

Insulation and Magnetic Properties of Iron Powder Coated by Wet Chemical Method

Hyeon-Taek Son^{1,a}, Cheol-Ho Yun^{1,b}, Hyun-Rok Cha^{1,c}, Chang-Seog Kang^{1,d} and Jung-Chan Bae^{1,e}

¹Korea Institute of Industrial Technology, Chumdan P.O. Box #1, Wolgye-dong, Gwangsan-gu, Gwangju, 506-824, South Korea

^asht50@kitech.re.kr, ^byun34@kitech.re.kr, ^chrcha@kitech.re.kr, ^dcskang7@kitech.re.kr, ^ejcbae@kitech.re.kr

Abstract

In this study, the pure iron powder was treated with aqueous phosphoric acid to produce phosphate insulating layer on the surface. After drying the powder, it was compacted in a mold with a diameter of 20mm at 800MPa. The powder compacts were then heat treated at 500 °C for 1 hour. The results showed that insulated iron powder was obtained with uniform phosphate layer by chemical reaction. With increased amount of phosphate layer, the core loss and density of compacts were decreased. It was also found that the addition of ethyl alcohol during insulating reaction resulted in improved core loss value.

Keywords : powder metallurgy, soft magnetic material, wet chemical method, insulation

1. Introduction

Recently, powder cores for reactors and transformers and various kinds of motor have drawn great attention among researchers of magnetic and electronic industry. Laminated silicon steels have been generally used for the low frequency applications while ferrite core has been used for the high frequency applications. Since the powder metallurgy for producing soft magnetic components has certain outstanding advantages such as near net shape capability with high dimensional accuracy and maximum material savings, the process has drawn much attention.

As of the shape of products become more complex, the required degree of freedom in form becomes higher and therefore, more extensive researches need to be carried out on the sintered soft magnetic materials with superior magnetic characteristics especially at the high frequency range [1-3].

In order to improve core loss value, many researchers have been studied insulating the iron powder particles with an inorganic or organic layer [4-5].

Therefore, in this study, we investigated the insulation and magnetic properties of iron powder coated by wet chemical method and powder metallurgy technique.

2. Experimental and Results

For this research, the pure iron powders, manufactured by using water atomization method, Höganäs AB NC100.24, were prepared. In order to produce a thin insulating phosphorous coating on the surface, the pure iron powder was treated with phosphoric acid (H₃PO₄) which was diluted with distilled water and/or ethyl alcohol. Then, the iron powder in the solution was stirred for 2 hours for the chemical reaction and then cleaned with distilled water and

alcohol and dried in a vacuum dryer. The concentration of phosphorus in phosphoric acid was varied as 0.5, 1.0, 1.5 and 3.0wt% based on the weight of iron powder.

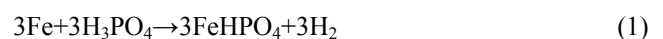
With 0.5wt% Zn stearate lubricant, the powder was mixed in ball mill for 1 hour. The 20g of insulated iron powder was cold pressed at 800MPa in pressure into 20mm diameter mold and then heat-treated. The heat treatment temperature was gradually increased with speed of 4°C/min up to 500°C and then held for 1 hour in atmospheric condition.

Microstructure analysis of both the as insulated powder and the heat-treated specimen were carried out by scanning electron microscopy (SEM, JSM6410). In order to analyze the core loss, a powder compact with an inside diameter of 10mm was machined out to form ring shaped and copper wire was wound. The measurement was performed with Iron loss & Hysteresis Characteristic Analyzer, MPG100D.

The scanning electron micrographs of the pure iron powders coated with different concentration of phosphorus are shown in Fig. 1. As can be seen from this figure, the increase in the concentration of phosphorus resulted in coarser and thicker surface coating on the iron powder.

With 1.0wt% of P, the surface started to reveal needle-like shaped oxides and the amount of the oxides on the surface was increased with increased amount of phosphorus concentrations, 1.5 and 3.0wt%.

The element mapping technique by using SEM was used to analyze the coatings on the iron powder. Fig. 2 shows the result for the element analysis of Fe and P by EDS dot mapping. After insulating treatment by wet chemical method, Fe and P were found to be well distributed throughout the surface of the iron powder, forming a Fe-P oxide layer. It is assume that a chemical reaction had taken place as follows.



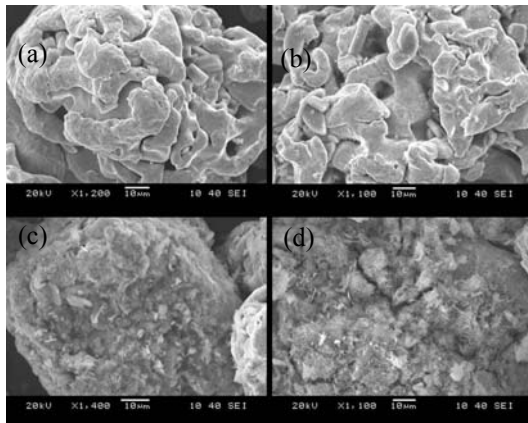


Fig. 1. SEM images of the insulated iron powder with different concentration of P, (a) 0.5wt%P, (b) 1.0wt%P, (c) 1.5wt%P, (d) 3.0wt%P.

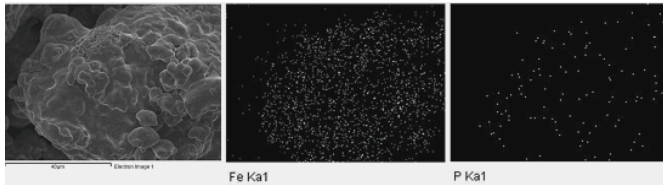


Fig. 2. Micrographs of EDS dot mapping of the insulated iron powder.

Table 1 shows the core loss results at 100Hz in frequency and 1.0Tesla in magnetic field of the compacts made from the insulated iron powder with differences in concentration of phosphorus. As phosphorus content was increased, the core loss decreased as a result of formation of thinner insulating layer. However, phosphorus content was more than 1.5wt%, the powder compacts exhibited large cracks after the heat treatment.

The core loss was affected by adding ethyl alcohol in the phosphoric acid and distilled water solution during insulating the iron powder. With increased amount of alcohol, the core loss value was slightly increased, where the effect of alcohol addition was considerable. As shown in Table 1, with the same amount of phosphorus content in the solution, 0.5wt%, addition of alcohol reduced the core loss value from 66.8 to 18.4W/Kg with distilled water and the alcohol ratio was 4 to 1. This suggests that the formation of iron oxides, such as Fe_2O_3 and Fe_3O_4 , was hindered by the presence of alcohol, and this hindrance resulted in more uniform $FeHPO_4$ oxide coating on the surface of the iron powder.

Table 1. The core loss of the powder compacts with different concentrations of phosphorus and distilled water and ethyl alcohol ratio solution (100Hz, 1.0Tesla)

| No | Content of phosphorus (wt%) | Amount of distilled water(ml) | Amount of alcohol(ml) | Core loss (W/Kg) |
|----|-----------------------------|-------------------------------|-----------------------|------------------|
| 1 | 0.5 | 200 | 0 | 66.8 |
| 2 | 1.0 | 200 | 0 | 55.0 |
| 3 | 1.5 | 200 | 0 | - |
| 4 | 3.0 | 200 | 0 | - |
| 5 | 0.5 | 200 | 50 | 18.4 |
| 6 | 0.5 | 200 | 100 | 19.6 |
| 7 | 0.5 | 200 | 150 | 21.0 |

3. Summary

1. This Insulation coating layer, $FeHPO_4$, was successfully formed on the surface of pure iron powder by wet chemical reaction by using phosphoric acid, distilled water and ethyl alcohol solution.
2. The oxide layer coated with more than 1.5wt%P became very brittle and thick, causing large cracks to form in the powder compact after the heat treatment.
3. The addition of ethyl alcohol in the solution considerably decreased reduction of core loss value compared to the solution without alcohol addition. It was assumed that the alcohol addition hindered the formation of iron oxides and allowed more uniform $FeHPO_4$ oxide insulating coating on the surface of the iron powders.

4. References

1. Erdal Bayramli, Ozgur Golgelioglu and H. Bulent Ertan : Journal of Materials Processing Technology 161 (2005) 83-88
2. E. Enescu, P. Lungu, A. Bara, V. Buharu : Romanian Reports in Physics 56(3) (2004) 487-493.
3. R. L. Orban : Romanian Reports in Physics 56(3) (2004) 505-516.
4. M. Godec, Dj. Mandrino, B. Sustarsic and M. Jenko : Surf. Interface Anal. 34 (2002) 346-351
5. Water A., Esper FJ, Gohl W., Scweikhardt J. : J. Magnet. Magnet Mater. 15 (1980) 1441-1442