

UDK 622.785: 615.849: 691.73

## Microwave and Conventional Sintering of Premixed and Prealloyed Cu-12Sn Bronze

G. Sethi<sup>1</sup>, A. Upadhyaya<sup>1\*</sup>, D. Agrawal<sup>2</sup>

<sup>1</sup> Department of Materials and Metallurgical Engineering, Indian Institute of Technology, Kanpur 208016, UP, India

<sup>2</sup> Materials Research Institute, The Pennsylvania State University, University Park, PA 16802, USA

**Abstract:** *The aim of the present investigation is to study the sintering behavior of the Cu-12Sn bronze system in both, a microwave furnace as well as a conventional furnace. The powders prepared by premixed and prealloyed routes were sintered in the range of solid state, transient and supersolidus liquid phase sintering conditions. The comparative analysis is based on the sintered density, densification parameter, hardness, macrostructures and microstructures of the samples.*

**Keywords:** *Microwave Sintering; Conventional Sintering; Bronze.*

**Резюме:** *Цель данных исследований – изучение процесса спекания Cu-12Sn бронзы в микроволновой и конвенциональной печах. Исходные порошки, предварительно смешанные и легированные, спекали в условиях твердофазного, переходного и в присутствии жидкой фазы. Сравнительный анализ основан на изучении изменения плотности спеченных образцов, параметров плотности и твердости, макроструктуры и микроструктуры образцов.*

**Ключевые слова:** *Микроволновое спекание; конвенциональное спекание; бронза.*

**Садржај:** *Циљ ових истраживања је проучавање процеса синтеровања бронзе система Cu-12Sn у микроталасној и конвенционалној пећи. Полазни прахови, претходно мешани и легирани, синтеровани су у условима чврстофазног, прелазног и синтеровања у присуству течне фазе. Упоредна анализа је заснована на проучавању промене густине синтерованих узорака, параметара згушњавања и чврстоће, макроструктуре и микроструктуре узорака.*

**Кључне речи:** *Микроталасно синтеровање; конвенционално синтеровање; бронза.*

### 1. Introduction

Microwave processing of materials has been in context from 1950s and since then the materials processed have been continuously evolving [1]. The materials range from wood, food items to ceramics, semiconductors and now metals [2]. Initially, it was shown that bulk

---

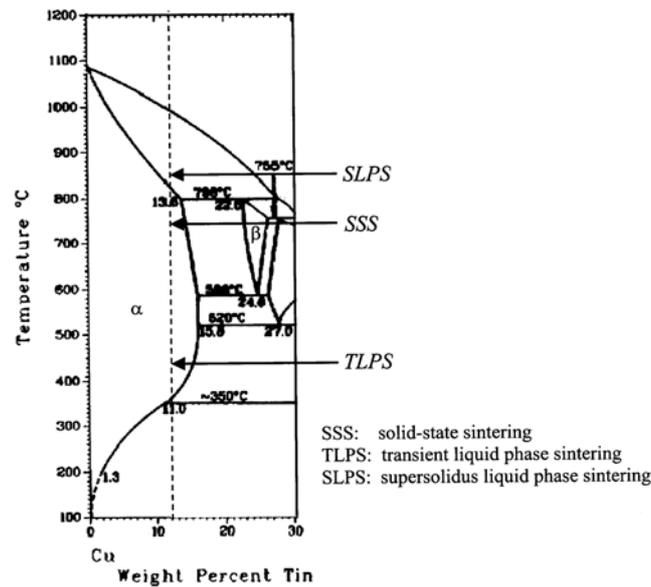
\* Corresponding author: anishu@iitk.ac.in

materials do not couple with microwaves because they reflect them. In general, they are not heated significantly by microwaves [1, 3]. Later, Walkiewicz *et al.* [4] reported modest heating of metals powders, ranging from 120°C for Mg to 768°C for Fe. Cheng [5] first showed that a combination of metal and hard materials could be sintered. He was effectively able to sinter WC with 6 and 12 % Co, which exhibited better mechanical properties, finer and uniform microstructure than the conventional parts. Porada *et al.* [6] were also able to sinter tungsten carbide-cobalt composites through normal sintering as well as reaction sintering through microwaves. Nishitani [7] made use of small amounts of electrically conducting powders like aluminum to show enhancement of heating rates of refractory ceramics, but no statement was made on microwave sintering of pure metals powders. Whittaker and Mingos [8] reported microwave induced synthesis of metal sulphides by highly exothermic reactions of metals powders with sulphur. All the above researchers stated heating of metal powders through microwaves, but did not clearly mention about sintering of pure metal powders. Roy *et al.* [2] showed experimentally for the first time, coupling of pure metals with microwaves in the form of powder. They had not only sintered Fe-Ni-C and Fe-Cu-C systems but powders of pure metals were also sintered using microwaves. Subsequently, microwave sintering of P/M steel was conducted [9-10]. Anklekar *et al.* [9] demonstrated higher sintered density, hardness and flexural strength in microwave sintered Fe-2Cu-0.8C (FC0208) steels as compared to conventional sintered. Porada [10] discussed the heating and sintering behavior of steel powders based on different material parameters such as homogeneity of alloying elements distribution and powder microstructure using different methods for adjusting the density, conductivity and microstructure of the steel powder compacts, independent of DC electrical conductivity.

Microwave heating is fundamentally different from conventional heating. The microwave heating involves energy conversion whereas conventional heating involves energy transfer. In the microwave process, the heat is generated internally within the material instead of originating from external sources, and hence there is an inverse heating profile. Microwave heating is much more uniform at a rapid rate resulting in reduction of processing time and energy consumption. Also rapid heating leads to finer microstructure enhancing the mechanical properties. Clark and Sutton [1] cited many other benefits of the process such as precise and controlled heating, environmentally friendly etc. Microwave heating is a very sensitive function of the material being processed and depends on several factors, such as sample size, its mass and geometry [11]. Though there have been attempts to explain microwave heating of metal powders, still there is not yet any consensus on a comprehensive theory to explain the mechanism [12].

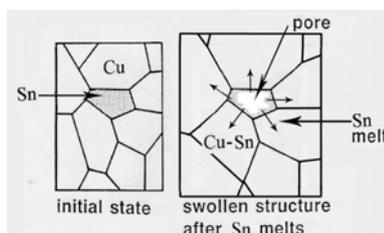
Till now most of the metallic systems studied using microwaves were complex and require very complicated chemistry, which has resulted in enormous confusion and skepticism [13]. Thus in order to understand the interaction of microwaves with metal powders and compare the response of a metal powder compact in a microwave *vis a vis* conventional sintering, experiments are required with a simpler system. Copper-tin based bronze is one such alloy system. Fig. 1 shows the phase diagram of the Cu-Sn system [14]. Powder metallurgy is an established processing technique for consolidating Cu-Sn bronzes [15-20]. Tailored bronze compositions can be prepared by both Cu-Sn premixed as well as prealloyed powder. Sintered bronzes are typically used for self-lubricating bearing and porous filters. Both these applications require controlled porosity in the sintered component. This is achieved by sintering the Cu-Sn alloys to a temperature above the melting point of Sn

(232°C), typically between 450-650°C. At these temperatures, Sn-melt diffuses into Cu and leaves behind porosity whose size and volume fraction corresponds to Sn powder size and content, respectively. This phenomenon is known as transient liquid phase sintering and requires premixed Cu-Sn powders (Fig. 2).

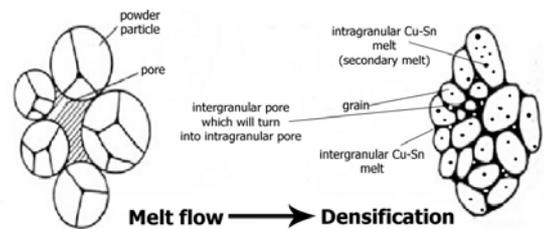


**Fig. 1** Phase diagram of Copper rich end of Cu-Sn system.

Nowadays, P/M bronzes are also being consolidated for structural applications by employing prealloyed powders. Use of prealloyed powder eliminates transient melt formation. The prealloyed powders are usually sintered in solid-state to relatively higher temperatures (up to 780°C) through supersolidus liquid phase sintering (SLPS) (Fig. 3).



**Fig. 2** Schematic illustration of the swelling in the Cu-Sn system associated with melting of tin.



**Fig. 3** Schematic illustration of the densification in the Cu-Sn system associated with Supersolidus liquid phase sintering.

From the above account, it is evident is that Cu-Sn alloys not only have a simpler chemistry, but also offer a unique opportunity to investigate the microstructural evolution in response to various sintering modes at a lower processing temperature (< 1000°C). Also at present, porosity is considered to be a major contributor in coupling of powdered metals with microwaves [12]. This study therefore aims at investigating the sintering response of Cu-Sn compacts as a function of powder condition, compaction pressure during microwave heating and comparing the same with conventional sintering.

## 2. Experimental Procedure

Table I summarizes the experimental parameters.

**Tab. I** Sintering parameters

Condition	Premixed and Prealloyed		
Compaction pressure, MPa	150, 300, 450, 600		
Sintering temperature and conditions	Sintering temperature, °C	Condition for Premixed	Condition for Prealloyed
	450	TLPS	SSS
	775	SSS	SSS
	830	SLPS	SLPS
Sintering time, min	30		

\* SSS: Solid State Sintering; TLPS: Transient Liquid Phase Sintering; SLPS: Supersolidus Liquid Phase Sintering.

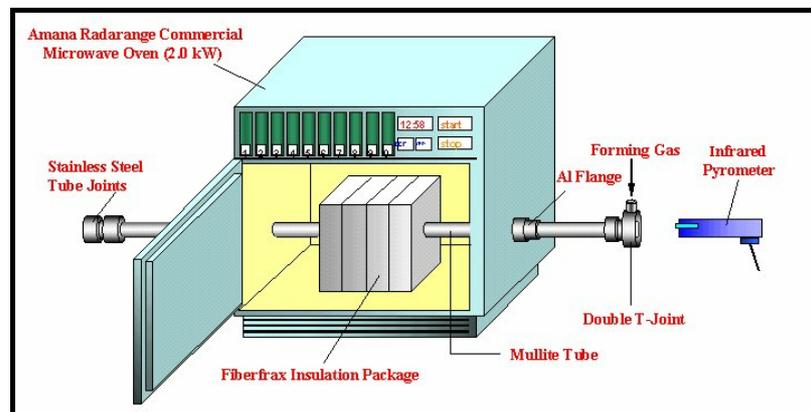
For the present study, a Cu-12 wt.% Sn composition was selected. The water-atomized Cu powder had an average size of 25  $\mu\text{m}$  whereas the Sn powder (gas atomized) was spherical in shape with an average size of 15  $\mu\text{m}$ . The details of powder characteristics are given elsewhere [21]. Both powders were mixed together in requisite proportion in a Turbula mixer (Type 2C, Bachofen AG, Switzerland) for about 30 min. The composition so prepared is referred to as ‘premixed’ Cu-12Sn. In the second set of experiments, gas-atomized ‘prealloyed’ Cu-12Sn powder was chosen for the investigation. The prealloyed Cu-12Sn powders had an average size of about 20  $\mu\text{m}$ . Both the premixed and prealloyed powders were pressed to cylindrical compacts (12.7 mm diameter) in a 20 tons capacity hydraulic press (Apex Ltd., UK).

### 2.1. Sintering

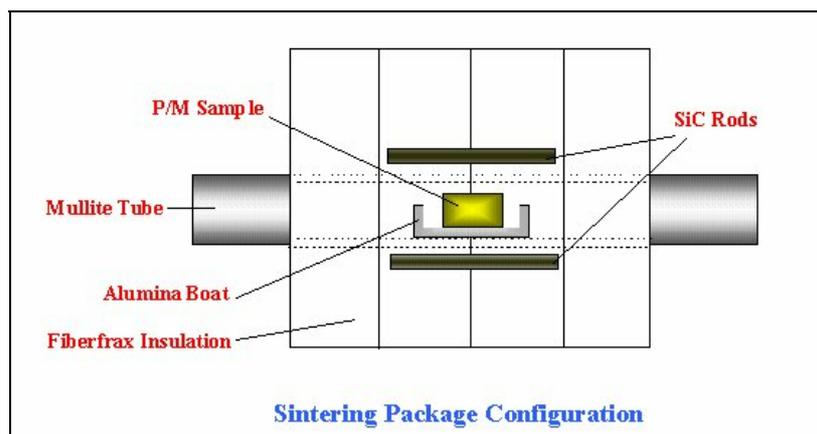
The as-pressed compacts were ‘conventionally’ sintered in a SiC-heated horizontal tubular furnace (Bysakh & Co., Kolkata, India) in forming gas (95N<sub>2</sub>-5H<sub>2</sub>) mixture. The furnace had a hot zone of about 75 mm. As described earlier, depending on the processing temperature, Cu-Sn alloys can be consolidated by solid-state, transient liquid phase or by supersolidus liquid phase sintering. Consequently, the Cu-12Sn alloys in the present study were sintered at 450, 775 and 830°C, respectively. In conventional sintering the compacts were heated at a constant heating rate of 5°C/min. A hold for 30 min was provided at the sintering temperature. The temperature was controlled to the accuracy of  $\pm 3^\circ\text{C}$ .

In another set of experiments, a 2 kW commercial microwave f/c (Amana Radarange, model RC/20SE, USA) with a 2.45 GHz multimode cavity was used to sinter the premixed and prealloyed Cu-12Sn alloys. The microwave f/c was modified to keep the external body temperature of the oven close to the ambient by circulating cold water through the copper tubes fixed at the top and the sides of the double jacketed oven by brazing. A mullite tube 31.8 mm in diameter and 914.5 mm in length was positioned at the center of the oven, by

drilling holes on the side-faces, with ends projecting on both sides. The schematic diagram of the microwave setup is shown in Figs 4a-b. As shown in Fig. 4b, a mullite based insulation package made from Fiberfrax™ boards was used to surround the mullite tube at the center of the cavity for containing the heat from dissipation during sintering. The design is made in such a way that it could be used both with and without the use of susceptor or secondary coupler, which are either SiC rods or graphite coating on the outer surface of the alumina boat. The susceptors usually couple very well with the microwaves and are used for initially raising the temperature of the bronze to about 200°C. Above this temperature, the Cu-12Sn compacts start to couple with the microwaves. For the present investigation, graphite coating on the alumina boat was used as the susceptor. Thermocouples cannot be used for temperature measurement in microwave furnaces. Instead, the temperature of the sample was monitored using an infrared pyrometer (Raytek, Marathon Series) with a circular crosswire focused on the sample cross-section. The pyrometer is emissivity based; therefore temperatures below 350°C could not be measured. The infrared pyrometer was coupled to a data acquisition and display software on a personal computer. Further details of the experimental setup of microwave sintering are described elsewhere [9].



**Fig. 4a** Schematic diagram of the microwave furnace used for the present study.



**Fig. 4b** Schematic diagram of the configuration of the sample during microwave sintering.

## 2.2. Post-Sintering Characterization

The sintered density was obtained by dimensional measurements. The degree of densification during sintering was measured using a densification parameter,  $\Psi$ , which is expressed as:

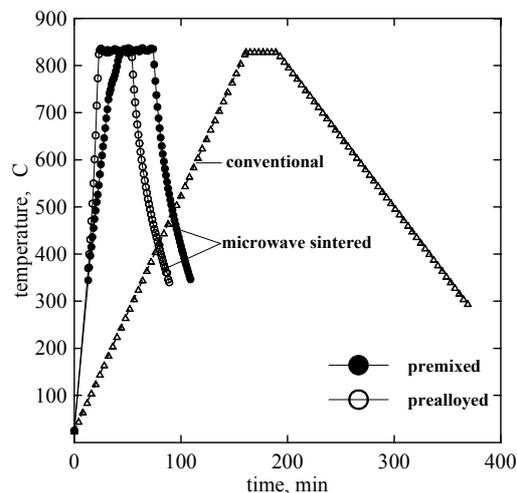
$$\Psi = \frac{\text{Sintered density} - \text{Green density}}{\text{Theoretical density} - \text{Green density}}$$

For hardness, Vickers macrohardness of the polished specimens was measured on a (Leco V-100-C1, USA) hardness tester. The machine was automatic and time for indentation was pre-programmed. The load of 500 g and indentation time of 10 s was maintained.

Hot mounting of the samples was done for the optical and scanning electron microstructural studies. The compacts were polished over 1/0, 2/0, 3/0 and 4/0 emery papers of the Lunn Major Unit of Struers followed by fine wet wheel polishing. Acidic  $\text{FeCl}_3$  was used as the etchant. Composition of the etchant was: 5 g  $\text{FeCl}_3$ , 50 ml  $\text{HCl}$  and 100 ml  $\text{H}_2\text{O}$ . Etching was done for 15-20 s. The microstructures of selected etched samples were observed using a JEOL, JSM – 840 A, Scanning Electron Microscope. Selected non-etched samples were observed in an optical microscope.

## 3. Results and Discussion

Fig. 5 compares the thermal profile of P/M bronze samples under microwave and conventional heating. Excluding the cooling time, it takes about 3 h to perform sintering in a conventional furnace, whereas in a microwave furnace the sintering time is reduced by more than 50 %. As the thermal mass is less during microwave heating, the microwave sintered samples cooled faster as compared to the conventionally sintered ones.



**Fig. 5** Thermal profile of conventional and microwave sintered Cu-12Sn alloy.

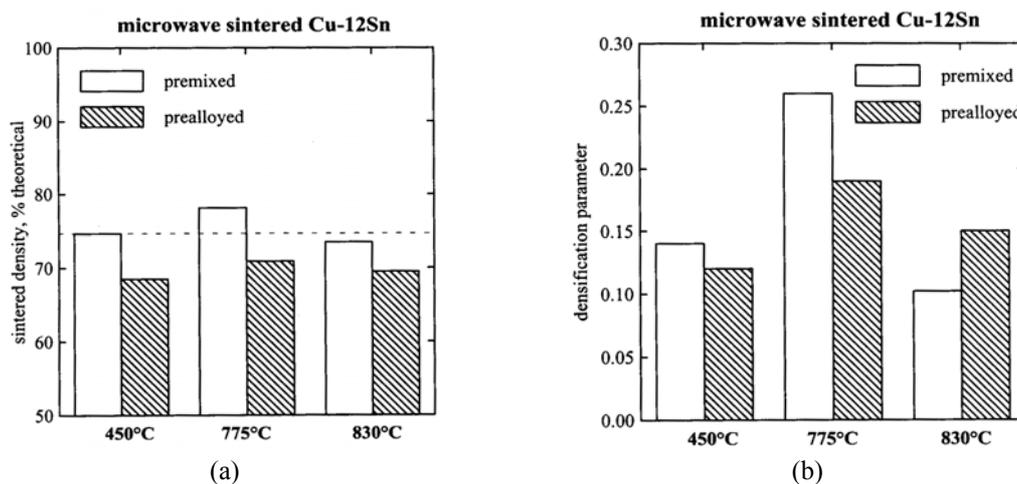
From Fig. 5, it is quite evident that both the premixed and prealloyed bronze samples couple with the microwave and do get heated up. Interestingly, the prealloyed samples start to heat up slightly faster than the premixed samples, which could possibly be attributed to different levels of coupling of the samples due to the ‘microwave effect’. Roy *et al.* [22] attributed the ‘microwave effect’ to anisothermal heating in the case where the system

comprises of more than two different components. The difference in the absorption factors of the powders causes the temperature gradient between the powders to be high which leads to enhanced reaction rates as was also observed in our case. Generally, the metal powders would be in an oxidized state. At a high temperature the reduction kinetics of the oxide layer would be lesser than the enhanced sintering kinetics. Hence anisothermal heating plays a major role in enhancing the sintering kinetics through the secondary system component or the oxide layer. In the case of premixed samples the mixed elements, Cu and Sn, would possibly cause 'anisothermal' heating leading to changes in the sintering kinetics. We observed high coalescence of particles in the case of premixed samples that could be explained by this theory. In the case of prealloyed samples the two elements are alloyed and hence the sintering kinetics would be different than the premixed microwave sintered bronze and would be much higher than conventionally sintered samples.

### 3.1. Effect of Sintering Temperature

#### 3.1.1. Sintered Density and Densification Parameter

Figs. 6a-b show the variation in the sintered density and densification parameter with temperature during microwave sintering. For all three sintering temperatures, the sintered density of premixed compacts is slightly higher than that of prealloyed ones. This can be attributed to the initially higher green density of premixed bronze as compared to the prealloyed sample at 150 MPa. Because of the solid-solution formation, the prealloyed bronze powders usually have lower compressibility as compared to the premixed ones. Sukanta [21] has shown that the green density of premixed bronze is about 68 % at 150 MPa, whereas that of prealloyed samples is around 60 %. Both the premixed and prealloyed microwave sintered samples show highest density at 775°C. During supersolidus liquid phase sintering (at 830°C), there is a density drop in both samples. For premixed compacts, this can be attributed to the formation of transient Sn melt and its diffusion in the Cu matrix [18-20].

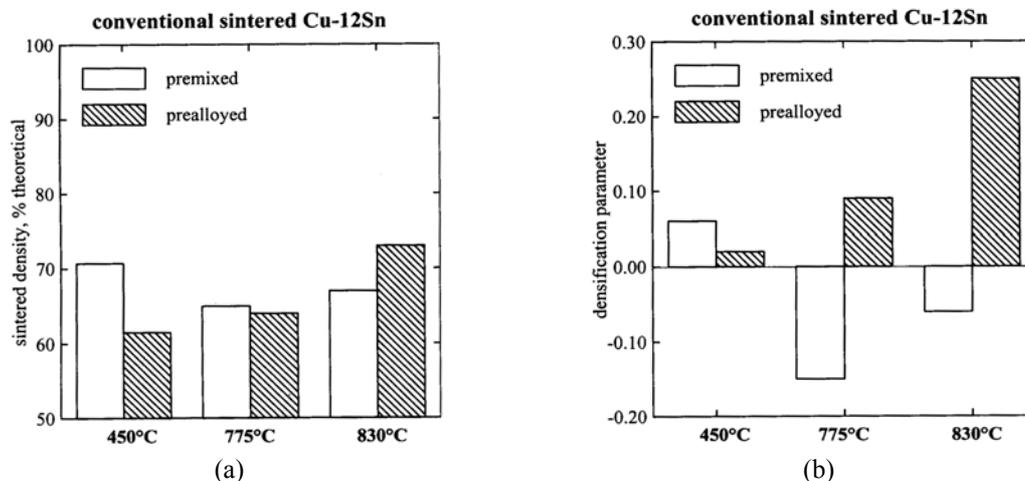


**Fig. 6** Effect of varying sintering temperature on the (a) density and (b) densification parameter of microwave sintered premixed and prealloyed Cu-12Sn alloy.

It is hypothesized that due to rapid heating in the microwave, there is very little time available for diffusion of Sn into Cu. Hence, most of the diffusion may be taking place during the sintering hold at 830°C. However, during supersolidus liquid phase sintering at 830°C,

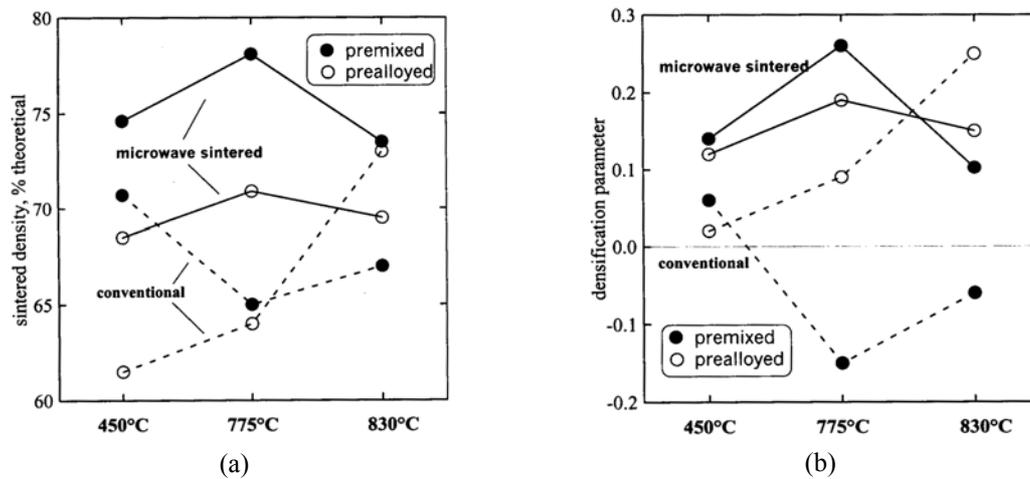
there will be a formation of Cu-Sn melts at the grain boundary and within the grains. Unlike the primary melt, this 'secondary' melt-formation will promote compact densification. Thus, during microwave sintering of premixed bronze at 830°C, compact density will depend on the nature of the melt and its volume fraction. In the case of prealloyed bronze, no primary (Sn) melt formation is expected at any stage, therefore, the compact is expected to show continued densification with increasing temperature. However, just like premixed bronze, the density of the microwave sintered prealloyed bronze is also lower at 830°C as compared to 775°C. This is possibly due to decoupling between microwaves and the bronze compact because of the formation of the secondary melt. The trend in densification parameter variation with temperature (Fig. 6b) is similar to that of the sintered density.

Figs. 7a-b compare the variation in the density and densification parameter with temperature for conventionally sintered premixed and prealloyed bronze. Unlike microwave sintering, both the sintered density and densification parameter of conventionally sintered prealloyed bronze increases with increasing temperature and is highest for SLPS conditions. For premixed bronze, the sintered density at 775 and 830°C is lower than that at 450°C (Fig. 7b). The densification parameters of premixed bronze at these two temperatures are negative giving rise to compact swelling. As discussed earlier, compact swelling is attributed to the diffusion of the primary or transient Sn-melt into Cu and therefore is a characteristic of premixed samples only. As indicated by the densification parameter, the compact swelling at 830°C is lower than that at 775°C. The decrease in compact swelling of premixed samples at 830°C is because of the supersolidus sintering conditions, which aids in densification, and thereby minimizes the swelling tendency.



**Fig. 7** Effect of varying sintering temperature on the (a) density and (b) densification parameter of conventionally sintered premixed and prealloyed Cu-12Sn alloy.

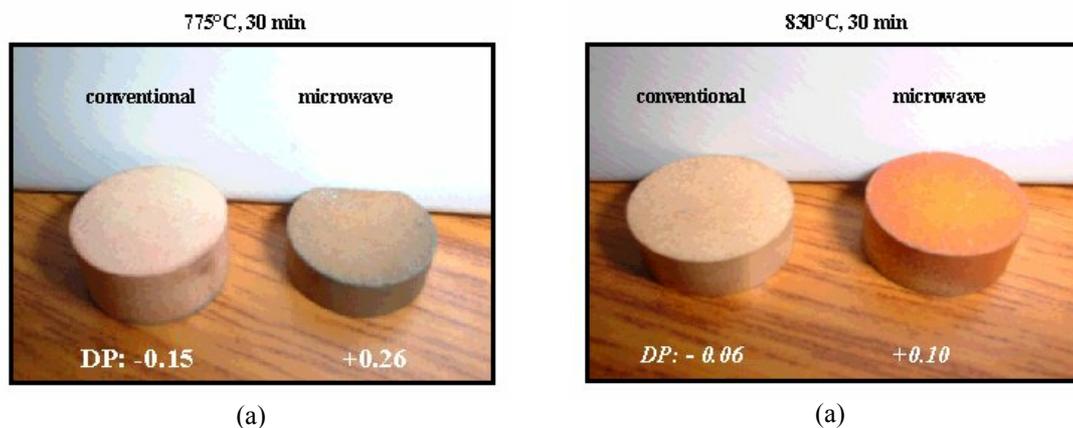
Figs. 8a-b summarize the effect of powder condition (premixed *vs* prealloyed), sintering mode (conventional *vs* microwave) and temperature on the sintered density and the densification parameter, respectively. It is interesting to note for 150 MPa green compacts, none of the microwave sintered samples show dilation, whereas the conventionally sintered premixed samples do so.



**Fig. 8** Effect of varying sintering temperature and composition preparation route on the (a) density and (b) densification parameter of conventionally and microwave sintered Cu-Sn alloy.

### 3.1.2. Macrostructural Studies

Figs. 9a-b show the photographs of premixed Cu-12Sn compacts microwave and conventionally sintered at 775 and 830°C, respectively. Note that the microwave compacts are slightly smaller than the conventionally sintered ones. This is supported by a negative densification parameter in the conventionally sintered premixed bronze.



**Fig. 9** Photographs of the premixed Cu-12Sn compacts, conventionally and microwave sintered at (a) 775°C and (b) 830°C for 30 min compacted at 150 MPa.

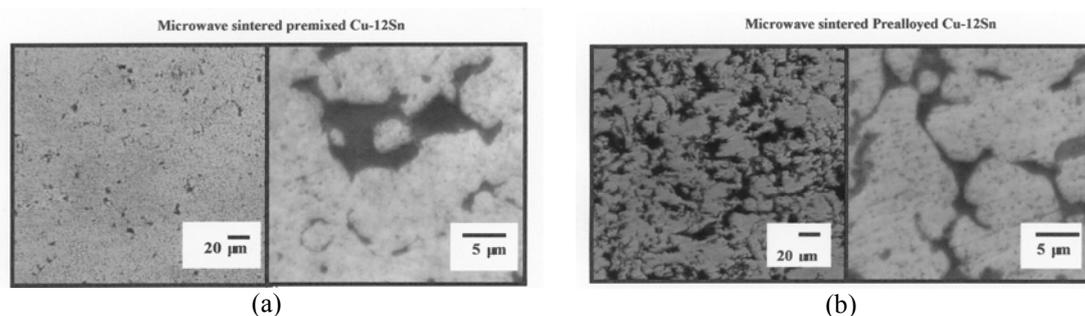
Fig. 10 shows the photograph of prealloyed Cu-12Sn sintered conventionally and in microwaves at 830°C for 30 min. Unlike the premixed samples, both samples show densification, as indicated by their positive densification parameter. The microwave sintered sample has lower density as compared to the conventionally sintered samples.



**Fig. 10** Photograph of the prealloyed Cu-12Sn compacts conventionally and microwave sintered at 830°C for 30 min compacted at 150 MPa.

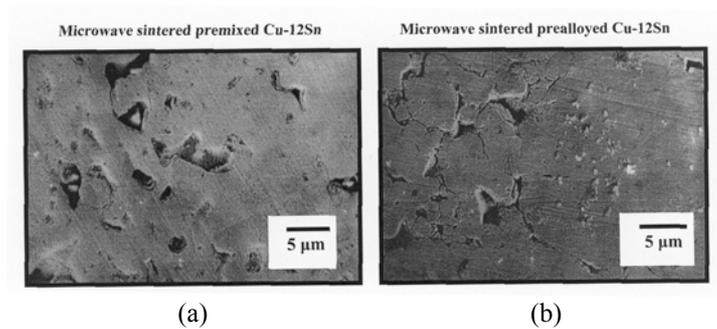
### 3.1.3. Microstructural Studies

Figs. 11-12 show the microstructures of microwave sintered premixed and prealloyed bronze compacted at 150 MPa and sintered at 775 and 830°C, respectively. At 775°C, premixed bronze has higher porosity than the prealloyed counterpart. As this is a transient liquid phase sintering condition for premixed bronze there is enhanced diffusion of Sn into Cu due to the ‘microwave effect’ leading to higher coalescence of particles. This can be seen from Fig. 11a, in which the grain boundaries are practically absent. The ‘microwave effect’ occurring due to anisothermal heating causes a large increase in diffusion kinetics of Sn in Cu and later on higher coalescence due to the presence of an oxide layer. In the case of prealloyed samples this is standard solid state sintering and hence no such effect is observed. Fig. 11b shows the clear presence of pores at the particle boundaries.



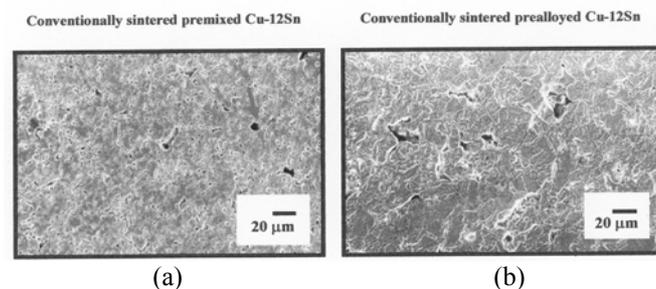
**Fig. 11** Optical photomicrographs of microwave sintered premixed (a) and prealloyed (b) Cu-12Sn alloy at 775°C compacted at 150 MPa.

At a higher sintering temperature, 830°C (Fig. 12), it is seen that the premixed sample has higher melt quantity as compared to the prealloyed ones. The higher melt content is due to formation of both primary as well as secondary intergranular melt during SLPS. Comparing Figs. 11 and 12, it is obvious that there is a higher quantity of melt in premixed bronze and for prealloyed bronze the microstructure has not changed much as even though the formation of intergranular melt could aid densification, the decoupling of microwaves due to the same factor prevents it.



**Fig. 12** SEM photomicrographs of microwave sintered premixed (a) and prealloyed (b) Cu-12Sn alloy supersolidus sintered at 830°C compacted at 150 MPa.

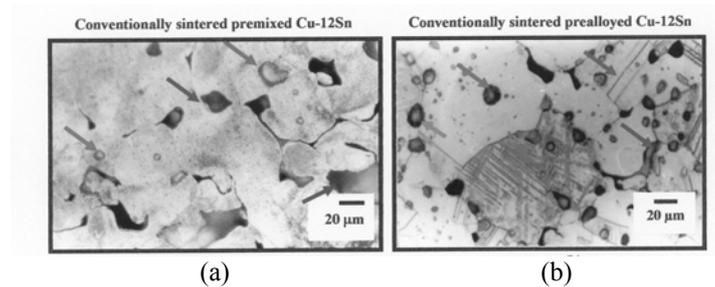
Figs. 13-14 show the microstructures of premixed and prealloyed bronze conventionally sintered and compacted at 150 MPa at 775°C and 830°C, respectively. In the case of conventional samples, the situation is very different from microwave sintered samples. At 775°C, uniform distribution of Sn melt is seen in the case of premixed bronze (Fig. 13a). The microstructure shows the presence of porosity at the grain boundary due to the diffusion of Sn into Cu. During solid-state sintering of prealloyed bronze, a homogenous microstructure was obtained which showed normal densification through rearrangement (Fig. 13b). Pores are of a uniform size because of the absence of melt formation. At 830°C, which is a condition for supersolidus liquid phase sintering, melt formation does take place, but the nature of the melt (which is ‘primary’ (pure Sn melt) and ‘secondary’ (Sn-Cu melt)) of the premixed sample would be different from that of the prealloyed one. Exactly the same thing is revealed by these micrographs.



**Fig. 13** Optical photo micrograph and SEM micrograph of conventionally sintered premixed (a) sample and SEM micrograph of prealloyed (b) sample sintered at 775°C compacted at 150 MPa.

The melt content is much higher in the premixed case (Fig. 14a) that can be attributed to the formation of both ‘primary’ (pure Sn melt) and ‘secondary’ (Sn-Cu melt) melt in premixed bronze. Further, intragranular melt globules are smaller in size than the intergranular melt. Tin powders are of different sizes. Here the kinetics has dominance over the thermodynamics due to small powder size. The smaller the particle size higher is the chemical reactivity. So, small tin particles melt at a faster rate and alloy very fast at the grain boundaries, in such a way that intergranular melts are bigger in size. However, tin inside the Sn-Cu alloyed grain can’t diffuse much faster. Intragranular melt globules are smaller in size because of this reason. In the case of the prealloyed sample, it is almost homogeneous. During heating the melt formation takes place at the same rate all over the structure. So, no tin rich phase should occur. Pores are naturally well distributed and finer compared to the premixed samples. Prealloyed samples have a higher tendency to form twins as compared to premixed

ones (Fig. 14b). Tin decreases the stacking fault energy of copper after alloying and thereby increases the twin band formation [21]. The micrographs confirm that the structure is a single phase one.

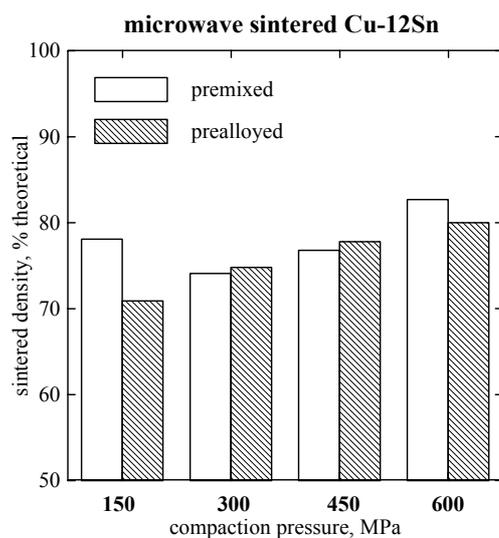


**Fig. 14** Optical photo micrographs of conventionally sintered premixed (a) sample and prealloyed (b) sample sintered at 830°C compacted at 150 MPa.

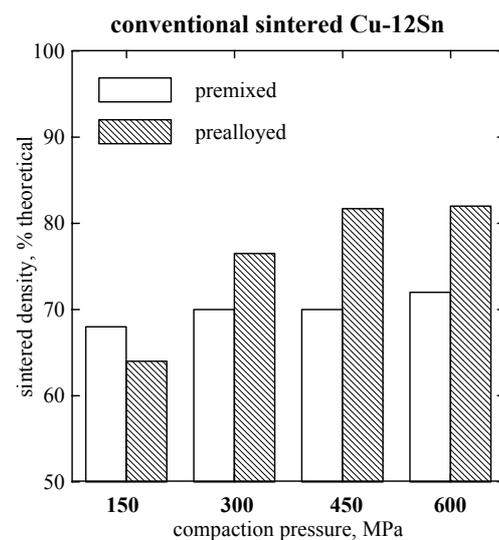
### 3.2. Effect of Compaction Pressure

#### 3.2.1. Sintered Density and Densification Parameter

Fig. 15 shows the effect of compaction pressure on the sintered density of premixed and prealloyed bronze microwave sintered at 775°C for 30 min. As the compaction pressure increases, the green density of Cu-12Sn alloy gradually increases. However, at higher pressures, there will be more variation in the green density of the compact from one region to another. From Fig. 15, the sintered density variation for both premixed and prealloyed samples is  $\pm 3\%$ , which is of the same order of magnitude as the variation in the green density. Hence, comparison of just the sintered density may give confusing results. It is therefore more appropriate to compare the trend in densification parameter with varying compaction pressure for microwave sintered premixed and prealloyed samples. For microwave sintered prealloyed samples the densification parameter slightly increases with increasing pressure (Fig. 17).



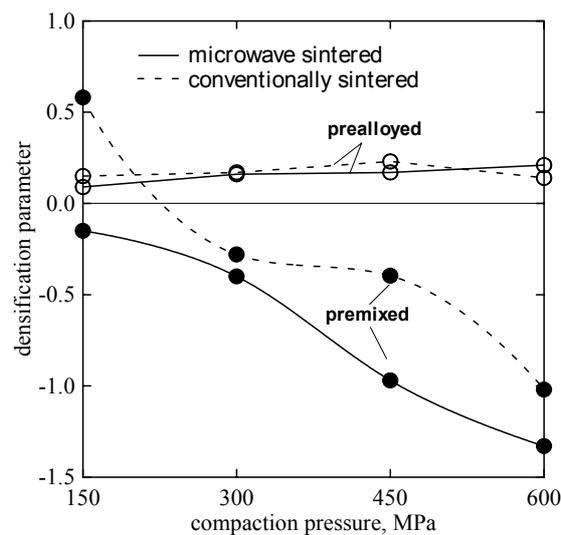
**Fig. 15** Effect of varying compaction pressure on the sintered density of premixed and prealloyed Cu-12Sn alloy microwave sintered at 775°C for 30 min.



**Fig. 16** Effect of varying compaction pressure on the sintered density of premixed and prealloyed Cu-12Sn alloy conventionally sintered at 775°C for 30 min.

Quite interestingly, all premixed microwave sintered samples show swelling, which increases with increasing compaction pressure, in accordance with the well-documented observations [19-20, 23]. A high compaction pressure results in lower porosity in green samples. Hence, the likelihood of Cu-Sn contact increases. During melt formation, this favors more effective diffusion of Sn-melt (primary liquid) into the Cu-matrix. This explains the increasing swelling in premixed bronze with pressure. In addition, as the pore fraction reduces, the melt induced capillary stress also decreases proportionately [24]. Thus, such compacts will have lower structural rigidity and will be more prone to swelling. Since microwave heating is more uniform as compared to conventional furnace heating, it results in homogeneous melt formation and causes greater swelling of premixed Cu-12Sn compacts as compared to their conventionally sintered counterparts.

Similar results are also observed in the density and densification parameter variation in conventionally sintered premixed bronze as shown in Figs. 16-17. The sintered density of conventionally sintered prealloyed Cu-12Sn increases with increasing compaction pressure (Fig. 16). However, this increase is due to the higher initial, as-pressed compact density. As evident from Fig. 17, there is no significant variation in the densification parameter of prealloyed bronze with pressure during conventional sintering. The premixed bronze on the other hand swells with increasing pressure.



**Fig. 17** Effect of varying compaction pressure on the (a) sintered density and (b) densification parameter of premixed and prealloyed Cu-12Sn alloy conventionally sintered at 775°C for 30 min.

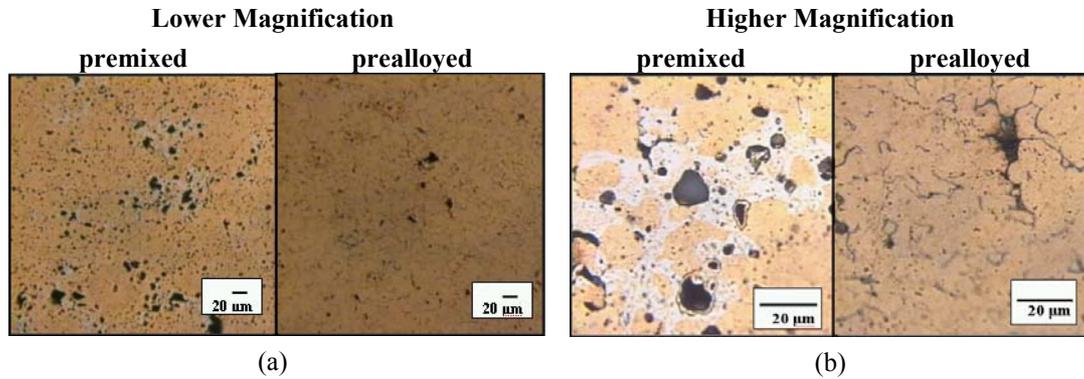
### 3.2.2. Microstructural Studies

Figs. 18-21 show micrographs of the microwave and conventionally sintered samples, with variation in green compaction pressure.

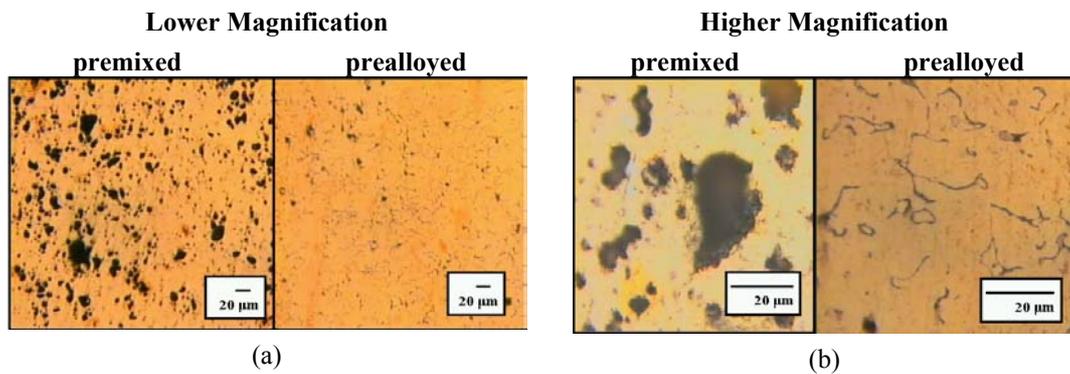
For the microwave sintered samples, the micrographs confirmed to the observations made for the sintered density. The variation of porosity is the same as before. At higher magnifications, we can clearly observe the tin rich areas near the pores. (Figs. 18b, 19b and 20b). The pores were uniformly distributed in the microstructure and rounded in morphology.

For samples conventionally sintered at 775°C, (Fig. 21) microstructures are not very different for premixed and prealloyed samples. Compaction pressure variation has very little effect on the microstructures. Pores are uniformly distributed through the microstructure. But

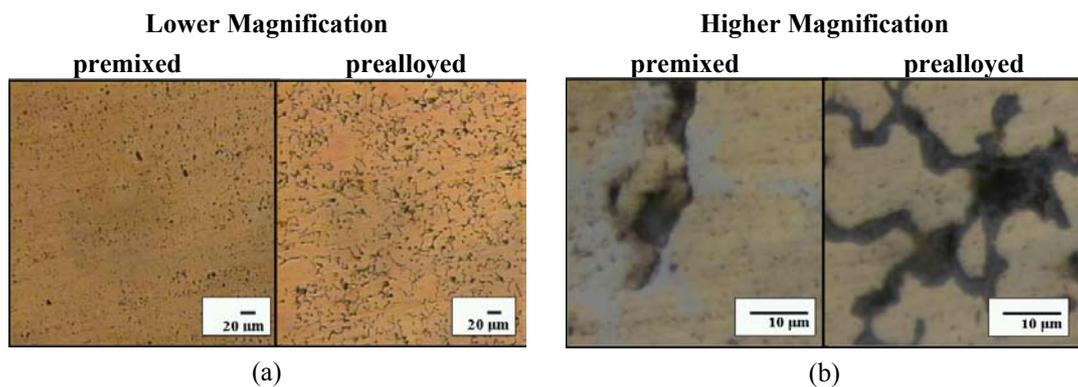
for premixed compacts, as the compaction pressure is increased, pore morphologies are changed as the amount and nature of Sn diffusion is dependent on the compaction pressure. The higher the compaction pressure the higher the contact area between Cu-Sn and hence diffusion of Sn into Cu would be enhanced.



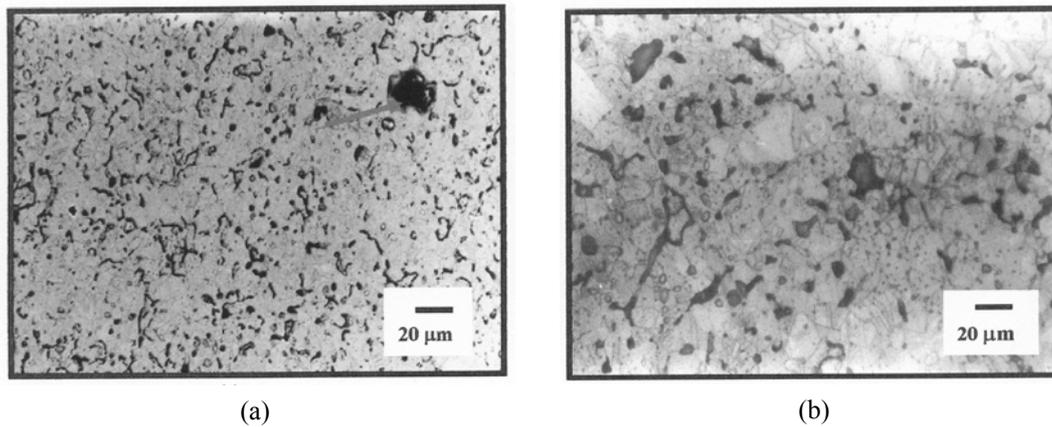
**Fig. 18** Split optical photomicrographs of microwave sintered samples, premixed and prealloyed bronze at 775°C, compacted at 300 MPa.



**Fig. 19** Optical photo micrographs of microwave sintered samples, premixed and prealloyed bronze at 775°C, compacted at 450 MPa.



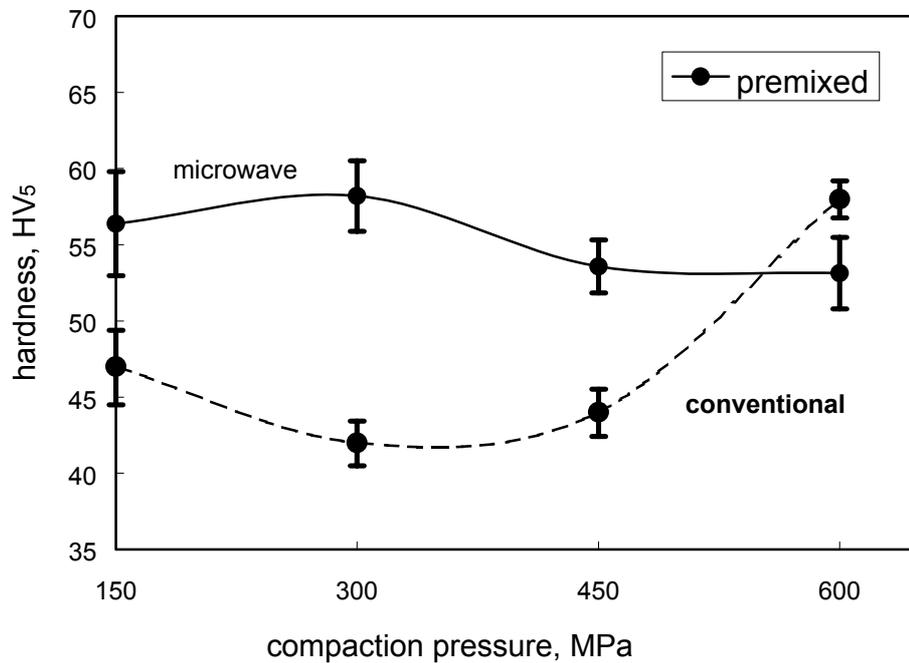
**Fig. 20** Optical photo micrographs of microwave sintered samples, premixed and prealloyed bronze at 775°C, compacted at 600 MPa.



**Fig. 21** Optical photo micrograph of conventionally sintered premixed (a) & prealloyed (b) sample sintered at 775°C.

### 3.2.3. Hardness

Fig. 22 shows the variation of Vickers macro-hardness with compaction pressure. In the case of premixed samples the microwave sintered samples have much higher hardness than the conventionally sintered samples except at 600 MPa. At such a high compaction pressure, as explained earlier, the enhanced diffusion and lowered capillary stress lead to lower structural rigidity. The higher hardness is attributed to the higher coalescence observed in the case of microwave premixed samples.



**Fig. 22** Effect of varying compaction pressure and composition preparation route on the hardness of Cu-12Sn alloy sintered in a conventional as well as a microwave furnace at 775°C for 30 min.

#### 4. Conclusions

For the first time, compacts prepared by using both premixed and prealloyed Cu-Sn bronze were sintered in a microwave furnace for temperatures corresponding to transient, solid-state, and supersolidus sintering. The conclusions of the results are as follows.

As compared to conventional sintering, bronze was microwave sintered in significantly less time. During conventional sintering, the premixed bronze compacts swell at 775 and 830°C, whereas no swelling occurs during microwave sintering of both premixed as well as prealloyed compacts compacted at 150 MPa. Hardness of the premixed microwave samples is higher than for the corresponding conventional premixed samples. Usually, the application of conventionally sintered premixed bronzes is restricted to making filters and bearings. However, the lack of swelling in premixed bronze, higher hardness during microwave sintering and uniform microstructure offer an opportunity of extending their use for structural applications as well.

The high coalescence observed in microwave premixed bronze is attributed to the 'microwave effect' because of anisothermal heating. In the case of prealloyed bronze, the degree of densification remains unaffected by varying porosity (green density) for both microwave as well as conventional sintering. The premixed bronze samples show an increasing tendency to swell as the initial (green) porosity is reduced by increasing the compaction pressure. The swelling is expressed for microwave sintered samples as compared to conventionally sintered ones. The microstructure in the case of microwave sintered samples is more uniform. Therefore, for fabricating bearing and filters with higher porosity it is recommended to compact the premixed bronze at higher pressure, followed by microwave sintering.

#### Acknowledgements

The authors thank Prof. R. Roy of Penn State, USA and Dr. G. Swaminathan of Bharat Heavy Electrical Ltd. (BHEL), Hyderabad, India for their helpful suggestions. The microwave sintering studies were partially supported by the grant from the Defense Advanced Research Projects Agency (DARPA) and Office of Naval Research (ONR). The research pertaining to conventional sintering was financially supported by the All India Council for Technical Education (AICTE), Department of Science and Technology (DST), Ministry of Human Resource and Development (MHRD), India.

#### References

1. D. E. Clark, W. H. Sutton, *Annu. Rev. Mater. Sci.*, **26** (1996) 299.
2. R. Roy, D. Agrawal, J. Cheng, S. Gedevisanishvili, *Nature*, **399** (1999) 668.
3. K. J. Rao, P. D. Ramesh, *J. Mater. Sci.*, **18** (4) (1995) 447.
4. J. W. Walkiewicz, G. Kazonich, S. L. McGill, *Miner. Metall. Proc.*, **5** (1988) 39.
5. J. P. Cheng, PhD Thesis, Wuhan University of Technology, China, 1991.
6. M. W. Porada, T. Gerdes, K. Rödiger, H. Kolaska, *Metal.*, **50** (1996) 744.
7. T. Nishitani, Method of sintering refractories and an apparatus therefore, US Patent No. 4147911, 1979.
8. A. G. Whittaker, D. M. Mingos, *J. Chem. Soc. Dal. Trans.*, (1995) 2073.

9. R. M. Ankelkar, D. K. Agrawal, R. Roy, *Powd. Metall.*, **44** (4) (2001) 355.
10. M. W. Porada, *Microw.: Theor. Appl. Mater. Proc. V, Ceram. T. III*, 2000, p. 459.
11. R. M. Hutcheon, M. S. De Jong, F. P. Adams, *Microw.: Theor. Appl. Mater. Proc. III, Ceram. T., Am. Ceram. Soc.*, **59** (1995) 215.
12. M. W. Porada, *Microw.: Theor. Appl. Mater. Proc. III, Ceram. T., Am. Ceram. Soc.*, **80** (1997) 153.
13. R. M. German, John Wiley, New York, NY, USA, 1996, p. 409,
14. ASM Metals Reference Book, American Society of Metals, Materials Park, OH, USA, 1981.
15. H. E. Hall, *Met. Alloys*, **10** (1939) 297.
16. D. F. Berry, *Powd. Metall.*, **15** (1972) 247.
17. G. Dowson, *Met. Powd. Rep.*, **2** (1984) 71.
18. A. B. Backensto, *Mod. Dev. Powd. Metall.*, P.U. Gummesson and D.A. Gustafson (eds.), MPIF, Princeton, NJ, USA, **19** (1988) 641-652.
19. K. Das, J. A. Bas, *Adv. Powd. Metall. Particul. Mater.*, MPIF, Princeton, NJ, USA, **6** (1992) 23-34.
20. N. N. Acharya, P. G. Mukunda, *Int. J. Powd, Metall.*, **31** (1) (1995) 63.
21. S. Ghosh, M. Tech. Thesis, Indian Institute of Technology, Kanpur, 2001.
22. R. Roy, D. K. Agrawal, J. P. Cheng, *Microw.: Theor. Appl. Mater. Proc. V, Ceram. T. III*, 2000, p. 459.
23. E. Deegan, A. D. Sarkar, *Metallurgia Met. Form*, **40** (8) (1973) 148.
24. J. Liu, A. L. Cardamone, R. M. German, *Powd. Metall.*, **44** (4) (2001) 317.

### Terminology

1. Primary Pores: As- pressed pores.
2. Secondary Pores: Pores created in the as-pressed compact during sintering by diffusion of Sn-melt in Cu.
3. Primary Melt: Sn melt.
4. Secondary melt: Cu-Sn melt created during SLPS.