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TENSILE BEHAVIOR OF TIN SINTERED USING MICROWAVES AND RADIANT HEATING

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ABSTRACT

Results of open literature search indicates that no information is available on the tensile properties of pure tin processed using powder metallurgy (PM) route which is widely used as the principal element in electronic solders. Accordingly, in the present study, tensile properties of pure tin were assessed in microwave sintered and radiant heat sintered conditions following hot extrusion. Room temperature tensile properties characterization revealed that microwave sintered and extruded samples exhibited better combination of 0.2% yield strength, ultimate tensile strength, failure strain and work of fracture than conventionally sintered and extruded samples.

Keywords: Pure Tin, Microwave Sintering, Tensile Properties.

1. INTRODUCTION

Tin is one of the earliest metals known to mankind and was used in bronze implements as early as 3500 BC due to its excellent hardening effect on copper [1]. China, Malaysia, Indonesia, Thailand, Bolivia, Brazil, Nigeria, United Kingdom and Australia are the major producer of tin ore [2]. Tin is mainly used in electronic/electrical soldering [3], food canning [4], bearing materials, wires and window glasses [5] worldwide. A survey report published online on 19 October, 2006 stated that " Global tin consumption is expected to increase by more than 10% this year and the first half of 2006 shows a stunning year-on-year growth rate of 18.6% and this increment is as high as 26.8% in Asia" [6]. One third of the tin produced globally is being used as solder materials in electronic packaging industries and its demand is gradually increasing with the rising demand of lead-free solders due to environmental concerns in recent years. Non-toxicity and high corrosion resistance also made it one of the most suitable candidates for food canning industries.

Powder metallurgy (PM) is one of the most common processing techniques in producing high performance metallic materials for various applications [7, 8]. Sintering in powder metallurgy process plays a major role in realizing the end properties of the metallic materials [9] by improving bonding between the powders and minimizing porosity. Sintering can be done by traditional methods of heating such as radiant heating [8, 9] or by the more recently introduced method of using microwaves [10–14]. In a typical resistance heating furnace, the direction of heating is from outside to inside of the powder compact, while for microwaves the direction of heating is from inside to outside of the powder compact. The former results in the poor microstructural characteristics of the core of the powder compact while the latter results in the poor microstructural characteristics of the surface [15]. These drawbacks can be minimized using two-directional rapid microwave sintering [16].

Powder metallurgy processing route has been used for the development of tin containing alloys for different purposes like reducing the sintering temperature of iron powder [5, 17], copper/tin in frangible bullets [18] and Cu/Sn-SiC MMCs [19]. However, the result of open literature search indicates that no research attempt is made so far to process tin using the technique of powder metallurgy and particularly adopting microwave sintering process. Accordingly, in the present study, pure tin was synthesized using two-directional rapid microwave sintering and radiant heat sintering assisted powder metallurgy route followed by hot extrusion. Room temperature tensile characterization was done on extruded samples which was the main focus of this study.

2. EXPERIMENTAL PROCEDURE

2.1 Material

In the present study, pure tin powder with -325 mesh size (less than 44 micron) of 99.9% purity (supplied by NOAH Technologies Corporation, Texas, USA) was used in PM process.

2.2 Primary Processing

The powder metallurgy technique was used to synthesize pure tin in this study. Preweighed metal powder was compacted to 40mm height and 36 mm diameter billets by hydraulic compaction using a 150 tonne press. The billets were coated with colloidal graphite and sintered using an innovative microwave assisted hybrid sintering technique for 7 min to 226 °C in a 900 W, 2.45 GHz SHARP microwave oven using SiC as the microwave susceptor material (see Fig. 1) [16, 20]. SiC powder (contained within a microwave transparent ceramic crucible) absorbs microwave energy readily at room temperature and is heated up quickly, providing the radiant heat to heat the billets externally while the compacted billets absorbs microwaves and are heated from within. This hybrid heating method results in a more uniform temperature gradient within the billets and circumvents the disadvantage of heating using either radiant heating or microwaves only. In order to compare the results with that of radiant heat sintering, tin billets processed in identical way were radiant heat sintered at 156 °C (0.85T_m) for 2h in argon atmosphere.

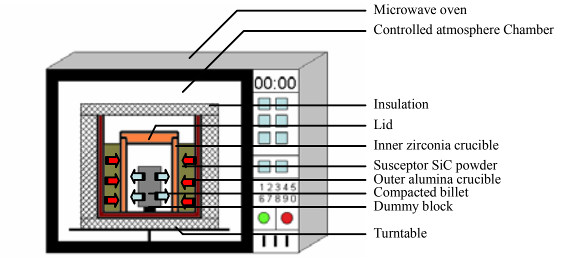


Fig 1: Schematic diagram of microwave set-up showing hybrid sintering.

2.3 Secondary Processing

The sintered billets were hot extruded using an extrusion ratio of 26.5: 1 on a 150 tonne hydraulic press. Extrusion was carried out at 230°C. The billets were soaked at 230°C for 5 minutes in a constant temperature furnace before extrusion. Colloidal graphite was used as lubricant. Rods of 7 mm diameter were obtained following extrusion.

2.4 Microstructural Characterization

Microstructural characterization studies were conducted on metallographically polished extruded samples to investigate morphological characteristics of grains and pores. The etching solution was made using 5% HCl into ethanol to reveal the grain boundaries for microstructural analysis [21]. Hitachi FESEM-S4300 equipped with EDS was used to capture micrographs. Image analysis using the Scion system was carried out to quantify the microstructural features.

2.5 XRD Studies

An automated Shimadzu LAB-X XRD-6000 diffractometer was used for phase analysis of extruded samples. The samples were polished and exposed to Cu Kα radiation (λ = 1.54056 Å) using a scanning speed of 2 deg/min.

2.6 Microhardness Testing

Microhardness measurements were made on the polished samples in accordance with the ASTM standard E384-99. Vickers microhardness was measured using Shimadzu-HMV automatic digital microhardness tester using 10 gf-indenting loads and a dwell time of 15 seconds.

2.7 Tensile Testing

The smooth bar tensile properties of the extruded pure tin samples were determined in accordance with ASTM test method E8M-01 using MTS 810 tensile testing machine with a crosshead speed set at 0.254 mm/min on round tension test specimens of 5 mm diameter and 25 mm gauge length.

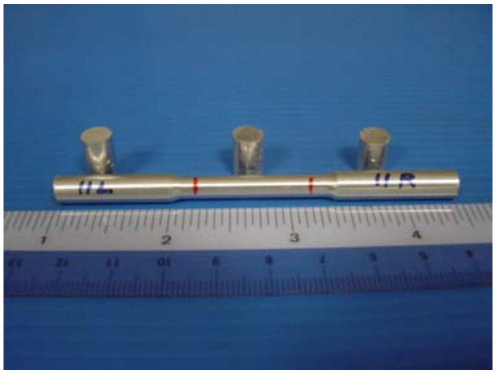


Fig 2: Macrograph showing samples for microstructural and tensile properties characterizations.

2.8 Fracture Behavior

Fracture surface characterization was done on the tensile fractured samples in order to provide insight into fracture mechanisms operating in pure tin under tensile loading. FESEM-S4300 equipped with EDS was used for this purpose.

3. RESULTS

3.1 Macrostructure

The result of macrostructural characterization conducted on sintered pure tin did not reveal presence of macropores or any other defects. Following extrusion, there was also no evidence of any macrostructural defects in any of the extruded rods.

3.2 Microstructural Characterization

Microstructural studies conducted on the extruded samples showed near equiaxed grain morphology (see Table 1). Average grain size was slightly larger for microwave sintered samples and aspect ratio was same as that of conventionally sintered samples. Considering standard deviation, the difference in grain size was statistically insignificant. However, the distribution of grain size for microwave sintered samples showed that around 70% grains felt within the range of 1.84 – 3.69 μm while for the case of radiant heat sintered samples, this amount of grains shifted to 1.53 – 3.07 µm range (see Fig. 3). Smaller average pore size was exhibited by microwave sintered samples than radiant heat sintered samples (see Table 1 and Fig. 4). Radiant heat sintered samples also showed sharp-edged pores and this can be clearly observed from the aspect ratio distribution (see Fig. 5). This distribution revealed more than 73% pores felt in the range of 1.00 – 1.98 while for the case of radiant heat sintered samples, only 16% pores are in that range.

Table 1: Results of grain and pore morphology of pure tin.

Materials*	Grain characteristics [a]		Pores characteristics [b]	
	Size (µm)	Aspect ratio	Size (nm)	Aspect ratio
Sn/M.Sint.	3.0 ± 1.0	1.5	144	1.5
Sn/C.Sint.	2.6 ± 1.0	1.5	353	3.5

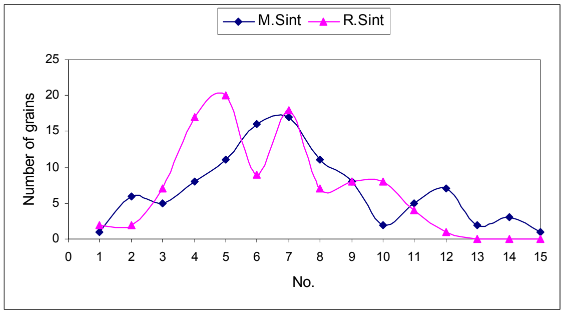
* M.Sint. = Microwave Sintered, R.Sint. = Radiant heat Sintered.

[a] 103 grains were quantified.

[b] 55 pores were quantified.

3.3 X-ray Diffraction

Table 2 shows the X-ray diffraction results obtained from pure tin processed under different sintering conditions. These results showed absence of tin oxide phase as all peaks matched with standard tin peaks only.



No.	Grain size range(µm)	M.Sint	R.Sint
1	0.90 - 1.21	1	2
2	1.22 - 1.52	6	2
3	1.53 - 1.83	5	7
4	1.84 - 2.14	8	17
5	2.15 - 2.45	11	20
6	2.46 - 2.76	16	9
7	2.77 - 3.07	17	18
8	3.08 - 3.38	11	7
9	3.39 - 3.69	8	8
10	3.70 - 4.00	2	8
11	4.01 - 4.31	5	4
12	4.32 - 4.62	7	1
13	4.63 - 4.93	2	0
14	4.94 - 5.24	3	0
15	5.25 - 5.55	1	0

Fig 3: Grain size distribution.

3.4 Microhardness

The result of microhardness measurement showed that radiant heat sintered samples exhibited better microhardness values than microwave sintered samples (see Table 2).

Table 2: Results of X-ray diffraction and microhardness measurement of pure tin.

Materials	Matching peaks		Microhardness (HV)
	Tin	Tin oxide	
Sn/M.Sint.	11 [3]	0	10.8 ± 0.1
Sn/R.Sint.	11 [3]	0	12.7 ± 0.3

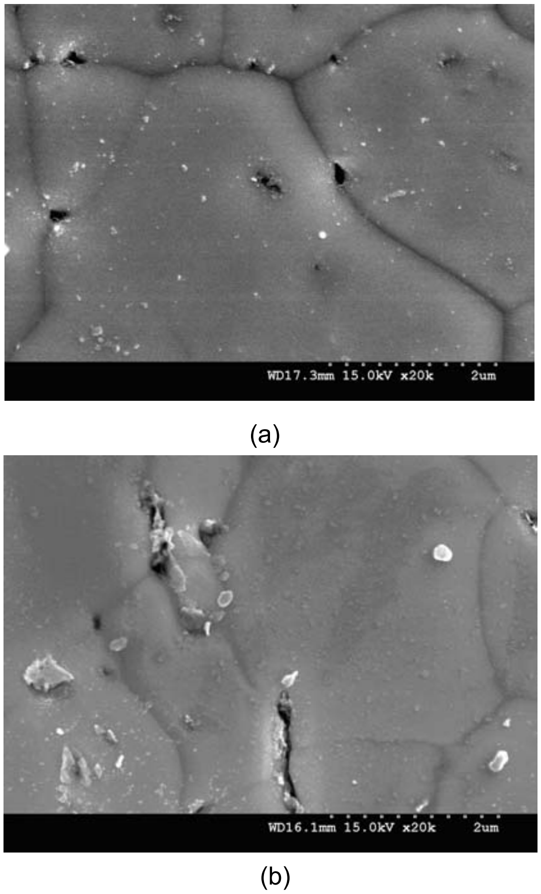


Fig 4: Representative FESEM micrograph showing pore morphology of pure tin in the case of: (a) microwave sintered and (b) radiant heat sintered samples.

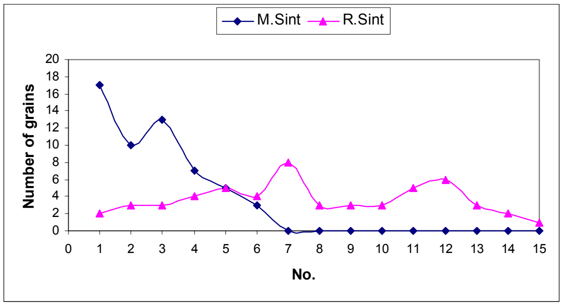
3.5 Tensile Characteristics

The results of ambient temperature tensile tests revealed better combination of tensile properties for microwave sintered samples than radiant heat sintered samples. Microwave assisted rapidly sintered samples revealed 30% and 26% higher yield strength (YS) and ultimate tensile strength (UTS), respectively than radiant heat sintered samples. An increment of 12% failure strain

(FS) and 56% work of fracture (WoF) were observed for microwave sintered samples over radiant heat sintered samples.

3.6 Fracture Behavior

Fractographs of the microwave assisted rapidly sintered tensile samples showed dimples which is a clear evidence of ductile failure while conventionally or radiant heat sintered samples revealed predominance of intergranular cracks (see Fig. 6).



No.	Aspect Ratio	M. Sint	R. Sint
1	1.00 - 1.32	17	2
2	1.33 - 1.65	10	3
3	1.66 - 1.98	13	3
4	1.99 - 2.31	7	4
5	2.32 - 2.64	5	5
6	2.65 - 2.97	3	4
7	2.98 - 3.30	0	8
8	3.31 - 3.63	0	3
9	3.64 - 3.96	0	3
10	3.97 - 4.29	0	3
11	4.30 - 4.62	0	5
12	4.63 - 4.95	0	6
13	4.96 -5.28	0	3
14	5.29 - 5.61	0	2
15	5.62 - 5.94	0	1

Fig. 5. Distribution of aspect ratio of pores.

4. DISCUSSION

4.1 Microstructural Behavior

The microstructural characterization conducted on extruded samples revealed the presence of near-equiaxed grains (see Table 1). Considering standard deviation, the variation in grain size can be said to be independent of sintering type.

Metallography of the extruded samples also revealed the variation in pores morphology as a function of sintering type (see Table 1 and Fig. 4 and 5). Lower average pore size was observed for microwave assisted sintered samples. Radiant heat sintered samples exhibited larger pore size and with higher aspect ratio suggesting the predominance of lenticular pores. Higher aspect ratio of the pores in the case of radiant heat sintered samples suggests the intermediate stage of sintering realized even when the holding duration was 2 hours. The absence of lenticular pores in the case of microwave sintered samples suggests that a 7 minute sintering duration in microwaves can bring the sintering into its final stage [9].

Table 3: Results of room temperature tensile properties of pure tin.

Materials	0.2% YS (MPa)	UTS (MPa)	FS (%)	WoF (MJ/m^3)
Sn/M.Sint.	35 ± 2	39 ± 3	10.0 ± 0.5	6.1 ± 0.7
Sn/R.Sint.	27 ± 5	31 ± 5	8.9 ± 0.8	3.9 ± 0.5

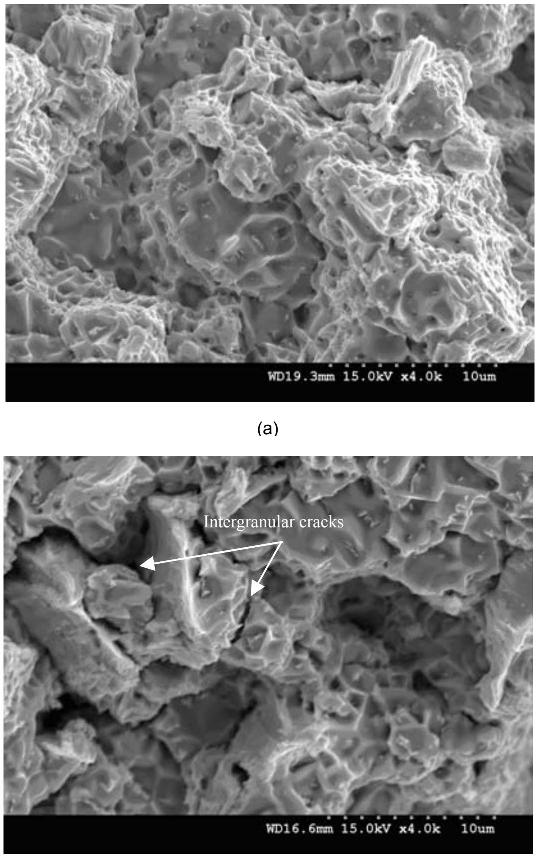
4.2 Microhardness

Microhardness measurement showed that radiant heat sintered samples exhibited higher values of microhardness than microwave sintered samples (see Table 2). This can be attributed to the lower average grain size, exhibited by radiant heat sintered samples. Lower grain size is associated with large grain boundary area leading to higher hardness [22]. Besides average grain size, grain size distribution of the radiant heat sintered samples was also shifted to lower grain sizes (see Fig. 3).

4.3 Tensile Behavior

The results of room temperature tensile tests revealed that microwave sintered samples exhibited better combination of tensile properties than radiant heat sintered samples. Microwave assisted rapidly sintered samples revealed 30% and 26% higher 0.2% YS and UTS respectively than radiant heat sintered samples (see Table 3). In general, the stress required to operate the dislocation sources is the yield stress of a material and is governed primarily by the dislocation density that restricts the motion of dislocations. Under the applied stress, the grain boundary areas act as the main obstacles to the dislocation movement for pure metals. Even though the variation of grain size was marginal considering standard deviation, for radiant heat sintered samples, lenticular pores along the grain boundaries reduced the total grain boundary area as well as the dislocation density and might have contributed to lower strength compared microwave sintered samples (see Tables 1 and 3 and Fig. 4). Lower strength values for radiant heat sintered samples can also be explained by the pore morphology where large and sharp-edge pores acted as stress concentration sites that led to premature failure. Fig. 6b clearly shows the predominance of intergranular cracks on the fracture surface of radiant heat sintered samples supporting this hypothesis (see also Fig. 4b).

An increment of 12% FS and 56% WoF were observed for microwave sintered samples over radiant heat sintered samples. Based on microstructural and fracture surface observation, this can primarily be attributed to the pore morphology where microwave sintered samples showed near equiaxed pores with smaller size while radiant heat sintered samples revealed sharp-edge pores (higher aspect ratio) with much larger size (see Table 1 and Fig. 4 and 5). These results are also consistent with the work of other investigators who observed an increase in toughness and ductility of Fe-based materials and attributed the increase to the presence of round-edged porosity [23].



(a)

(b)

Fig. 6. Representative FESEM fractographs showing: (a) dimples in the case of microwave sintered samples and (b) predominance of intergranular cracks in the case of radiant heat sintered samples.

4.4 Fracture Behavior

Fractographic analysis on microwave sintered fracture samples revealed the extensive presence of dimple like features, indicative of relatively higher ductility (see Table 3 and Fig. 6a) [24]. The observation made on the fracture surface of radiant heat sintered tensile samples revealed predominance of intergranular cracks with limited evidence of dimple like features indicative of relatively lower ductility (see Fig. 6b). Observations made on the fracture surfaces of the samples are consistent with the results of tensile properties such as failure strain and work of fracture (see Table 3) and in particular the pore morphology (see Table 1 and Figs. 4 and 5).

5. CONCLUSIONS

The following conclusions can be made from the present study:

1. Pure tin can be successfully synthesized using innovative microwave and radiant heat sintering in powder metallurgy route.
2. Microstructural characterization revealed that radiant heat sintered samples showed lenticular porosity while microwave sintered samples revealed near equiaxed porosity.
3. Tensile characterization revealed that microwave assisted rapid sintering route leads to better room temperature tensile properties than radiant heat sintering.

6. ACKNOWLEDGEMENT

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