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ORIGINAL PAPER

Effect of Iron Particles Size on the High-Frequency Magnetic Properties of Iron-Borosilicate Soft Magnetic Composites

T. Gheiratmand[1] · H. R. Madaah Hosseini[1] · F. Shalbaf[1] · M. Mohhebali[1] · M. A. Mozaffari[1] · S. H. Arabi[1] · F. Farzanegan[1]

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Abstract Iron-borosilicate magnetic composites could be applied as a soft magnetic material in high temperature and high frequency applications. In this research, the magnetic properties of soft magnetic composites with different iron particle sizes made by spark plasma sintering have been investigated. Different magnetic properties such as permeability, loss factor, and quality factor were examined up to frequencies in the order of kilohertz. The microstructural observations indicated the distribution of borosilicate on the iron grain boundaries. The results revealed that the loss factor is smaller for composites with fine particles at high frequencies. In addition, the magnetic impedance for smaller particles was greater. It was also found that the permeability and quality factor of composites with coarse particles are larger than those of fine particles. Indeed, when the particles become coarse, the density of porosities and consequently, the demagnetizing fields decrease which result in the increase of permeability. Furthermore, when the size of particles reduces, the density of grain boundaries enhances which is the main reason of lower loss factor achieved in the composites with fine particles.

Keywords Iron-borosilicate · Soft magnetic composite · Permeability · Loss factor · Quality factor

1 Introduction

Soft magnetic composites (SMC) are superior to the silicone-laminated steels owing to their isotropic magnetic properties, ease of production, and very low eddy current loss at high frequencies (in the order of MHz). Although the saturation flux density of laminated steels are around 2 T, their low permeability ($\sim 10^3$) causes a serious energy concern [1, 2]. SMCs are used in the transformers, alternating current (AC) motors and generators, switch reluctance power supplies, antilock braking sensors, magnetic field shielding, electromagnetic actuation devices, resonant inductors, frequency filters, DC output chokes, and in high-temperature applications like aircraft engine electric components [3].

The basis of SMCs is coating the iron powder particles with an insulating material which causes a barrier for eddy current flow at high frequencies [1]. Borosilicate with the trade name of Pyrex could decrease the eddy current loss at high temperatures as well as the high frequencies [2].

Actually, by modifying the particles size, it is possible to design the SMCs for specific magnetic performance [4]. Different powder metallurgy processes have been applied to produce SMCs up to now [1, 3, 5]. The best technique under which the highest density has been obtained so far is the spark plasma sintering (SPS) [2]. In this technique, heating is carried out simultaneously from both inside and outside of the sample allowing the material to be heated homogenously [6, 7]. Since the softening point of Pyrex is ~ 820 °C, the temperature of sintering could be raised up to this temperature, hence, the annealing could be carried out simultaneously to reduce the residual stress [2, 8, 9].

In this study, soft magnetic composites with different initial particles sizes of iron and also with addition of 2 wt% of borosilicate have been fabricated using spark plasma

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✉ T. Gheiratmand
t.gheiratmand@yahoo.com

✉ H. R. Madaah Hosseini
Madaah@sharif.edu

[1] Department of Materials Science and Engineering,
Sharif University of Technology, Tehran, 1458889694, Iran

sintering. The effect of iron particles size on the magnetic properties at high frequencies (such as permeability, magnetic impedance, quality factor, and loss factor) has been investigated.

2 Experimental Procedure

The initial powders were iron and borosilicate. The iron powders were screened into the three different particles sizes of $d < 45$, $45 < d < 125$, and $d > 125$ μm. Borosilicate powder was attained by milling of Pyrex glass; particles smaller than 63 μm were employed. Iron powders were mixed with 2 wt% of borosilicate in a Turbula mixer; they were then consolidated using spark plasma sintering technique under the pressure of 40 MPa at ~800 °C for 20 min. To remove the carburized layer and graphite paper from the surface, the samples were sanded and polished with the solution containing Al_2o_3 particles. The results of density measurements and some electrical and magnetic studies have been given in full detail in our previous publication [2].

The microstructure observation of samples has been performed using a field emission scanning electron microscope (MIRA3-TESCAN). To determine the magnetic properties at high frequencies, the samples were twined with copper wire. The permeability, loss factor, magnetic impedance, and quality factor of samples were measured by means of an inductance/capacitance/resistance, LCR, meter (8110G, Instech). To determine the permeability, the real and imaginary parts of permeability were calculated using the measured values of inductance and AC resistance according to the following relationships:

$$\mu' = \frac{L_s}{L_0} = \frac{L_s l_e}{\mu_0 N^2 A_e} \tag{1}$$

$$\mu'' = \frac{R_{\mathrm{ac}}}{\omega L_0} = \frac{R_{\mathrm{ac}} l_e}{\omega \mu_0 N^2 A_e} \tag{2}$$

$$\mu = \mu' + i\mu'' \tag{3}$$

where L_s and L_0 represent the inductance of the solenoid with and without a soft magnetic core, R_{ac} is indicative of AC resistance of the magnetic core, N stands for the number of turns of the cupper coil, ω (2πf) is the angular frequency, A_e is the cross section's area, and l_e is the path of mean flux density of the sample. It should be noted that L_0 could be estimated from the geometrical relations of sample as well [5, 10].

3 Results and Discussion

Figure 1 displays the secondary and back-scattered images from the microstructure of samples with different iron particle sizes which were consolidated by SPS technique. As could be seen, the borosilicate phase has been distributed on the boundaries of iron particles. The EDX analysis taken from the regions with different contrasts in the back-scattered images revealed that the dark contrast is attributed to the borosilicate phase while the regions with light contrast are ascribed to the iron. In the secondary image of sample with particles smaller than 45 μm, Fig. 1a, some pores and porosities could be observed. These porosities are distinguished with the dark contrast in the back-scattered image (Fig. 1b) as well.

Moreover, it is observed that by increasing the particles size, the amount of porosities decreases. The near full densities of 99.90 and 99.80 were obtained for samples with iron particles size of $45 < d < 125$ and $d > 125$, respectively [2], which confirms this claim.

Figure 2 shows the change of permeability as a function of frequency for samples with different particles sizes. As could be seen, the permeability of samples with coarse particles is higher than that of samples with small particles. In fact, when the size of particles decreases the fraction of defects, pore and grain boundaries in the same volume of material enhances. Thus, the ratio of free surface to the volume increases. Accordingly, based on the magnetostatic effect, the demagnetizing fields which retard the magnetization process enhance and consequently, the permeability decreases.

Generally, in alternating current (AC) electromagnets, the magnetic field is continuously changing. This causes energy losses in their magnetic cores that are dissipated as heat in the core. The dissipations are typically categorized into the three different groups: (1) hysteresis loss, (2) eddy current loss, and (3) residual loss [3]. If a circuit with a magnetic core be considered, the total loss resistance (R_s) is a combination of four different components: winding (R_c), residual (R_r), hysteresis (R_h), and eddy current (R_e) resistances. The total loss could be written as follows:

$$\tan\delta_t = \frac{\mu''}{\mu'} = \frac{R_{\mathrm{ac}}}{\omega L_s} = \frac{R_c}{\omega L_s} + \frac{R_h}{\omega L_s} + \frac{R_e}{\omega L_s} + \frac{R_r}{\omega L_s} \tag{4}$$

where ω is the angular frequency ($2\pi f$) and L is the inductance of solenoid [5, 11–13]. It should be mentioned that the residual losses arise from the different sources depending on the operational frequency. At high frequencies, they are the result of domain wall resonance and ferromagnetic resonance. While at low frequencies, the micro eddy currents developing across the domain walls are the main source for residual losses [13].

The variation of total loss factor of samples with different particle sizes has been depicted in Fig. 3. It is obviously seen that the maximum value of loss factor is obtained at low frequencies, thereafter, it decreases to the minimum value and afterwards by increasing the frequency increases gradually. In fact, the winding loss is the dominant factor at low

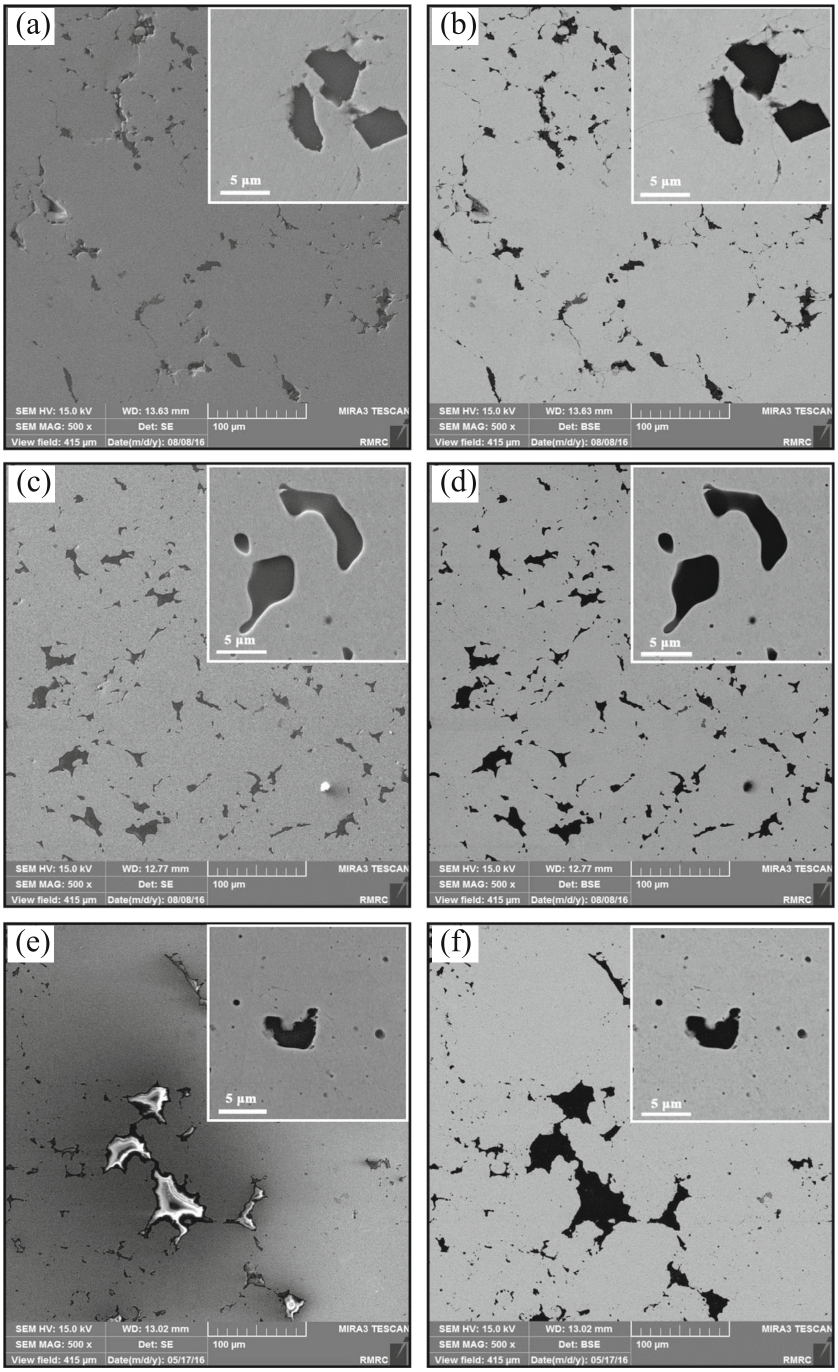

Fig. 1 The secondary and back-scattered images of microstructure of composites with initial iron particles size of **a**, **b** $d < 45$, **c**, **d** $45 < d < 125$, and **e**, **f** $d > 125$ μm

frequencies which determines the total loss. This factor is proportional to the reciprocal of frequency; therefore, it is expected that the maximum loss is attained at low frequencies. At high frequencies, the contribution of winding loss is very low that it could be neglected. Instead, the eddy current losses which are proportional to the second power of frequency [3, 8], become important.

As could be observed, the total loss factor of sample with coarse particles is slightly greater than that of fine particles. This result is due to the higher density of grain boundaries existing in the sample with fine particles. The boundaries act as a barrier for flow of eddy currents. Hence, they reduce the eddy currents and consequently, the total loss. Furthermore, it was indicated in our previous publication that the sample with coarse particles has the greater DC electrical resistivity owing to the larger open pores filled by borosilicate flow after sintering [2] , albeit the increment was rather insignificant. It is known that the eddy current loss is inversely proportional to the resistivity. Inasmuch as the loss factor of sample with fine initial particles was smaller, it could be

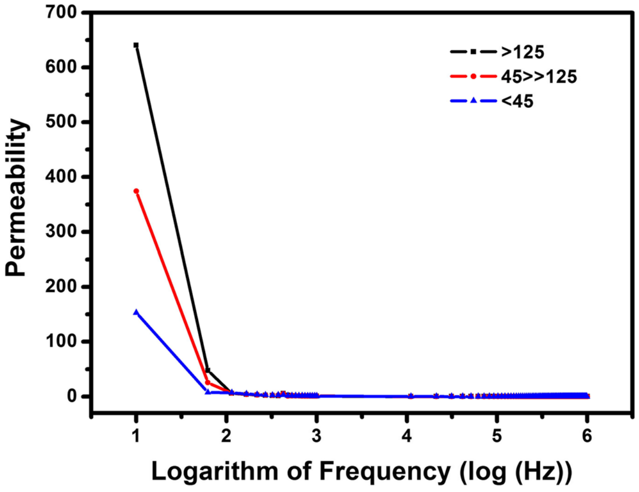

Fig. 2 Permeability for iron-borosilicate composites with different iron particles sizes

concluded that the existence of the higher density of grain boundaries are more effective on reducing the eddy currents than the larger pores filled by borosilicate flow which increased the DC resistivity slightly.

Figure 4 indicates the change of magnetic impedance as a function of frequency for composites with different iron particles sizes. The impedance is a dependence of frequency according to the following relationship:

$$z = R_{\mathrm{ac}} + jX_L = R_{\mathrm{ac}} + j2\pi f \times L_s \tag{5}$$

here, X_L is called the inductance resistance $(2\pi f)$ [14]. Since both R_{ac} and X_L increase with increase of frequency, the magnetic impedance increases as well. As it is clear in Fig. 4, the impedance of sample with coarse iron particles is higher than that of sample with fine particles. As it was explained before, when the size of particles decreases, the density of grain boundaries enhances. Thereby, more barriers are developed which could avoid the flow of eddy currents at high frequencies. Thus, the total resistivity of the material, which is indicative of its opposition to the increase of frequency enhances. This result was also obtained when discussing on the effect of particles size on the loss factor.

In a number of applications such as narrow-band filters, the eliminating of unwanted frequencies and signals is required. The quality (Q) factor is a parameter representing the ratio of inductive reactance to the resistance. This parameter is expressed by the following equation [15, 16]:

$$Q = \frac{\mu'}{\mu''} = \frac{\omega L_s}{R_{\mathrm{ac}}} \tag{6}$$

The quality factor of samples with different particles sizes has been exhibited in Fig. 5. As it is evident, by increasing the particles size, the Q factor increases. Actually, the greater the Q factor is, the narrower the band width will be. Higher Q factor indicates a lower rate of energy loss relative to the stored energy of the resonator; in other words, the oscillations die out more slowly. This implies that the samples with high Q factor oscillate within a smaller range of frequencies and are certainly more stable.

As it was shown in our previous publication [2], the samples with coarser particles reach to the higher densities after consolidation. This result is the consequence of existence of larger open pores and porosities between the coarse particles which could be filled by the flow of borosilicate after sintering. Another reason for achieving the lower densities in the samples with finer particles is the inter-particle friction and bridging effects which occur more pronouncedly for

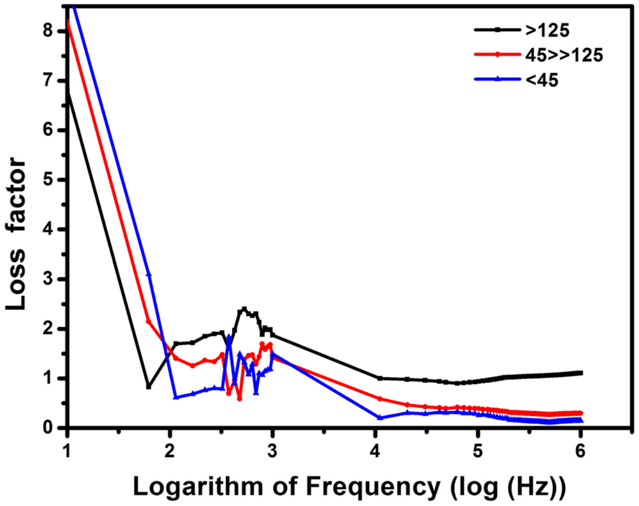

Fig. 3 Loss factor of iron-borosilicate composites with different initial particles sizes

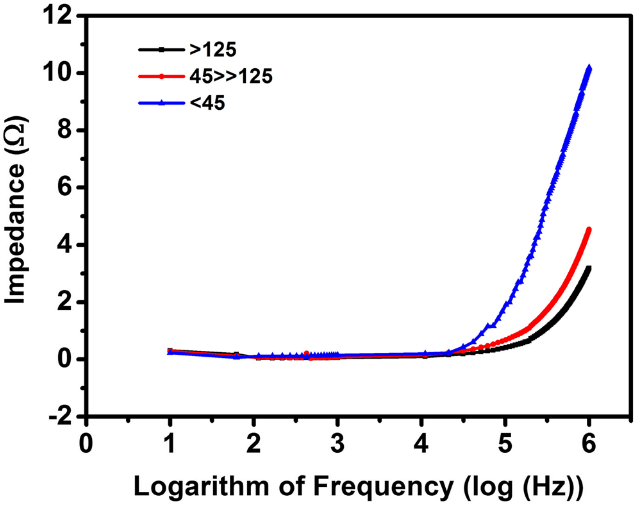

Fig. 4 Magnetic impedance for iron-borosilicate composites with different iron particles sizes

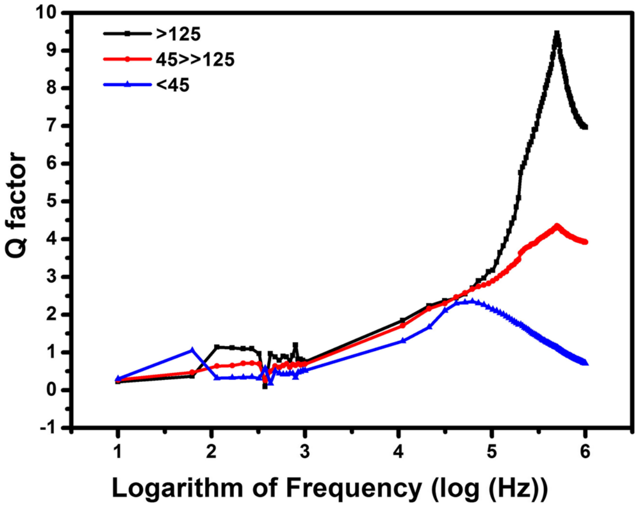

Fig. 5 Quality factor of iron-borosilicate composites with different initial particles sizes

fine particles. Indeed, the higher density of samples means that the higher amounts of borosilicate have filled the pores and air gaps between iron particles; thus, more barriers have been developed for the flow of eddy currents. Accordingly, the DC electrical resistivity of samples with coarse particles was a bit higher than that of composites with fine particles. However, the results of loss factor measurements showed that the existence of high density of grain boundaries is more effective in increasing the electrical resistivity (R_{ac}) at high frequencies.

As a result, the total resistance of composites with coarse particles at high frequencies is lower and Q factor which is inversely proportional to resistivity is greater. It should be mentioned that the reciprocal of Q factor is also called the dissipation factor which represents the loss-rate of energy of an oscillation.

4 Conclusion

In this work, the effect of initial particles size of iron on the high frequency magnetic properties of iron-borosilicate soft magnetic composites has been studied. The microstructural observation showed that the borosilicate is distributed on the boundaries of iron particles. By increasing the initial size of iron particles, the permeability increased. This result was a consequence of lower amount of porosities and defects exists in the sample with coarse grains which leads to the lower demagnetizing fields. Moreover, it was found that the loss factor of composite with fine particles is smaller than that of sample with coarse particles. The magnetic impedance was also higher for fine particles. The quality factor showed the inverse behavior, this means that it was greater for composite with coarse particles. Actually, these results are the consequence of greater AC electrical resistivity (R_{ac}) of samples with fine particles at high frequencies which is due to the high density of grain boundaries.

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