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Influence of mixing technique on sintering response of binary aluminium alloy powders

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ABSTRACT

Aluminium powder metallurgy (Al-PM) alloys are finding promising applications in many areas such as household appliances, automotive vehicles and many other allied sections where reduction in weight is the primary constraint. In the proposed research, two binary alloy systems Al-X were studied by mixing elemental powders of Aluminium (Al), Tin (Sn) and Magnesium (Mg). Two types of mixing techniques were followed. In one case, elemental powders were blended in a mechanical mixer without any mixing media (A) and another case powders were blended with ball to powder ratio of 10:1 (B). The content in binary premix was varied from 0.4 to 0.8 % Sn (by wt.) and 0.5 to 2 % Mg (by wt.) with base of Aluminium. The blended powders was compacted at 450 MPa followed by sintering in ultra high purity nitrogen atmosphere in tubular sintering furnace at 600°C. The mixing technique B showed significant influence on increase in hardness by two-fold and reduction in dimensional growth. Copyright © 2013 VBRI press.

Keywords: Powder metallurgy; aluminium; liquid phase sintering; Al-Sn system; Al-Mg system; mixing techniques.

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Introduction

Aluminium P/M parts are currently receiving rapid attraction by automotive industry due to cost effective nature of the process. But the major setback is due to the lack of commercially available alloys for particular application. Up to last decade, there were only two alloys mostly preferred viz. AC2014 (Al–4.5Cu–0.8Si–0.5Mg) and A6061 (Al–1Mg–0.6Si–0.25Cu). This limited number of commercial P/M alloys corresponds to a narrow range of mechanical properties [**1**]. Mechanical properties of aluminium alloys can be improved either on a micro (≤ 0.5 wt. %) or macro (1–20 wt. %) scale with small additions of some alloying elements such as Cu, Mg, Zn, Si and specifically trace additions of Sn along with high sintering temperatures and modified heat treatments [**2-3**]. The use of liquid phases is an alternative to solid state sintering. During transient liquid phase sintering, the amount of liquid formed and its duration is influenced by the process variables like heating rate, final sintering temperature, compaction pressure, sintering atmosphere, impurity levels and starting powder characteristics. These variables should be optimized in order to get maximum sintered properties [**4**]. Addition of tin and magnesium facilitates liquid phase sintering where magnesium addition is necessary for reduction of oxide layer on aluminium powder particle [**5-7**]. The increasing content of Sn in the spray formed alloys decreases the wear rate [**8**]. The controlling

atmosphere is the key issue in the sintering of aluminium. The dimensional growth varies with the type of atmosphere chosen; hence pure dry nitrogen atmosphere is commonly used for getting favourable sintered properties as compared to other atmospheres [**9-11**]. In this research work, the sintering response of Al-Sn system (0.4 to 0.8% Sn by wt) and Al-Mg system (0.5 to 2% Mg by wt) was examined to evaluate the impact of mixing techniques on sintered properties of these binary premix alloy systems.

Experimental

Selection of powders

The elemental powders viz. Aluminium (Al), Tin (Sn) and Magnesium (Mg) were used for making premix alloy. The morphology of powders used in this work is shown in **Fig. 1** and their corresponding physical and chemical properties are listed in **Table 1**.

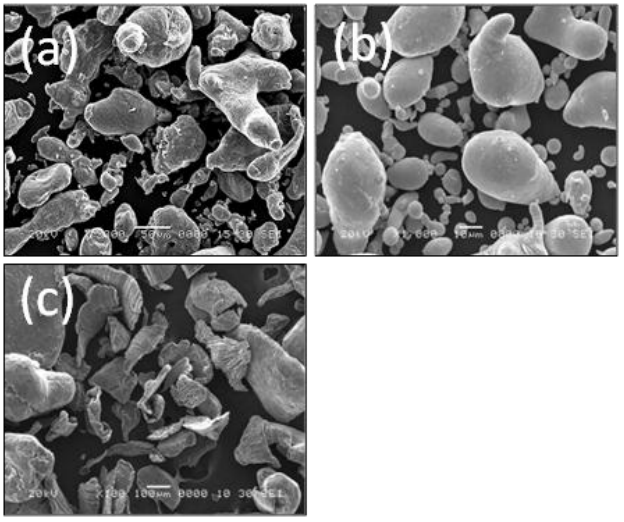

Fig. 1. SEM morphology of powder (a) Irregular-aluminium (b) Ellipsoidal-tin and (c) Flaky-magnesium.

Table 1. Physical and chemical properties of powders.

Sr. No	Type of Powders	Apparent density, g/cc	Purity, %	Particle size/ Mesh (ASTM)	Sources of Powders
1	Aluminum (Al)	1.16	99.60	170 (90 µm)	Komal Atomizer, Mumbai
2	Tin (Sn)	4.05	99.50	325 (45 µm)	Vivek Agencies, Mumbai
3	Magnesium (Mg)	0.64	99.50	270 (53 µm)	Research Lab, Mumbai

Blending and fabrication of sintered compacts

Two binary Al-X alloy systems were prepared by powder metallurgy route viz. a) Al-0.4 to 0.8% (by wt) Sn with an incremental addition of 0.2%; and b) Al- 0.5 to 2% Mg (by wt) with an incremental addition of 0.5%. Premix powders of Al, Mg and Sn were blended by two techniques. In one case, steel balls were used as mixing media with ball to powder ratio of 10:1 (by wt) for 2 hr called as mechanical grinding and designated by B. Other type of blending was without the mixing media referred as free mixing and designated by A. Both these techniques of blending were followed for both these binary premix powders. The blended powders were pressed in Universal Testing Machine (UTM) uniaxially at compacting pressure of 450 MPa to form green compacts of size 12 mm diameter and 8 mm height. The samples fabricated in this work were free from any lubricant addition. Zinc stearate was used for die wall lubrication and applied manually before each run to reduce wall friction. The green compacts were sintered in dry nitrogen gas purified through purification train consisting of pyrogollal acid and silica gel to eliminate any traces of oxygen and moisture respectively. Sintering was carried out in a tubular sintering furnace at 600^0C for 30 min followed by quenching in water.

Characterization

The green and sintered density was measured using Archimedes's principle and compared with the theoretical density calculated by using Rule of Mixture (ROM). Specimens were polished by using electrolytic polishing machine (make- Eletropol Metatech) in an electrolyte solution consisted of Methanol-730 mL, Butyl Cellosolve-98mL, Perchloric acid-78 mL and Distilled water- 100 mL. Additionally, the etching was carried out using Keller's reagent for revealing grain boundaries of matrix. Microstructures were observed using optical microscope (make Carl Zeiss) and Scanning electron microscope (SEM) and Energy Dispersive Spectroscopy (EDS) techniques (make-JOEL, Japan) for spot analysis. The phase analysis was done by XPERT-PRO X-ray diffractometer (Copper target, K-alpha-1.5406^{o}A). Bulk hardness was measured on Rockwell H-scale (HRH) with a 60 kg load and 1/8 inch ball indenter. The dimensions (height and diameter) were measured using Mitutoyo digital micrometer having least count of 1 µm. For cylindrical samples, diameter was checked at three positions top, middle and end along longitudinal direction. The hardness and dimensional changes reported in this paper were resulted from average of three readings per sample for total three specimens.

Results and discussion

Sinterability of Al- Sn premix alloy

Tin is a low melting point (232^{o}C) element which is useful in enhancing liquid phase sintering. Al-Sn system has been widely studied since this system exhibits almost all of the features of an ideal system [**5, 13**]. **Fig. 2** (**a**) shows microstructure of Al-0.8% Sn depicts tin rich region as confirmed by EDS spectra (**Fig. 3**).

Fig. 2 (**b**) shows complete homogenization of tin due to mechanical mixing (B) which results into fine grains as compared to technique (A) in which isolated spot of tin rich phase (**Fig. 2**a). Such fine grains are attributed to phenomena akin to mechanical alloying [**12**]. It may be noted from **Fig. 4** (**a**) that premix powders was blended in a mixer which induces the plastic deformation in the

particles and hence show low green density (B) due to poor compressibility than without mechanically grind powder (A). In contrast to this, the sintered density of both the types of mixes is almost same as depicted in **Fig. 4** (b).

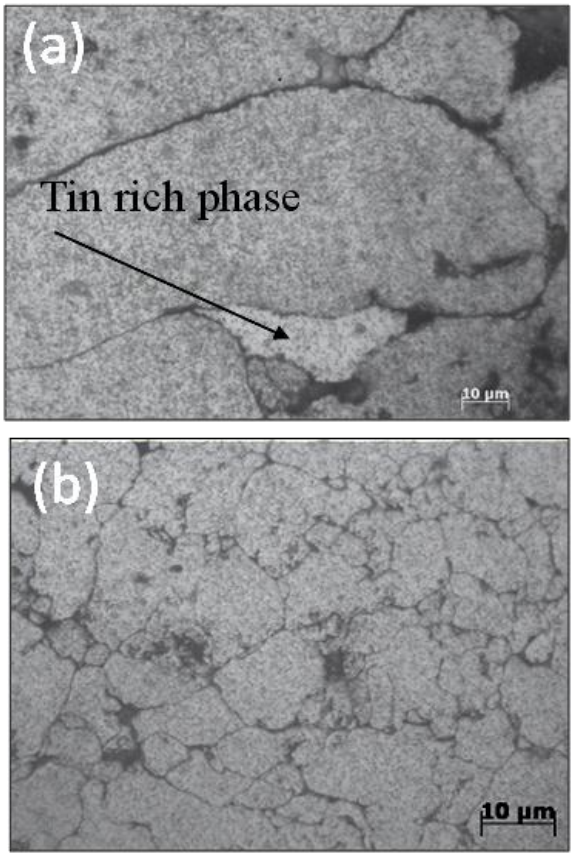

Fig. 2. Optical microstructure of Al-0.8%Sn sintered at 600^{0}C showing (a) tin rich region due to mixing technique A and (b) without segregation of tin due to mixing technique B.

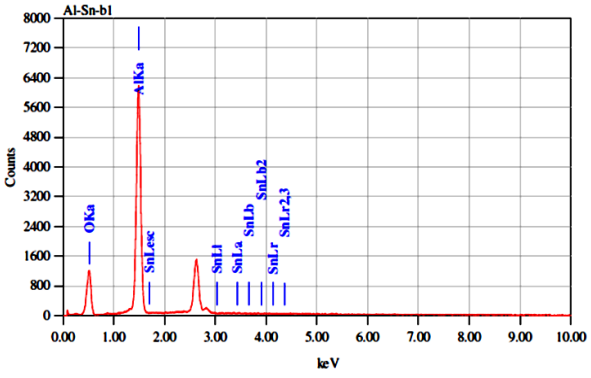

Fig. 3. EDS spot analysis of tin rich region marked in Fig. 2 (a).

The mechanically mixed powder by mixing technique B has high driving force for sintering due to residual strain in the particles. It is observed from **Fig. 5** that the mechanical mixing (B) exhibits low dimensional growth as compared to other mixing technique (A). This is evident from **Fig. 2** that the complete mixing of tin is essential as Al-Sn exhibits mutual solid insolubility and complete solubility in the liquid phase [**13**]. Even though, there is overlapping sintered density (**Fig. 4b**) for the types of mixing practices, the hardness of mechanically grind (B) practice demonstrate almost twice the hardness than mixing technique (A).

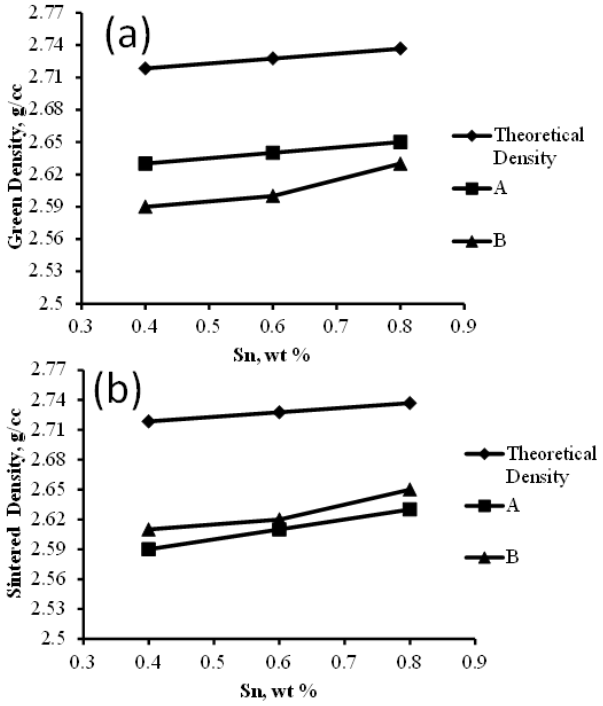

Fig. 4. Effect of Sn addition and mechanical mixing on (a) Green density and (b) Sintered density.

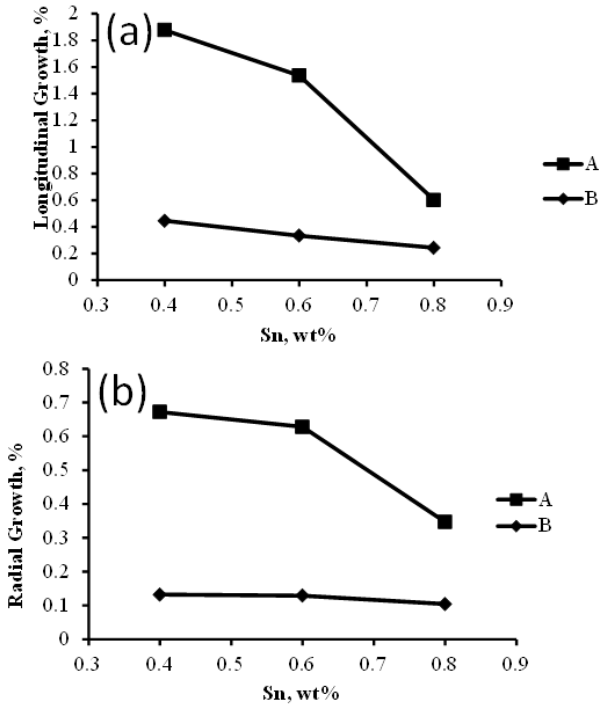

Fig. 5. Effect of Sn and mixing techniques on longitudinal growth (a) and radial growth (b).

Hence the increase in hardness (**Fig. 6**) could be attributed to solid solution strengthening as liquid tin (M.P. 232^{o}C) promotes faster diffusion of atoms across the particle-particle interface and other reason may be attributed to fine grain size of the sintered compact.

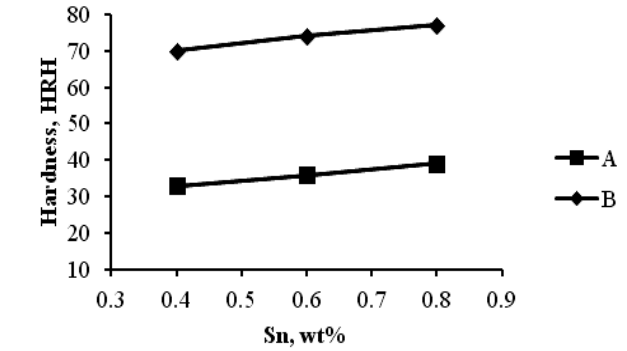

Fig. 6. Influence of Sn and mixing techniques on hardness variation.

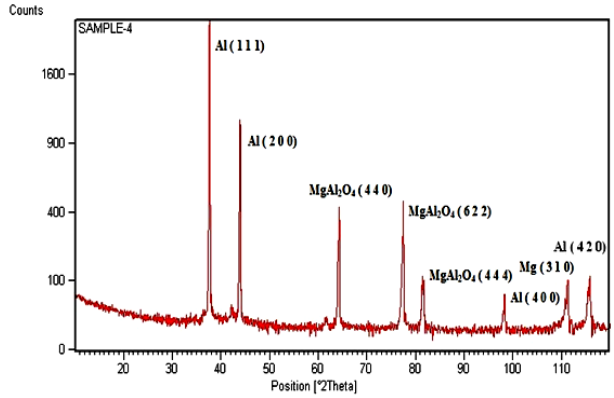

Fig. 7. XRD result of Al- 2% Mg showing presence of spinel $MgAl_20_4$.

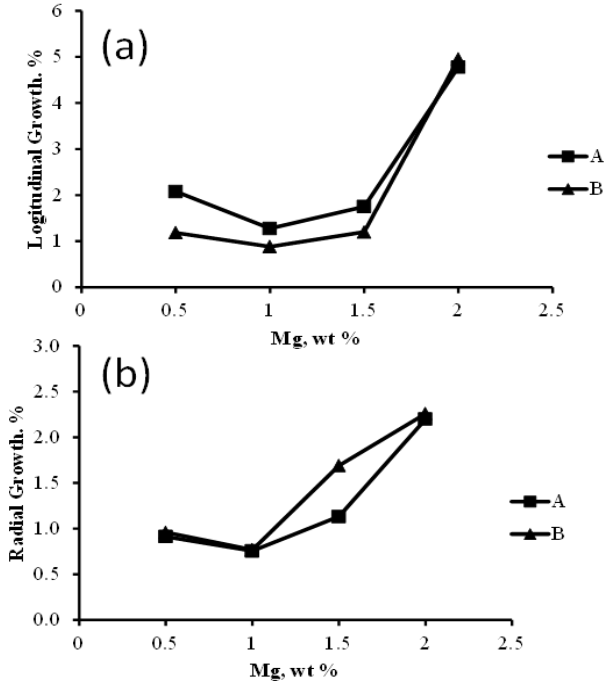

Fig. 8. Effect of magnesium on variation in dimensional change.

Sinterability of Al-Mg premix alloy

Magnesium is useful in the aluminum sintering which reduces the oxide film on aluminium powder particles by forming complex spinels of Mg with Al_20_3 [7]. **Fig. 7** shows the XRD result of Al- 2%Mg confirming presence of spinel $MgAl_20_4$. The spinel presence indicates that magnesium disrupt the oxide film sufficiently to facilitate diffusion, wetting and sintering of aluminum particles [**5-7**].

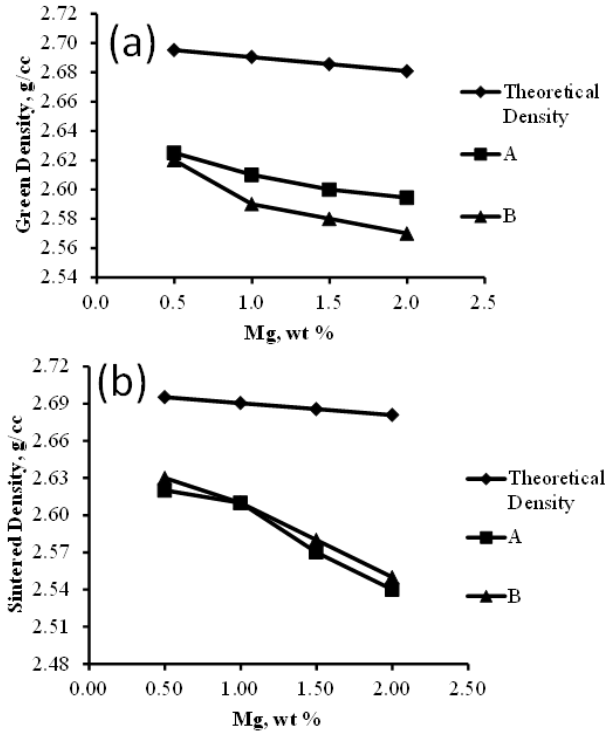

Fig. 9. Effect of Mg content on a) green density and b) sintered density.

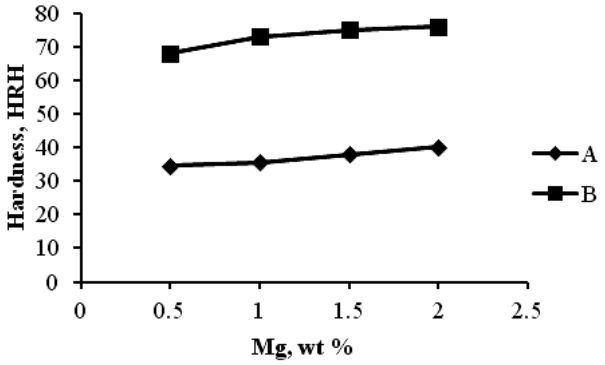

Fig. 10. Effect of Mg content on hardness variation of Al-Mg alloy.

Fig. 8 shows the lowest dimensional growth noticed for less than 1% Mg which can be attributed to the contraction of the matrix due to spinel formation. Beyond 1% Mg, the excess magnesium may cause expansion of the matrix by the Kirkendall effect and solid solution hardening [**5-7**]. It is noted that higher magnesium addition deteriorates the surface finish of samples by way of black dots on the surface. There is a decrease in sintered density observed as shown in **Fig. 9** which can be attributed to low density of magnesium.

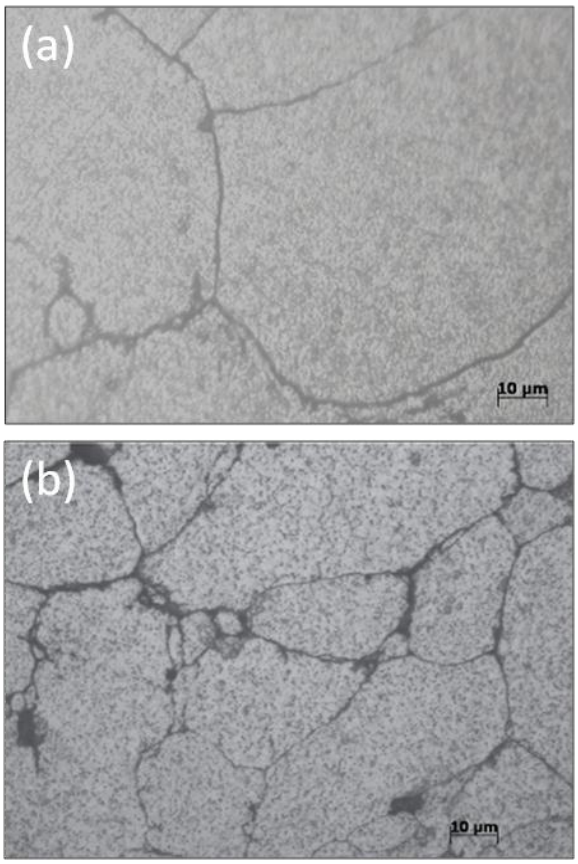

Fig. 11. Microstructure shows (a) coarse grain due to mixing technique –A and (b) fine grains due to mixing technique-B.

The hardness of premix powder compact prepared by technique B shows almost twice the hardness than the premix powder prepared by technique A as evident in **Fig. 10**. This rise in hardness is obviously the result mechanical alloying in addition to solid solution strengthening due inherently large solubility of Mg in the aluminum matrix [**13**]. **Fig. 11** depicts premix powder processed by technique B gives significant decrease in grain size than mixing technique A.

Conclusion

The sinterability of Al-Sn and Al-Mg alloy was analyzed with reference to types of mixing practice adopted, that is, mechanical mixing of premix (B) and other was without application of mixing media (A). Based on the foregoing discussion, the following conclusions can be drawn -

1) The induced residual strain in the mechanically grind premix powder is the main driving force for enhancing atomic diffusion of the alloying elements. Except dimensional growth and hardness, the sintered density shows overlapping pattern in both the mixing practice adopted.
2) The mechanical mixing has significant impact on enhancing solid solubility of tin in aluminium matrix. Such premix powder makes homogeneous distribution of tin in aluminum matrix and hence gives rise to limited dimensional growth. The hardness shows almost two fold increase.
3) Magnesium addition helps to reduce oxide film on aluminium by forming spinel of $MgAl_2O_4$ as confirmed by XRD as this is attributed to decrease dimensional growth till threshold value of 1% Mg and thus it limits the addition of magnesium in powder premix. Beyond this critical content, the surface finish of sintered compacts deteriorates.

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