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Research Article

Properties Evolution during Transient Liquid Phase Sintering of PM Alumix 431

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This paper reports a study on densification and microstructural changes, which were occurred during transient liquid phase sintering of Alumix 431. The objective was to find the optimum sintering temperature for obtaining higher densities and improved mechanical properties. For this purpose, the commercial Al powder mixture ECKA Alumix 431 was pressed at 400 MPa and sintered under pure nitrogen in a broad range of temperature between 580 and 620°C for 30 minutes in N_2. The effect of sintering temperature was evaluated by measuring sintered density, transverse rupture strength, dynamic Young's modulus, and macrohardness. The optical microscopy and scanning electron microscopy used for studying microstructural characterization and fracture surface of the resulting materials, respectively. It could be observed that the sintered density increases with higher sintering temperature up to a certain limit, rearrangement as well as pore elimination due to transient liquid phase sintering being helpful. On the other hand, too high sintering temperatures are unwelcome either apparently as a consequence of Zn evaporation and microstructural change such as grain growth. It was found that the maximum sintered density for Alumix 431 is attained by sintering at the range of 610°C when the amount of liquid phase is satisfactory.

1. Introduction

Lightweight metals are finding increased use in the P/M industry due to their unique physical and mechanical properties. Aluminum P/M parts because of high strength-to-weight ratio, corrosion resistance, and superior finishing properties, are used in business machine, automotive, aerospace, and appliance applications [1].

Sintering of aluminum is complicated by the presence of a thermodynamically stable oxide layer, which limits diffusion and hinders wetting and liquid spreading. The powder used in this work was produced by Eckart-Dorn under the designation of Ecka Alumix 431 while it corresponds to the 7XXX series. Zinc and magnesium are the main alloying elements in this powder; high Zn: Mg ratios produce the best strength and response to heat treatment, together with the highest susceptibility to stress corrosion. Low ratios produce the best weld ability and the lowest quench sensitivity [2].

Magnesium is known to react with the oxide, and it therefore plays a major role in the sintering of aluminum. The atmosphere is also known to be important, and nitrogen is widely regarded as necessary [3–6].

For studying the densification of bulk powder, one should include all manufacturing steps in a careful analysis, starting from pressing the metal powder in a die; another attempt should be done to analyses changes in the compact taking place during presintering and sintering. Densification due to liquid phase sintering is typically considered to be combination of rearrangement, solution-reprecipitation, contact flattening, pore filling, and solid state sintering. The progression of sintering and the final porosity in this system is also dependent on the process variables such as additive particle size, heating rate, and final sintering temperature [7].

In this paper the densification behavior of EA431 at different sintering temperature was investigated and the optimum sintering temperature was obtained.

2. Materials and Methods

The material used in this work is the commercial prealloyed Al powder (ECKA Alumix 431) whose characteristics are summarized in Table 1. The powder mixture was cold pressed at 400 MPa in rigid steel dies. The resulting green parts are bars with 12 mm in height and 12 mm in width and 100 mm in length. A minimum of three samples was tasted for each sintering temperature to ensure data reproducibility. To remove the lubricant, samples were delubricated at 400°C for up to 30 minutes in a high-purity nitrogen atmosphere. Sintering was conducted in flowing N_2 of 99.999% purity (flow rate 2 L/min) at different temperatures between 580°C and 620°C for 30 minutes. The effect of sintering temperature was evaluated by measuring sintered density, transverse rupture strength, dynamic Young's modulus, and apparent (= macro-) hardness. Densities of green compacts were determined from measurements of the mass and the dimensions of the compacts, while those of the sintered compacts were determined using Archimedes principle (DIN ISO 3369).

The transverse rupture strength was determined in 3-point bending, with the distance between supports being 25.4 mm, using an universal testing machine Zwick 1474. The apparent (= macro-) hardness was measured on an EMCO M4U-025 tester. Metallographic sections were prepared following standard procedure. To make sure that the pore structure was properly shown, the specimens were resin impregnated before polishing. The sintered microstructure was investigated by optical microscopy. Fracture surface analysis was done on a scanning electron microscope JEOL 6400. The dynamic Young's modulus was determined using a resonance system, and evaluated according to ASTM E 1876-99.

3. Results and Discussion

The optimum temperature for sintering Alumix 431 was defined on samples that were sintered for 30 minutes in N_2 at different temperatures, with criteria being the resulting properties such as sintered density, sintered transverse rupture strength, dynamic Young's modulus, and apparent (= macro-) hardness of the sintered samples. The obtained sintered properties are listed in Table 2. They give a good reference to the process taking place at the sintering step.

3.1. Density and Microstructure. In Figure 1 the sintered density of samples prepared for measuring the transverse rupture strength and dynamic young module is shown graphically. The average green density of uniaxially compacted alumix 431 at 400 MPa is 2.65 g/cm^3. Maximum obtained sintered density is 2.76 g/cm^3, which indicated that the densification value (Φ) as relative to the pore-free density (ρ_t) is 35.14%:

$$\Phi = \frac{\rho_s - \rho_g}{\rho_t - \rho_g} * 100. \quad (1)$$

At lower sintering temperatures, in the range of 580°C up to 610°C, the sintered density increases. The maximum

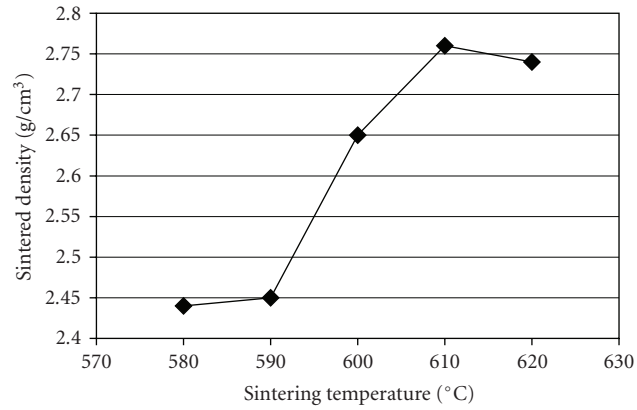


Figure 1: Sintered density as a function of sintering temperature (sintering 30 minutes in N_2).

increase in sintered density is found at the range of 610°C sintering temperature. It stands out clearly from the obtained microstructures (Figure 2) that the highest sintered density is attributed to the transient liquid phase sintering that occurs at 610°C. Finally, a negligible decrease in sintered density is seen after sintering at 620°C.

Here the main obstacle against densification seems to be Zn evaporation, the increases of volume fraction of liquid, and consequently grain growth (Figure 2).

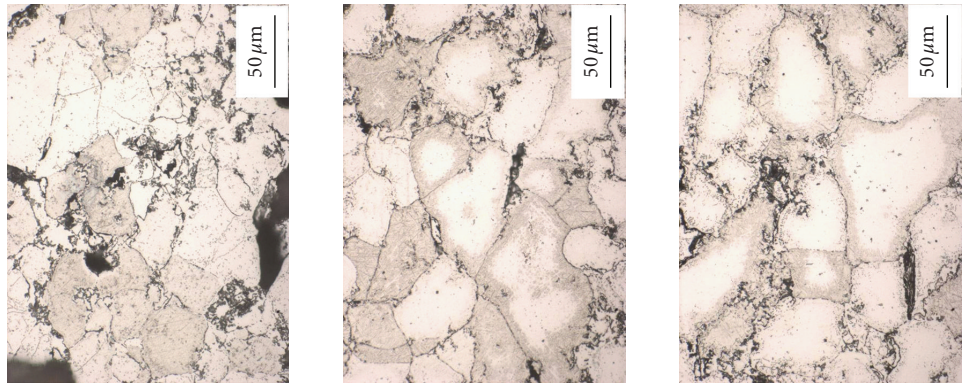
The results demonstrate that high sintered densities are possible with certain amounts of liquid formation during sintering [8]. When studying the microstructure of sintered samples, the main interest was focused on the pore elimination, grain growth, and shape accommodation. Generally with increasing sintering temperature the amount of liquid phase increases. Created liquid phase leads to fragment rearrangement, coarsening, and eventual pore elimination.

The microstructures corresponding to sintered EA431 at 610°C (Figure 2), illustrates the steps associated with transient liquid sintering. At sintering temperatures above 610°C, the microstructure has large grain size and limited porosity. As shown in Figure 2, relatively thick layers are seen at grain boundaries around the course grains arising from transient liquid phase sintering and subsequently precipitation of some alloying elements.

3.2. Fractography. The obtained fracture surface from TRS test specimens gives a good reference to the corresponding properties achieved in consequence of pore elimination at different sintering temperatures. Significant changes in fracture morphology occur with increasing sintering temperature. Comparing the fracture surfaces of sintered Alumix 431 at different temperatures (in the range of 580 up to 620°C) showed that the sintered sample at 610°C has smoother surface than the others. Such smooth fracture surface as the consequence of sintering process at 610°C can be related to created proper amount of liquid phase and pore elimination (Figure 3).

Table 1: Typical chemical composition and physical characteristic of Alumix 431.

Typical chemical composition							
Material	Alloy	Cu%	Mg%	Si%	Zn%	Wax lubricant	ECKA aluminium
Alumix 431	AlZnMgCu	1.5	2.5	—	5.5	1.2	Remainder
Typical physical characteristic							
Apparent density [g/cm^3]	Tap density [g/cm^3]		Flowability [s/50 g/5,0 mm]		Green strength [N/mm^2]		Sieve Analys [%]
1.05	1.35		<30		>8.0		<0.2 mm >97%



590°C (a) 610°C (b) 620°C (c)

Figure 2: Microstructure of EA431, compacted at 400 MPa and sintered at indicated temperatures for 30 minutes in flowing N_2.

Table 2: Mechanical properties of as-sintered Alumix 431 at different sintering temperatures (580–620°C).

Sintering temperature (°C)	Sintered density (g/cm^3)	Dynamic Youngs modulus (GPa)	Transvers rupture strength (MPa)	Macro hardness (HV 30)
580	2.44	49	331	79
590	2.45	49	345	90
600	2.65	60	462	95
610	2.76	67	564	119
620	2.74	64	473	106

3.3. Mechanical Properties

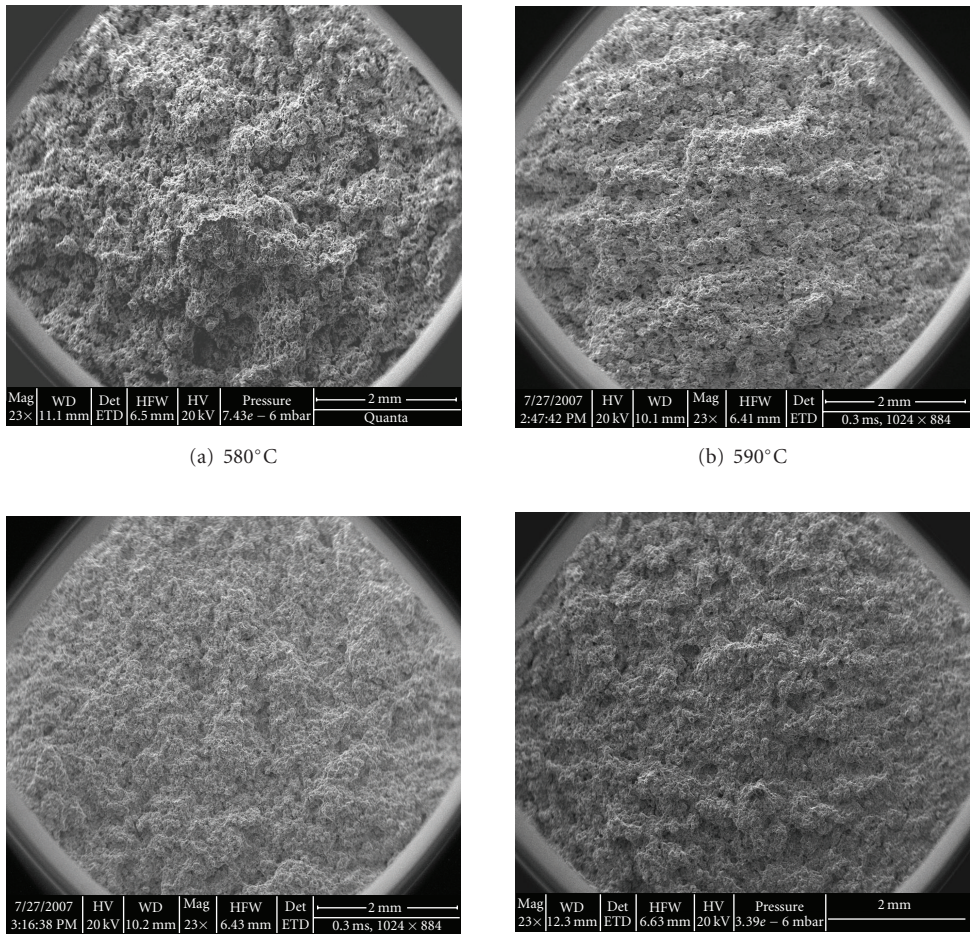
3.3.1. Transverse Rupture Strength. The obtained transverse rupture strength values of sintered samples at different temperatures are listed in Table 2, and its changes with sintering temperature are graphically shown in Figure 4. With rising sintering temperature, up to 610°C, the transverse rupture strength of sintered Alumix 431 increases evidently similar to their density. The strengthening is due to the increase of interparticle contact areas. The TRS attains its maximum after sintering at about 610°C, it is around 560 MPa. Such response of the transverse rupture strength to sintering at the mentioned temperature can be attributed to liquid phase formation followed by pore elimination.

At sintering temperature above 610°C slumping occurs. Seemingly, it is due to coarsening and a greater separation between grains.

3.3.2. Dynamic Young's Modulus. The results of dynamic young's modulus measurement for sintered samples are shown in Table 2 and are graphically depicted in Figure 5.

Figure 5 shows the effect of sintering temperature (580–620°C) on DYM of Alumix 431. The response of DYM to the sintering temperature is similar to that of density. At lower sintering temperature, in the range of 580–590°C, the DYM does not change obviously and the results are approximately the same.

Above 590°C to around 610°C the DYM increases as a result of decreasing porosity. The maximum increase in DYM is found at the range of 610°C sintering temperature. The phenomenon is due to the fact that at such higher sintering temperature the pores will eliminate, their surface becomes smooth, and consequently the load bearing cross section gets higher which also contributes to this effect [7]. In principle the DYM is dependent on the amount of hard phase and the contiguity of the hard phase. Thus the DYM will be reduced by a high volume fraction of matrix (liquid) phase [8]. Accordingly, as shown in Figure 5 with rising sintering temperature above 610°C, when high volume fraction of liquid phase creates, the DYM decreases negligibly.



(a) 580°C (b) 590°C

(c) 610°C (d) 620°C

Figure 3: Fracture surface of alloy EA431 after sintering at different temperatures.

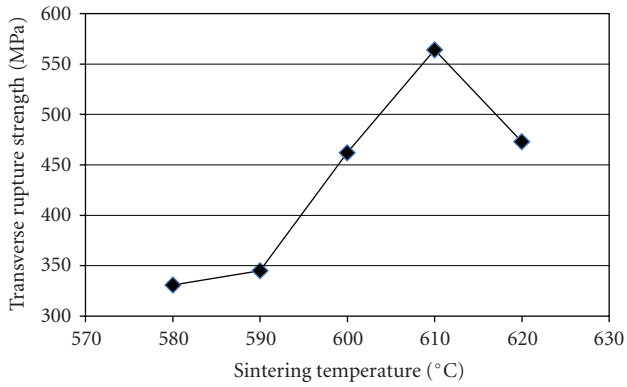


Figure 4: TRS as a function of sintering temperature.

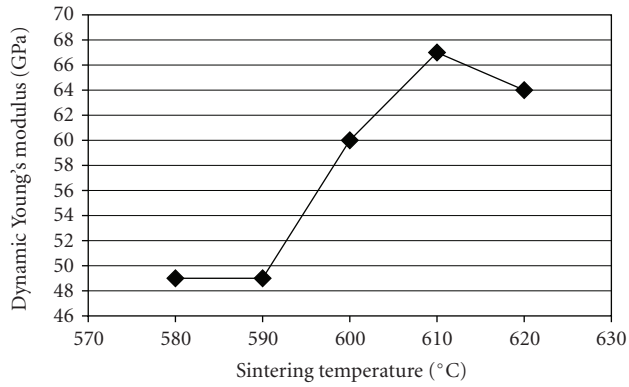


Figure 5: DYM as a function of sintering temperature.

3.3.3. Hardness. The results of macrohardness measurement with a load of 30 kg (HV 30) for sintered samples are presented in Table 2 and for better comparison are also shown in Figure 6. It is seen that when increasing the sintering temperature up to 610°C the hardness increases, seemingly following the same trend observed by their density. It is due to the fact that a high contiguity, as a consequence of optimum liquid phase sintering and greater rigidity from the solid-solid contacts, aids the hardness. On the other hand, too high sintering temperature (>610°C) should be avoided either, since grain growth as a consequence of increasing sintering temperature is detrimental, resulting in decreasing hardness.

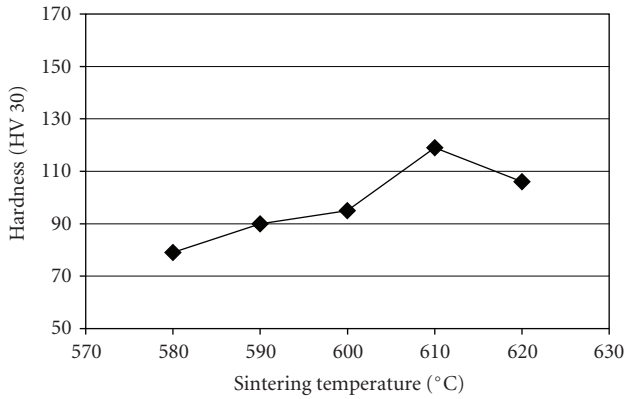


Figure 6: Effect of sintering temperature on macrohardness of sintered Alumix 431.

4. Conclusions

In manufacturing of sintered Alumix 431, liquid phase formation as a consequence of sintering temperature plays an important role in densification and resulting improved properties. This phenomenon can be explained from two view points.

(i) Regarding insufficient amount of created liquid phase: lower sintering temperatures (<610°C), is unsatisfactory since there are insufficient liquid to wet the interparticle contacts

(ii) Regarding extra amount of created liquid phase: higher sintering temperatures are also unwelcome while, at higher temperatures (>610°C) increased volume fraction of liquid increases the grain size and leads to slumping. It confirms that higher sintering temperatures can be detrimental.

(iii) Between these two antithetical extremes there is an optimum state to achieve the highest sintered density and improved mechanical properties. In this investigation, the maximum sintered density for Alumix 431 has been attained at the range of 610°C sintering temperature when the amount of liquid phase is satisfactory. The study of metallographic sections and fracture surfaces gives a good reference to the mechanism taking place during sintering.

The curves relating sintering temperature to sintered density, dynamic Young's modulus, transverse rupture strength and macrohardness are qualitatively similar and suggest that all four properties are of about equal value for predicting optimum sintering temperature. According to the resulted density, hardness, and TRS values, optimum sintering temperature for Alumix 431 was obtained around 610°C.

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