processing by Clark and Sutton,¹ Schiffman,² Katz,³ and Sutton,^{4,5} describe its potential use for a wide range of materials from wood, bacon, and potato chips to rubber, ceramics, and semiconductors.

The Microwave Research Group at the Materials Research Institute of the Pennsylvania State University, however, first made the step function advance in the microwave sintering of many traditional and advanced ceramic materials, such as alumina, mullite, and hydroxyapatite, by demonstrating very rapid sintering in time intervals varying from 3–20 min, leading to transparency and almost full density.⁶ This same step function advance in ceramic processing has been demonstrated in other commercial ceramics such zirconia, zinc oxide, perovskites, and silicon nitride.^{7–12} Microwave heating results in near theoretical densities in almost all cases in less than 30 min. The use of microwave processing has been most fully developed in the authors' laboratory and elsewhere to cemented carbide parts used in cutting and drilling tools.^{13–15}

The applicability of microwave sintering to metals has been simply ignored for the good reason that most metals are 'known' to reflect microwaves. The sintering community has explicitly ignored even the possibility of their sintering using microwaves.¹⁶ Very few papers have been found reporting the microwave sintering of powder metal alloys¹⁷ although a couple of papers do report the modest heating using microwave energy of some metal powders.^{18,19} The first publication of some of the preliminary results on microwave sintering of the powdered metals appeared recently from this group.¹⁶

Ferrous alloys are widely used in powder metallurgy (PM) for various applications, the predominant being for automotive uses. The PM copper and nickel steels are very extensively used for their high strength and excellent dimensional control.^{20,21} It has been reported that copper addition improves powder compressibility and green density, desired for near net shape processing. It also provides the liquid phase during sintering necessary for good densification and improving the mechanical properties. The present paper reports the experimental results of the systematic study undertaken for the sintering of PM copper steel (MPIF FC-0208) modulus of rupture (MOR) bar samples using microwave technique.

MICROWAVE SINTERING SETUP

A 2.0 kW commercial microwave oven (Amana Radarange, model RC/20SE) with 2.45 GHz multimode cavity was used to sinter PM copper steel samples in the present investigation. The microwave setup was modified in house to keep the external body temperature of the oven close to the ambient by circulating cold water through the copper tube fixed at the top and the sides of the double jacketed oven by brazing. The compressed air was circulated inside the cavity to keep the sensor positioned near the magnetron



Sintering Package Configuration

1 Schematic diagram of microwave sintering set up

from getting heated and thereby stopping the power supply to the magnetron. A mullite tube, 31.8 mm (outer diameter). $\times 25.4 \text{ mm}$ (inner diameter) $\times 914.5 \text{ mm}$ in length, was positioned at the centre of the oven, by drilling holes on the side faces, with ends projecting on both the sides. The mullite tube was supported mechanically by fixing two aluminium tubes with flanges from inside to provide support at the two ends. The aluminium flange interface with the side faces was sealed from inside using an aluminium foil for avoiding any microwave leakage. The schematic diagram of the microwave sintering set up used is shown in Fig. 1.

A mullite based insulation package made from Fiberfrax boards was used to surround the mullite tube at the centre of the cavity for containing the heat from dissipation during sintering. The package was so designed that it could be used both with and without the use of a susceptor or secondary coupler. Two different types of susceptor materials were used in the present study, namely. (i) SiC rods, typically four in number of 127 mm length each, inserted inside the insulation package, and (ii) a carbon based special coating material on the outer surface of the alumina tube, which couples very effectively with the microwaves. The details of the exact composition and the configuration used are reported in a patent application filed recently.²² The role of susceptor, whether in SiC rod or special coating form, is to mainly keep the temperature of the samples more uniform and also to provide initial heating of the sample before it starts coupling with the microwaves.

The samples were placed at the centre of the tube and the power to the oven was controlled manually using a standard variac (dimmerstat). The reducing atmosphere of flowing forming gas (5% $H_2 + 95\% N_2$ mixture) which has a dewpoint of -60° C was maintained inside the mullite tube for preventing oxidation of the samples, by fitting standard stainless steel connectors and T joints with 'O' rings at both ends of the tube. The gas flowrate inside the mullite tube was controlled using a pressure regulator and a gas flow meter. The temperature of the sample was monitored using an infrared pyrometer (Raytek, Marathon Series) with the circular crosswire focussed on the sample crosssection. The infrared pyrometer was integrated to a dedicated personal computer for accessing the temperature data as a function of time. The temperature was also measured using a conventional optical pyrometer (Leeds and Northrup) as a reference and in case of any temperature discrepancy; the latter was used as the correct temperature.

Sieve size, Mesh	Sieve aperture, µm	Weight fraction, %
+60	>250	Trace
-60+70	250-212	0.2
-70 + 100	212-150	9.9
-100 + 140	150-106	15.5
-140 + 200	106-75	20.2
-200 + 230	75-63	9.4
-230 + 325	63-45	17.2
-325	<45	27.6

EXPERIMENTAL PROCEDURE Starting powder characterisation

Atomet AXD 3401, an admixed copper steel alloy powder, sourced from Quebec Metal Powders Limited, Canada was used for fabricating the MOR bar samples, in the present investigation. The sieve analysis and chemical analysis of the base steel powder, along with the nominal composition of the admixed copper steel alloy powder used is given in Tables 1-3. The powder had an apparent density of 2.89 g cm⁻³ (ASTM B212) and a flowrate of 35s/50g (ASTM B213), both measured using the Hall Apparatus. The lubricant used was ethylene bis-stearamide (Acrawax C, a trade name of Algroup Lonza, Fine Chemicals and Specialties). The powdered metal lubricant offered high performance in terms of free flowing powder characteristics, low die wear, easy part removal from the die, reduced part distortion, and excellent green and sintered properties. Also, being clean burning and 100% organic, it was completely combustible and left no residue on sintering. No metallic or corrosive by products were formed during processing, leading to furnace corrosion and environmental pollution.

Powder compaction and binder removal

The MOR bar samples were fabricated by compacting ~20 g of ready to press admixed copper steel alloy powder using the automatic double action hydraulic press (Carver) with the floating die configuration. The die cavity was filled with powder and a load of ~27210 kg (equivalent to 60000 lb, corresponding to the maximum load of the hydraulic press) was applied for 2 min, which amounted to a compaction pressure of ~656 MPa. The dimensions of the green MOR bars were in the range of $31.85 \times 12.80 \times 6.95$ mm ±0.10 mm. The green density of the bar samples, as measured by direct measurements, was in the narrow range of 6.97-7.07 g cm⁻³.

The bar compacts were delubricated in a stainless steel muffle furnace fitted with a quartz tube by heating slowly

 Table 2
 Chemical analysis of base steel powder

Constituent	wt-%	
Carbon	0.005	
Oxygen	0.11	
Sulphur	0.008	
Manganese	0.19	
Iron	Balance	

Table 3 Nominal composition of admixed copper steel alloy powder

Constituent	wt-%	
Iron (ATOMET 1001)	97.20	
Copper	2.00	
Carbon (Graphite, SW-1651)	0.80	
Lubricant (Acrawax C, Atomised)	0.75	

to 500°C in 200 min in a reducing atmosphere of forming gas (95% N_2 + 5% H_2 mixture) having a dewpoint of -60° C. The temperature was controlled using a calibrated chromel-alumel thermocouple fitted to the above muffle furnace. The gas flowrate was controlled using a calibrated flow meter and the rate was maintained at 600 mL min-The samples were soaked for 6 h at 500°C to remove the binder completely. The weight loss after the delubrication process was measured and found to be $\sim 0.75\%$, which corresponded to the lubricant content of the admixed copper steel alloy powder, indicating almost complete binder burnout. The removal of binder prior to microwave sintering in a separate step was done to avoid the contamination of the microwave system. The soaking time of 6 h to remove the binder is very typical of these systems in a conventional process to ensure complete binder burn out. The measurement of the sample dimensions after the delubrication process, showed no change with respect to the original dimensions of the samples, thereby indicating no distortion. The slow heating during binder removal also resulted in no blister or crack formation. The samples were later stored in a desiccator to prevent corrosion.

Sintering of MOR bar samples

The delubricated MOR bar samples were sintered using both conventional and microwave heating techniques. Five sets of samples were sintered for each of the fixed parameters studied for the possible variations in processing and for checking the reproducibility of the final properties measured. The conventional sintering was carried out in an alumina tube furnace (Lindberg) fitted with a proportional integral differential (PID) temperature controller (Eurotherm). Two sintering temperatures namely 1140 and 1260°C were employed and the samples were soaked for 20 min each in a reducing atmosphere of flowing forming gas (95% $N_2 + 5\%$ H₂ mixture) having a dewpoint of -60° C. The microwave sintering of MOR bar samples was carried out both with and without using a susceptor in a 2.0 kW Amana commercial oven operated at a frequency of 2.45 GHz in multimode operation.

Microwave sintering experiments with SiC rods as a susceptor were carried out at four different temperatures, namely 1100, 1140, 1260, and 1300°C with soaking times of 5, 10, and 20 min in a reducing atmosphere of forming gas with a dewpoint of -60° C. Microwave sintering was carried out using the special carbon based coating material as a susceptor at two temperatures, namely 1140 and 1260°C, for soaking time of 20 min in a reducing atmosphere of forming gas having a dewpoint of -60° C. The delubricated samples were placed in an alumina boat inside the mullite tube at the centre of the cavity and the infrared pyrometer focussed at the centre of the sample cross-section. The temperature of the microwave cavity was kept close to that of the ambient by circulating cold water and compressed air during the experiment. The temperature of the sample was controlled by manually varying the variac (dimmerstat) voltage with time and the voltage typically ranged from 140 to 170 V. The infrared pyrometer was integrated to the personal computer for acquiring the temperature data as a function of time. The sample was kept in the microwave cavity in flowing forming gas atmosphere till it was cooled almost close to the ambient temperature to prevent any oxidation or decarburisation of the sample surface.

Physical and mechanical properties evaluation

All the five sets of conventional and microwave sintered samples were characterised for sintered density using the Archimedes' principle. The exact volume was calculated using the liquid displacement method, employing distilled water in the present experiment. The results obtained represent the average value of the readings obtained on the



2 Typical temperature-time plots obtained for sintering at 1140° for 20 min soaking

five sets of samples investigated for each of the fixed parameters studied. The average value of scatter observed in the results obtained for each of the properties studied is given in the next section.

The carbon content was measured using the standard carbon analyser (Leco CS 244) coupled to a HF 100 induction furnace. The carbon content was determined by heating the sample in an oxygen atmosphere and detecting the amount of carbon dioxide generated using an infrared detector. The hardness measurements were carried out using the Rockwell hardness tester (Series 2000, Wilson/Shore Instruments, Instron Corp.) in B scale (HRB) with 1/16 inch (1.5875 mm) ball indentor and a load of 100 kg. The flexural strength was determined by 4 point bending technique using the universal testing machine (Instron, model 4206) integrated to a Gateway 2000 (Crystal scan) Pentium and loaded with Instron Series IX software. The microstructures and porosity distributions of the conventional and microwave sintered samples were carried out by an optical microscope (Olympus BX60M, Hitech Instruments) and the observations were made using reflected light bright field mode. The microstructural details of the fractured surface of the MOR test samples were observed using environmental scanning electron microscope (Electro Scan, model E-3).

RESULTS AND DISCUSSION

The typical temperature–time profiles, for conventional and microwave sintering of copper steel MOR bar samples, at 1140 and 1260°C for soaking time of 20 min, are shown in Figs. 2 and 3, respectively. It is clear from these figures that microwave sintering, using the special coating material as a susceptor, yielded the fastest heating rates as compared with any other heating methods used, and with considerable savings in time. The microwave sintering without a susceptor was faster than that using SiC rods as a susceptor, for both the temperatures studied, and took about 12 and 40 min less time for reaching 1140 and 1260°C, respectively. This can be explained by the fact that, when a susceptor is used, part of microwave energy is absorbed by the susceptor, and therefore, there is not as much microwave energy available as in the case of not using a susceptor.

Microwave sintering using the special carbon based coating material as a susceptor took only about 62–64 min, while microwave without a susceptor took about 162–170 min for the complete sintering cycle for both the temperatures used. However, there was marked difference in the times



3 Typical temperature-time plots obtained for sintering at 1260°C for 20 min soaking

taken for attaining these temperatures for the conventional and microwave sintering with SiC rods as a susceptor. Conventional sintering took 46 min more time while microwave sintering with SiC rods as a susceptor took 36 min more time for attaining 1260°C as compared with 1140°C. Interestingly, for attaining 1140°C, the temperature–time profile for conventional sintering matched well with that of microwave sintering without using a susceptor, while for 1260°C, the conventional sintering took almost the same time as microwave sintering with SiC rods as a susceptor.

The sintering conditions and physical properties of microwave sintered copper steel MOR bar samples using SiC rods as a susceptor are given in Table 4. The green density of the bar samples varied in a narrow range, namely $6.97-7.07 \text{ g cm}^{-3}$, while the sintered density varied in the range of $7.08-7.33 \text{ g cm}^{-3}$ for different sintering temperatures and times employed. The typical scatter in sintered density results was observed to be $\pm 0.05 \text{ g cm}^{-3}$, representing an average fixed value. The weight loss on sintering for all the samples was found to vary between 0.126 and 0.198%.

The sintering conditions and physical properties of the conventional and microwave sintered copper steel MOR bar samples are given in Table 5. The samples sintered at

 Table 4
 Sintering conditions and physical properties of microwave sintered* copper steel MOR bar samples

Sintering temperature, °C	Sintering time, min	Sintered density,† g cm ⁻³	Weight loss on sintering, %	Carbon content, %
1100	5	7.08	0.126	0.76
1100	10	7.11	0.150	0.75
1100	20	7.16	0.157	0.76
1140	5	7.12	0.149	0.77
1140	10	7.16	0.177	0.74
1140	20	7.22	0.179	0.75
1260	5	7.18	0.185	0.74
1260	10	7.24	0.185	0.75
1260	20	7.32	0.189	0.74
1300	5	7.20	0.194	0.72
1300	10	7.25	0.198	0.73
1300	20	7.33	0.197	0.71

* Microwave sintered using SiC rods as a susceptor.

† Sintered density measured by liquid (water) displacement method using the Archimedes' principle. The typical scatter was observed to be ± 0.05 g cm⁻³.



4 Sintered density of copper steel MOR bar samples sintered in microwave system using SiC susceptor, as function of sintering for different sintering temperatures

 1260° C for 20 min showed higher sintered density than those sintered at 1140° C for 20 min, in the case of both microwave sintering using different susceptor configurations and conventional sintering. The weight loss on sintering for all the samples was observed to be between 0.171 and 0.222%.

The sintered density of copper steel MOR bar samples processed in microwave using SiC susceptor, as a function of sintering time and for different sintering temperatures is shown in Fig. 4. The sintered density increased consistently both as a function of sintering temperature and time, however, the increase in sintered density for 20 min soaking time with reference to 10 min soaking time decreased as compared with the sintered density for 10 min soaking time with reference to 5 min soaking time.

It is important to note that the sintered density results showed clear cut trends, namely. the conventional sintered samples showed lower density in the range of 7.00-7.15 g cm⁻³. The microwave sintered samples with SiC rods as a susceptor, showed slightly higher sintered density in the range of 7.08-7.33 g cm⁻³, for all the various sintering temperatures and times used. The microwave sintered samples, both without SiC rods as a susceptor and using the special carbon based coating material as a susceptor, showed fairly higher sintered density in the range of 7.28-7.36 to 7.40-7.45 g cm⁻³, respectively. The sintered density obtained was maximum for the microwave sintered samples using the special carbon based coating material as a susceptor, sintered at 1260°C for 20 min in forming gas atmosphere (5% H_2 +95% N_2 mixture) having a dewpoint of $-60^{\circ}C$, and showed a typical value of 7.45 g cm^{-3} . The typical scatter in the sintered density results of the above samples was observed to be ± 0.05 g cm⁻³, representing an average fixed value.

The Rockwell hardness (HRB) of copper steel MOR bar samples, processed in microwave using SiC susceptor, as a function of sintering time, for different sintering temperatures, is shown in Fig. 5. The hardness values increased with increasing sintering temperature, for soaking time of 5 min. However, the increase in hardness with increasing sintering temperature, for soaking time of 10 min was not as prominent, and for 20 min soaking, the hardness values converged to a narrow range of 76-78. The hardness increased with temperature for the soaking times of 5 and 10 min, and thereafter for longer soaking times there was no substantial change in the hardness. The microwave sintered samples using SiC rods as a susceptor showed the hardness to be the highest with a typical value of 82 ± 2 for the sintering conditions of 1260°C, with a 5 min soaking in flowing forming gas atmosphere.

The flexural strength of copper steel MOR bar samples processed in a microwave oven using SiC rods as a susceptor, as a function of sintering time for different sintering temperature is shown in Fig. 6. The flexural strength of the samples was at a minimum for the samples sintered at 1100°C for



5 Rockwell hardness of copper steel MOR bar samples sintered in microwave system using SiC susceptor, as function of sintering time for different sintering temperatures



6 Flexural strength of copper steel MOR bar samples sintered in microwave system using SiC susceptor, as function of sintering time for different sintering temperatures

5 min soaking and had a typical value of 799 ± 10 MPa, while with increasing sintering temperature and for the same soaking time, the flexural strength was observed to increase and be in a narrow range of 957–990 MPa. However, the flexural strength increased with increasing sintering temperature for 10 min soaking, while for 20 min soaking the flexural strength was very close, namely 1021 ± 10 MPa and 1029 ± 10 MPa, respectively, for the sintering temperature of 1100 and 1260°C. The flexural strength increased with increasing sintering temperature for the same soaking time, except for 1140°C, 10 min and 1300°C, 20 min sintering conditions.

The comparison of sintered density of samples, using conventional, and microwave sintering with different susceptor configurations, for two different sintering conditions studied is shown in Fig. 7. It is clear from the figure that the sintered density of the MOR bar samples was higher for microwave sintering, for all three different susceptor configurations



7 Histogram showing comparison of sintered density for conventional and microwave sintering for two different sintering conditions

employed, as compared to conventional sintering for both temperatures of 1140 and 1260°C used. The conventional sintered samples showed the least sintered density, followed by microwave sintered using a SiC rod as susceptor. This was followed by microwave sintered without SiC rod as susceptor, and the microwave sintered with the coating susceptor showed the highest sintered density for both the sintering temperatures of 1140 and 1260°C employed.

The comparison of Rockwell hardness (HRB) for the conventional, and microwave sintering using different susceptor configurations, for two different sintering conditions is shown in Fig. 8. The Rockwell hardness of microwave sintered samples was higher for all different susceptor configurations studied, as compared to conventional sintered samples, for both the sintering temperatures investigated. The higher hardness values observed for microwave sintered samples as compared with conventional sintered samples, cannot be explained by slightly higher sintered density of the microwave sintered samples alone, and may be attributed to the novel microstructure, as discussed later in this section. The hardness values of microwave sintered samples at 1140°C were more or less same for all of the three different susceptor configurations studied. However, there was a trend showing an increase in hardness value for microwave sintered samples at 1260°C, using different susceptor configurations, namely from using SiC rods, to that without using SiC rods, to that using special coating as susceptor. The highest hardness value was achieved for microwave sintered samples using the coating as susceptor.

The comparison of flexural strength for the conventional and microwave sintering with different susceptor configurations, for two different sintering conditions studied

Sintering type	Sintering temperature, °C	Sintering time, min	Sintered density,† g cm ⁻³	Weight loss on sintering, %	Carbon content, %
Conventional	1140	20	7.00	0.194	0.74
Microwave (SiC Rods)	1140	20	7.22	0.179	0.75
Microwave (No SiC Rods)	1140	20	7.28	0.191	0.72
Microwave (Coating)	1140	20	7.40	0.171	0.72
Conventional	1260	20	7.15	0.222	0.72
Microwave (SiC Rods)	1260	20	7.32	0.189	0.74
Microwave (No SiC Rods)	1260	20	7.36	0.195	0.72
Microwave (Coating)	1260	20	7.45	0.185	0.71

Table 5 Sintering conditions and physical properties of conventional and microwave sintered copper steel MOR bar samples

 \dagger Sintered density measured by liquid (water) displacement method using the Archimedes' principle. The typical scatter was observed to be ± 0.05 g cm⁻³



8 Histogram showing comparison of Rockwell hardness for conventional and microwave sintering for two different sintering conditions

is shown in Fig. 9. The flexural strength values in the case of microwave sintered samples were significantly higher, typically as high as 15-30% as compared to the conventional sintered samples, and agreed well with what has been reported on similarly processed samples.¹⁷ The maximum flexural strength obtained was 1077 ± 10 MPa for microwave sintered samples at 1140°C for 20 min soaking in forming gas atmosphere using the coating as a susceptor. The trend for the flexural strength was found to be opposite for the sintering temperatures of 1140 and 1260°C, when compared with that observed for the Rockwell hardness (HRB). The higher flexural strength results for the microwave sintered samples as compared with the conventional sintered samples can be attributed to the higher sintered density values obtained using the Archimedes' principle of liquid displacement.

The optical microstructures and porosity distributions of the conventional and microwave sintered samples, using both SiC rods and the special carbon based coating material as susceptors, as a function of depth are shown in Fig. 10. Figure 10*a*, *b*, and *c* represents conventional sintering, while Fig. 10*d*, *e*, and *f* represents microwave sintering with SiC rods as a susceptor, and Fig. 10*g*, *h* and *i* represents microwave sintering using the special coating as



9 Histogram showing comparison of flexural strength for conventional and microwave sintering for two different sintering conditions

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a susceptor. It is clear from the optical micrographs of the porosity distribution (Fig. 10) that the conventional sintered samples showed maximum porosity, consisting mainly of large, angular, and non-uniformly distributed pores, while the microwave sintered samples using SiC rods as a susceptor showed intermediate porosity, consisting mostly of medium, rounded, and uniformly distributed pores. The microwave sintered samples using the special carbon based coating material as a susceptor showed minimum porosity, primarily consisting of very small, rounded, and uniformly distributed pores. However, the reason for this characteristic microstructure in case of microwave sintering as compared with conventional sintering is not yet completely clear and is presently being investigated from the view point of fundamental understanding of the various sintering mechanisms. This explains clearly the reason for the higher flexural strength values obtained for the microwave sintered samples as compared with the conventional sintered samples. These results also corroborate well with the higher sintered density values obtained using the Archimedes' principle method as well as with the optical porosity distribution results obtained.

Figure 10b, e, and h represents the core of each of the above samples while Fig. 10a, c, d, f, g, and i represents the top and bottom edges of the above samples. It is evident from Fig. 10a, b, and c that conventional sintering resulted in a non-uniform microstructure with dense edges and a slightly more porous core. This is typical feature observed for conventional sintering of PM parts. It is clear from Fig. 10d, e, and f that microwave sintering with SiC rods as a susceptor resulted in somewhat lesser degree of nonuniform microstructure and a clear cut reversal of the conventional situation, i.e. a denser core and slightly porous edges were observed. This is typical of most ceramics sintered in a conventional microwave system, indicating that heat transport is from inside out, and the interior of the material is generally hotter than the surface. It is quite obvious from Fig. 10g, h, and i that microwave sintering with the special carbon based coating as a susceptor resulted in very uniform microstructure, and both uniformly dense core and edges were observed. This proves the point that in a suitably modified microwave system very uniform microstructures can be achieved.

The SEM fractographs of the modulus of rupture test samples sintered at 1140 and 1260°C both by conventional and microwave techniques using different susceptor materials are shown in Figs. 11 and 12, respectively. From the fractographs it is clear that both conventional and microwave sintering studies resulted in similar type of microstructural features, for both the temperatures studied. However, the higher flexural strength values observed for microwave sintered samples, for all the three different susceptor materials/ configurations studied, can be attributed to the evolution of distinct porosity distribution consisting of small, rounded, and uniformly distributed pores as against large, angular, and non-uniformly distributed pores in case of conventional sintered samples.

CONCLUSIONS

The modulus of rupture bar samples of copper steel (MPIF FC-0208 composition) were successfully sintered by microwave technique to obtain higher sintered density, Rockwell hardness (HRB), and flexural strength, thus yielding equivalent and at times even superior mechanical properties than conventional sintering. Some of the important results are as follows.

1. The maximum sintered density obtained was $7.45 \pm 0.05 \text{ g cm}^{-3}$ for the microwave sintered samples using the special carbon based coating material as a susceptor, and sintering at 1260°C for 20 min in forming gas atmosphere (5% H₂ + 95% N₂ mixture) with a dewpoint of -60° C.



10 Optical micrographs showing porosity distribution of conventional sintered (a-c), microwave sintered with SiC susceptor (d-f), and microwave sintered with coating susceptor (g-i) unetched samples. Centre micrographs (b, e, h) represent core, while rest of micrographs represent edges of copper steel MOR bar samples

2. The highest Rockwell B hardness (HRB) of 82 ± 2 was obtained for microwave processed samples using SiC rods as a susceptor, and sintered at 1260°C for 5 min soaking in flowing forming gas atmosphere.

3. The maximum flexural strength of 1077 ± 10 MPa was obtained for microwave sintered samples at 1140° C for 20 min soaking in forming gas atmosphere using the coating as a susceptor.

4. The microwave sintered samples using the special carbon based coating material as a susceptor showed minimum porosity, consisting of very small, rounded, and

uniformly distributed pores as against conventional sintered samples, which showed maximum porosity, mainly consisting of large, angular, and non-uniformly distributed pores. The microwave sintering using SiC rods as a susceptor, showed intermediate level of porosity, primarily consisting of medium, rounded, and uniformly distributed pores.

5. The microwave sintered samples using the special carbon based coating material as a susceptor showed very uniform microstructure with minimum porosity, as against conventional sintered samples, which showed non-uniform microstructure, namely denser edges and a porous core.



a conventional; *b* microwave with SiC susceptor; *c* microwave without SiC susceptor; *d* microwave with coating susceptor 11 SEM fractographs of copper steel MOR bar samples sintered at 1140°C, 20 min



a conventional; b microwave with SiC susceptor; c microwave without SiC susceptor; d microwave with coating susceptor

12 SEM fractographs of copper steel MOR bar samples sintered at 1260°C, 20 min

The microwave sintering using SiC rods as a susceptor showed the reverse behaviour, i.e. a denser core and slightly porous edges, but more uniform microstructure.

6. The improved mechanical properties of the microwave sintered samples using different susceptor materials may be attributed to the evolution of small, rounded, and uniformly distributed pores as against large, angular shaped, and non-uniformly distributed pores in conventional sintering in addition to the effect of higher sintered densities observed.

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