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Sintering of Al-Si-Fe-Cu-Mg-SiC powder prepared by gas atomization process

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Aluminum powder metallurgy (P/M) composites offer high mechanical properties, low coefficient of thermal expansion and good weight strength ratio with homogeneous distribution of the reinforcement phase. This work aims to investigate the mechanical properties of pre-mixed aluminum matrix composites with different chemical compositions. Mixed powers of Al-14Si-2.5Cu-0.5Mg and Al-14.5Si-1.85Cu-2.85Fe-0.8Mg with 10% volume fraction of SiC (12 μm) were used as starting powders. Reaction during sintering and chemical composition of the starting powders played important role on the sintered properties. T6 heat-treatment was carried out to produce fine precipitates to impede the movement of dislocation to increase their mechanical properties. The heat treatment involved solutionizing materials followed by water quenching and artificial aging.

Key words: Aluminum powder, Composite, Sintering, Mechanical properties, T6 Heat treatment.

Introduction

Aluminum alloys have attracted to the attention for automotive applications because of their good mechanical properties with light weight [1-3]. For the most part, recent applications about of Al alloys tended to be centered on the use of casting or wrought method [4-5]. Relatively little research has been carried out on the sintering of aluminum alloys which is still in its early stage. Aluminum metal matrix composite (MMC) with ceramic particles are also generally produced by the casting methods [6-7]. In these methods, the ceramic reinforcements occur in intra grains or inter-grain boundaries with the inhomogeneous distribution in the aluminum matrix. Therefore, there are some limitations to get enhanced mechanical properties.

During the sintering of aluminum powder, the role of nitrogen gas and magnesium needs to be considered importantly. Aluminum particles always covered with thin aluminum oxide layer and this oxide cannot be reduced by the sintering atmosphere. Several sintering researches on Al alloys were carried out through the formation of a liquid phase that disrupts the stable aluminum oxide in nitrogen atmosphere [5-8]. The interaction between the aluminum powders and nitrogen gas have been analyzed, but the reaction mechanism with nitrogen gas and aluminum powders to form AlN still remains unclearly [9, 10]. It was reported that magnesium plays an important role to break up the stable alumina layers and make a liquid aluminum. Magnesium can help to break up the alumina layers on the surface of the aluminum powders through the formation of a spinel phase $MgAl_2O_4$ [11]. For these reasons, it is possible to increase the sinterability and mechanical properties of aluminum P/M alloys.

Although the previous researches offered the possibility of P/M aluminum for the commercial applications, more researches on the enhancement of wear property with the reinforcement are needed. On liquid phase sintering of aluminum MMC with SiC as the reinforcement, the wettability and the uniform distribution of SiC particles in aluminum alloy are related to the mechanical properties such as wear and tensile strength. The possible formation of Al_4C_3, resulted from the reaction between SiC and Al-Si alloys, is crucial to understand wetting behavior in the alloying materials. This reaction is generally deleterious due to poor corrosion resistance and brittleness of Al_4C_3. Concentration of Si on the alloy and temperature play important role in this reaction [7].

To enhance the mechanical properties of aluminum P/M composite alloys, increasing the sinterability and put the reinforcements with intra-granular structure have to be realized. This paper aims to evaluate the sinterability and mechanical properties of Al-14.5Si-1.85Cu-2.85Fe-0.8Mg with 10 vol% SiC (12 μm) alloys prepared by pressureless sintering. A new type of Al-Si-Cu-Fe-Mg with 10 vol% SiC (12 μm) alloy powders were prepared by a gas atomization process and sintered without pressure in a furnace.

Experimental Procedure

Gas atomized Al-14.5Si-1.85Cu-2.85Fe-0.8Mg composite powder with 10 vol% SiC (12 μm), which is called by VNS alloy system, was prepared. The composite

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Table 1. The sample notations of the powders with different composition and their theoretical densities

Sample Notation	Materials	Theoretical Density (g/cm^3)
VNS	VNS	2.78
75V15A	75 wt%VNS + 25 wt% Alumix231	2.75
50V50A	50 wt%VNS + 50 wt% Alumix231	2.72
25V75A	25 wt%VNS + 75 wt% Alumix231	2.70
Alumix231	Alumix231	2.67

powder contains Si, Cu, Fe and Mg to improve the sinterability and mechanical properties. Cu could be reacted with Al to form the strengthening precipitates during heat treatment. $CuAl_2$, Si and SiC help to are the elements to improve mechanical properties, especially wear resistance. Mg is aimed to eliminate oxide layer on aluminum powder to form spinel, Al_2MgO_4. After analyzing the sinterability of the composite powder, Alumix 231 (pre-mioxed powder, Ecka Granules, Germany) was added to increase its sinterability. Alumix 231 has chemical composition of Al-14Si-2.5Cu-0.5Mg with 1.5 wt% lubricant. Both powders were mixed together with different compositions. Theoretical density also changed with the mixing ratio. Table 1 illustrates the sample notations of the powders with different composition and their theoretical densities.

The mixed powders were compacted at 700 MPa with dual action press with the cylindrical dies of 16 mm in diameter and about 5 mm in height. After measuring the green densities of the powder compacts, they were then sintered at the temperature range of 550~565 °C for 1 hour under flow of ultra high purity nitrogen gas in a tube furnace at a heating rate of 10 °C/min. Before reaching sintering temperature, the powder compacts were de-waxed at 400 °C for 30 min. Sintered density was measured by Archimedes method.

Rockwell hardness was performed to measure the mechanical properties of the sintered and heat treated materials. For sintered materials, the hardness was measured with Rockwell B hardness scale with 100 kgf and 1/16 in diameter of diamond ball indenter. To improve the mechanical properties of sintered samples, the precipitation strengthening was carried out. The sintered samples were heated to form the solid solution at 512 °C for 30 minutes, followed by water quenching and artificially aging for 10 hours, and finished by air cooling. In the case of T6 heat-treated materials, the hardness was measured with Rockwell D hardness scale with 100 kgf load and brale indenter. Tensile test was also carried out to measure their tensile strength.

Optical microscopy and x-ray diffraction (XRD, Rigaku Diffractometer with Cu Kα radiation ($\lambda = 0.154$ nm) in the range of 2θ from 20-80 ° by the step and scanning speed for 0.02 ° and 5 °/min.) analysis were used to characterize the composite materials.

Results and Discussion

Fig. 1 shows the effect of the mixed ratio of the powders on the green density. Green density of VNS is about 86.5% and increases with increasing the added amount of Alumix 231 powder into VNS powder. The 25V75A sample possess the highest green density of 92.5%, indicating that commercially available Alumix 231 improves the compressibility of mixed powder.

Fig. 2 shows the variation of the sintered density and hardness with the sintering temperature for all the samples. The sintered densities of the 50V50A and

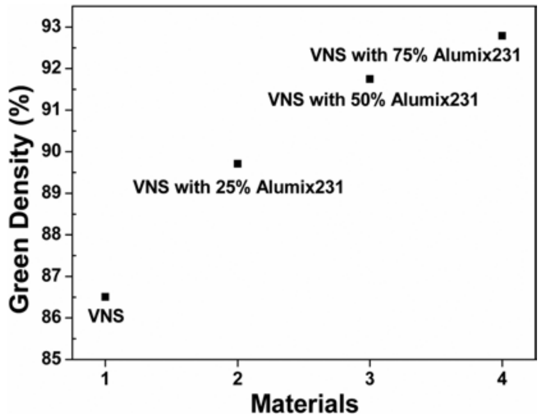

Fig. 1. Green Density of the mixed powders compacted at the pressure of 700 MPa.

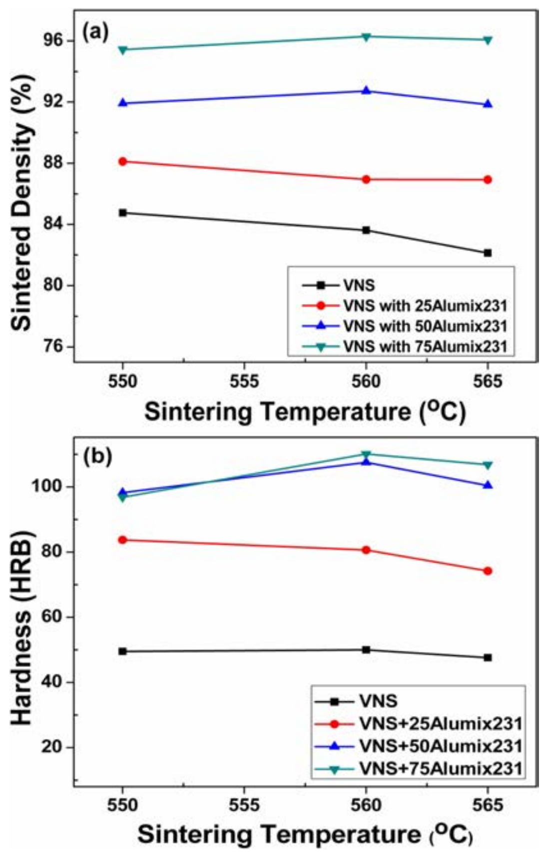

Fig. 2. (a) Sintered density and (b) hardness of the composite materials depending on the sintering temperature.

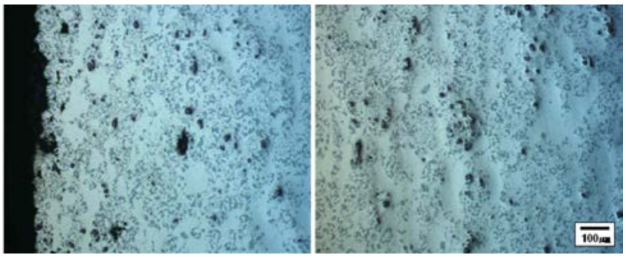

Fig. 3. Optical micrographs of the 25V75A samples sintered at 560 °C for 1 hour.

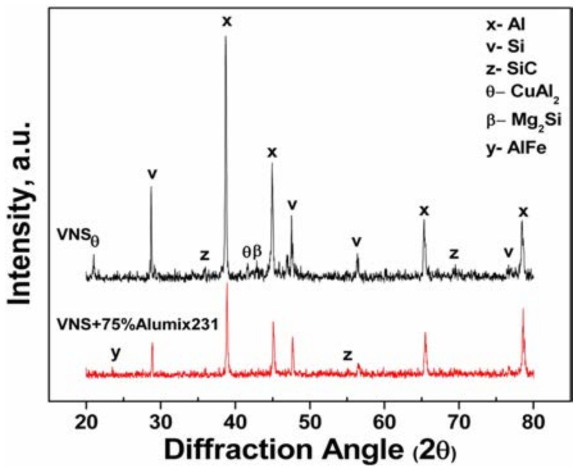

Fig. 4. X-ray diffraction patterns of the VNS and 25V75A samples. They were sintered at 560 °C for 1 hour.

25V75A samples increased with the sintering temperature until 560 °C and decreased at 565 °C but those of the VNS and 75V25A samples decreased with the sintering temperature, which may be attributed to the melting point of VNS alloy which is lower than Alumix 231. In VNS alloy system, the amount of Alumix 231 has important role to improve the sinterability of VNS alloy. In this work, the 25V75A composite sample significantly revealed the improved sinterability to reach the relative density of 96.2% at the sintering temperature of 560 °C. The hardness also shows the same tendency to the sintering density (Fig. 2(b)). The highest hardness was obtained for over than 100 HRB in the composite of 25V75A sample. It is seen that Alumix 231 influences greatly to increase the mechanical properties of the VNS alloy. Optical micrographs of the sintered composite also confirm the high densification of the materials as shown in Fig. 3.

XRD analysis was carried out to observe possible precipitates formed during sintering. Fig. 4 shows XRD pattern of the VNS and 25V75A samples sintered. It is found that both of the patterns look similar and precipitates of $CuAl_2$, Mg_2Si and AlFe were formed in both samples. Al, Si and SiC were also found as a strongest peak.

To obtain high sintered density, the wetting liquid is necessary. The wettability of a solid by a liquid is

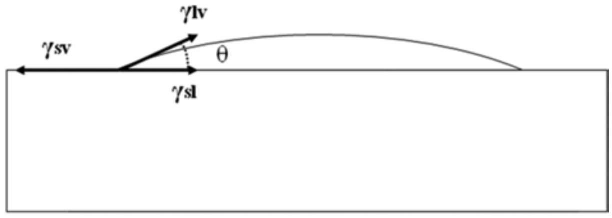

Fig. 5. Wetting angle of the liquid phase sintering process.

determined by the work of adhesion, Wa, :

$$W_a = \gamma_{lv}\ (1 + \cos\theta) = \gamma_{sv} + \gamma_{lv} - \gamma_{sl} \tag{1}$$

Where γ_{lv} is the surface tension of the liquid-vapor interface, γ_{sv} is the surface tension of the solid-vapor interface and γ_{sl} is the solid-liquid interfacial tension and è is the contact angle [13]. A liquid is said to wet a solid when $\cos\theta > 0$ as illustrated on Fig. 5.

The reaction between SiC and Al-Si alloys and the conditions for $A1_4C_3$ formation at the interface are of particular importance to understand the wetting in this system. This reaction is generally considered deleterious because of the poor corrosion resistance and brittleness of $A1_4C_3$. The chemical reaction is described by the equation:

$$3SiC + 4Al \rightarrow Al_4C_3 + Si \tag{2}$$

The critical silicon concentration needed to avoid $A1_4C_3$ formation. In this research, Al_4C_3 was not found within the XRD detection limit. From the previous work, Al_4C_3 was not formed at temperature below 1000 °C but, when Si concentration is more than 12 wt.%, there is possibility to form Al_4C_3 at higher temperature because it is difficult to take control of carbon activity on this condition [14].

Not only to impede formation of Al_4C_3, liquid phase sintering also helped to remove the oxide layer on aluminum particles. Pieczonka et. al showed that, at the first stage of sintering, Mg atoms move to the surface of aluminum particle and react with aluminum oxide, Al_2O_3 to produce the spinel phase, Al_2MgO_4 [15].

T6 heat-treatment was employed for the strengthening precipitates formation of the sintered composite powders. Fig. 6(a) shows that the hardness of T6 heat-treated composite materials increases with increasing amount of Alumix 231 powder to have the highest hardness of about 60 HRD. The XRD analysis was preformed to identify the possible formation of strengthening precipitates. Fig 6(b) shows the XRD pattern of T6 heat-treated 75V25A sample. It was found that the strongest peak for strengthening precipitate is $CuAl_2$ phase. Another possible strengthening precipitate, Mg_2Si, was also found. Mg_2Si was possibly formed by the eutectic reaction and the phase appeared in black color. Fig 7 shows that tensile strength is increase with

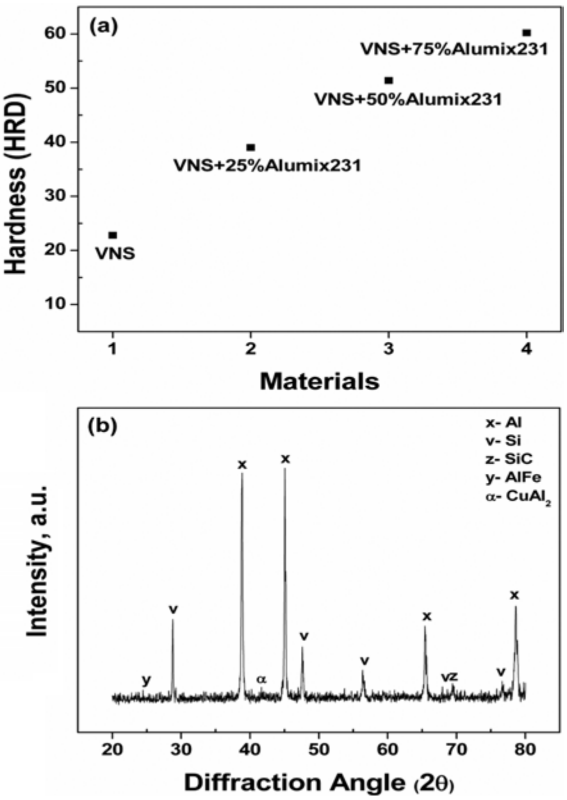

Fig. 6. (a) Hardness of T6 heat-treated materials and (b) X-ray diffraction pattern of heat- treated materials.

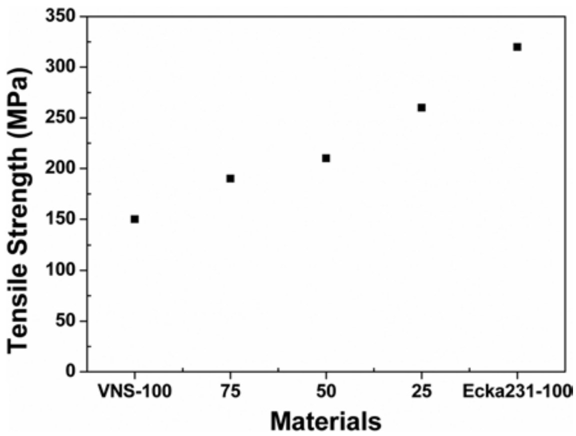

Fig. 7. Tensile strength of VNS and Ecka Alumix 231 with different compositions.

increasing amount of Alumix 231 in the composite. Pure VNS alloy has a tensile strength of 150 MPa and pure Alumix 231 has 320 MPa. It can be summarized that the high sinterability of Alumix 231 plays important role in this work to improve the mechanical properties.

Summary

The sinterability of VNS alloy system significantly were improved by adding wt.75% of Alumix 231. The sintered density increased with increasing sintering temperature up to 560 °C. Considering lower melting point of VNS alloy than that of Alumix 231, the sintering temperature of higher amount Alumix 231 would be higher. Wettability of SiC contains in aluminum alloy powder is very important during liquid phase sintering to obtain higher density. According to the wt% amount of Si inside the alloys and low sintering temperature, Al_4C_3 was not found as a secondary reaction between SiC and Al. T6 heat-treatment was found to successfully increase the mechanical properties of these materials and the highest hardness was 60 HRD with the presence of strengthening precipitate of $CuAl_2$ as confirmed by the XRD analysis.

Acknowledgement

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