The effect of compaction parameters and dielectric composition on properties of soft magnetic composites

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Abstract. This paper investigated the effect of compaction parameters and dielectric composition on mechanical, magnetic and electrical properties of iron-organosilicon epoxy resin soft magnetic composites. In this work, iron powders with high purity were covered by an organic material (organosilicon epoxy resin) and then by coupling agent (KH-550). The coated powders were then cold compacted at 600, 800 and 1000 MPa and cured under vacuum respectively. The results show that the saturation magnetic flux density and electrical resistivity are dependent on compaction pressure and resin content. Increase in the organic phase content leads to decrease of the saturation magnetic flux density, while increase of the electrical resistivity. Furthermore, the samples with 0.9 wt% resins + 0.1 wt% coupling agent at compaction pressure of 800 MPa shows better properties than the others.

1. Introduction
Soft magnetic composites (SMCs) can be described as pure iron powder particles coated with organic electrically insulating layers. SMCs have unique properties such as three-dimensional (3D) isotropy, low eddy current loss, high resistance (typically 70-1000 μΩ·m) and flexible shape design for low cost [1]. Although SMCs have many advantages, now it is seldom used in industrial production for its lower mechanical strength and permeability [2].

In order to be used in high speed rotors and meet the requirements of electrical motors and magnetic bearings, it is important that the mechanical strength of the SMCs should be increased without weakening the magnetic and electrical properties. For this reason, it is necessary to cover the iron particle with a very thin insulating layer to improve the densification and Vickers hardness. In this area, several researchers have investigated the effect of epoxy resin, phenol resin, polyeopxy on the properties of iron-epoxy SMCs [2,3,4]. In this paper, organosilicon epoxy resin was chosen for its good thermal stability and mechanical property. The object is to investigate the effects of compaction pressure and dielectric composition on mechanical, magnetic and electrical properties of SMCs.

2. Experimental
2.1 Raw materials

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The iron powder was supplied by Fucheng chemistry factory. The purity of Fe was 98%, containing 0.1% sulphuric acid insoluble, 0.06% SO₄, 0.005% N, 0.005% Cu and 0.03% water soluble. The iron powders were coated by organosilicon epoxy resin (OER) and KH-550 coupling agent supplied by Xi’an resin factory.

2.2 Preparation of the coated powders
Firstly, the iron particles were degreased in acetone and dried in a vacuum furnace for 30 minutes. Then, the cleaned iron powders were mixed with the OER in acetone in a container by a spiral mixer for 20 minutes, and manual mixing for about 10 minutes. At last, the coated powders were obtained after the evaporation of the acetone, and then heated at 150°C for 25 minutes.

2.3 Production
The powders coated with the OER were cold compacted at 600, 800 and 1000 MPa in a cylindrical die with a diameter of 10 mm and kept for 5 minutes respectively. The glycerol was used as the lubricant for the die wall when compacting. Finally, the samples were cured at 200 °C under vacuum for 35 minutes.

2.4 Characterization
The composition of coated powders was analyzed by a Fourier transform infrared spectrometer (FTIR) with the scan range from 400 to 4200 cm⁻¹. The microstructure and the powder morphology were examined by the scanning electron microscopy (JEOL JSM-7000F SEM/EDS). The average density was measured by the principle of Archimedes. The hardness was tested by a Vickers hardness tester. The value of the strength is three times more than that of Vickers hardness, which can reflect the strength to a certain extent, but can not be replaced. The saturation magnetic flux density was measured by vibrating sample magnetometer (VSM). The electrical resistivity was measured using four-point probe method. Then the density, Vickers hardness, saturation magnetic flux density and resistivity of the samples were measured and compared.

3. Results and discussion
3.1 Structure and morphology of coated powders and SMCs
In order to find the optimum proportion of resin and coupling agent, and to study its effect on mechanical, magnetic and electrical properties of SMCs, six different types of specimens were prepared as shown in Table 1. The samples are classified into two groups according to whether the coupling agent is used. The first three samples in group 1 have no coupling agent. The proportions of coupling agent in the last three samples in group 2 are 0.5, 0.3 and 0.1 wt% respectively. Furthermore, the corresponding samples in group 1 and group2 (sample 1 and 4, 2 and 5, 3 and 6) have the same percentage of iron powders.

| Group | Series No. | Fe wt% | Resin wt% | Coupling agent wt% |
|-------|------------|--------|-----------|-------------------|
| 1     | 1          | 95     | 5         | -                 |
|       | 2          | 97     | 3         | -                 |
|       | 3          | 99     | 1         | -                 |
| 2     | 4          | 95     | 4.5       | 0.5               |
|       | 5          | 97     | 2.7       | 0.3               |
|       | 6          | 99     | 0.9       | 0.1               |

Figure 1. FTIR spectra of coated powders
The FTIR spectra of sample 2 and 5 are shown in figure 1. The main differences between sample 2 and 5 are the peaks at 700-800 cm$^{-1}$, 950 cm$^{-1}$ and 1634 cm$^{-1}$. The peak at 950 cm$^{-1}$ indicates the condensation reaction between silanol groups and iron surface hydroxyl group occurred in figure 2 [4,5] and the peaks 700-800 cm$^{-1}$ in sample 5 is related to the stretching vibration of Si-C bond[6]. Additionally, the N–H bending vibration of primary amine is observed at 1634 cm$^{-1}$ in sample 5. According to [6], the peaks at 1080 cm$^{-1}$ may be considered as Si-O-C and cyclic ether in sample 2, at 1083 cm$^{-1}$ in sample 5 is Si-O-Si. At around 1367 cm$^{-1}$, there is no distinct difference between two samples, may be caused by C-H bending vibration (-CH$_2$-, C-CH$_3$-) in organosilicon epoxy resin. The peak at 3444 cm$^{-1}$ indicates the iron powder surface may be oxidized during degreasing process.

![Figure 2.](image)

**Figure 2.** Condensation reaction between silanol groups and iron surface hydroxyl group

SEM micrographs of sample 6 on 800 MPa and coated powders are shown in figure 3. In figure 3a, it can be seen that porosities exist at the boundary of different iron particles and the morphology of iron powders in figure 3b is not regular. When the irregular powders were compacted together, the probability of the porosities is more at risk than that of regular powders. Thus, it can be considered that the densification of sample may be related to the morphology of iron powders.

![Figure 3.](image)

**Figure 3.** SEM micrograph of sample 6 on 800Mpa (a) and coated powders (b)

![Figure 4.](image)

**Figure 4.** EDS spectrums in the scanning electron microscope (figure 3) of sample 6 at 800Mpa (a) and coated powders (b)
EDS spectrum (figure 4) shows that the composition of the porosity (figure 3a) and the surface layer (figure 3b) mainly consist of iron, oxygen and carbon elements. The sulfur in figure 4a is because of impurity of the iron powders. The silicon in figure 4b is from the resin element. Therefore, it confirms the existence of the insulating layer on the surface of the iron powders.

3.2 Mechanical properties

The densities of each sample at different pressures are shown in figure 5. With the increase of the iron content, the density of the sample increases. When the iron content is lower, the density increases with increase of the pressure from 600 to 800 MPa and as the pressure gets to 1000 MPa, the density starts to decrease. While the iron content is higher, the density has been increasing from 600 to 1000 MPa. Because iron particles are work hardened during compaction [7], heat treatment relieves the stresses and strains. Since the stresses between particles become larger when the pressure gets to 1000 MPa, the particles will spread out to reduce the stress during the heat treatment. This leads to the growing of the whole volume, and therefore the density will be decreased. The densities of sample 3 and 6 continue to increase for the total content of resin and coupling agent is less than other samples and heat expansion coefficient of the resin (6.0E-5/K) is greater than that of iron (about 1.2E-5/K). Thus, the volume decreasing during compaction (compared with 800 MPa) is more than the volume expansion in heat treatment process. Furthermore, by increasing the amount of epoxy resin, the density at the same pressure decreases, because the density of epoxy resin (about 1g/cm^3) is much smaller than that of pure iron. Coupling agent will increase the density of the sample, which is related to the reactions of silane coupling agent with iron particle surface [4].

The Vickers hardness (HV) of the SMCs was measured using four point method with a load 20 g for 10s on the polished samples and the results are shown in figure 6. With the increase of the pressure and iron content, the HV increases. This is according to the increase of density, work hardening in the grains and decrease of porosities. Furthermore, the HV of the same iron content sample with coupling agent is higher than that without it. This is confirmed by the cross linking reactions among iron particles, resin and coupling agent. To samples without coupling agent, there is only mechanical combination of iron particles and resin, no reaction with each other. Comparing with Somaloy 500 + 0.5 wt% Ken [7], the HV of samples with 97 wt% and 99 wt% Fe at 600 MPa are higher.

3.3 Magnetic properties

Figure 7 shows the saturation magnetic flux density increases with the increase of the pressure from 600 to 1000 MPa. The saturation magnetic flux densities of the SMCs are lower than that of
pure iron sample (about 2T). The soft magnetic properties are related to microstructure change during compaction and heat treatment process. On the one hand, the internal stress is generated during compaction process. This stress, having a significant effect on metal magnetization, results in giant magneto impedance effects [8]. When eliminating the stress below the Curie temperature, it promotes the magnetization and improves the magnetic properties. On the other hand, there are also other microstructure changes such as a decrease in the defects from 600 to 800 MPa, which also improve the soft magnetic properties. The coupling agent has no obvious effect on the saturation magnetic flux density.

![Graph of saturation magnetic flux density](image1)

**Figure 7.** Saturation magnetic flux density as a function of pressure for different resin contents

3.4 Electrical properties

![Graph of electrical resistivity](image2)

**Figure 8.** Electrical resistivity as a function of pressure

Figure 8 depicts the electrical resistivity of the samples on different pressures. Firstly, in both groups, the resistivity decreases as the content of iron powder increases at the same pressure, and the resistivity of sample with coupling agent is smaller than that without coupling agent. Since the resistivity of resin ($1.0e17 \, \Omega \cdot m$) is much greater than that of pure iron ($1.0e-7 \, \Omega \cdot m$), the resistivity increases as the proportion of resin increase because that better insulating layer between iron particles may be obtained. Furthermore, the resistivity of SMCs strongly depends on the pressure. With the increase of the pressure, the resistivity decreases. Because under very high pressure, the distance between atoms becomes smaller, and the morphology, electronic structure, Fermi surface, energy band structure and electron scattering mechanism in inner defect will be changed, which might finally affect the resistivity of the SMCs. The relationship between resistivity and pressure can be written as [9]:

\[ \rho = \rho_0 \left( \frac{P}{P_0} \right)^n \]
\[ \rho = \rho_0 (1 + \psi p) \]  

where \( \rho_0 \) is the resistivity in vacuum, \( \psi \) is the pressure coefficient (negative), \( p \) is the pressure (Pa). It can be seen from this formula that the relationship between the resistivity and pressure is linear. However, since only three sets of data were obtained in the experiment, it is not enough to confirm the linear relationship.

4. Conclusion

In this paper, the effect of compaction parameters and dielectric composition on mechanical, magnetic, and electrical properties of SMCs is investigated. The results show that the insulating layer was prepared successfully and the optimum compaction pressure is about 800 MPa and proportion of content is 0.9 wt % resins + 0.1 wt % coupling agent. The saturation magnetic flux densities of the SMCs fabricated from iron and resin are lower than 1.1 T for lower permeability of resin and defects in SMCs. In particular, the coupling agent improves the density and Vickers hardness of the SMCs as the proportion of resin is decreased.

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