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Annealing Effect on Magnetic Properties and Electromagnetic Absorption Behaviors for Fe-Cr Alloy Powder-Polymer Composites

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We investigated annealing effect of microforged powders on magnetic properties and electromagnetic absorption behaviors for ferromagnetic Fe-Cr metal alloy powder-polymer composites. The coercive properties greatly decreased with annealing temperature and the magnetic permeability had significantly increased after microforging and subsequent annealing treatment, due to a reduction in lattice strain of the microforged powders. The power loss in the far field regime also had greatly increased after microforging and subsequent annealing treatment in frequency range from 50 MHz to 6 GHz. As a result, the electromagnetic absorption of ferromagnetic Fe-Cr alloy metal powder-polymer composites was highly improved because of the relaxation of the internal strain during annealing process.

Keywords : electromagnetic shielding, soft magnetic properties, permeability, composites, radio frequency

1. Introduction

For the shielding of electromagnetic noise, soft magnetic metal alloy powder-polymer composites have been studied because of their excellent soft magnetic properties such as relative permeability and saturation magnetization [1-3]. The magnetic metal alloys with a random mixture of ferromagnetic and antiferromagnetic interactions have recently attracted a lot of attention because of the discovery of giant magneto resistance. Similar to Fe-V transition metal alloys, the Fe-Cr alloy system also shows ferromagnetism over a wide range of Fe compositions. Although a lot of papers have been reported for the granular Fe-Cr alloy system [4, 5], magnetic properties of Fe-Cr plate-like powders with Fe rich composition were rarely reported. The magnetic properties of the Fe-Cr alloy system are very sensitive to heat treatment [6]. In addition, the internal stress developed in the Fe-Cr plate-like powders during microforging process can severely deteriorate magnetic properties. Thus, subsequent annealing for stress relaxation is necessary to achieve excellent soft magnetic properties. For the necessities, we investigated annealing effect of the microforged powders on soft magnetic properties and electromagnetic absorption behaviors of ferromagnetic Fe-Cr metal alloy powder-polymer composites.

2. Experimental Procedure

Plate-like Fe (89 wt.%)-Cr (11 wt.%) alloy powders that have a thickness of about 2 mm and a large aspect ratio were produced by microforging of the alloy granule powders in a high energy mechanical mill. The microforged powders were annealed in a vacuum furnace from 500°C to 800°C for 30 minutes under argon gas atmosphere, respectively. The annealed powders and silicone rubber were mixed in an agate mortar. A weight ratio of powder mixture and binder was kept constant at a ratio of 90:10. These mixtures were compacted to toroidal shaped samples with 7 mm outer diameter and 3 mm inner diameter using the uniaxial press. Magnetic properties of the powders were measured at room temperature using the vibrating sample magnetometer (VSM). The powders were also characterized by X-Ray diffraction (XRD) using Cu$K\alpha$ radiation (λ=0.154178 nm). The reference powders were

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Fe-Cr granules before microforging process. The internal strain was obtained using the Williamson-Hall plot [7] given in Eq. (1).

$$\delta(2\theta)\cdot\cos\theta_0 = 0.9\lambda/L + 4\ e\cdot\sin\theta_0 \qquad (1)$$

Where, the instrumental-broadening-corrected line profile breadth $\delta(2\theta)$ of each reflection 2θ was calculated from the parabolic approximation correction given equation [8]. θ_0 is Bragg angle of the analyzed peak, λ the incident X-ray wave length in nanometer, L the crystallite size in nanometer, and e the strain.

The high-frequency electromagnetic measurement setup consists of a Hewlett-Packard Vector Network Analyzer (VNA) 8753ES (1 kHz-6 GHz) with a synthesized sweep oscillator and scattering parameters (S_{11}, S_{21}) test set. The coaxial line sample holder was connected to the VNA. With the holder and the VNA, we measured the S parameters in the frequency range from 50 MHz to 6 GHz. For the measurement toroidal samples with inner and outer diameters of 3 mm and 7 mm were used. The thickness of toroidal samples was about 1 mm. Material parameters such as the complex permeability and the permittivity were calculated from the measured S parameters by using the simulation program of Reflection/Transmission Nicolson-Ross model. The transmission power loss was calculated by Eq. (2) and Eq. (2) is given by,

$$P_{(\mathrm{loss})}/P_{(\mathrm{in})} = 1-(|\Gamma|^2+|T|^2) \qquad (2)$$

where, Γ and T are reflection coefficient and transmission coefficient, respectively

3. Results and Discussion

Fig. 1 shows a SEM image with microforging time for the Fe-Cr magnetic powders. The thin plate-like Fe-Cr soft magnetic powders with a thickness of about 2 μm and a large aspect ratio were produced by mechanical microforging of the alloy granule powders. X-ray diffraction patterns of the plate-like powders microforged for 6 hours and annealed at different temperature for 30 minutes are shown in Fig. 2. With increasing annealing temperature, the relative intensity of the (200) diffraction peak of α-Fe crystalline phase increased and the width of peak was narrowed. The decrease in the full-widths at half maximum (FWHM) of α-Fe peak with increase of annealing temperature indicates development of refined crystallization and relief of internal strain. Fig. 3 shows the internal strain calculated from the Williamson-Hall plot at different annealing temperature. With increasing annealing temperature, the lattice strain was relieved to the level of the un-microforged granules by annealing at 800°C for 30 minutes.

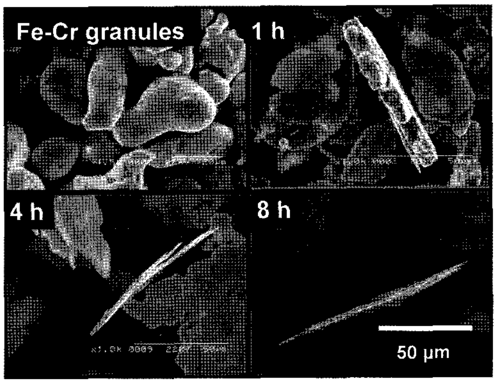

Fig. 1. SEM image of Fe-Cr powders with different microforging time.

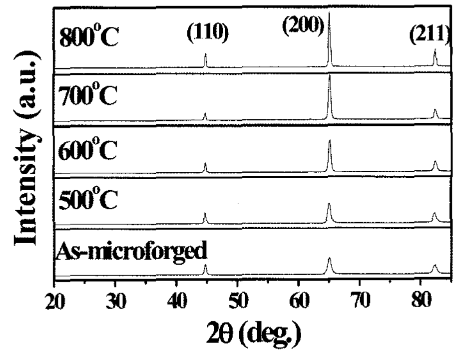

Fig. 2. X-ray diffraction patterns of microforged powders annealed at different annealing temperature.

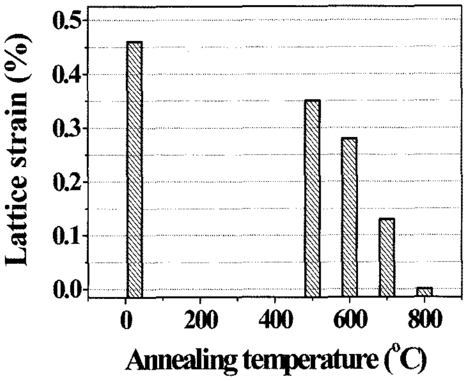

Fig. 3. Lattice strain of microforged powders annealed at different annealing temperature.

The relaxation of the internal strain shows that lattice defects decreased in the annealed powder.

The magnetic properties of the plate-like powders were measured by using a VSM in the magnetic field range from −2000 G to 2000 G. Fig. 4 shows a variation of the coercive force (Hc) and the saturation magnetization (Ms) with an annealing temperature. The annealed Fe-Cr plate-like powder at 800°C for 30 minutes considerably increased

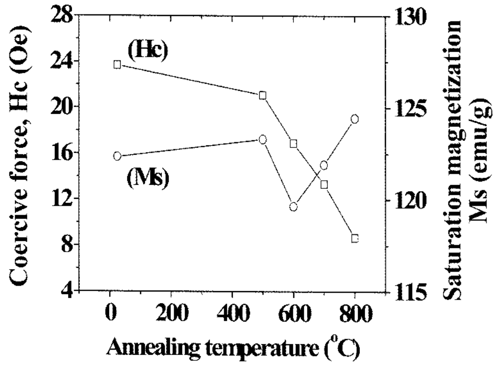

Fig. 4. Variation of the coercive force (Hc) and the saturation magnetization (Ms) with annealing temperature.

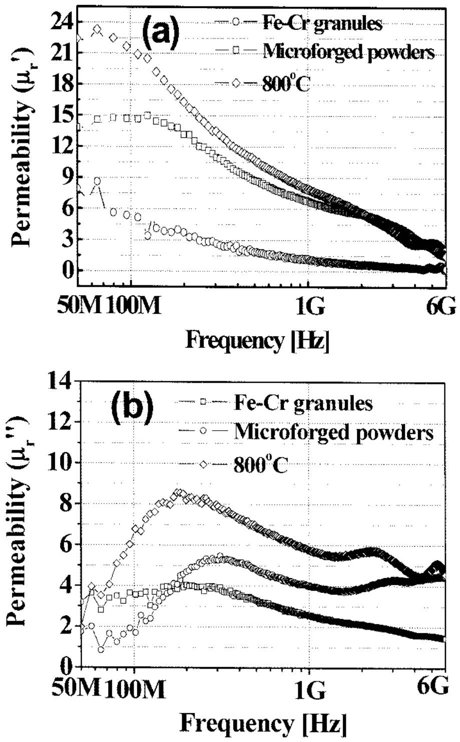

Fig. 5. Real (a) and imaginary (b) parts of magnetic permeability for the soft magnetic Fe-Cr powder-polymer composites.

soft magnetic properties. Especially, coercive force (Hc) of the annealed Fe-Cr plate-like powder was about 67% smaller than that of the as-microforged powder. As a result, the soft magnetic properties of the annealed powder were greatly improved by the relief of internal strain.

The frequency response of the real part (a) and imaginary part (b) of relative initial magnetic permeability for the soft magnetic Fe-Cr powder-polymer composites was shown in Fig. 5. The real part of initial permeability for the un-microforged Fe-Cr granule-polymer composites was very low value of ~8. The initial permeability for the as-microforged Fe-Cr powder-polymer composites was improved by the increase of the induced shape anisotropy but it was still low due to the development of the internal strain in the as-microforged powder. Owing to microforging process and subsequent annealing treatment, the magnetic permeability considerably increased in the measured frequency range. The real part of magnetic permeability for the magnetic composites consisting of the annealed powder at 800°C was 60% higher than that for the magnetic composite consisting of the as-microforged powder. The imaginary part of magnetic permeability had also significantly increased after annealing treatment. The real part of magnetic permeability of all the magnetic composites decreased rapidly above ~100 MHz. The frequency dependence of complex magnetic permeability is related to the relaxation processes of domain wall motion and magnetic spin rotation. Although the low frequency range behavior corresponds to reversible bowing of domain walls pinned at the initial permeability, it is not applicable because the domain walls unpinned and irreversibly displaced for the high frequency range in this study. However, the result of Fig. 3 clearly shows that the increase of magnetic permeability is very closely related to the decrease of lattice defects in the annealed powder. Therefore, the slight shift of relaxation frequency to lower frequency by annealing indicates due to the fact that the relaxation by domain wall motion induced from the pinning at lattice defects contributes to the magnetic permeability as well as magnetic spin rotation. K. Kawano et al showed that both spin rotation and domain wall can contribute to the magnetic permeability in the MHz frequency [9]. The contribution of the domain wall might be considered from intergranular movement between grains, because it is well-known that the intragranular domain wall movement does not contribute to the initial permeability [10, 11].

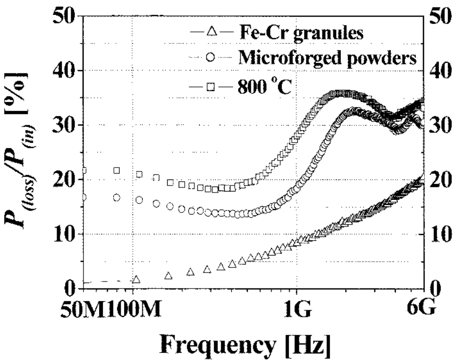

Fig. 6. Power loss of the soft magnetic Fe-Cr powder-polymer composites.

Fig. 6 showed the power loss of the soft magnetic Fe-Cr composites at different powder condition. As compared to the as-microforged powder composites, the power loss of the 800°C annealed powder composites improved over 20% higher due to annealing treatment in below 2 GHz frequency range. Consequently, the enhancement in power loss after annealing treatment was attributed to the increase in magnetic permeability by the relief of the internal strain of the as-microforged powder.

4. Conclusions

The coercive properties greatly decreased with annealing temperature and the real part and imaginary part of magnetic permeability significantly increased, due to the reduction of the lattice strain of the as-microforged powders. The power loss in the far field regime was improved by microforging and subsequent annealing treatment in frequency range from 50 MHz to 6 GHz. As a result, the electromagnetic absorption of ferromagnetic Fe-Cr alloy metal powder-polymer composites was highly improved because of the relaxation of the internal strain during annealing treatment.

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