Effects of compaction and heat treatment on the soft magnetic properties of iron-based soft magnetic composites

Yifan Pan, Jingguang Peng, Liwei Qian, Zhen Xiang and Wei Lu

1 School of Material Science and Engineering, Tongji University, Shanghai, 200092, People’s Republic of China
2 Shanghai Automotive Powder Metallurgy Co, Shanghai, 201900, People’s Republic of China

E-mail: jingguangp@shautopm.com.cn and wellu@tongji.edu.cn

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Abstract

Iron-based soft magnetic composites (SMCs) are promising substitutes for laminate steels in electromagnetic applications due to their excellent magnetic properties and productivity. However, the preparation process is a key factor in deciding the magnetic performance of SMCs. In this work, the Fe-based soft magnetic composites with improved soft magnetic properties were achieved by optimizing the compaction and the annealing process. Results showed that the core–shell structure of powders which would directly have an impact on the permeability and the core loss of the SMCs could be affected by the compaction and the annealing process. In addition, the magnetic properties were enhanced by tuning the microstructure. As a result, the optimal magnetic performance of the compact with high permeability and low total core loss was obtained. The real part of the permeability of the soft magnetic composites could reach a maximal value of 336.8 and a rather low core loss of 2.5 W Kg\(^{-1}\) (measured at 50 mT and 5 kHz). Therefore, soft magnetic composites with enhanced magnetic properties were obtained by optimizing the powder metallurgy (PM) process in this study.

Introduction

Soft magnetic composites (SMCs) are promising substitutes for laminate steels in electromagnetic applications such as transformers, generators and motors [1] due to their core–shell structure [2] and unique properties including high magnetic saturation, high magnetic permeability, high resistivity, low core loss, high degree of productivity and three-dimensional (3D) isotropy [3–5]. Basically, the SMC cores are produced by traditional powder metallurgy techniques including mixing soft ferromagnetic powders with insulating materials and lubricants, compacting and pressing them to the desired shapes, as well as the subsequent annealing treatment [3, 6] to relieve stress. However, insulating layers would be easily destroyed under too high pressing pressure during the compaction process so that the adjacent iron powers would connect directly with each other and the eddy-current loss would increase sharply as a result [7]. On the other hand, much pore would be introduced if pressing pressure is not sufficient and they would act as dislocations to impair the magnetic performance [8]. As is known that much dislocation and internal stress would be introduced during the compaction process to pin both the rotation and the movement of the magnetic domain wall so as to deteriorate the magnetic performance. Therefore, heat treatment is critical to reduce the pinning effect which would decrease magnetic permeability and increase core loss [3], neither too much high temperature nor the low temperature would benefit the magnetic properties of SMCs [9, 10]. Above all, a combination of a suitable compaction process and a proper annealing process would have a great contribution to the soft magnetic properties of the SMCs. Perigo et al [11] fabricated a type of phosphorus-added iron-based alloy under the pressure of 400–800 MPa at room temperature and found that the compact with the highest density exhibited the highest maximum permeability, but the particle deformation caused large coercive field. Evangelista et al [12] compacted SMC rings by commercially available Somaloy 3 P 700 (Höganäs AB) powders and investigated the relationship between the optimal operating frequency and the annealing temperature. Results showed that surface coatings of SMC cores
degraded during the high-temperature annealing process and the optimal operating frequency differed as the frequency increased. To prepare high-performance SMCs, selecting suitable coating materials, coating methods and PM techniques are critical important[13]. Thus, many attempts have been carried out to enhance the magnetic properties of SMCs by coating suitable materials on matrix powders and optimizing the annealing treatment. Whereas, limited studies on the effect of PM processing parameters which include the compaction temperature, temperature and atmosphere of the annealing treatment on the magnetic properties of soft magnetic composites (SMCs) are available in the literature.

Somaloy is an isotropic and high resistive soft magnetic composite material with low loss and high magnetic permeability. Besides, the unique 3D flux properties and net-shaping opportunity could be used to design innovative, compact and powerful electromagnetic applications, especially promising to replace the steel and to manucacture the lightweight car. In this work, commercially available Somaloy 5P (Höganäs AB) powders whose particle size was mainly between 75 μm to 250 μm and more detailed, the weight percent of 60 mesh particle is approximate 21.75%, 80 mesh particle is approximate 28.5%, 100 mesh particle is approximate 20.9%, 200 mesh particle is approximate 27.45% were used as raw materials and compacted into ring-shaped components with different processing parameters and then components were annealed under various conditions. Effects of the compaction and the annealing parameters on the soft magnetic properties of SMCs were systematic studied, and the optimal processing parameters for Somaloy 5P-based components were revealed.

**Preparation and characterization**

The raw material were commercially available Somaloy 5P (Höganäs AB) powders. Uniaxial pressing followed by the annealing process were carried out to prepare ring-shaped samples with an outer diameter of 40 mm and a square cross section of 8 mm * 8 mm. For powder compaction process, powders were compacted under 800 MPa at room temperature, 85 °C and 100 °C respectively. The reason why we selected 800Mpa and 85 °C and 100 °C is that the Somaloy 5 P is a type of Fe-based soft magnetic composites which is relative soft, and the ordinary pressure of Fe-based SMC powders is range from 400Mpa to 1200Mpa and 800Mpa is the most commonly used. Besides, tolerance of die was also considered. So we selected 800Mpa. As for the temperature of compaction, the DSC experiment of 5 P powders were measured and the result showed that there was a peak in the temperature of approximate 105 °C, it might be the melting point of the lubricant. So we select the 85 °C and 100 °C which was below the melting point of lubricant for not melting it and therefore deteriorate the properties And compacts whose density varied from 7.2 g cm⁻³ to 7.7 g cm⁻³ were also prepared. After the compaction procedure, samples were submitted to heat treatment cycles represented in figure 1.

The initial magnetic permeability, maximum permeability, saturation magnetization and coercivity of samples were measured by the magnetometer (MATS-2010SD, Linkioin, China) under a maximum applied field of 10 000 A m⁻¹. The amplitude permeability, real part of permeability, imaginary part of permeability and total core loss of toroid samples were obtained from a B-H looper (MATS-2010SA/500, Linkioin, China) at maximum flux density $B_m = 20$ mT~50 mT, over a frequency range from 5 kHz to 100 kHz.
Results and discussion

The effect of the compaction process

The apparent density and the flow velocity of powders are two of vital properties which are related to the compaction process, the apparent density and the flow velocity of Somaloy 5P were tested and the results were 3.216 g cm$^{-3}$ and 34.9 s/50 g respectively. High density and flow velocity ensure the possibility of achieving compacted samples. The changes of the amplitude permeability ($\mu_a$) and the core loss of samples compacted at different temperature as a function of the measurement of frequency are shown in figure 2.

It can be seen from figure 2(a) that the amplitude permeability ($\mu_a$) of the cold-pressed sample is lower than that of the warm-compacted samples, however, it exhibits excellent frequency stability from 5 kHz to 100 kHz while the amplitude permeability ($\mu_a$) of warm-compacted samples decreased sharply as frequency increases, the amplitude permeability ($\mu_a$) of samples become almost the same at the frequency of 100 kHz. On the one hand, the warm-compaction process could not only just improve the plastic deformation capacity of iron powders so that the contact points of powders could bond stronger but also to contribute the rearrangement of particles which benefits decreasing the porosity. Therefore the amplitude permeability ($\mu_a$) of warm-compacted components are higher because of the lower porosity, stronger bonding between particles and higher density. On the other hand, though the stronger bonding between particles makes the component more compacted, it also create cracks in the surface of particles which destroys the integrity of the insulating layers and decreases the resistivity of the components. The skin depth of the conductor could be written as:

$$\Delta = \frac{\rho}{\sqrt{\pi f \mu}}$$

(1)

Where $\rho$ represents the resistivity of the material, $f$ represents the operation frequency and $\mu$ is the permeability. Cracks that were introduced during the warm compaction process in the surface of particles make parts of particles connect directly and therefore the resistivity decreased which result in the reduce of skin depth, as a result, the effective cross section of the conductor reduces which lead to lower inductance and $\mu_a$.

Figure 2(b) shows the relationship between the core loss of components and the operation frequency. The increasing tendency of total loss of SMCs with the increasing frequency could be observed from figure 2(b). The core loss of all the components are almost the same at low frequency, however, the core loss of warm-compacted components rapidly increase with the frequency and are much higher at high frequency. The total loss of the SMCs could be separated into three parts which are the hysteresis loss ($P_h$), the eddy current loss ($P_e$) and the residual loss ($P_r$). Among them, hysteresis phenomenon that appeared in various magnetic field leads to the hysteresis loss ($P_h$). The eddy current which can be divided into two parts of intra-particle and inter-particle eddy current and are induced by the changing magnetic field cause the eddy current loss ($P_e$). And the relaxation and resonant loss constitute the residual loss ($P_r$). However, it could be ignored compared to the other two parts. Therefore the total loss can be written as [3, 13, 16]:

$$P_{total} = P_h + P_e + P_r = f \int H dB + \frac{CB^2f^2d^2}{\rho}$$

(2)

where $f$ is the operating frequency, $H$ represents to the magnetic field strength, $B$ is the excited magnetic field of test, $C$ is the factor of proportionality, $d$ and $\rho$ refer to the thickness and the resistivity of the material respectively. It can be known from the equation (2) that both $P_h$ and $P_e$ are in positive correlation with frequency, therefore

![Figure 2. The effect of compaction temperature on (a) the amplitude permeability and (b) the total loss of samples versus frequency.](image-url)
$P_{\text{total}}$ increases sharply with increasing frequency. The hysteresis loss ($P_h$), which is strongly affected by the pore, impurities and other dislocations [13] in the material plays a key role at low frequency, whereas the exponential coefficient of $f$ in the eddy current loss ($P_e$) part is two, so the influence of the eddy current loss on the total loss is more important at high frequency. We have known that the warm-compacted samples own lower porosity and higher density but possess lower resistivity than the cold-pressed one, so the total loss of components at low frequency are almost the same. However, the eddy current loss of warm-compacted components sharply increases because of the lower resistivity caused by the cracks introduced in the compaction process and the total core loss becomes much higher as a result.

Figure 3 shows the amplitude permeability ($\mu_a$) and the total core loss versus the frequency of components with various density. As the density of the compacts increases, the amplitude permeability ($\mu_a$) gradually increases and then decreases. The compact with the density of 7.5 g cm$^{-3}$ shows the highest permeability. The higher ratio of the magnetic phase and the lower ratio of the porosity in the denser compact benefit the improving permeability. However, too much pressure would create cracks on the surface of the particles and incomplete insulating layer would volatilize during heat treatment. As a result, much pore appears inside the compact as non-magnetic phase. The lower amplitude permeability ($\mu_a$) of the components with the density of 7.7 g cm$^{-3}$ may be ascribed to the excessive compaction process and the pores that appear during the annealing process.

Figure 3(b) shows that the core loss of compacts with variable density are almost the same at low frequency. However, at high frequency, the core loss decreases first and then increases as the density of samples increase. The compact with the density of 7.5 g cm$^{-3}$ shows the lowest core loss. As we have discussed above that more air gaps exist in lower-density composite compacts, which would act as pinning sites to restrict both the rotation and the movement of magnetic domain wall and lead to the increasing of hysteresis loss. In addition, with the increasing of density, particles in compacts would contact more closely and resistivity would increase. As a result, inter-particle eddy current and the total loss would decrease. For the composite compact with the density of 7.7 g cm$^{-3}$, some iron powders would connect directly because of the incomplete insulting layers caused by the high pressure. Therefore, inter-particle eddy current and total loss increases.

The effect of the annealing process

The surface composition of the iron-phosphate based soft magnetic composites annealed in air has been investigated by Oikonomou et al [17]. However, the nitrogen and the steam are also commonly used as the heat-treatment atmosphere in industries. Therefore, studying the effect of different atmosphere on the magnetic properties of SMCs is valuable.

Table 1 shows the DC magnetic properties of annealed samples in different atmosphere. It could be seen that the DC magnetic properties of the nitrogen-treated compact and the air-annealed compact are similar and superior to the steam-annealed compact. It is known that the saturation magnetization is strongly related to the ratio of magnetic phase [7] and magnetic dilution effect of non-magnetic phases would decreases the saturation magnetization [18]. Lubricant could be removed in air and Nitrogen while the steam would restrain the lubricant extraction during the annealing process. As a result, the organic lubricant act as non-magnetic phase to reduce the saturation magnetization of the steam-treated compact. As we have mentioned that pore and impurities would deteriorate the permeability and improve the coercivity. Organic lubricants that are not removed from the steam-annealed compact not only act as impurities but affect the distribution of pore, therefore, the permeability decreases and the coercivity increases.
Figure 4 shows the amplitude permeability ($\mu_a$) and the total loss of compacts that annealed in the different atmosphere versus frequency. From figure 4(a), it is obvious that the annealing process significantly contribute to the amplitude permeability ($\mu_a$) at low frequency and the compact treated in the air exhibits the highest $\mu_a$ of 341 at 5 kHz. The main reason is that the annealing process is efficient to remove the lubricant and release the internal stress. The steam would restrict the extraction of lubricants and the amplitude permeability ($\mu_a$) of steam-annealed compact is relatively lower as a result. Whereas, the frequency stability of the annealed compacts are worse than that of the green compact and the nitrogen-annealed compact shows relatively the best frequency stability among the annealed compacts while the steam-annealed compact is the worst with a decrease of approximate 80 percent from 250 at 5 kHz to 50 at 100 kHz. It could be explained that both air and nitrogen are relatively inactive and would not react with the insulating layers of the powders. Oikonomou et al [17] found that the diffusion of the oxygen in air would transform the surface coating to a mixture of iron phosphates and iron oxides which are in both form of divalent and trivalent to decreases the permeability and prevent further oxidation. As to the steam-annealed compact, the steam would restrain the extraction of lubricants and destroy the surface coating. As a result, the permeability would decrease and much pore would be introduced between the particles. Both incomplete surface coating and high degree of porosity dramatically deteriarated the permeability of the steam-annealed compact.

As it can be seen from figure 4(b) that the total core loss of the green compact, the air-annealed compact and the nitrogen-annealed compact are almost the same, while the core loss of steam-annealed compact is obviously higher than the others and sharply increases with increasing frequency. We have discussed above that the insulating layers are still integrated after being annealed in air and nitrogen so that the core loss are almost the same as without-annealed compact. Whereas, owing to the breaking of coatings by steam, matrix of powders would contact directly and the inter-particle eddy current which contribute to the eddy current loss would increases dramatically as a result.

Metallography images of the cross section of compacts that annealed in nitrogen at different temperature are shown in figure 5. It could be seen from figures 5(a) and (b) that some pores distribute in grain boundaries. It could mainly be explained by the realtive poor flowability of the magnetic powders during the compaction process and the insufficient annealing temperature. Compared with the green compact and the compact annealed at 600 °C, less pores are observed in the cross section of the compact annealed at 650 °C, which is shown in figure (c). It could be explained that pores could be eliminated by efficient temperature. However, we could see from figure 5(d) that part of the insulating layers disappear and iron matrix connect directly, even sintering neck appears in some parts when the annealing temperature is 700 °C.

The resistance of green compact was 970 $\mu\Omega$.m and it slightly decreased with the annealing temperature increasing to 680 °C, the resistance of 680 °C annealed sample was 600 $\mu\Omega$.m. Increasing the annealing further

**Table 1.** DC magnetic properties of annealed samples in different atmosphere ($H_m = 10$ kAm$^{-1}$).

| Atmosphere | $\mu_i$ | $\rho_{nn}$ | $B_s$(T) | $H_c$(A m$^{-1}$) |
|------------|--------|------------|--------|------------|
| Nitrogen   | 164.41 | 430.19     | 1.28   | 124.5      |
| Air        | 184.76 | 425.19     | 1.23   | 136.7      |
| Steam      | 142.54 | 380.59     | 1.19   | 162.3      |

Figure 4. The effect of the atmosphere on (a) the amplitude permeability and (b) the total loss of samples versus frequency.
would cause the dramatic decreasing of resistance, the resistance of 700 °C annealed sample was 103 μΩ.m. This result could be explained by the fact that the resistance of SMCs is greatly affected by the thickness of insulating layers. Metallography images showed that the insulating layers became thinner and thinner as annealing temperature increased, and it even disappeared when the temperature reached to 700 °C.

Besides the excellent magnetic properties, good mechanical strengthen like high yield strength, compressive strength, transverse strength and Youngs modulus, bending strength, etc are also some of the many unique properties of Somaloy. The bending strength and, compressive strength of the green compacted were tested and the results were 8.31Mpa and 531Mpa respectively. It may increase after proper annealing process because of the existence of less pore in the annealed samples as figure 5 shows and relief of stess. Other mechanical strengthen like yield strength, and Youngs modulus might reach to 20Mpa and 150Gpa [19] after annealing process.

Figure 6 shows the effect of annealing temperature on the soft magnetic properties of compacts. The real part of permeability increases firstly and then decreases with increasing temperature. The compact annealed at 650 °C exhibits the highest permeability of approximate 325 at 5 kHz while the permeability of the compact annealed at 700 °C is severely deteriorated. We have known that internal stress and pores would impair the permeability. According to figures 5(a)–(c), heat treatment is efficient to reveal stress and eliminate pores to reduce the pinning effect so that the permeability would improve as temperature increases. However, as is shown in figure 5(d) that surface coating would decompose above a critical temperature which depends on the material of insulating layer and sintering necks appear so as to severely deteriorate the permeability.

Figure 6(c) shows the change of the core loss as a measurement of frequency. Core loss of the compacts annealed below 680 °C are almost the same and are obviously lower. However, it dramatically increases when the compact is annealed at 700 °C. It could be explained by the loss separation and the microstructure shown in figure 5(a). The total loss of SMC samples could mainly be separted into two parts which are the hysteresis loss and the eddy current loss. The hysteresis loss is related to the magnetic hysteresis and it could be written as follows:

![Figure 5. Metallography images of the cross section of samples (a) without annealed; (b) annealed at 600 °C; (c) annealed at 650 °C; (d) annealed at 700 °C.](image)
The magnetic hysteresis is caused by the hinder of domain wall motion which could by pinned by the impurities and stress regions and the $H_c$ value is the vital for hysteresis loss. The annealing process could effectively relieve the stress and minimize the pinning effect, the $H_c$ value listed in table 1 was low, so the hysteresis was low. As for the eddy current loss, it could be calculated by equation (2). The thickness of a specific sample before and after heat treatment is almost the same and the proper annealing temperature would not destroy the insulating layers which could be seen in figure 5(a). So the sample could keep a high resistivity and exhibited low eddy current loss. As a result, the total core loss could be reduced to ultra low.

Dependence of the loss factor $\tan \delta$ on measurement frequency are shown in figure 7 to investigate the effect of annealing temperature on the core loss of compacts further.

Further, in order to investigate the effect of annealing temperature on the core loss of compacts, the dependence of the loss factor $\tan \delta$ on measurement frequency are shown in figure 7. The total loss factor of the magnetic core can be written as [20–23]:

$$P_h = \int H dB$$  \hspace{1cm} (3)

where $K_1$ is the hysteresis loss coefficient and $K_2$ represents to the eddy current loss coefficient. It could be clearly seen from figure 7 that elevating the annealing temperature below 680 $^\circ$C only causes slight increasing of the total loss coefficient. However, the total core loss factor almost doubles when the annealing temperature is 700 $^\circ$C. It could be known from the equation (4) that the hysteresis loss coefficient is a constant and the eddy current loss coefficient is a liner function of frequency [24], therefore the eddy current loss coefficient ($K_2$) and the hysteresis loss coefficient ($K_1$) could be obtained by calculating the slope for each line and deriving the line to a frequency of zero respectively.

Figure 8 depicts the hysteresis loss coefficient and the eddy current loss coefficient of SMC core samples annealed at different temperature. It can be seen from figure 7 that the hysteresis loss coefficient decreases slowly with the increasing annealing temperature. It can be explained by the fact that the annealing process could effectively release the residual stress and reduce the dislocation density in samples which were introduced during the compaction process. However, the relationship between the eddy current loss coefficient and the annealing temperature is opposite which increases slightly first with the increasing annealing temperature and then
dramatically decreases when being annealed at 700 °C as a result of the disappearance of insulating layers shown in figure 5(d).

**Conclusions**

In summary, iron-based soft magnetic composites with enhanced soft magnetic properties were obtained by optimizing the compaction process and the annealing process. The room-temperature compaction process could improve the relative density of the compact without damaging the insulating layer, while the moderate heat treatment under the nitrogen could remove the lubricant and the internal stress without deteriorating the core–shell structure of powders. The SMC cores annealed under nitrogen at 650 °C exhibit the optimal magnetic performance with the real part of the magnetic permeability of 336.8 and the low core loss of 2.5 W Kg$^{-1}$ (measured at 50 mT and 5 kHz). Metallography images showed that excessive annealing temperature would cause the disappearance of surface coatings and introduce sintering necks inside compacts which were negative for the soft magnetic performance of the core.
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ORCID iDs

Yifan Pan  
https://orcid.org/0000-0001-5674-9924

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