Improving Sinterability of Aluminium Alloy (Al-Si-Cu-Mg) by Adjusting Sintering Conditions

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Abstract

In this work, pre-alloyed Al-Si-Cu-Mg powders were compacted and sintered using different sintering conditions by adjusting the level of vacuum applied before heating, the atmospheric controlling conditions, the purity of nitrogen gas, sintering chamber and chamber capacity. The results suggested that the sinterability can be improved by employing higher purity of nitrogen gas. In addition, a sufficient flow of gas compared to the chamber capacity of furnace is highly important to ensure proper sintering. Well sintered Al-Si-Cu-Mg aluminium alloy has more homogeneous dispersion of silicon phase and less pores. Furthermore, the self-gettering phenomenon is the result of poor sintering at the specimen surface. The optimum sintering condition had the lowest dew point temperature, which was -38°C. This optimum sintering condition produced sintered parts with the best properties, i.e. the highest sintering density (2.62 g/cm^3), the highest tensile strength (131MPa) and the highest hardness (76 HRF).

Keyword: Aluminium-Silicon based alloy, Powder metallurgy, Sinterability

1. Introduction

The internal combustion engine is a significant source of CO_2 emissions. Light weight is one of the most effective routes to reduce CO_2 emissions from vehicles. It was found that 100 kg weight saving in a car is equivalent to a reduction of 9 grams of CO_2 per kilometer [1]. Therefore, this leads to the increased usage of light-weight aluminium alloys in the automotive industry.

Al-Si alloy powder metallurgy (P/M) is a high wear resistance light-weight alloy. The mechanical properties of the P/M parts are comparable to wrought or cast alloy [2]. However, sintering of aluminium and its alloys powder is difficult due to the presence of the extremely stable aluminium oxide layer covering the powder. Hence, the atomic diffusion between each particle is prevented. The compact is said to have "poor sinterability" as the powders are weakly bonded with each other. Poor sinterability results in low sintered density and poor mechanical properties [3].

However, it is still possible to sinter and improve the sinterability of aluminium. There are several factors that affect the sinterability of aluminium, for example, the presence of magnesium in an alloying composition. It was found that addition of less than 1%wt. of magnesium will facilitate the sintering of aluminium by disrupting the oxide film through the formation of spinel ($MgAl_2O_4$) [4]. Sintering parameters, such as sintering atmosphere and moisture can also influence the sinterability.

Therefore, this work investigated the effects of sintering conditions on the sinterability of Al-Si-Cu-Mg alloy by varying the sintering conditions. Properties indicating sinterability such as sintered density, dimensional change characteristic, hardness and microstructure are shown and discussed.

2. Experiments

The pre-alloyed aluminium powder used in this study was Alumix 231 (Al-14.9Si-2.4Cu-0.55Mg), which was air-atomised and supplied by ECKA Granulate Velden GmbH, Germany. The powder was admixed with 1.5%wt. of Amide wax (Ethylene bisstearamide). Scanning electron image of this powder is shown in Fig. 1. The powders have irregular shape and they are agglomerated by the admixed lubricant. The particle size distribution is listed in Table 1.

Alumix 231 powder was compacted into tensile specimens. In order to obtain the green specimens with the green density of 2.50 g/cm^3, or 93.6% theoretical density (TD), compaction pressure of 700 MPa was needed [7]. It is noted that the theoretical density of Alumix 231 is 2.67 g/cm^3.

After compaction, dewaxing was carried out in nitrogen atmosphere at 420°C for 60 minutes [7]. Subsequently, sintering was continued in nitrogen atmosphere, at 560°C for 60 minutes [7]. The heating rate for these thermal processes was 5°C/min.

Different sintering conditions were achieved by adjusting the level of vacuum applied before heating, the atmospheric controlling conditions, the purity of nitrogen gas, sintering chamber and chamber capacity as shown in Table 2. Moisture within the furnace during sintering was also measured.

As-sintered density was measured by Archimedes's method according to the Metal Powder Industrial Federation (MPIF) standard no. 42. Dimensional change of the specimen was measured and compared to the corresponding green state. Hardness test in HRF scale according to the MPIF standard no. 43 was carried out using Instron Wolpert 930/250 hardness tester. In addition, the tensile test according to the MPIF standard no. 10 was examined using the Instron 8801 universal mechanical testing machine. Microstructure was observed using the optical microscopy. The samples were cut, mounted, mechanically ground with silica papers and finally polished with diamond suspension solution respectively.

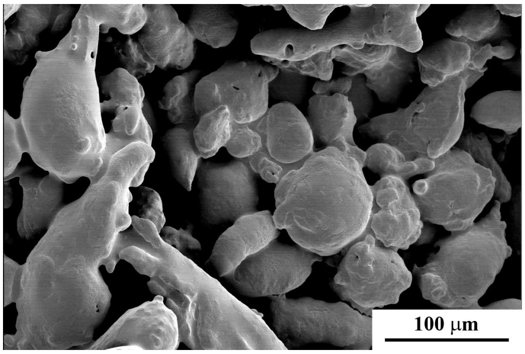


Fig. 1 Scanning electron image of Alumix 231 powder.

Table 1 Powder size distribution of Alumix 231 [7].

Size (μm)	%
< 45	35.6
45-63	24.9
63-100	25.3
100-160	12.0
160-200	1.7
200-250	0.5

Table 2 Sintering conditions.

Conditions	Level of vacuum applied before heating	Atmospheric controlling	Purity of nitrogen gas (%)	Nitrogen flow rate (l/min)	Sintering chamber	Chamber capacity (litres)
A	Low	Pressure	99.99	6	Graphite box	165
B	Low	Pressure	99.999			
C	High	Pressure	99.999			
D	-	Flow	99.999	4	Ceramic tube	5.3

3. Results and Discussion

3.1 Effects of Sintering Conditions on Dew Point Temperature

From the different sintering conditions shown in Table 2, the corresponding dew point temperatures, which are related to the level of moisture within the furnace, were obtained and shown in Fig. 2. It suggests that sintering with high purity nitrogen (99.999%, condition B) results in lower dew point temperature than those sintered in 99.99% purity (condition A). Higher purity nitrogen gas has lower concentration of water vapour in the gas mixture. Moreover, applying high level of vacuum before the heating started (condition C) did not significantly improve the dew point temperature when compared with condition B. However, it was found that the dew point temperature was significantly improved by using the ceramic-tube furnace with the permanent flow of nitrogen gas as shown condition D.

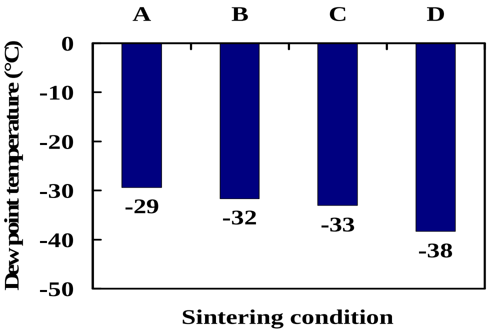


Fig. 2 Furnace dew point temperatures of the nitrogen atmosphere corresponding to the various sintering conditions.

Flow controlling atmosphere gave lower dew point temperature than pressure controlling atmosphere due to the water vapour in the atmosphere being continuously swept out by the permanent flow of gas. In contrast, for pressure controlling atmosphere, the pressure of the sintering atmosphere was always kept constant. Therefore, the rate of sweeping out of water vapour was less, although the gas flow rate was lower in condition D.

Applying a higher vacuum state before heating up did not significantly decrease the dew point temperature during sintering because of the limitation of the sintering equipments. It was suggested that for the furnace with the 42.5-litre chamber, the suitable gas flow rate in order to maintain a -40 to -50 °C furnace atmosphere dew point, should be 28-47 l/min [8]. However, the gas flow rate through the chamber was only 6 l/min compared to the chamber size of 165 litres as shown in Table 2.

3.2 Effects of Sintering Conditions on Physical Properties

The effects of these sintering conditions on the density of the sintered specimens are shown in Fig. 3. It shows that the sintered density of about 2.51 g/cm^3 (94%TD) can be obtained for the specimens sintered in condition A with the dew point of -29°C. The sintered density increased as the dew point temperature decreased. When the specimens were sintered in condition D with the dew point of -38 °C, a 2.62 g/cm^3

(98%TD) was obtained. The increase in density after sintering suggests that shrinkage occurred.

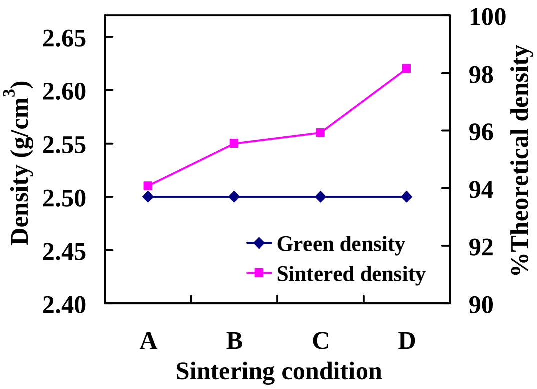


Fig. 3 Variations of sintering conditions on the sintered density of Alumix 231.

Consequently, the shrinkage of the specimens was evaluated by comparing the dimensional change of the sintered specimens and their corresponding green state. Fig. 4 shows the shrinkage at indicated positions and compares with others from different sintering conditions. The specimens sintered in condition A shows the least shrinkage at any position. The specimens sintered in conditions B and C show similar shrinkage and the largest shrinkage was observed for the specimens sintered in condition D. Hence, the shrinkage is in good agreement with the as-sintered density.

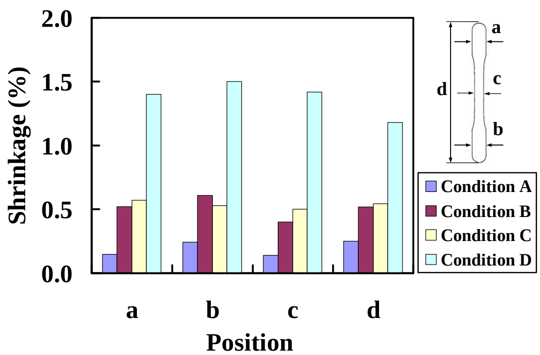


Fig. 4 Shrinkage of the sintered specimens from their green dimension.

3.3 Effects of Sintering Conditions on Mechanical Properties

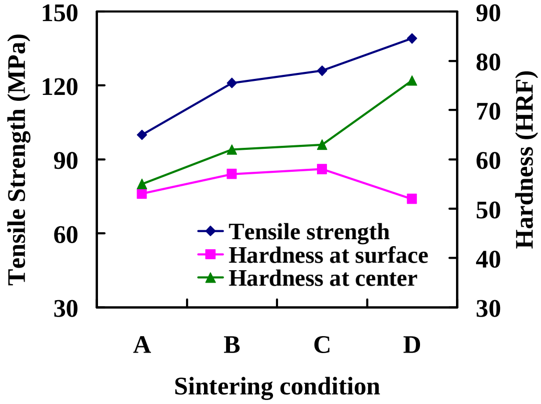


Fig. 5 Tensile strength and hardness tested at the surface and centre of the specimens sintered in various conditions.

Fig. 5 shows that the effect of sintering conditions on tensile strength of the specimens. The best and worst tensile strength were observed on specimens sintered in condition D (lowest dew point) and condition A (highest dew point) respectively. The decrease in dew point temperature caused the powders to densify and shrink more. Although the tensile results varied with the sintering conditions, hardness of the specimens measured at the specimen surface was nominally constant. The results of hardness are contradicted with other properties, especially for the specimens sintered in condition D. This can be explained later by the microstructural study and the measurement of hardness at the centre of the specimens.

3.4 Effects of Sintering Conditions on Microstructure

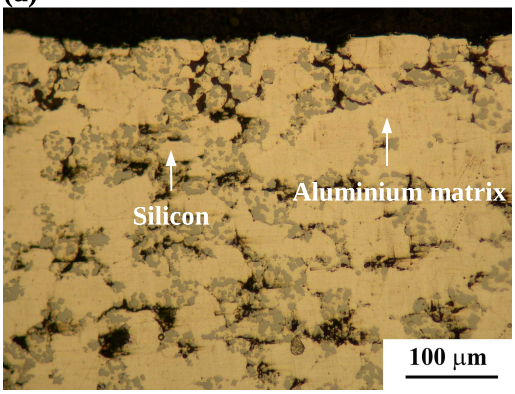
From the microstructural images of Alumix 231 specimens sintered in various sintering conditions shown in Fig. 6, the microstructure consists of a silicon phase dispersed in an aluminium matrix. It is suggested that this aluminium alloy shows the “duplex” microstructure [9], which occurs by the penetration of the master alloy through the aluminium matrix during sintering.

All observed microstructures in this study show that the sinterability was relatively poor near the specimen surface (Fig. 6 (a), (c), (e), and (g)), resulting in the lower hardness, while the centre of the specimens showed better sinterability (Fig. 6 (b), (d), (f), and (h)). The observation is in good agreement with results by Casellas, *et al.* [10], who observed a porous layer near the surface of sintered aluminium alloy.

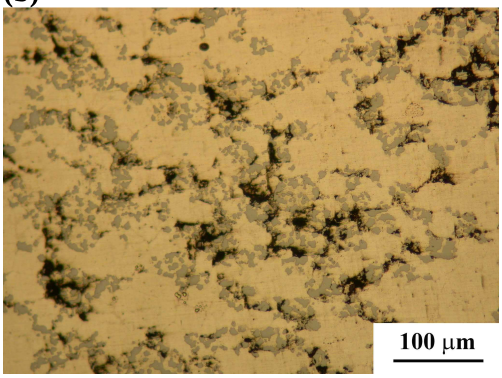
It was suggested that during sintering of aluminium powder, the aluminium powders at the surface layer acted as self-gettering [5]. During sintering, the gas flow rate inside the compacted specimen was much lower than at the surface. Hence, the powders at the surface consumed more oxygen causing the lower oxygen partial pressure in the inner section. This will allow the powders in the inner section to sinter better.

From Fig. 6, it can also be seen that as the dew point temperature decreased, the sinterability improved. This can be observed by the reduction of pores and the dispersion of silicon phase in the aluminium matrix. The specimens sintered in the highest dew point atmosphere (Fig. 6 (b)) had pores distributed throughout the specimens and the shape of the pores was irregular. This indicated that there was low level of densification for the compacts. The specimens sintered in lower dew point atmosphere (Fig. 6 (d) and (f)), had small and rounded pores, and higher densification. However, the dispersion of silicon phase within the aluminium matrix in these two specimens was poor. Fig. 6 (h) shows the microstructure of the specimens sintered in the lowest dew point atmosphere. It shows the compact with good sinterability, low porosity and well-dispersed silicon phase.

(a)



(b)



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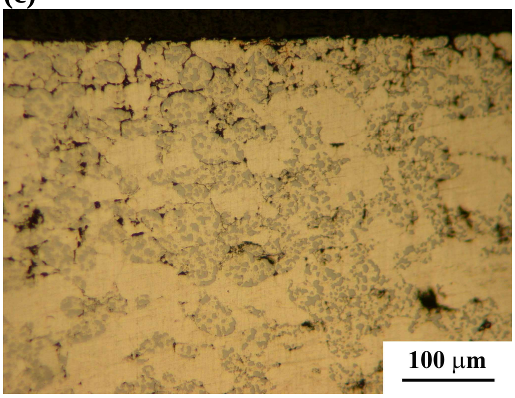
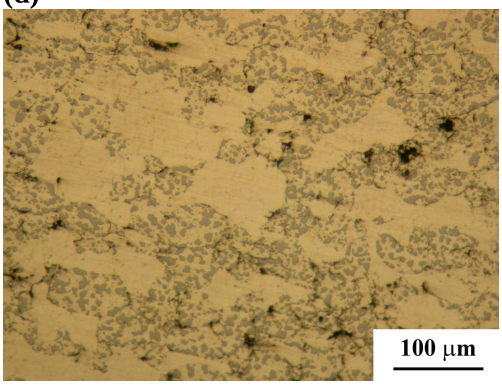


(d)



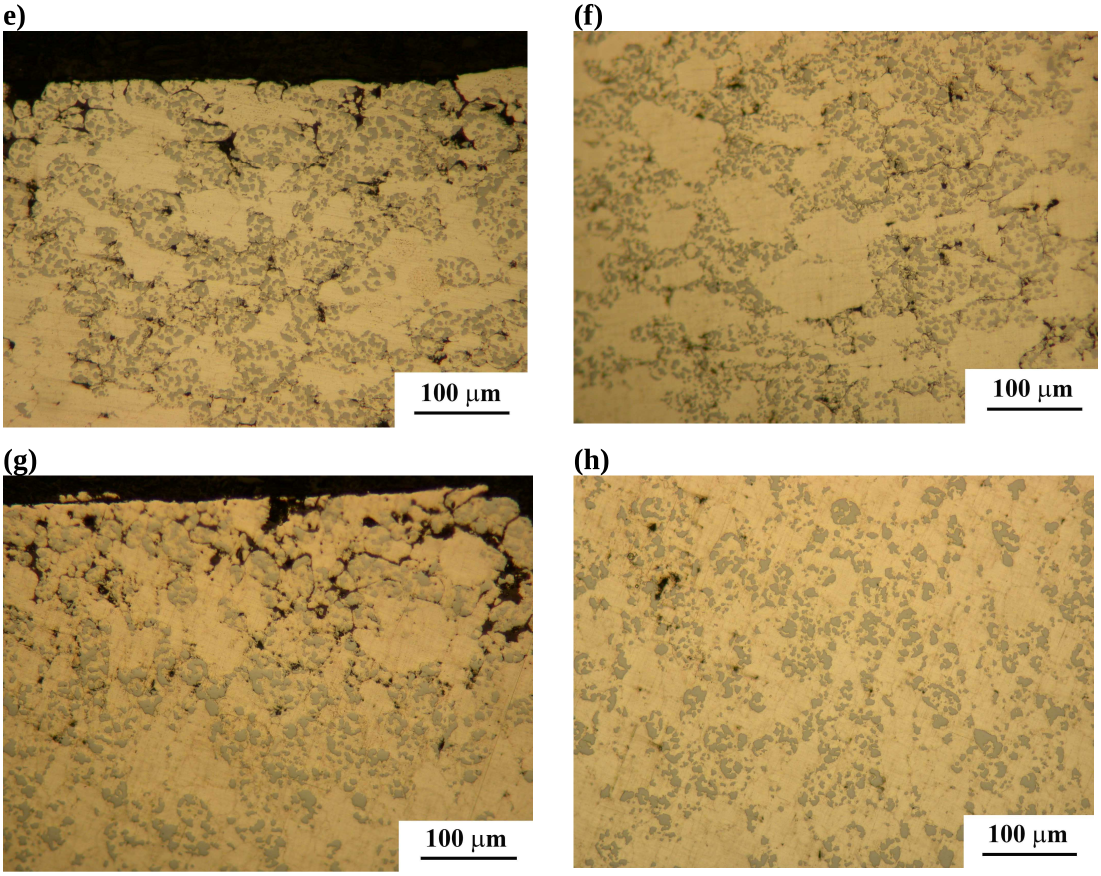


Fig. 6 Microstructural images of Alumix 231 specimens sintered in various conditions; condition A (a) and (b), condition B (c) and (d), condition C (e) and (f), and condition D (g) and (h). The left images show the microstructure at the area near the specimen surface, while the right images show the microstructure at the centre of specimens.

The unexpected lower hardness at the specimen surface observed in Fig. 5 for condition D could be explained as the result of the low sinterability of aluminium alloy powder at the surface region as shown in Fig. 6 However, in the central region, it shows better sinterability. Therefore, the hardness of the specimens measured at the centre of the specimen is also shown in Fig. 5 For all sinter conditions, the hardness at the specimen centre was higher than those at the surface associated with the self-gettering phenomenon. It is noted that the trend of hardness measured at the specimen centre is similar to the trend of the tensile strength. Hence, the hardness at the specimen centre is a better representative. In addition, the specimens sintered in the lowest dew point atmosphere (condition D) showed the highest hardness of 76 HRF, while the lowest hardness was observed to be 55 HRF for the specimens sintered in the highest dew point atmosphere (condition A). Sintering of Alumix 231 powder in a lower dew point atmosphere caused the more effective dispersion of hard phase (silicon) in the aluminium matrix. Therefore, higher hardness result was observed.

4. Conclusion

Sintering conditions can affect furnace dew point temperature and hence can significantly affect the sintered properties of aluminium alloy. The best sintering condition for Al-Si-Cu-Mg alloys was 99.999% purity nitrogen atmosphere permanently flowing at sufficient flow rate. This condition resulted in the lowest dew point temperature and improved the final properties, such as density, tensile strength, and hardness. Microstructure of the Al-Si-Cu-Mg alloys showed poor sinterability of the powders at the surface region due to the self-gettering phenomenon. However, at the centre section, the sinterability was better. Well sintered parts showed microstructure with more homogeneous dispersion of silicon phase and less pores throughout the specimens.

5. Acknowledgement

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6. References

[1] European Aluminium Association, Aluminium in Cars, [Downloadable] http://www.eaa.net/en/applications/automotive/aluminium-in-cars/, 2009.

[2] Vukcevic M., and Delijic K., Some New Directions in Aluminium-Based PM Materials for Automotive Applications, Materiali in Tehno-logije, Vol. 30, pp. 101-105, 2002.

[3] Tang F., Anderson I. E., and Biner S. B., Solid State Sintering and Consolidation of Al Powders and Al Matrix Composites, Journal of Light Metals, Vol. 2, pp. 201-214, 2002.

[4] Lumley R. N., Sercombe T. B. and Schaffer G. B., Surface Oxide and the Role of Magnesium During the Sintering of Aluminum, Metallurgical and Materials Transactions, Vol. 30A, pp. 457-463, 1999.

[5] Schaffer G. B. and Hall B. J., The Influence of the Atmosphere on the Sintering of Aluminum, Metallurgical and Materials Transactions, Vol. 33A, pp. 3279-3284, 2002.

[6] Schaffer G. B., Hall B. J., Bonner S. J., Huo S.H., and Sercombe T.B., The Effect of the Atmosphere and the Role of Pore Filling on the Sintering of Aluminium, Acta Materialia, Vol. 54, pp. 131-138, 2006.

[7] Technical Data Sheet. Certificate of Analysis. ECKA Granulate Velden GmbH., 2008.

[8] ASM Handbook of Powder Metallurgy, ASM International, Vol. 7, p. 381, 1984.

[9] Neubing H. C., Gradl J., and Danninger H., Sintering and Microstructure of Al-Si P/M Components, Adv. in Powder Metallurgy and Particulate Materials, Vol. 13, pp. 128-138, 2002.

[10] Cassellas D., Beltran A., Prado J. M., Larson A., and Romero A., Microstructural Effects on the Dry Wear Resistance of Powder Metallurgy Al-Si Alloys, Wear, Vol. 257, pp. 730-739, 2004.