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Original Research Paper

Structural and mechanical properties of hypereutectic AlSiFe powders and a new method for determination of sintering temperature

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ABSTRACT

In this study, the effect of Si amount on the microstructure, crystal structure and some mechanical properties of Al-(20,25,30 wt%)Si–5Fe powder mixtures produced by high energy ball milling method was investigated by determining the sintering temperature. In the X-ray diffraction analysis, no inter-metallic phases were found except for the Al, Si and Fe phases of the powder mixtures. The X-ray diffraction patterns were analyzed by the Rietveld method to determine amounts of the Al, Si and Fe elements in the powder mixtures. From the scanning electron microscopy analysis, it is seen that as the amount of Si increases, there is a marked decrease in the grain size of powders. In the micro-indentation tests performed at room temperature, the hardness and elastic modulus values of the pelletized powders were found to increase with the amount of Si. It was also found that the Al-20Si-5Fe powder mixture with the lowest Si content had the highest damping capability. The high temperature micro-indentation tests showed that the resulting Al-(20,25,30 wt%) Si-5 Fe powder mixtures started to consolidation at 200 °C and completed the consolidation at 400 °C. This method can be used as an alternative method in the determination of the sintering temperature of materials.

1. Introduction

Al-Si alloys were classified as hypoeutectic (<12%Si), eutectic (12–13%Si) and hypereutectic (14–25%Si) depending on the silicon concentration. Hypereutectic Al-Si alloys are preferred in many areas because they have low density, high rigidity and high temperature resistance, high abrasion resistance and low thermal expansion coefficient [1]. The microstructure of hypereutectic Al–Si alloys is composed of primary silicon particles and eutectic structure of -Al and Si. The high strength and wear resistance of these alloys are mainly attributed to the presence of hard silicon particles (both primary Si and eutectic Si). The addition of a third element to the Al-Si hypereutectic alloys allows the secondary phases to be thinned, thereby enhancing the strength, wear resistance and thermal stability. In particular, Fe addition provides high chemical homogenization and thermal stability due to high diffusivity in the liquid state and low diffusivity in the solid state [2,3]. Hypereutectic Al-Si-Fe alloys produced with powder metallurgy (PM) are extensively used in high-tech applications such as the automotive, military and aerospace industries due to their high strength/weight ratio, low coefficient of thermal expansion, high wear and corrosion resistance and moderate mechanical properties [4–6]. Ball milling is a very effective, easy and inexpensive method for increasing the homogeneity of microstructure in Al-Si-Fe powder mixtures and for reducing the size of coarse Si and Fe-containing inter-metallic compounds [6,7]. However, consolidation of powder mixtures is a very challenging aspect and numerous researchers have put so much effort into this subject [8–11]. There are many sintering parameters, namely temperature, pressure, time, working atmosphere, etc. that affect the mechanical properties of consolidated powder mixtures [12,13]. It is important to determine the proper sintering temperature in order to a fully compact product with desired properties. In the literature, the sintering of Al-Si-Fe powder mixtures takes place at a wide temperature range. Sastry et al. have consolidated mechanically milled Al-17Si-5Fe-3.5Cu-1.1 Mg-0.6Zr (wt.%) powders using field assisted sintering method and achieved fully dense compacts at 400 °C of sintering temperature and 283 MPa of compaction pressure [14]. By Shim et al., the Al-10Si-5Fe-1 Cu-0.5 Mg-1Zr (wt.%) powder mixtures produced with gas atomization method were compacted using cold isostatic pressure (CIP) and then sintered at 600 °C for 1 h. They investigated the effects of temperature and strain rate on

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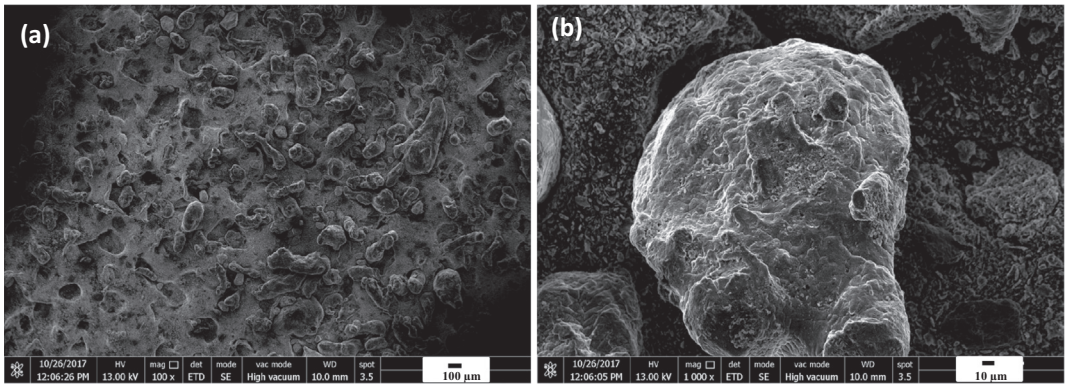

Fig. 1. SEM image of initial powder of Al-20Si-5Fe (a) 100× and (b) 1000×.

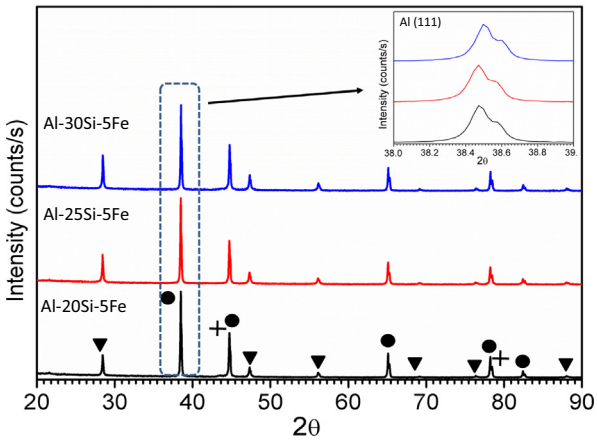

Fig. 2. X-ray diffraction patterns of powder mixtures. The inset image shows Al (1 1 1) peak of the powder mixtures.

porosity variation of sintered powders [15]. Rajabi et al. produced Al-20Si-5Fe alloy by gas atomization and melt spinning and consolidated by hot pressing at 400 °C under 250 MPa for 1 h. They have observed that coarsening of primary silicon particles and precipitation of β-Al_5FeSi phase during consolidation process [16]. Lee et al. have prepared the Al–Si–Fe based bulk alloy by using different pressing step consisting of pre-compaction of the powders and subsequent forging in the temperature range of 380–524 °C and reported that the alloy produced by cold pressing and then hot forging exhibited finest microstructure [17]. As can be understood from above, many attempts have been made to determine the proper sintering temperature by testing of different sintering temperatures. There are several approach for determining the sintering temperature in literature such as dilatometer [18], combined differential thermal analysis and thermogravimetric analysis [19], thermal conductivity analysis [20], electrical conductivity [21], thermochemical equilibrium calculations [22] or yield stress [23]. Recently Schimpke et al. proposed a new procedure called as cold compression strength (CCS) test on ash pellets for the determination of initial sintering temperature. This process is based on the combination of the tests, TG, DSC, HT-XRD and SEM-EDX [24]. All these reported approaches consist of few steps which are time consuming. Here, we will propose a new one-step method for determining sintering temperature using high temperature micro-indentation test.

In this new one-step method, the high temperature micro-indentation test is used to perform the indentation process on the powder mixtures under different temperatures and constant load. After the indentation process, it is determined in which temperature the consolidation takes place by examining the photographs of the trace formed on the powder mixtures. The depth of penetration for each load is also recorded by the system and the hardness and elastic modulus values of the powder mixture at each temperature can be determined by using this data. It is also possible to have information about the consolidation of the powder mixtures in the light of this data. The sintering temperature of the powder mixtures is determined much more rapidly and more properly than other methods by considering the values of both trace photographs and mechanical properties (hardness and elastic modulus).

In this study, the authors of this manuscript produced Al-(20,25,30 wt%) Si-5Fe powder mixtures by High Energy Ball Milling (HEBM) technique and determined sintering temperature of the powders by high temperature micro-indentation test. The powders were consolidated at the sintering temperature determined by micro-indentation test and their microstructure and some mechanical properties were investigated by XRD, SEM, depth sensitive micro-indentation analysis.

2. Experimental

The initial powders supplied from Alfa Aesar company were Al powder with purity of 99.97% (44–149 µm), Si powder with purity of 99.999% (<149 µm) and Fe powder with purity of 98% (<149 µm). The SEM images of initial powder of Al-20Si-5Fe is depicted in Fig. 1. Al-(20,25,30)Si-5Fe powder mixtures were produced using 0.5 mm diameter balls in a stainless steel chamber in a high-energy ball milling system (HEBM, P100). In the milling process, the ratio of the ball to the powder is 30:1; rotation speed 800 rpm; rotation times were selected as 10 min for powder mixtures. Stearic acid (5 wt%) was added into to hopper to prevent clumping and sticking of the alloys to the chamber walls during production. The placement of the alloys in the steel hopper was carried out in an argon atmosphere glove box. The powder mixtures were cold pressed at a pressure of 760 MPa and polished with Metkon automatic head Forcipol 2V polishing device for hardness and microstructure analysis. The composition of the acid solution used in the chemical etching of the alloys was selected as 75 ml of Hydrochloric acid (HCl), 25 ml of nitric acid (HNO3), 5 ml of Hydrofluoric acid (HF1) and 50 ml of distilled water. SEM photographs were taken under a SEM Quanta FEG 450 model Scanning Electron Microscope with a potential of 20 kV. The diffraction patterns were obtained with XRD Emperiance brand XRD instrument under 40 kV and 30 mA current and monochromatic Cu Kα

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