A Study of Debinding Behavior and Microstructural Development of Sintered Al-Cu-Sn Alloy

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Abstract. A new approach is explored to achieve sintered aluminium alloy from metallic powder mixtures without compression or adding Mg. In this approach, mixtures of micron-sized aluminium powder (average size of 2.5 μm) and nano-sized alloying elemental powder of Cu and Sn (less than of 70nm), at appropriate proportions to compositions of Al-6wt%Cu, Al-6wt%Cu-3wt%Sn with and without adhesive binder were prepared by magnetic stirring. Then, the powder mixture was poured into a crucible and heat treated at a temperature of 600°C for 11 hours in inert atmosphere of N_2 or Ar. In this paper, we investigate the debinding behavior of loosely packed Al-based powder mixture and the microstructural development and mechanical property sintered parts using a combination of thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), X-ray diffractrometry (XRD) and hardness test.

Introduction

Aluminium alloys are widely used in many engineering applications due to its unique features of lightweight and high strength properties. Metallic injection moulding (MIM) technology has been successful in making precision components out of metallic powders. However, MIM is known to undergo severe dimensional change after sintering. Furthermore, aluminium powder is known in the powder metallurgy area to be the most difficult material for sintering, because the surfaces of powder particle are covered by tenacious oxide layers that cannot be broken or removed by heating[1-2] due to the thermodynamic stability of the oxide. To achieve an unstable oxide layer and to reduce its thickness, it requires a dew point below -140°C[3] or an oxygen partial pressure of less than 10^{-50} atm at 600°C[4]. However, these conditions can not easy to obtain.

In general, in aluminium powder metallurgy, the powder is compressed in a container to cause the oxidized surface layers to rupture, which in turn induces deoxidisation by adding Mg in the aluminium as prealloyed or blended mixture of powder to improve sinterability[5-7]. In aluminium powder metallurgy, Cu and Sn is commonly used as alloying additions for strengthening and enhancing sinterability of aluminium powders. Although such methods are effective, it is difficult to implement them in some aluminium prototyping and especially in the fabrication of microcomponents, for example, where soft moulds are used and the miniaturization requires the use of ultrafine or nano-sized metal powder.

Currently, there is only few studies on the sintering of loosely packed metal powders of sizes ranging from nanometer to few micrometers. This paper presents preliminary studies on the effect of addition of binder, nano-sized powder of Cu and Sn on the sintering of loosely packed micron-sized Al powder together with debinding behavior.

Experiment methods

As-supplied materials. Table 1 shows a list of Al, nano-sized Cu and Sn, supplied by AlPoCo, UK and Shenzhen Junye, China , respectively.

Table. 1. Particle sizes and sources of the powders used.

Powder	Particle size	Purity	Shape	Source
Al	mean scale 2.5 μm	99.9%	Spherical	Alpoco
Cu	less than 70 nm	99.9%	Spherical	Shenzhen Junye
Sn	less than 70 nm	99.9%	Spherical	Shenzhen Junye

Preparation of powder mixtures. Appropriate proportions of nano-sized Cu and Sn were added to Al powders to produce the nominal required composition of Al-6wt%Cu and Al-6wt%Cu-3wt%Sn with overall sample weight of 3 gram. About 30ml of ethanol was then added to this mixture to prevent the airborne of these ultrafine powders. The mixing was done for 2 hours by using a magnetic stirrer (Hanna Instruments HI180H/D). After sufficient mixing, the mixture was dried inside an oven with the temperature maintained at 50°C for 4 hours to remove the ethanol.

Preparation of powder mixture with adhesive binder. 80wt% of Al powder, 5wt% of nano-sized Cu powder and 15wt% of adhesive (Bostik) together with about 30ml of acetone were mixed together as a slurry. The slurry of powder mixture was dried in an oven at similar conditions as before in section.

Sintering of loosely compacted Al, Al-Cu and Al-Cu-Sn. In order to study the effect of adding nano-sized Cu and Sn on the sintering of Al powder, a control sample was prepared from pure elemental Al powder. Sintering of samples was performed using a Carbolite tube furnace maintained at temperatures between 630°C and 600°C for up to 11 hours for pure Al and Al alloy mixture powders, respectively, in a dynamic flow of either Ar or N_2 gas. Fig. 2 shows the list of sintering conditions used in this study.

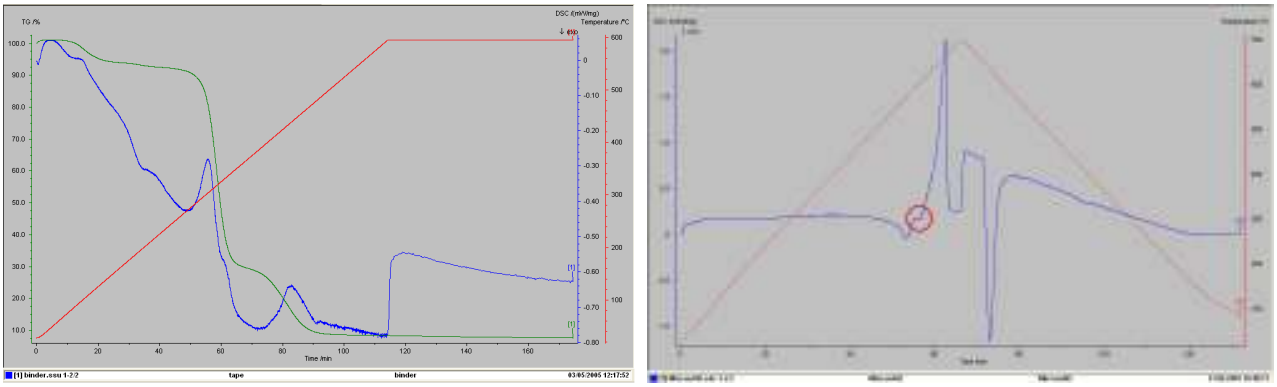
Table. 2. List of sintering conditions used in this study.

Sample	Binder	Sintering Temperature	Sintering Time	Atmosphere
Pure Al	No	630°C	2 hr	Ar
Al-6wt%Cu	No	600°C	1hr, 11hr	N_2
Al-6wt%Cu-3wt%Sn	No	600°C	1hr, 11hr	Ar and N_2
Al-5wt%Cu-15wt%Adhseive	Yes	600°C	1hr, 11hr	Ar

Material characterization. The microstructure and hardness of sintered specimens were investigated using SEM (FEI Co. Strata DB235, Philips XL30 and JEOL JSM-7000F), optical microscopy (Carl Zeiss Jena, SL100) and micro vickers hardness tester (Mitutoyo, MVK-H1).

Thermal stability of powder mixture. Phase transformation during the heating of the powder mixture was investigated using a Netzsch Simultaneous Thermal Analyser (STA) 449C. Each powder mixture of 10mg was heated at a rate of 10°C/min up to 700°C under a dynamic flow of Ar.

Results and discussion



(a) (b)

Fig. 1. (a) TG & DSC traces of binder in Ar-gas, (b) DSC trace of Al-Cu powder mixture.

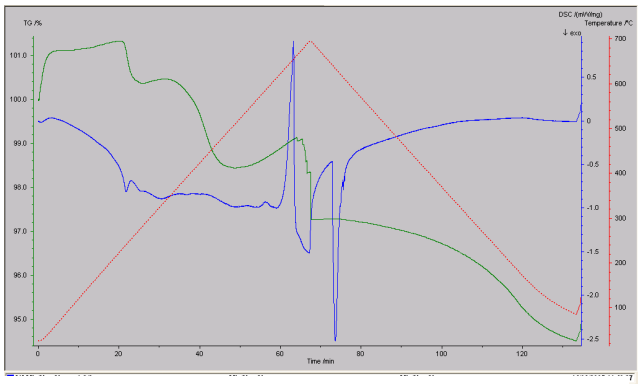


Fig. 2 shows TGA and DSC traces of Al-5wt.%Cu with15wt.% binder mixture.

STA of Powders. To understand the high temperature behavior of the Al, Cu, powders and binder mixture, thermal analysis was performed by STA. Figure 1(a) shows the Thermogravimetry (TG) result of adhesive binder (Bostic). We found that over 90% of the binder weight evaporated during the temperature ramping up to 450°C in Ar-gas. This evaporation of binder makes the sintering process simpler as no additional debinding process is required. That is, the binder becomes evaporated in-situ during sintering cycle inside the furnace.

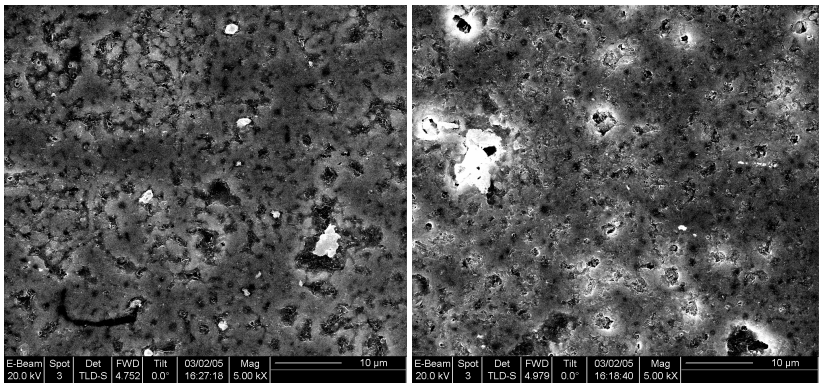
Fig. 1(b) show DSC traces of Al-6wt%Cu powder mixture. It consists of an exothermic peak that appear at around 500°C followed by a small endothermic peak at 550° and a large endothermic peak at 640°C, which indicates that Al_2Cu intermetallic formed, melting of Al_2Cu and melting of the remaining Al phase, respectively, and this is in good agreement with binary equilibrium Al-Cu phase diagram[8]. Figure 2 shows the TGA and DSC traces of Al-5wt%Cu-15wt%binder powder mixture. TGA trace shows a weight loss of around 3% which suggests that the binder was not completely been removed during the heating cycle. DSC trace was similar to those found in binary Al-6wt%Cu powder mixture as shown in Fig 1(b).

Sintering of loosely packed powders. In pure Al powder, the specimen remained as loose powder after sintering at 630°C for 2 hours in Ar. No sintering process was carried outI for this specimen. Al-6wt%Cu powder without adhesive binder was sintered at various time. The porosity level showed to decrease when sintering time elapsed from 1hr to 11hrs as shown in Fig. 3.

The change of sintering atmosphere from N_2 to Ar, significantly reduced the porosity in Al-6wt%Cu-3wt%Sn composition, as shown in Fig. 4.

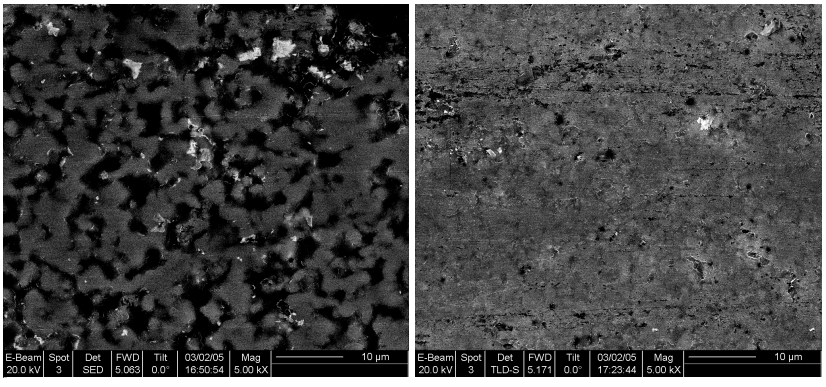
Among the various conditions for sintering, the best powder mixing ratio is Al-6wt%Cu mixture. Therefore, this mixture was used with adhesive binder. For more details on the study of the microstructures analysis of these types of Al alloy, see the literature [9].

An adhesive binder was introduced into the powder mixture in order to increase the bonding strength of green structures. Al-6wt%Cu powder mixture was used to analayze the effect of adding adhesive binder on sintering of loosely packed Al powders. The powder mixture with binder was then sintered at 600˚C for 11hr at Ar atmosphere. Fig. 5



(a) **(b)**

Fig. 3. SEM images of Al-Cu after (a)1hr in N_2, (b) 11hr in N_2.



(a) **(b)**

Fig. 4. SEM images of (a)Al-Cu-Sn sintered for 1hr in N_2, (b)Al-Cu-Sn for 1hr in Ar.

shows sintered mictostructure of this sample. The microstructure consists of Al_2Cu intermetallic in Al matrix, which was confirmed by X-Ray diffractrometry spectrum (see Fig. 6). The average vickers hardness of this specimen was found to be 58.

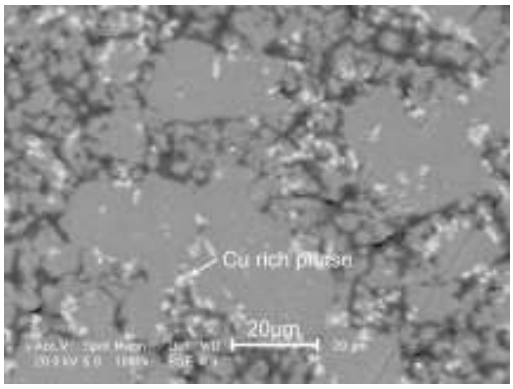


Fig. 5. The sintered microstructures of Al-5wt%Cu-15wt%adhesive binder at 600˚C in Ar, for 11hr.

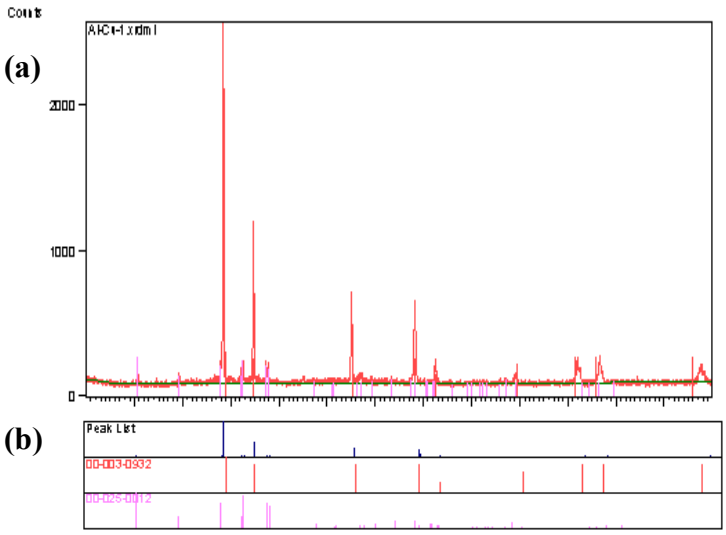


Fig. 6. (a) XRD patterns of sintered Al-5wt%Cu-15wt%adhesive binder at 600˚C in Ar, for 11hr and (b) peak position of samples, pure Al, and Al_2Cu standards.

Conclusions

Loosely packed Al alloy powder sintering was studied in different composition, sintering time and atmosphere with and without adhesive binder. Based on our findings, the following conclusions can be summarized as:

1. The sinterability of Al-6wt%Cu and Al-6wt%Cu-3wt%Sn mixtures were investigated and processing optimum conditions were proposed.
2. Sintering process was simplified by using adhesive binder system.
3. Loosely packed Al-5wt%Cu-15wt% binder mixture was successfully sintered.
4. The microstructure of sintered Al-5wt%Cu-15wt% binder powder mixture consisted of Al_2Cu intermetallic in Al matrix.

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