MICROWAVE SINTERING OF ALUMINUM ALLOYS

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ABSTRACT

Until 2000 almost all research in the microwave sintering area was confined to non-metallic materials. However, after the first report by Penn State in 1999[1] on full sintering of steel powders in microwave, now in the last few years there has been increasing interest in applying microwave energy for processing of variety of metallic materials. The present study is an extension of this work and relates to the sintering behavior of aluminum alloy powders. Blended 2712 (Al-Cu-Mg-Si-Sn) and 6711 (Al-Mg-Si-Cu) alloy powders were consolidated by microwave sintering through temperature range of 570 to 630 °C for 1 hr in vacuum, nitrogen, argon and hydrogen atmospheres. The influence of sintering temperature and atmosphere on densification response were investigated in comparison with conventional sintered parts. Microwave sintering enhanced the densification response in shorter times and lower sintering temperature in turn leading to better properties.

Keywords: P/M Al alloys; microwave sintering, vacuum atmosphere, phase analysis, swelling.

INTRODUCTION

The application of P/M aluminum alloys has been increasingly used in automotive industry, where low cost and light weight materials are the essential design criteria. The P/M aluminum alloys are replacing small Al die casting which requires precision net shaping and better material utilization [1]. Sintering of aluminum alloys were considered to be very difficult due to the presence of the thermodynamically stable oxide layer on powder particle surface, which required an oxygen partial pressure of $<10^{-50}$ atm at 600°C to be reduced [2]. However, it has been demonstrated that nearly full density can be obtained by the newly emerging technology of microwave sintering due to its inherent uniqueness and advantages [3]. Microwave heating has several advantages over conventional sintering process such as rapid heating rates, short cycle time, improved product quality and environmentally friendly [4]. Microwave sintering has been extensively used for consolidation of ceramics and hard metals, until Roy *et.al* [4] for the first time showed effective coupling of microwaves and metals in powder form. Later on many researchers have reported microwave sintering of many powdered metal compacts and alloy compositions (steel, stainless steel, Cu, Al, Ni, Ni, Mo, Co, Ti, W, WC, Sn etc) to nearly full dense bodies in turn better properties [5-14]. W. Wong *et.al* [15] have reported the two-directional heating for sintering of materials like Mg, Al and lead free solder for short time. In the present work, an attempt has been made for effective sintering to take place by combining protective atmosphere and novel microwave sintering technique has been employed for improving densification and properties.

EXPERIMENTAL DETAILS

The prealloyed 6711 and 2712 Al alloy powders with particle size [d_{50} = 83.58 and d_{50} = 104.75] respectively was supplied by AMPAL Inc., U.S.A. The typical SEM micrograph of aluminum alloy powder resulted in spherical morphology as it was processed by gas atomization process as shown in Fig.1a. The alloy powders were compacted at 200MPa using a uniaxial press (model: CTM-50; supplier: FIE, Icharanji, India) to cylindrical pellets (diameter: 16 mm and height: 6 mm to green densities of 96.42 ± 0.97. Before sintering, delubrication was carried out in conventional vacuum furnace at 350°C for 6hrs in vacuum range of 10^{-2} to 10^{-3} Torr. The microwave sintering was carried out using 2.54Hz, 6kW multimode furnace in vacuum, nitrogen, argon and hydrogen, respectively at temperature ranging from 570 to 630 °C for 1 hr. Conventional sintering of green compacts were carried out in a SiC heated horizontal tubular sintering furnace (model: OKAY 70T-7, supplier: Bysakh, Kolkata, India) at constant heating rate of 5°C/min at above mentioned atmosphere and temperatures. The thermal cycle profiles for microwave and conventional sintering of aluminum alloy is shown in Fig.1b. The sintered densities of samples were measured using Archimedes' method. The densification parameter (D.P) is expressed as in equation (1):

$$D.P = \frac{(\text{sintered density - green density})}{(\text{theoretical density - green density})} \qquad (1).$$

The TGA analysis was carried on the green compacts before and after delubrication at 10°C/min in nitrogen atmosphere at 700°C, which shows the complete removal of lubricant as shown in Fig.1(c).

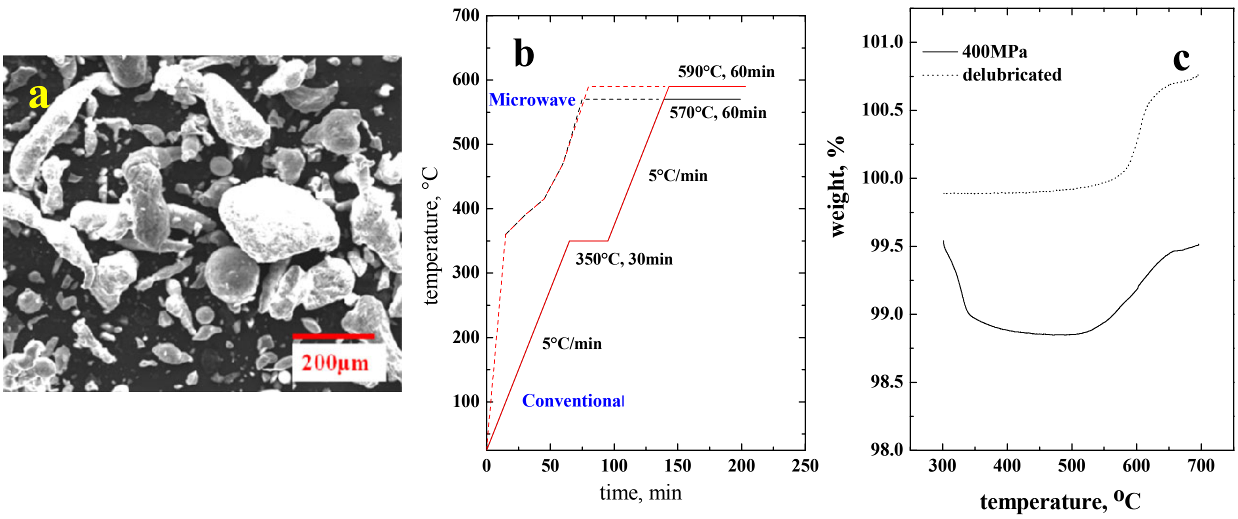

Fig.1 (a) SEM of typical Al alloy powder (b) Thermal cycle for microwave and conventional sintering of Al alloy (c) TGA analysis of Al alloy compact before and after delubrication.

RESUTLS AND DISCUSSION

Fig 2(a) shows the densification response of conventional and microwave sintered samples of Al-3.8Cu-1Mg-0.8Si-0.3Sn (2712) alloy. It can be seen that sintered density increases with sintering temperatures for all cases in microwave in vacuum atmosphere as compared with conventional parts. The vacuum sintering normally produces higher densities, since sintered alloy does not emit any vapor to fill pores or no decomposition occurs, and the densification happens by unimpeded trapped gases [16]. Vacuum sintered are hypothesized to have closed pores and less porosity which can be confirmed on further metallography investigations.

The higher sintered density was observed for microwave nitrogen sintered samples at 590°C and thereafter a decreasing trend, whereas conventional samples resulted in better densification at higher temperatures (630°C). At lower sintering temperature, Al-Cu alloys experiences either swelling or shrinkage behavior. The higher sintering temperature results in initial swelling; followed by shrinkage such that alloy attains the solid-liquid state region [17]. It can be seen that microwave sintered samples in N_2 decrease in density after 590°C, due to the faster heating rate during microwave heating, Cu, Mg, Si have less time to diffuse into Al lattice and therefore the spreading of liquid phase reduces causing to slow down the homogenization process[18]. Hence it can be hypothesized that microwave sintering in N_2 at 590°C has attained an alloy homogenization, and contained sufficient volume fraction of permanent liquid.

Conventional and microwave samples sintered in Argon atmosphere showed an increasing trend in density with sintering temperatures, except at 630°C for microwave case. The samples sintered in H_2 also followed similar trend as nitrogen sintered ones, with higher density at 590°C, thereafter decreasing trend in density. The densification parameter was observed to be positive for vacuum sintered samples for conventional mode at 630°C and at temperatures higher than 590°C for microwave mode as shown in Fig.2 (a). Also nitrogen microwave sintered samples at 590°C resulted in slighter swelling as compared to other sintering temperatures and heating mode. The argon and hydrogen sintered samples exhibited more or less the similar swelling behavior slightly higher than nitrogen and vacuum sintered samples in both heating modes.

The swelling behavior of above alloy can be well explained by dilatometric experiments as seen in Fig.3 (a). From the dilatometeric curve for 2712 alloy, initially compacts undergo swelling at lower temperature and therafter additional amounts of liquid formation occurs on melting of Mg particles, dissolution of Cu and Si in liquid, and formation of intermetallics [19]. The maximum swelling started occurring at 510°C; which may be due to the transient liquid formation, followed by diffusing of the alloying elements Cu an Mg into aluminum matrix leaving behind the secondary pores leading to additional compact swelling behavior [16].

Another eutectic reaction occurred at 548°C which is not accompanied by sample swelling, which led to change in slope of dilatometry curve. The shrinkage occurred thereafter due to large amount of liquid phase formation [17]. At higher sintering temperatures (590°C), larger volume fractions of liquid results in enhanced densification. The densification mechanism of vacuum sintered differs from nitrogen sintered samples in case of both heating modes; where densification occurs by pore filling mechanism in later stages of sintering for latter case [2]. The reduced density of argon and hydrogen sintered samples in both heating modes, was mainly due to the presence of entrapped gases inside the pores, leading to inhibition of densification and inferior properties. Microwave sintered 2712 alloy resulted in enhanced densification in vacuum atmosphere followed by nitrogen, argon and hydrogen as compared to conventional.

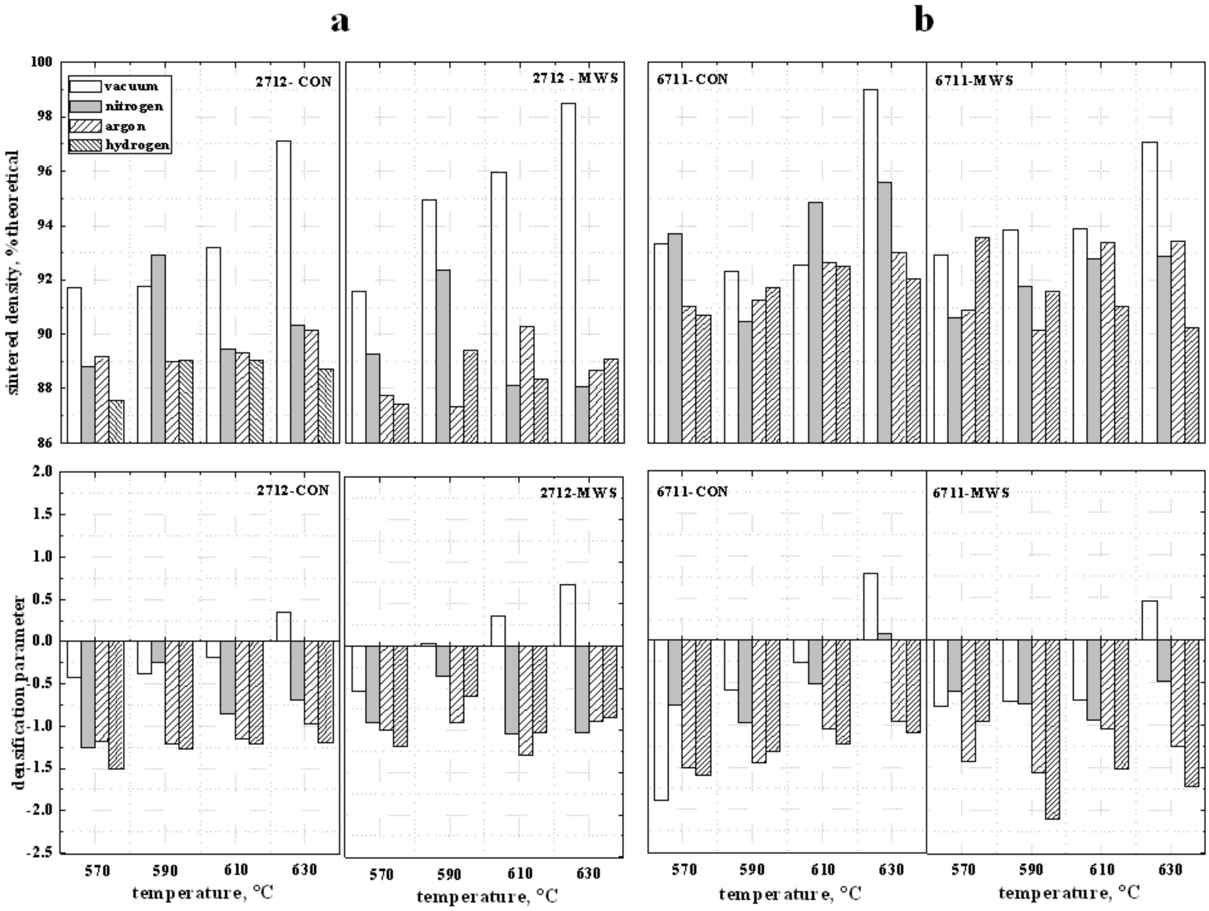

Fig.2 Effect of sintering atmosphere and sintering temperature on variation in sintered density and densification parameter of microwave sintered compared with conventional (a) 2712 alloy (b) 6711 alloy.

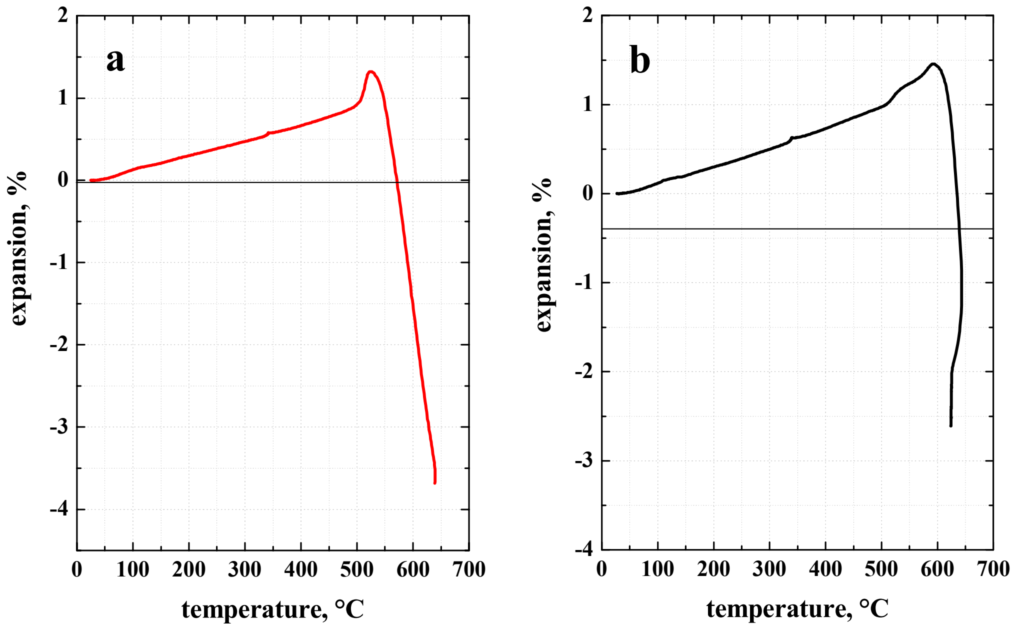

Fig 3 Dilatometric curves for Al alloys conducted at 3°C/min in nitrogen atmosphere (a) 2712 alloy (b) 6711 alloy respectively.

Densification response of 6711 alloy as shown in Fig.1 (b), vacuum sintered (conventional and microwave heating mode); showed an increasing trend in density with sintering temperature. The nitrogen sintered samples showed an increasing trend in density with sintering temperature, with higher density at 630°C. Microwave vacuum sintered samples exhibited slightly lower density as compared to conventional parts. The argon sintered samples in both heating modes, showed increased density with sintering temperatures, where microwave and conventional sintered samples showed more or less same density. In hydrogen sintered conventional samples also followed an increase in density upto 610°C and slight decrease therafter; whereas microwave sintered ones showed higher density at lower temperature (570°C) and thereafter decreasing density as compared with conventional.

The shrinkage behavior was observed for vacuum and nitrogen sintered samples in case of both heating mode at 630°C, except for nitrogen microwave sintered samples. The densification behavior followed a similar trend as sintered density, swelling behavior was observed for almost all samples. The microwave argon and hydrogen sintered samples was found to have slightly more compact swelling as compared to conventional parts. The swelling behavior can be well explained from the dilatometric studies as seen in Fig. 2(b).

From Al-Mg phase diagrams; initial swelling behavior occurred at lower temperature due to the formation of eutectic liquids at 450°C and 437°C, and formation of intermetallics $Al_{12}Mg_{17}$ and Al_3Mg_2. The swelling may also be contributed due to the dissolution of Mg in Al solid solution, where secondary porosity forms at sites of Mg particles leading to additional swelling [20]. It was found that at 550-595°C, shrinkage of alloy continued showing a change in slope of the dilatometeric curve, replacing swelling, may be attributed to formation of Al_3Mg_2 intermetallics. The shrinkage become prominent, as temperature was increased to 630-640°C [21].

CONCLUSONS

For the first time, Al-Cu-Mg-Si-Sn and Al-Mg-Si-Cu alloys have been successfully sintered in microwave furnace. The microwave sintering of aluminum alloys resulted in saving of sintering time up to 50% as compared with conventional sintering. The absence of swelling in microwave vacuum sintered samples combined with higher sintering temperature showed the further chance of extending their use in industrial applications.

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