

Enhancing Mechanical and Structural Properties of Pewter Alloy Using Microwave Sintering

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97%Sn 2%Cu 1%Sb (pewter) alloys were examined to determine the effect of green density, sintering time and sintering temperature on the mechanical and structural properties of the conventional and microwave sintered compacts. Two compaction loads; 30kN and 40kN were used to produce the samples with different green densities. Eight different time-temperature combinations were used for each heat treatment. Samples with a higher green density resulted in a higher sintered density and higher hardness. Longer sintering time and higher sintering temperatures resulted in higher densities, larger grain size and higher hardness for both sintering methods. However, the microwave sintered samples in general have finer microstructures, higher densities and higher hardness compared to the conventional sintered samples in a much shorter duration. Better mechanical and structural properties were achieved by microwave sintering in 15 minutes compared to conventional sintering which took 120 minutes.

Keywords: Microwave sintering, conventional sintering, pewter, mechanical and structural properties

1. Introduction

Microwave sintering is a method of heating that involves energy conversion which is different from the conventional sintering that concerns energy transfer. In microwave sintering, the heat is generated internally within the material instead of originating from external sources. In the process of microwave heating, the materials absorb microwave energy themselves and then transform it into heat within the sample volume [1]. The energy is directly transferred to the material through the interaction of electromagnetic waves with molecules leading to heating [2]. Microwave sintering is much more uniform at a higher heating rate. This results in a reduction of processing time and energy consumption.

Microwave heating is a function of the material being processed, and there is almost 100% conversion of electromagnetic

energy into heat, largely within the sample itself, unlike conventional heating where there are significant thermal energy losses [3]. While it is well recognized that bulk metals are opaque to microwaves and good reflectors, metallic materials in powder form are very good absorbers of microwaves and can be heated very rapidly since unsintered alloys will couple in a microwave field very efficiently and effectively to produce highly sintered bodies [4].

Pewter has been traditionally produced by the casting process where tin, copper and antimony are melted and mixed in the liquid phase to form the pewter alloy. This does consume a tremendous amount of energy, cost and time for the furnace.

This paper investigates the possibilities of modern pewter production through a powder metallurgy process and

extends this by exploring the possibilities of implementing microwave sintering as a substitute for conventional sintering.

2. Experimental method

2.1 Mixing of powder

Tin powder with 99.5% purity (-100 mesh), copper powder with 99% purity ($<75\mu\text{m}$) and antimony powder with 99.5% purity (-100 mesh) were used to prepare samples of 97%wt Sn 2%wt Cu 1%wt Sb powder. These powders were weighed accordingly and placed into their respective containers which were then evacuated in a glove box to remove oxygen up to about 40 ppm. They were then mixed using a roller mixer (ABB:ABS 100) for about 12 hours at a frequency of 40Hz.

2.2 Preparation of green compacts

Sixteen samples from 80g of powder were prepared from the same die to produce samples with a cross section of 10.1 mm in width and 30.8 mm in length with the height of 42mm in average. Two different pressing pressures were used to prepare samples for this study. Samples were compacted using a 30kN load and a 40kN load in a 10 ton Hydraulic Floor Press Machine (D2003K) with a holding time of 5 minutes. Between these limits, samples were defect-free and had sufficient green strength for handling.

2.3 Sintering

8 samples were sintered using the vacuum furnace and the other 8 sintered using the microwave furnace with varying experimental conditions at two different temperatures (160°C and 220°C) and different durations.

A conventional furnace with cavity size of 5cm x 110cm under vacuum condition was used. A heating rate of 6°C/min was maintained for the conventional sintering. The vacuum pressure was always allowed to reach 10^{-6} MPa before sintering.

A Panasonic Thermwave Mod.111 multimode microwave system (1.3kW,

2.45GHz, 47cm x 61cm x 64 cm) with water cooling system designed for the high temperature processing of materials in a laboratory or small-scale industrial manufacturing was used for this study. The green compacts were placed in a cylindrical thermal pod made from ceramic fibre.

The sample was sintered with graphite pellets which were used as susceptors to ensure two-directional sintering process took place and that excessive heat and energy did not build up in the system. It helps to give a better control of temperature at the low sintering temperatures used.

The crucible was filled with argon gas prior to sintering and maintained a flow rate of 50mL/min during sintering. The power dial at the controller was adjusted to 50% input energy level and the microwave unit was set to 70% output power level to allow high and uniform heating rates of 15°C/min.

2.4 Mechanical Analysis

The density of as-pressed pellets was calculated from the sample mass and volume. Meanwhile, a liquid displacement method, Archimedes' technique, was used to determine the density of the sintered samples.

The hardness of the sintered samples was measured using the Vickers Microhardness Tester (LM 700) with a 25 kg test load and a dwell time of 15 seconds to give a micro-indentation.

2.6 Microstructural Analysis

Scanning Electron Microscopy (S-4000 Hitachi) was used to generate digital images from the specimens. The SEM images were also used to produce a virtual elemental map of a sample's surface. X-Ray Diffraction (XRD) was performed using Philips X'PERT System. The patterns were used to characterize the sintered samples and to observe if any changes in phases occurred.

3. Results and Discussion

3.1 Mechanical Properties

The green compacts pressed at 30kN load had an average green density of 80.5% while those pressed at 40kN load had an average green density of 84.4%. Higher green densities produced samples with higher bulk density and less porosity.

Table 1 clearly shows that by increasing the sintering time from 60 to 120 minutes for the same compaction load, the bulk density had increased by about 3% for the conventional sintering. However, for the microwave sintering, the bulk density had increased by 1 to 2% when the sintering time had increased from 15 to 30 minutes under the same compaction load.

Both microwave sintering and conventional sintering produced higher density samples when the sintering temperature was increased from 160°C to 220°C. Nevertheless, the densities for microwave sintered samples at 160°C were still higher when compared to conventional sintering at 220°C. By increasing the sintering time from 60 to 120 minutes for the conventional sintering at 220°C, the bulk

density had increased by about 5 to 7%. However, for the microwave sintering at 220°C, the sintering time did not have a significant impact on the increase in density.

Microwave sintering had produced samples with higher hardness values compared to conventional sintering even at lower temperature (160°C). The hardness value for microwave sintering was generally about 25 to 28% higher than conventional sintering. The conventional sintering had only an average increase of about 12% in hardness values under varying conditions while microwave sintering had an increase of about 13 to 30% in hardness values under varying conditions.

As the compaction load, sintering temperature and sintering time increased, higher hardness were achieved. This is a result of diffusion of Cu into Sn which was clearly observed from X-ray maps shown in Figure 2. Furthermore, XRD data shows that the degree of Cu diffusion into Sn increases as sintering time and temperature increases. Moreover, microwave sintering has further enhanced the diffusion process compared with conventional sintering.

Table 1: Summary of Sintered Samples

Sample Name	Compaction Load	Sintering Temperature	Sintering Time	$\rho(\text{sintered})$ (g/cm ³)	%Theoretical density	Hardness (HV)	Grain Size(μm)
97CS1	30kN	160°C	60 min	6.1	83.29	12.92	23
97CS2	30kN	160°C	120 min	6.33	86.52	13.98	25
97CS3	30kN	220°C	60 min	6.53	89.25	14.24	26
97CS4	30kN	220°C	120 min	6.92	94.52	17.18	27
97CS5	40kN	160°C	60 min	6.19	84.62	14.88	23
97CS6	40kN	160°C	120 min	6.41	87.52	15.96	26
97CS7	40kN	220°C	60 min	6.59	90.06	16.92	27
97CS8	40kN	220°C	120 min	7.18	98.14	19.14	29
97MW1	30kN	160°C	15 min	6.78	92.63	17.32	16
97MW2	30kN	160°C	30 min	6.83	93.35	17.86	17
97MW3	30kN	220°C	15 min	6.88	94.00	20.16	20
97MW4	30kN	220°C	30min	6.96	95.02	22.10	24
97MW5	40kN	160°C	15 min	6.74	92.13	19.70	16
97MW6	40kN	160°C	30 min	6.89	94.11	23.30	18
97MW7	40kN	220°C	15 min	7.11	97.15	23.72	22
97MW8	40kN	220°C	30min	7.22	98.57	23.96	25

3.2 Structural Properties

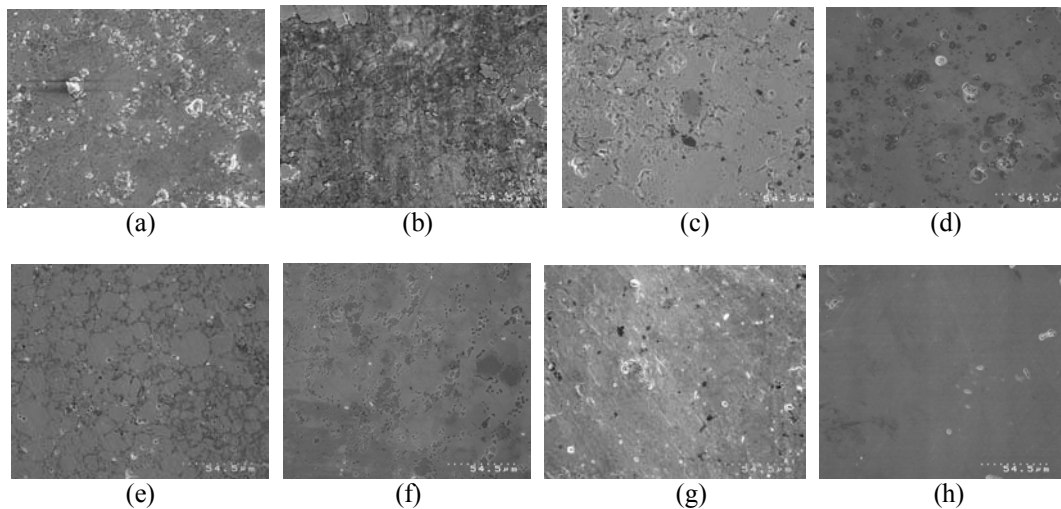


Figure 1: SEM images of: (a) CS3 (30kN, 220°C, 60min) (b) CS4 (30kN, 220°C, 120min) (c) CS7 (40kN, 220°C, 60min) (d) CS8 (40kN, 220°C, 120min) (e) MW3 (30kN, 220°C, 15min) (f) MW4 (30kN, 220°C, 30min) (g) MW7 (40kN, 220°C, 15min) (h) MW8 (40kN, 220°C, 30min)

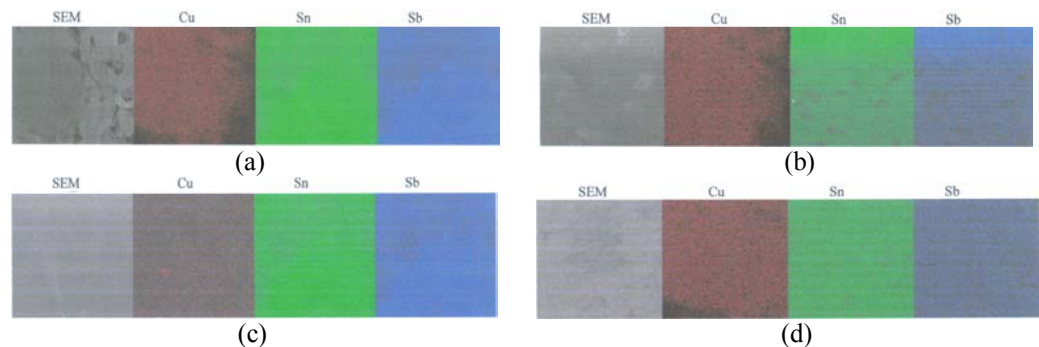


Figure 2: X-Ray map of: (a) CS4 (30kN, 220°C, 120min) (b) CS8 (40kN, 220°C, 120min) (c) MW4 (30kN, 220°C, 30min) (d) MW7 (40kN, 220°C, 15min)

The samples with lower green densities, when conventionally sintered, had somewhat more porosity and larger sized pores. The irregular shaped pores were scattered as shown in Figure 1. The conventional sintered samples with higher green densities had less porosity and smaller sized pores. As the sintering temperature increased, the porosity decreased for both compaction loads. Doubling the sintering time from 60 minutes to 120 minutes, increased the density by 3% and 5% after sintering at 160°C and 220°C respectively.

The microwave sintered samples with lower green densities produced fewer larger sized pores when sintered at 160°C compared with conventional sintering.

However, when microwave sintered at 220°C, the size and quantity of pores significantly decreased for both compaction loads.

Microwave sintering produces samples with fewer pores and more uniformly distributed porosity compared with conventional sintering, even at a lower temperature. Moreover, the pores were more regularly shaped. As the sintering temperature increased from 160°C to 220°C, the sample appeared to have minimal porosity and to have almost achieved full density; 97.15% and 98.57% at 15 and 30 minutes of sintering time respectively.

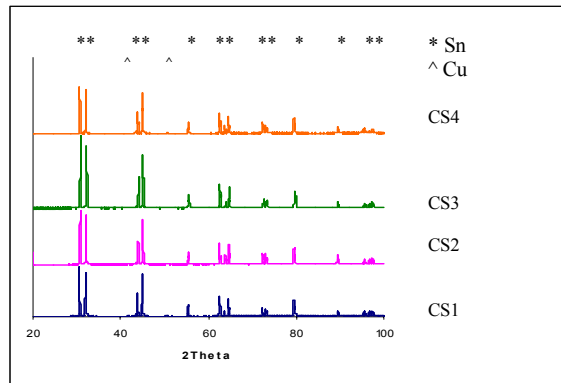


Figure 3: XRD peaks for CS at 160°C

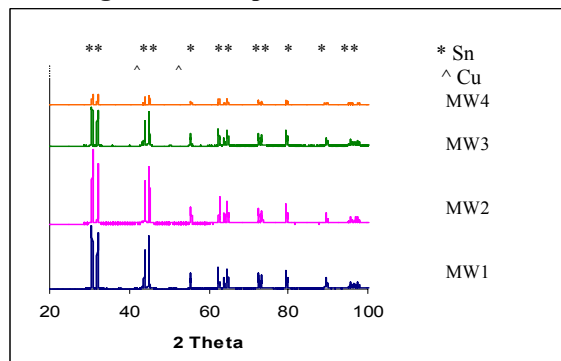


Figure 5: XRD peaks for MW at 160°C

CS: Conventional Sintering
 MW: Microwave Sintering

X-Ray analysis showed that with increasing sintering time and temperature, Cu and Sb peaks gradually broaden and eventually disappear. Sn peaks were broadened and their intensities increased and decreased accordingly. Sb atoms were shown to have partially diffused into the Sn lattice early on during the blending process and were completely diffused later during sintering. This explains why Sb peaks were not visible at all. Meanwhile Cu atoms gradually diffused into the Sn lattice and formed a Sn based solid solution with tetragonal structure.

4. Conclusion

Microwave sintering produced better mechanical and structural properties at lower processing temperature and with shorter processing time. Higher green strength, longer sintering time and higher sintering temperature resulted in improved densities, porosities and hardness values for both conventional and microwave sintering. Microwave sintering gave finer microstructure,

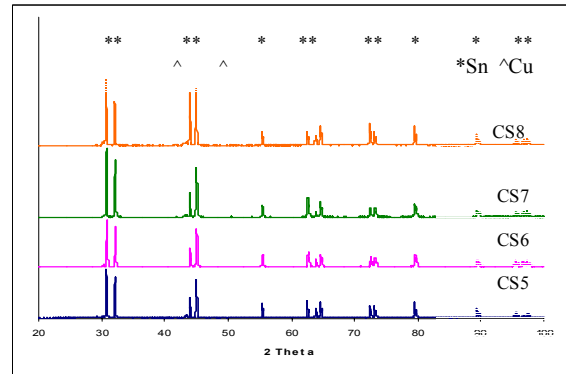


Figure 4: XRD peaks for CS at 220°C

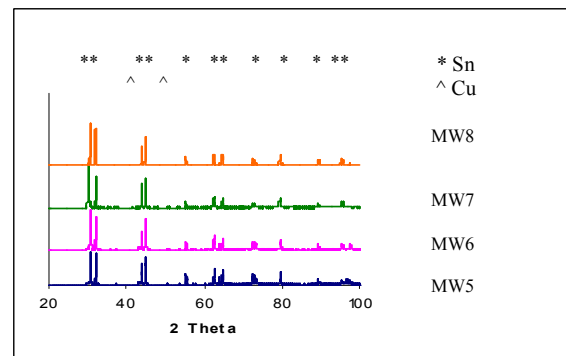


Figure 6: XRD peaks for MW at 220°C

better diffusion of Cu, improved mechanical and structural properties in 15 minutes compared to 120 minutes in conventional sintering.

References

- [1] M. Sorescu, L. Diamandescu, R. Peelamedu, R. Roy and A. Daly and Z. Bruno, "Evidence for microwave-induced recrystallization in NiZn ferrites", *Materials Chemistry and Physics*, Volume 101, pp 410-414, 15 February 2007.
- [2] T. Ebadzadeh and M. Valefi, "Microwave -assisted sintering of zircon", *Journal of Alloys and Compounds*, In Press, 11 February 2007.
- [3] D.K. Agrawal, "Microwave processing of ceramics", *Current Opinion in Solid State and Materials Science*, Volume 3, Issue 5, pp 480-485, October 1998.
- [4] D.K. Agrawal, *Microwave Sintering Developments Spur Emergence of New Materials & Technologies*, Materials Research Inst., Penn State University, 7 June 2005.