Paper

Effect of Si Addition on the Soft Magnetic Properties in High Frequency Range for Fe-Ni Alloy Powder

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ABSTRACT

Recently, soft magnetic metal powders are widely used for electric components such as inductors. These are required further improvement of magnetic properties. Fe-Ni soft magnetic alloys are called “Permalloy” and are known as high magnetic permeability alloys. In particular, “Permalloy B (JIS)”, Fe-45~50Ni, is expected for application of inductors because of high saturation magnetic flux density. We researched the effect of Si addition into “Permalloy B” on the magnetic properties in high frequency range. Powders were prepared by gas atomization process and mixed with resin and lubricant, and then compacted into toroidal shape cores. These cores were heat-treated for hardening resin and release of internal stress by compaction. Effective permeability was measured by LCR meter up to 100 MHz and core loss was measured by B-H analyzer up to 5 MHz. As a result, permeability was slightly decreased but core loss was reduced by Si addition. Core loss was also reduced by using finer powders (mean particle diameters are less than 10 μm). Moreover, the permeability increased by the heat treatment of core after compaction. We found the availability of soft magnetic metal powders in high frequency over 3 MHz for using inductors.

KEY WORDS

soft magnetic powder, Fe-Ni alloy, gas atomization, core loss, inductor

1 Introduction

Soft magnetic powders are widely used for electric components such as transformers, reactors and inductors. These are required further improvement of magnetic properties for miniaturization, improving performance and efficiency. In the past, soft ferrites have been used in high frequency of over 1 MHz because they have high electrical resistivity. Recently they have been replaced with metallic materials with high magnetic flux density. In particular, metal composite inductors which are built-in coil have been used for mobile devices at 1~3 MHz in frequency. These are expected to be applied higher frequency range in a few years later.

Fe-Ni alloys called “Permalloy” are known as high magnetic permeability alloys in the soft magnetic alloys¹⁾. In particular, “Permalloy B(JIS)”, Fe-45~50Ni, is expected for application of inductors because of high saturation magnetic flux density compared with “Permalloy C(JIS)”, Fe-79Ni-5Mo. As past research about frequency characteristics of Fe-Ni alloys, Ichinose reported decline of permeability to high frequency was suppressed by small amount of Si addition to Fe-50Ni alloy²⁾. Fukuda et al. reported permeability remained nearly constant until 1 GHz by addition of Si to Fe-50Ni alloy, and control of particle diameter and filling ratio

of powders³⁾. But these reports were related to permeability, there were little reports about core loss at high frequency over 1 MHz. Therefore, we investigated the effect of Si addition to Fe-Ni alloy and control of particle size on the permeability and the core loss in the MHz range.

2 Experiment

2.1 Effect of Si addition

For the purpose of increasing electrical resistivity, Fe-48 mass% Ni-X mass% Si (X = 0, 1.0, 1.5, 2.0) alloy powders were prepared by gas atomization in Ar atmosphere melting. These powders were sieved less than 75 μm , then measured mean particle diameters by laser diffractometry.

3.0 mass% of silicone resin was added as insulation and binder to Fe-Ni-Si powders, and then 0.5 mass% of zinc stearate was added as lubricant to them. These powders were compacted into toroidal shape cores by uniaxial press in the size of 21 mm in outer diameter, 13.5 mm in inner diameter, and 1.7 mm in thickness at 980~1078 MPa. The packing fraction of powder was adjusted to be equal control by compaction pressure. Toroidal cores were heat-treated for 1 h at 150 °C in the atmosphere to harden the silicone resin. These samples were measured the permeability and the core loss.

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Effective permeability μ_e' was calculated by equation (1) from measuring results of inductance of the toroidal cores with 10 turns in the winding⁴⁾. These were measured at 1~100 MHz in the range of frequency by LCR meter 4287A and test fixture 16093A (Agilent Technologies, Inc.).

$$\mu_e' = \frac{(L_{\text{eff}} - L_w) \cdot l_m}{\mu_0 \cdot n^2 \cdot A} \quad (1)$$

where L_{eff} is effective inductance of core, L_w is inductance of coil, l_m is magnetic pass length, μ_0 is permeability in vacuum, n is turn of coil and A is cross sectional area of core.

Samples were wound 20 turns in the primary winding and 10 turns in the secondary winding. Core loss P_c was measured at 1~5 MHz in the range of frequency for 0.01 T in the magnetic flux density by B-H analyzer SY-8218 (Iwatsu Test Instruments, Co.).

2.2 Effect of mean particle diameter

Gas atomized powders were classified to 10 μm in mean particle diameter by inertial classification. Furthermore, we investigated magnetic properties of cores which consisted of powders less than 10 μm in mean particle diameters. We prepared water atomized powders of same composition because it was difficult to obtain the powders less than 10 μm by gas atomization. Water atomized powders were classified to 3~10 μm in mean particle diameters, and then prepared toroidal cores at 1225~1421 MPa and measured the magnetic properties.

2.3 Effect of heat treatment of the magnetic core

The cores were heat-treated for 1 h at 400, 600, 700, 750 and 800 $^{\circ}\text{C}$ in Ar atmosphere. And then these cores were measured the magnetic properties.

3 Results

3.1 Effect of Si addition

The chemical compositions and the particle diameters of Fe-48Ni-Si gas atomized powders are shown in Table 1. The compaction pressures and the packing fractions of toroidal cores for gas atomized powders were shown in Table 2. As results of the magnetic measurements, the frequency characteristics of μ_e' and P_c for the powders are shown in Fig. 1 and Fig. 2, respectively. And the effects of Si addition to Fe-48Ni on μ_e' at 5 MHz and P_c at 5 MHz for 0.01T are shown in Fig. 3 and Fig. 4, respectively. μ_e' was slightly decreased with increasing the amount of Si addition. P_c was slightly reduced by Si addition.

Table 1 The chemical compositions and the particle diameters of Fe-48Ni-Si gas atomized powders.

	Ni [mass%]	Si [mass%]	Fe [mass%]	D ₁₀ [μm]	D ₅₀ [μm]	D ₉₀ [μm]
48Ni	47.87	—	Bal.	15.6	30.5	51.1
48Ni-1Si	47.93	0.97	Bal.	13.5	30.1	51.2
48Ni-1.5Si	48.30	1.42	Bal.	12.2	30.0	51.8
48Ni-2Si	47.70	1.86	Bal.	14.0	30.5	50.5

Table 2 The compaction pressures and the packing fractions of toroidal cores for Fe-48Ni-Si gas atomized powders.

	Compaction pressure [MPa]	Packing fraction [vol%]
48Ni	980	76.4
48Ni-1Si	980	74.3
48Ni-1.5Si	1078	75.0
48Ni-2Si	1078	75.3

Fig. 1 Frequency characteristics of μ_e' for Fe-48Ni-Si powders.

Fig. 2 Frequency characteristics of P_c for Fe-48Ni-Si powders.

Fig. 3 Effect of Si addition to Fe-48Ni on μ_e' at 5 MHz.

Fig. 4 Effect of Si addition to Fe-48Ni on P_c at 5 MHz for 0.01 T.

Table 3 The chemical compositions of Fe-48Ni-1.5Si gas and water atomized powders.

	Ni [mass%]	Si [mass%]	Fe [mass%]
Gas Atomized Powders	48.30	1.42	Bal.
Water Atomized Powders	48.85	1.47	Bal.

Table 4 The particle diameters of Fe-48Ni-1.5Si gas atomized powders.

	D_{10} [μm]	D_{50} [μm]	D_{90} [μm]
30 μm	12.2	30.0	51.8
10 μm	4.9	10.2	18.1

From these results, there were no significant differences in the range of 1~2 %Si addition, so we considered it would not to be expected for drastic improvement of magnetic properties in high frequency by small amount of Si addition.

3.2 Effect of mean particle diameter

The chemical compositions of Fe-48Ni-1.5Si gas and water atomized powders are shown in Table 3. The particle diameters of the gas atomized powders are shown in Table 4, and SEM images of each powder are shown in Fig. 5. The compaction pressures and the packing fractions of toroidal cores for gas atomized powders were shown in Table 5. As results of the magnetic measurements, the frequency characteristics of μ'_c and P_c for each powder are shown in Fig. 6 and Fig. 7, respectively. The frequency characteristics were significant differences in high frequency over 10 MHz. μ'_c at

Table 5 The compaction pressures and the packing fractions of toroidal cores for 48Ni-1.5Si gas atomized powders.

	Compaction pressure [MPa]	Packing fraction [vol%]
30 μm	1078	75.0
10 μm	1127	75.6

Fig. 6 Frequency characteristics of μ'_c for 48Ni-1.5Si gas atomized powders.

Fig. 7 Frequency characteristics of P_c for 48Ni-1.5Si gas atomized powders.

5 MHz were no significant differences but P_c at 5 MHz for 0.01 T were drastically reduced by using finer powder.

The particle diameters of Fe-48Ni-1.5Si water atomized powders are shown in Table 6, and SEM images of each powder are shown in Fig. 8. The compaction pressures and the packing fractions of

Fig. 5 SEM images of Fe-48Ni-1.5Si gas atomized powders: (a) 30 μm and (b) 10 μm .

Table 6 The particle diameters of Fe-48Ni-1.5Si water atomized powders.

	D_{10} [μm]	D_{50} [μm]	D_{90} [μm]
10 μm	4.4	10.1	18.6
9 μm	4.1	9.1	18.0
8 μm	3.8	8.0	14.9
7 μm	3.6	7.0	12.7
6 μm	3.0	5.9	10.7
5 μm	2.6	5.0	8.9
4 μm	1.8	4.0	6.3
3 μm	1.3	3.1	5.2

Table 7 The compaction pressures and the packing fractions of toroidal cores for Fe-48Ni-1.5Si water atomized powders.

	Compaction pressure [MPa]	Packing fraction [vol%]
10 μm	1225	77.4
9 μm	1225	77.0
8 μm	1225	77.1
7 μm	1274	77.5
6 μm	1274	77.4
5 μm	1323	77.1
4 μm	1421	77.0
3 μm	1421	76.6

toroidal cores for water atomized powders were shown in Table 7. As results of the magnetic measurements, the frequency characteristics of μ_e' and P_c for each powder are shown in Fig. 9 and Fig. 10, respectively. And the effects of mean particle diameters of the powders on μ_e' at 5 MHz and P_c at 5 MHz for 0.01 T are shown in Fig. 11 and Fig. 12, respectively.

From these results, μ_e' slightly decreased but P_c was gradually reduced with decreasing the mean particle diameter of powders.

3.3 Effect of heat treatment of the magnetic core

The effects of heat treatment temperature for Fe-48Ni-1.5Si water atomized powder on μ_e' at 5 MHz and P_c at 5 MHz for 0.01 T are shown in Fig. 13 and Fig. 14, respectively. μ_e' was improved until 750 °C with increasing heat treatment temperature, but extremely worsened at 800 °C compared with other conditions. P_c were no significant differences until 600 °C, but worsened at over

Fig. 9 Frequency characteristics of μ_e' for 48Ni-1.5Si water atomized powders. Fig. 10 Frequency characteristics of P_c for 48Ni-1.5Si water atomized powders.

700 °C. P_c at 5 MHz for 0.01 T could not be measured at 800 °C.

4 Discussion

μ_e' was slightly decreased by Si addition. It was considered that Si behaved as impurity for ferromagnetic phase.

In Fig. 6, difference in the frequency characteristics of mean particle size were caused by skin depth. Skin depth s is given by equation (2)⁵.

Fig. 8 SEM images of Fe-48Ni-1.5Si water atomized powders: (a) 10 μm , (b) 9 μm , (c) 8 μm , (d) 7 μm , (e) 6 μm , (f) 5 μm , (g) 4 μm and (h) 3 μm .

Fig. 11 Effect of mean particle diameter of Fe-48Ni-1.5Si water atomized powder on μ_e' at 5 MHz.

Fig. 12 Effect of mean particle diameter of Fe-48Ni-1.5Si water atomized powder on P_c at 5 MHz for 0.01 T.

Fig. 13 Effect of heat treatment temperature for Fe-48Ni-1.5Si water atomized powder on μ_e' at 5 MHz.

$$s = \sqrt{\frac{\rho}{\pi \cdot f \cdot \mu_r \cdot \mu_0}}, \quad (2)$$

where ρ is electrical resistivity, f is frequency and μ_r is relative permeability.

Fig. 14 Effect of heat treatment temperature for Fe-48Ni-1.5Si water atomized powder on P_c at 5 MHz for 0.01 T.

In the past researches, electrical resistivity of 50Ni-Fe was $40 \times 10^{-8} \Omega \cdot m$ and increased about $10.5 \times 10^{-8} \Omega \cdot m$ by the addition of 1 %Si²⁾. So, ρ of 48Ni-1.5Si is about $55.8 \times 10^{-8} \Omega \cdot m$. Skin depth at 20 MHz calculated by equation (2) was 18.0 μm . Mean particle diameter of 30 μm was larger than skin depth, but that of 10 μm was smaller. Therefore it was considered that the frequency characteristics of μ_e' were different.

Core loss P_c is given by equation (3)⁶⁾.

$$P_c = P_h + P_e$$

$$P_c = k_h \cdot B_m^{1.6} \times f + k_e \cdot B_m^2 \cdot f^2, \quad (3)$$

where P_h is hysteresis loss, P_e is eddy current loss, k_h is hysteresis loss coefficient, B_m is magnetic flux density and k_e is eddy current loss coefficient.

P_h and P_e separated from the results of Fig. 2 by equation (3) are shown in Fig. 15. These results show the eddy current loss is significantly influence on core loss in higher frequency range. So, we consider the reduction of eddy current loss is very important in high frequency range. The eddy current loss is given by equation (4)⁶⁾.

$$P_e = \frac{\pi^2 \cdot d^2 \cdot B_m^2 \cdot f^2}{C \cdot \rho}, \quad (4)$$

where d is particle diameter and C is shape coefficient.

From the section 3.1 and 3.2, core loss is improved by Si addition or using finer powder. In equation (4), magnetic flux density and frequency are determined by applications. From the point of view of materials, the eddy current loss depends on the particle diameter and the electrical resistivity. Comparison of the measurement results and the calculation values at 5 MHz for 0.01 T with regards to Si addition and mean particle diameter are shown in Fig. 16 and Fig. 17, respectively. In Fig. 16 and Fig. 17, the calculation values were calculated from measurement result of the value for 0 % in Si addition and 30 μm in mean particle diameter by equation (4), respectively. From Fig. 16 and Fig. 17, the

Fig. 15 P_h and P_e at 5 MHz for 0.01 T separated from the measurement results of Si addition.

Fig. 16 Comparison of the measurement results and the calculation values at 5 MHz for 0.01 T with regards to Si addition.

Fig. 17 Comparison of the measurement results and the calculation values at 5 MHz for 0.01 T with regards to mean particle diameter.

measurement results and the calculation values were not matched, but it was confirmed that P_e decreased by increasing electrical resistivity and decreasing the mean particle diameter. Furthermore, the particle diameter had a greater effect on the core loss than electrical resistivity.

In Fig. 10, P_e of 3~5 μm in the mean particle diameters decreased at 5 MHz. From the results of the considerations, we concluded that measurements were carried out correctly. However, causes of these results remained unknown.

From the section 3.3, release of internal stress by heat treatment occurred to improve permeability of powder, therefore μ'_e of core was improved. μ'_e of the core heat-treated at 400 °C was no significant difference from resin hardening because the heat treatment temperature was not sufficient to obtain these effects. At the temperature of 800 °C, it was considered that the insulation of silicone resin was destroyed by heat. The results of the core loss were no significant difference until 600 °C in heat treatment temperature. At the temperatures of 700 and 750 °C, it was considered that the insulation of silicone resin was partly destroyed by heat and then interparticle eddy current loss was generated, therefore the core loss was worsened.

5 Conclusion

In this work, we investigated the effects of Si addition and the mean particle diameter on the magnetic properties in high frequency over 1 MHz. The results indicated the mean particle diameter affected the core loss in high frequency drastically.

In case of 3 μm in the mean particle diameter of powder and 600 °C in the heat treatment temperature of core, we obtained excellent results that μ'_e at 5 MHz was 27 and P_e at 5 MHz for 0.01 T was 220 kW/m³.

From these results, we found the availability of soft magnetic powders in high frequency over 3 MHz for using inductors.

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