Improved mechanical properties and microstructural development of microwave sintered copper and nickel steel PM parts


The application of microwave technology to a diverse range of materials and processes has resulted in a wide spectrum of materials that are commercially processed using microwaves, from the heating of food to the vulcanisation of rubber to the sintering of specialty ceramics. Microwave sintering of elemental or alloy metal powders has gained significance in recent times as a novel processing method since it offers many advantages over the conventional sintering method. Despite substantial R&D investment in this area in the past two decades, no competitive microwave technology has yet emerged for powder metallurgy (PM) sintering. In sharp contrast, because it is 'obvious' that microwaves are reflected by metals, it is not uncommon to be unable to locate many journal papers or literature, wherein metal powders have been sintered in a microwave field. This paper reports the improved mechanical properties and microstructural development of microwave sintered copper and nickel steel PM parts as compared with that obtained using conventional sintering technique. The paper describes the fabrication details of the FC-0208 and FN-0208 composition steel PM parts, and the in-house modified commercial microwave oven used for sintering. Microwave sintering resulted in higher sintered density and improved mechanical properties for both Cu and nickel PM parts as compared with that processed using conventional sintering under identical conditions. The improved mechanical properties can be attributed primarily to more uniform distribution of the alloying elements, which resulted in greater material homogeneity at the nano- and microlevels as revealed by the Cu and Fe X-ray maps using high spatial resolution scanning transmission electron microscopy (STEM). The optical micrographs of both the etched and unetched samples clearly showed development of novel sintered microstructures having distinct characteristics for the porosity distributions: smooth and rounded pores with low stress concentration regions for microwave sintering as against sharp, triangular and wedge shaped pores with high stress concentration regions for conventional sintering. PM/1099

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Keywords: Microwave Sintering, FC-0208 and FN-0208 Steel PM Parts, Mechanical Properties, Sintered Microstructures, Porosity Distribution, Cu and Fe X-ray Maps

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INTRODUCTION

Microwaves can be defined as that part of the electromagnetic radiation spectrum having a wavelength typically ranging from about 1 mm to 1 m in free space, and the frequency ranging from about 300 MHz to 300 GHz. However, only narrow frequency bands centred at around 915 MHz, 2-4.5 GHz, 26-30 GHz and 80-81 GHz are actually permitted for microwave research purposes. Microwave processing has gained a lot of significance in recent years for high temperature processing and synthesis of a diverse range of materials, mainly because of its intrinsic advantages such as rapid heating rates, shorter processing times, uniform temperatures with minimal thermal gradients, higher energy efficiency, improved mechanical properties, novel finer microstructures, reduced atmospheric interaction and lesser environmental hazards as compared with the conventional sintering process.

Microwave heating of materials is fundamentally different from conventional heating, in that the heat is generated internally within the whole volume of the material, unlike from an external source in a conventional process, and subsequent heat transfer involving a thermal conductivity mechanism. Microwave heating is a very sensitive function of the material being processed and depends on a variety of factors such as size, geometry, mass and dielectric property of the sample. As a matter of fact, the sample itself becomes the source of heat during processing in a microwave field.

The dielectric constant \( \varepsilon_r \) or the relative permittivity \( \varepsilon_r \), the dissipation factor \( \tan \delta \), and the dielectric loss \( \varepsilon_r \times \tan \delta \) of the material being processed, and their dependence on temperature generally dictates to a large extent the microwave power absorption characteristics during microwave sintering of ceramic materials. Materials that have a high dielectric loss couple well with microwaves at room temperature while the poor lossy materials require initial heating using some kind of secondary susceptors. For most powder metals, no susceptor (preheater) is needed, but it is mainly used to provide uniform temperature distribution throughout the sample to obtain uniform sintering.
Recent reviews on microwave processing by Clark and Sutton,1 Schiffman,2 Katz,3 Sutton,4,5 and Agrawal,6 describe the potential uses of microwave technology for a wide range of diverse materials from wood, bacon and potato chips to rubber, semiconductors and ceramics. The microwave research group at Materials Research Institute of the Pennsylvania State University has made significant advancement in the sintering of many traditional and advanced ceramic materials such as alumina, mullite and hydroxypatite by demonstrating rapid sintering in very short times typically 3-20 min, leading to transparency and full density. These advances in ceramic processing have been demonstrated in other commercial ceramics, such as zirconia, zinc oxide, silicon nitride and other perovskite structures.7-13 Microwave sintering has been reported to result in uniform heating and near theoretical densities in almost all cases in less than 30 min of total cycle time.8-13

The use of microwave sintering has been most fully developed in the authors' laboratory and elsewhere to WC/Co cemented carbide composites with improved mechanical properties.14-16 The cemented carbides are widely used in metal cutting, metal forming and mining tools, and as wear parts. The applicability of microwave sintering to metals has been simply ignored for the good reason that all the commonly known metals are well known to reflect microwaves. The powder metallurgy (PM) community has explicitly ignored even the possibility of sintering the metal powders using microwaves.17 Very few papers and patents have been reported for the sintering of metal alloy powders, although a couple of papers report only the modest heating of some elemental metal powders.22,23 Some of the preliminary results on the microwave sintering of powder metals were first published by this research group.17 In our recent publication, the microwave group has reported a substantial improvement in the mechanical properties obtained for the microwave sintered copper steel PM modulus of rupture bars over that obtained in the conventional sintering process.20

Ferrous PM alloys are widely used for a variety of structural applications, the predominant ones being for the automotive industry. The copper and nickel steel PM parts are used extensively for properties such as high mechanical strength and excellent dimensional control.24 It has been reported that alloying with copper and nickel improves the powder compressibility and the green density that is desired for near net shape processing. It provides the desired liquid phase during sintering necessary for good densification, and also improves the dynamic mechanical properties such as impact strength, fracture toughness and fatigue strength. The present paper reports the experimental results obtained from a systematic study undertaken for the microwave sintering of ferrous PM fabricated parts, such as modulus of rupture (MOR) bars, cylindrical tubular samples, tensile bar specimens and pinion gears using Fe-2Cu-0-8C (FC-0208) and Fe-2Ni-0-8C (FN-0208) composition steel powders as per MPIF Standard 35. The development of improved mechanical properties and novel microstructures for microwave sintered copper and nickel steel PM parts has been compared with that obtained by a conventional sintering method.

EXPERIMENTAL PROCEDURE
Fabrication of ferrous PM parts and binder removal
In the present investigation, an admixed alloy powder of copper steel, Atomet AXD 3401 sourced from Quebec Metal Powders Limited, Canada, was used to fabricate the FC-0208 copper steel PM parts, namely modulus of rupture (MOR) bars, cylindrical tubular samples, tensile bar specimens and pinion gears.18 The fabrication details of the MOR bars and the pinion gears using Fe-2Cu-0-8C composition steel powder as well as the binder removal details has been reported in earlier papers.18,20

The tensile bar specimens and the cylindrical tubular samples were supplied in the unsintered or green condition by Keystone Powdered Metal Company, Inc. (St Marys, PA). The tensile bar specimens were fabricated from Fe-2Ni-0-8C steel powder mixed with 1% ethylene bistearamide (trade name Acrawax C) at a compaction pressure of 760 MPa. The cylindrical tubular samples were fabricated from FC-0208 and FN-0208 steel powders mixed with 1% ethylene bistearamide at a compaction pressure of 608 MPa. The tensile bar specimens and the cylindrical tubular samples were subsequently sintered at 750°C for 20 min in a conventional belt furnace using the reducing forming gas atmosphere (95%N2+5%H2 mixture).

The digital camera photographs of ferrous PM parts fabricated in the present investigation, employing FC-0208 copper steel and FN-0208 nickel steel powders, namely modulus of rupture bars, cylindrical tubular samples, tensile bar specimens and pinion gears using both microwave and conventional sintering techniques under identical conditions are shown in Fig. 1.

Microwave sintering of ferrous PM fabricated parts
Details of the commercial microwave oven (Amana Radargate model RC20SE) used to microwave sinter the copper and nickel steel PM parts in the present investigation were reported in earlier papers.18,20 Figure 2 shows a schematic diagram of the microwave sintering setup used, showing the cross-sectional details of the sintering package configuration employed. The microwave sintering of FC-0208 and FN-0208 PM parts was performed under identical conditions to those used in conventional sintering for comparison purposes.

The delubricated FC-0208 copper steel MOR bars were fabricated using both conventional and microwave sintering methods.20 Conventional sintering was carried out in an alumina tube furnace (Lindberg) fitted with a proportional integral derivative (PID) Eurotherm temperature controller. The sintering temperatures employed for MOR bars were 1140°C and 1260°C, and the samples were soaked for 20 min at the maximum temperature. Sintering was carried out in a reducing atmosphere of flowing forming gas consisting of a mixture of 95%N2+5%H2 with a dewpoint of −60°C.

Microwave sintering of MOR bars was carried out in a commercial Amana microwave oven having a maximum power rating of 2-0 kW and equipped with a multimode cavity for obtaining uniform heating of the parts. The microwave sintering experiments were carried out at 2.45 GHz using silicon carbide rods as the susceptor (preheater). In the present study, silicon carbide was used as a susceptor material for microwave sintering of different fabricated ferrous PM parts as it is known to couple efficiently with microwaves at room temperature. The susceptor basically helps in providing the uniform temperature distribution throughout the sample needed to obtain uniform sintering.

The delubricated FC-0208 copper steel and FN-0208 nickel steel cylindrical tubular samples were conventionally sintered at 1260°C for 20 min soak time while the delubricated FN-0208 nickel steel tensile bar specimens and the delubricated FC-0208 copper steel pinion gears,18 were conventionally sintered at 1140°C and 1260°C for 20 min soak time in a reducing gas atmosphere of flowing forming gas (95%N2+5%H2 mixture) at Keystone Powdered Metal Company, Inc.

The delubricated FC-0208 and FN-0208 cylindrical tubular samples were microwave sintered at 1260°C for 20 min soak time. The delubricated FN-0208 tensile bar specimens and the delubricated FC-0208 pinion gears were
microwave sintered at 1140°C and 1260°C for 20 min soak time.18 The microwave sintering of the PM parts was carried out in a reducing atmosphere of flowing forming gas having a dewpoint of -60°C using silicon carbide rods as the susceptor. The commercial Amana microwave oven was operated at 2.45 GHz. The microwave oven had a maximum power rating of 2.0 kW and was equipped with a multimode cavity for obtaining uniform heating of the parts.

The delubricated copper and nickel steel PM fabricated parts were placed individually one at a time in a recrystallised alumina boat placed inside the mullite tube at the centre of the microwave cavity, and the infrared pyrometer was focused at the centre of the part cross-section. The microwave cavity was kept cool by circulating cold water and compressed air during the experiment. The temperature of the part was controlled by manually changing the variac voltage with time and the voltage typically ranged from 140 to 170 V. The infrared pyrometer was integrated to a personal computer to acquire the temperature data as a function of time. The part was kept in the microwave cavity in a reducing atmosphere of flowing forming gas until it was cooled to near ambient temperature to prevent any oxidation or decarburisation occurring on the surface of the part by coming into contact with oxygen in the air.

Evaluation of mechanical properties of ferrous PM sintered parts

The FC-0208 and FN-0208 PM parts processed using both conventional and microwave sintering techniques were evaluated for sintered density and some of the typical mechanical properties. The sintered density of the ferrous PM parts was determined using the Archimedes principle as per MPIF Standard 42. The exact volume of the PM part was calculated using the liquid displacement method by employing distilled water. The sintered density, as well as the mechanical properties obtained represented an average value of readings for three to five samples for each of the different PM sintered parts studied in the present investigation, using different sintering temperatures and compositions.

The apparent hardness measurements of the as sintered modulus of rupture bars,20 cylindrical tubular samples and pinion gears,18 were determined as per MPIF Standard 43 by employing a Rockwell hardness tester (Series 2000, Wilson/Shore Instruments, Instron Corporation) using the B scale (HRB) and utilising a 1/16 inch (1.5875 mm) ball indenter and a load of 100 kg. The flexural strength of MOR bars was determined using the four point bending method by employing a universal testing machine (Instron, model 4206) integrated to a Gateway 2000 (crystal scan) Pentium and loaded with Instron Series IX software. The percentage elongation of the tensile bar specimens was determined using a universal testing machine (Tinus Olsen Super L) and an Epsilon Extensometer (model 3542-0100-020-ST) as per MPIF Standard 10.
The toughness parameter \( K \) measurements of the cylindrical tubular samples were carried out using the ductility test wherein the minimum load for failure, also known as the crushing strength, was determined by employing a universal testing machine (Tinus Olsen Super L). The toughness parameter of the cylindrical tubular samples, which is a measure of the ductility of the material, was calculated using the relation,

\[
K = \frac{P(D - t)}{4L^2}
\]

where \( P \) is crushing strength or minimum load for failure (N), \( L \) is length of the cylindrical tubular sample (m), \( D \) is outer diameter of the cylindrical tubular sample (m) and \( t \) is wall thickness of the cylindrical tubular sample (m).

Evaluation of microstructural and microcompositional analysis of PM sintered parts

The microstructural observations on the FC-0208 and FN-0208 cylindrical tubular samples sintered at 1260°C for 20 min soak time in a reducing atmosphere were made by employing an optical microscope (Olympus BX60M, Hitech Instruments) using the reflected light brightfield mode. The unetched samples processed by conventional and microwave sintering techniques under identical conditions were employed for comparing the microstructural development as well as the porosity distribution obtained for the two different heating methods.

The optical microstructures of the FN-0208 tensile bar specimens sintered at 1260°C for 20 min soak time in a reducing atmosphere were evaluated using an optical microscope (Nikon Epiphot 300). The polished and etched samples using both the microwave system and the conventional furnace were employed for obtaining the sintered microstructures and comparing the porosity distribution. The SEM fractographs of the FN-0208 tensile bar specimens were evaluated on the fractured surfaces of the microwave and conventionally processed samples sintered at 1140°C for 20 min soak time. The microstructures of the fractured surfaces were obtained after carrying out the elongation test using a scanning electron microscope (Philips, model XL-30 SEM).

The microcompositional analysis using high spatial resolution (HR) scanning transmission electron microscopy (STEM) was performed on FC-0208 copper steel samples processed by both the microwave and conventional sintering techniques under identical conditions. The STEM specimens were prepared by manual polishing of approximately 3 mm discs of the sintered FC-0208 samples to about 40 μm thick. The discs were then ion beam thinned at an angle of 4° using a Gatan precision ion polishing system.

The microstructural and microcompositional analysis was carried out using a VG HB603 FEGSTEM with a probe size of 14 nm (FWTM) and a beam current of 0.5 nA. The STEM used a windowless Si (Li) XEDS with a large detector solid angle of 0.3 sr. X-ray acquisition was carried out on a Oxford (Link) exil system, where the elemental windows were defined over the Kα lines of C, O, Al, Si, Cr, Mn, Fe, Ni and Cu. The elemental X-ray mapping had an acquisition time of 100 ms/pixel with a 128 × 128 pixels resolution. The experimental details and the microstructural and microcompositional analysis results on the FC-0208 samples have been reported in a recent publication.

**RESULTS AND DISCUSSION**

The comparison of sintered density and other mechanical properties obtained for the FC-0208 and FN-0208 composition steel PM fabricated parts processed using both the conventional and microwave sintering techniques under identical conditions, is shown in Table 1. It is clearly seen from the Table 1 that the sintered density values of both the conventional and microwave sintered parts were very close to each other for most of the fabricated parts. However, the sintered density of microwave processed parts was only slightly higher, by only a small percentage, than that of the conventionally sintered parts. This clearly suggests that microwave sintering resulted in good densification and dense microstructures for the microwave sintered PM parts, as do parts processed by conventional sintering.

The mechanical properties obtained for the microwave sintered parts, however, showed a marked improvement over those of the conventionally sintered parts. This consistent improvement in mechanical properties for the different microwave sintered PM parts investigated has been correlated primarily to the development of novel microstructures resulting in characteristic porosity distributions that yielded better mechanical properties. The new

| PM parts         | MPIF designation | Sintering conditions, °C* | Mechanical property | Conventional sintering | Microwave sintering | Percentage increase
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<td>rupture bars</td>
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* All were carried out for 20 min.

* Refers to percentage increase in the mechanical property value obtained for PM parts processed using microwave sintering as compared with conventional sintering under identical conditions.
3 Ductility test illustrating minimum load for failure of FC-0208 cylindrical tubular samples sintered at 1260°C for 20 min soak time in reducing atmosphere

findings with regard to the improved mechanical properties and the development of novel microstructures for the microwave sintered PM parts are discussed in detail in the present paper.

It is important to note from Table 1 that the FC-0208 copper steel MOR bars fabricated using microwave sintering showed consistently higher flexural strength and Rockwell hardness (HRB) values as compared with those of the conventionally sintered MOR bars for the different sintering temperatures employed. The improvement in flexural strength and Rockwell hardness values for microwave sintered parts has been specifically attributed to the evolution of a novel porosity distribution. The porosity distribution obtained typically consisted of small, rounded and uniformly distributed pores as opposed to large, angular and non-uniformly distributed pores observed for conventional sintering. This distinct feature of the porosity distribution has been reported for the microwave sintered MOR bars and used to explain the improvement in mechanical properties such as sintered density, Rockwell hardness and flexural strength, in comparison to those of the conventionally sintered PM parts.

The ductility test results obtained for the FC-0208 copper steel cylindrical tubular samples sintered at 1260°C for a soak time of 20 min using both microwave and conventional sintering techniques are shown in Fig. 3. The ductility test was used to determine the actual minimum load required for failure of the sintered cylindrical tubular samples. The typical crushing strength values determined for the microwave and conventionally sintered cylindrical tubular samples are shown in Fig. 3. The minimum load values obtained in the ductility test were used for calculating the toughness parameter of the cylindrical tubular samples. It is clear from Fig. 3 that not only was the minimum load for failure of the microwave sintered cylindrical tubular samples higher than that of the conventionally sintered samples but the manner in which both the microwave and conventional sintered samples failed by fracture was observed to have distinct characteristics. The microwave sintered samples fractured into two pieces only while the conventionally sintered samples fractured into four distinct pieces during the ductility test thereby indicating higher ductility related properties.

The sintered density, toughness parameter and Rockwell hardness (HRB) results obtained for the FC-0208 and FN-0208 cylindrical tubular samples fabricated using microwave and conventional sintering methods at a 1260°C for 20 min soak time are given in Table 1. It is evident from Table 1 that the both the toughness parameter and Rockwell hardness of the microwave sintered cylindrical tubular samples was found to be significantly higher in the ranges 7.7-34.3% and 2.0-34.4%, respectively, as compared with that of the conventionally sintered samples; the sintered density improvement was only in the range of 0.5-0.6%. This suggests that the improved mechanical properties obtained for the FC-0208 and FN-0208 steel composition cylindrical tubular samples can be directly correlated to the development of novel microstructures. The higher toughness parameter values observed for the microwave sintered samples can be directly correlated to the distinct porosity distribution obtained which lowered the stress concentration factor greatly resulting in failure by fracture at much higher loads in the ductility test as compared with that observed for the conventionally sintered samples.

The optical micrographs of the FC-0208 copper steel cylindrical tubular samples sintered at 1260°C for a 20 min soak time in a reducing atmosphere by microwave and conventional sintering techniques are shown in Fig. 4. Sintered samples in an unetched condition were employed for observing the typical porosity distributions obtained. The porosity distributions obtained clearly showed the presence of both interconnected and isolated pores having distinct characteristics: smooth and rounded pores with low stress concentration regions for the microwave sintered samples as opposed to the sharp, triangular and wedge shaped pores with high stress concentration regions for the conventionally sintered samples, as clearly seen from Fig. 4.

The percentage elongation values obtained for the FN-0208 nickel steel tensile bar specimens processed by both
microwave and conventional sintering at 1140°C and 1260°C for a 20 min soak time in a reducing atmosphere are given in Table 1. It is clear from Table 1 that the percentage elongation values for the microwave sintered samples are higher in the range of 7.5-26.7% while the sintered density values were higher only marginally by 0.3-0.4% as compared with those of the conventionally sintered samples for both the temperatures investigated. It is important to note that the percentage elongation values increased with an increase in sintering temperature for both conventional and microwave sintering.

The optical micrographs of the FN-0208 nickel steel tensile bar specimens sintered at 1260°C for a 20 min soak time in a reducing atmosphere using both microwave and conventional sintering techniques, are shown in Fig. 5. The polished and etched sintered samples were employed for obtaining the porosity distributions of the microstructures using ×250 magnification. It is clear from Fig. 5 that the overall microstructural porosity distribution of the microwave sintered sample showed finer and rounded pore characteristics as compared with the coarser and angular pore characteristics observed for the conventionally sintered sample, although the total porosity levels were more or less equivalent. The overall microstructure was also more homogeneous for the microwave sintered samples as compared with that of the conventionally sintered samples.

The SEM fractographs of the FN-0208 nickel steel tensile bar specimens sintered at 1140°C for a 20 min soak time in a reducing atmosphere using both microwave and conventional sintering techniques are shown in Fig. 6. The microstructures were obtained on the fractured surfaces using ×984 magnification after carrying out the elongation test on tensile bars. It is evident from Fig. 6 that the grain structure was much finer for the microwave sintered samples than that of the conventionally sintered samples. The higher percentage elongation values observed for microwave sintering can be explained clearly by correlating to the finer grain structure obtained.

The sintered density and Rockwell hardness (HRB) values obtained for FC-0208 copper steel pinion gears processed using microwave and conventional sintering techniques are given in Table 1. It is clear from Table 1 that both the sintered density and the Rockwell hardness (HRB) values were only slightly higher for the microwave sintered pinion gears than for the conventionally sintered samples processed under identical conditions. These results, showing only the marginal improvements in the mechanical properties of the pinion gears, can be correlated to the novel microstructures obtained for the microwave sintered pinion gear samples and these results were published earlier.14

The Cu and Fe X-ray maps obtained using high spatial resolution scanning transmission electron microscopy on the microwave sintered FC-0208 sample are shown in Fig. 7. These Cu and Fe X-ray maps are typical of the whole microstructure, although in some cases the Fe grains and the Cu precipitates were smaller than those shown in the Fig. 7. The conventionally sintered FC-0208 sample showed a microstructure similar to that of the microwave sintered sample for the majority of cases. However, for the conventionally sintered FC-0208 sample, there were some
7 Cu and Fe X-ray maps obtained using high spatial resolution scanning transmission electron microscopy of FC-0208 sample microwave sintered at 1260°C for 20 min soak time in reducing atmosphere. Distribution of Cu precipitates was found to be uniform throughout sample areas where there was a completely different microstructure observed. Such a typical one is shown in Fig. 8. The regions showing this type of completely different microstructures were not in abundance, therefore, it accounted for only a slight difference in the mechanical properties observed between the microwave and conventionally sintered samples, for instance those observed in the case of the pinion gears.26

In general, a more uniform distribution of the Cu phase was observed in the microwave sintered samples than in the conventionally sintered samples.26 The improved mechanical properties obtained for the microwave sintered samples can thus be attributed primarily to more uniform distribution of the alloying elements, which resulted in greater material homogeneity at the nano- and microlevels as revealed by the Cu and Fe X-ray maps using high spatial resolution scanning transmission electron microscopy.

CONCLUSIONS
In the present investigation, ferrous PM parts were fabricated from the FC-0208 and FN-0208 composition steel powders, and successfully sintered using both the microwave and conventional sintering techniques. Some of the important results obtained on the PM sintered parts can be summarized as follows.

1. The copper and nickel steel PM sintered parts produced by microwave sintering showed improved mechanical properties and novel microstructural development as compared with those obtained by conventional sintering.

2. The sintered density values obtained for microwave sintered parts exceeded slightly, by a small percentage, those of the conventionally sintered parts. This indicates good densification and dense microstructures can be realized in the microwave sintered PM parts as those attained by the conventional sintering method.

3. Microwave sintering resulted in higher flexural strength and Rockwell hardness (HRB) for FC-0208 modulus of rupture bars, higher toughness parameters and Rockwell hardness for FC-0208 and FN-0208 cylindrical tubular samples, higher percentage elongation for FN-0208 tensile bar specimens, and higher Rockwell hardness for FC-0208 pinion gears than in the conventionally sintered PM parts under identical conditions.

4. The optical micrographs of both the etched and unetched samples showed the development of novel sintered microstructures having distinct characteristics for the porosity distributions: smooth and rounded pores with low stress concentration regions for microwave sintering as opposed to sharp, triangular and wedge shaped pores with high stress concentration regions for conventional sintering.

5. The improved mechanical properties obtained for microwave sintered samples can be attributed primarily to a more uniform distribution of the alloying elements at the nano- and microlevels, as revealed by the Cu and Fe X-ray maps using high spatial resolution scanning transmission electron microscopy.

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