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Original Research Paper

Effect of particle size on densification of pure magnesium during spark plasma sintering

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ABSTRACT

The effect of particles size ranges (<38 μm, 75–150 μm, 270–550 μm) of atomized magnesium powders on densification mechanisms during spark plasma sintering (SPS) process was investigated. The intrinsic driving force, local pressure and current of Mg powders with different particle sizes were analyzed by theoretical calculation. The results obviously indicate that the densification of pure magnesium can be improved by the reduction of particle size, suggesting the intrinsic driving force, local pressure and current intensity are enhanced significantly by a decrease in the particle size at the same sintering conditions, which can promote shrinkage of pores, formation of the sintering neck and mass transportation in the SPS process. Not only that, rapid densification is also interpreted in term of mechanical movement of particles, Joule heating effect and plastic deformation. However, the mechanical movement of the large particles is higher than that of small particles due to high punch displacement, and plastic deformation, detected by scanning electron microscopy, plays a main role in densification for large particles in the case during the sintering. Joule heating effect is the key factor for densification of small Mg particles, and high densification degree can be obtained by sintering small particles.

1. Introduction

As an innovative sintering technology, Spark plasma sintering (SPS) makes it possible to enhance the densification of sintered samples in a very short period of time because of its a number of advantages, such as lower sintering temperature and voltage, rapider heating rate and shorter holding time [1,2]. The notion that presence of the plasma causes cleansing action and promoting bonding of particles has been proposed, even if the generation of plasma is still an open point [3–5]. In general, the contact points between the particles have high temperature when spark plasma generates at a gap, which results in the melting and evaporation on the surface of the particles [6–9]. Besides, the densification process during SPS of metal powders is involved in many mechanisms, such as mechanical movement of particles, Joule heating effect and plastic deformation [10–12]. These densification mechanisms of the SPS process are subjected to several factors, such as sintering temperature, applied pressure, holding time and so on. However, effect of particle size on the mechanical movement of particles, Joule heating effect and plastic deformation of pure magnesium has been seldom studied, and the relative contributions of these mechanisms to densification have not yet been explicitly illuminated.

Diouf [13,14] investigated the effect of particle sizes, pressure and the temperature on densification of as-atomized copper powder, and concluded that densification was enhanced with the particle sizes. Jabbara and Couret [15] prepared TiAl alloy by SPS and believed plastic deformation of the powder particles played an important role in densification, and the small particles were more deformed than the large ones in SPS process. And besides that, particle sizes also affect the specific surface area which is affiliated with the surface energy driving force, and local pressure and current intensity of sintered body varies with the particle sizes. In order to obtain full densification of samples by SPS, it is essential to understand the effect of particle size on densification.

The present work is aimed to research the influence of the particle sizes on densification mechanisms of pure magnesium. In this paper, three different particle sizes of Mg powders were sintered by SPS at the same sintering parameters. The results were examined by analyzing the punch displacement and temperature curves

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Nomenclature

List of symbols

ΔE	intrinsic driving force
E_p	surface energy of powder before sintering
E_d	surface energy of powder after sintering
γ_{sv}	solid-gas surface energy
W_m	molar mass of material
S_p	specific surface area of 1 g powder
n	number of particles of 1 g powder
S_{PT}	total specific area of 1 g powder
ρ	density of powder
R	particle radius
R'	equivalent radius of curvature
R_1 R_2	radius of contacted particles
σ	tensile stress of sintering neck
γ	surface tension
E_1 E_2	electric field
J	current density
G	electric conductivity
I	current
A	cross-sectional area
X	neck radius

during experiments, and the focus was on the punch displacement, local pressure, plastic deformation and current intensity during sintering process. Contribution of different mechanisms to densification of pure Mg was verified, and a theoretical analysis was propounded to quantify the effect of particle sizes on densification mechanisms during SPS process.

2. Experimental detail

Mg powders (Tangshan Weihao Magnesium Powder Co., Ltd., showed in Fig. 1) with a purity of 99.88% and three different particle sizes (<38 μm, 75–150 μm, 270–550 μm) were used in SPS process, and the average particle size of the three ranges were about 23.5 μm, 110.8 μm and 394.5 μm respectively through calculation. Sintered samples were represented by Mg_1, Mg_2 and Mg_3, respectively. An SPS-331LX sintering apparatus (Fuji Electronic Industrial Co., Ltd., Japan) and graphite die (40 mm height) with an internal diameter of 20 mm and an external diameter of 40 mm were employed, and graphite paper of 0.5 mm thickness was placed between graphite punches and the powder to prevent sticking during the sintering process. Mg powders were first pre-compacted under a pressure of 5 MPa, and then sintered by SPS at a temperature of 480 °C under a pressure of 40 MPa for 5 min, and heating rate was 50 °C/min. Sintering was performed in vacuum (residual pressure <10 Pa).

The cylindrical samples were with diameter of about 20 mm and 5 mm in thickness, the grain morphology and size were revealed by using an etching solution (picric acid 0.75 g, deionized water 50 ml) and examined by optical microscopy (OM, DM2700MRL) after polished by the metallographic abrasive paper in 2000 grit size. Microstructural characterization of the samples surface were observed by scanning electron microscopy (SEM, VEGA3 SBH) equipped with an energy dispersive spectrometer (EDS).

Relative density of the sintered samples were determined though Archimedes method by using an electronic balancer. The Vickers hardness was measured by hardness tester (HVS-1000A) with the load of 0.1 kg for 15 s after polished, and the average value of each specimen was gained by evaluating 7 points.

3. Results and discussion

3.1. Microstructure and hardness analysis

OM of the sintered samples is shown in Fig. 2. As seen in Fig. 2, the samples were sintered well and no obvious sintering defects, and the particle boundaries and grain boundaries can be observed clearly. Relative density, Vickers hardness and average grain size of samples are expressed in Table 1, the average grain sizes were about 4.8 μm, 14.3 μm and 36.1 μm, respectively, shown in Table 1. Different particle size can cause the difference of the sintering mechanisms for powders and this will be discussed below. The EDS results of sample Mg_2 are shown in Fig. 3, bright dots were observed clearly on the surface of the Mg_2. The bright dots were found, O and Mg were determined in the particles boundaries, which confirmed that the oxides still existed along the particle boundaries. The EDS analysis indicated that main elemental impurity on the surface of samples was O, and oxygen does exist on the surface by EDS analysis. There may be two reasons to account for this case: one is even sintering under vacuum environment, but

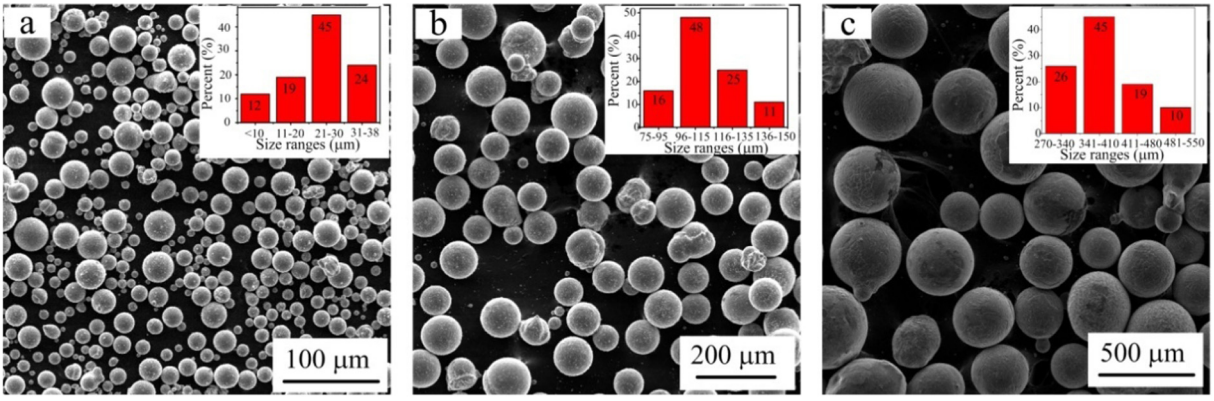

Fig. 1. SEM images of original Mg powders with particle size of (a) <38 μm, (b) 75–150 μm and (c) 270–550 μm.

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