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DENSIFICATION AND MAGNETIC PROPERTIES OF FE POWDER COMPACTS ACCORDING TO HEAT-TREATMENT CONDITIONS

In this study, the effects of heat-treatment conditions of Fe powder compacts on densification, microstructure, strength and magnetic properties were investigated. The prepared Fe powder was compressed in a mold of diameter 20 mm at a pressure of 800 MPa for 30 sec. This Fe powder compact was heat-treated under different atmospheres (air and 90% Ar + 10% H₂ and heat-treatment temperatures (300 and 700°C). The Fe powder compacts heat-treated in an Ar+H₂ mixed gas atmosphere showed a denser microstructure and higher density than the Fe powder compacts heat-treated in an air atmosphere. Oxygen content in the heat-treatment conditions played a significant role in the improvement of the densification and magnetic properties.

Keywords: Fe powder; Heat-Treatment; Microstructure; Density

1. Introduction

Soft magnetic composites (SMCs) composed of electrically insulated magnetic powders have become the strongest candidates for laminated ferrites, steel cores and electrical steels for electrical and electronic applications in the sustainable energy market [1,2]. It is an essential material for electronic components manufactured by coating an insulating layer on the surface of magnetic powder particles, and is widely used in nuclear reactors, transformers, and inverters [3].

SMC materials are produced using the established manufacturing technology provided by powder metallurgy technology, and are a very attractive option for users due to their efficient production route, material utilization, and high level of reproducibility.

As the shape of the product becomes more complex, high magnetic permeability and low core loss are required, so research on soft magnetic sintered compacts with excellent magnetic properties is required [4,5]. Therefore, in order to achieve high permeability and low core loss of SMC, a thin, dense and continuously distributed insulating layer is very important so that adjacent magnetic particles cut down the intergranular eddy currents and ensure continuity of magnetic flux [6,7]. Also, Defects and internal stresses generated in the compression process

increase the loss of SMC, so it is necessary to adopt suitable heat-treatment conditions [8].

In this study, the effects of heat-treatment conditions of Fe powder compacts on densification, microstructure, strength and magnetic properties were investigated.

2. Experimental

In the current study, pure iron powders were prepared using the water atomization method. In order to prepare the Fe powder, Fe chips were melted using high-frequency induction melting at a temperature of 200°C above the melting temperature. The molten alloy was atomized by a water stream at the pressure of 20 bar with a nozzle 5 mm in diameter. Subsequently, the received powder was sieved from 100 to 300 μ m. The prepared Fe powder was compressed in a mold of diameter 20 mm at a pressure of 800 MPa for 30 sec. This Fe powder compact was heat-treated under different atmospheres (air and 90% Ar + 10% H₂ and heat-treatment temperatures (300 and 700°C).

The microstructure of the Fe powder compact was analyzed by field emission scanning electron microscope (FE-SEM, JEOL, JSM-7000F). The density of the Fe powder compacts was measured using the Archimedes method. In order to evalu-

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ate the mechanical properties, a universal material test machine (UTM, Shimizn, AG-IS 50 KN) was used at room temperature. In order to analyze the core loss, 10 mm of the inside diameter of the Fe powder compact was machined out to form a ring shape and copper wire was wound around it. The measurement was performed using an iron loss and hysteresis characteristic analyzer, MPG100D.

3. Results and discussion

Fig. 1 shows the SEM image of the initial Fe powder and the EDS analysis result according to the heat-treatment. As shown in Fig. 1(a), the Fe powder had an irregular shape rather than a spherical shape. The size of the Fe powder has a range of

 $100 \sim 300 \ \mu\text{m}$. Fig. 1(b) and (c) shows the SEM images and EDS results of the Fe powder heat- treated in air and Ar + H₂ mixed gas atmospheres. As a result of EDS on the surface of the Fe powder, the oxygen element was found to be about twice as high in the case of the powder heat-treated in the air atmosphere compared to the powder heat-treated in the Ar + H₂ mixed gas atmosphere.

Fig. 2 shows the photographs according to the heat-treatment atmosphere and temperature of the specimens compressed to 800 MPa. In the case of the specimens heat-treated in an air atmosphere, dark colors were displayed compared to the specimens heat-treated in an $Ar + H_2$ mixed gas atmosphere. It is evident that the color of the heat-treated specimens changed with different heat-treatment atmospheres and temperatures owing to the formation (in air) and reduction (in $Ar + H_2$) via oxidation and hydrogen reduction at the specimen surface.



Fig. 1. SEM image (a) of initial Fe powder and EDS results of the Fe powder heat-treated in (b) air and (c) Ar + H₂ mixed gas atmosphere

Atmosphere gas / Density	Raw metal	300°C	700°C
Air	Room temp.		
Аг+H ₂ (9 : 1)	-		



Fig. 3 shows the changes in (a) density, (b) oxygen content and (c) core loss according to the heat-treatment conditions of the compressed specimens. Fig. 3(a) shows the density change of the compressed specimens. The density of the compressed specimen without heat-treatment was 7.37 g/cm³. In the case of the specimens heat-treated in an Ar + H₂ mixed gas atmosphere, the density slightly increased to 7.38 and 7.41 g/cm³ as the heattreatment temperature increased. The density of the compacts obtained using the Fe pow- der heat-treated in air was lower than that of compacts obtained using Fe powders heat-treated in $Ar + H_2$ mixed gas atmosphere owing to the formation of oxide layer at the surface of the Fe powder. Fig. 3(b) shows the oxygen content of the as-heat-treated the compressed specimens under different heat-treatment temperatures and atmospheres. The oxygen content of the compressed specimen without heat-treatment was 0.11%. In the case of heat-treatment in an air atmosphere, the oxygen content increased as the heat-treatment temperature increased, and in particular, it rapidly increased to 0.55% at 700°C. The oxygen content of the specimens heat-treatment in an $Ar + H_2$ mixed atmosphere was lower than that of the specimens heat-treatment in an air atmosphere. Compared to the initial powder, the oxygen content of the powder heat-treated in the $Ar + H_2$ mixed atmosphere was similar. However, at a high temperature of 700°C, the oxygen content slightly increased. Fig. 3(c) shows the core loss according to the heat-treatment atmosphere and temperature change at 1.0T. As presented Fig. 3(c), at the same heat-treatment temperature of 700°C, the core loss of the heat-treated powder with high oxygen content was lower than that of the powder heat-treated in $Ar + H_2$ mixed atmosphere. In both air and $Ar + H_2$ mixed atmosphere, iron loss increased as the heat-treatment temperature increased. The core loss of the powder heat-treated in $Ar + H_2$ mixed atmosphere with low oxygen content was the lowest.

The SEM images of the specimens under different heattreatment conditions in air and $Ar + H_2$ mixed atmosphere are shown in Fig. 4. In the case of the specimen heat treated at 300°C using air and $Ar + H_2$ mixed gas, large pores and powder boundaries were observed. All the specimens heat-treated with air and $Ar + H_2$ mixed gas showed a dense microstructure as the temperature increased from 300°C to 700°C.

Fig. 5 shows the change in compressive strength according to the heat-treatment atmosphere. The max compression strength of the compressed specimen without heat-treatment was 432.9 MPa. As for the maximum compressive strength, the air atmosphere and $Ar + H_2$ mixed atmosphere showed similar strength at 300°C, and the $Ar + H_2$ mixed atmosphere had higher maximum compressive strength than the air atmosphere at 700°C.

4. Conclusions

In this study, changes in microstructure, densification and magnetic properties according to the heat-treatment conditions of the Fe powder compacts were investigated. Obtained results are summarized as follows. The oxygen content of the Fe powder compacts heat-treated at 300°C showed similar values in the Ar + H₂ mixed atmosphere and the Air atmosphere. However, the oxygen content of the Fe powder compacts heat-treated at



Fig. 3. (a) Density, (b) oxygen content and (c) core loss of the as-heat-treated specimens under different heat-treatment temperatures and atmospheres



Fig. 4. SEM images for the specimens heat-treated under different heat-treatment atmospheres and temperatures



Fig. 5. Compression strength for the specimens heat-treated under different heat-treatment atmospheres and temperatures

700°C was lower in the $Ar + H_2$ mixed atmosphere than the Air atmosphere. The Fe powder compacts heat-treated in $Ar + H_2$ mixed atmosphere was higher than the density of the Fe powder compacts heat-treated in air atmosphere, and showed a dense microstructure. The oxygen content in the heat-treatment conditions may have played a significant role in the improvement of the densification and magnetic properties.

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