

## GHz Range Absorption Properties of $\alpha$ -Fe/Y<sub>2</sub>O<sub>3</sub> Nanocomposites Prepared by Melt-spun Technique

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Nanocomposite materials of  $\alpha$ -Fe/Y<sub>2</sub>O<sub>3</sub> prepared by melt-spun technique exhibited good electromagnetic wave absorption properties ( $RL < -20$  dB) in a 2.0–3.5 GHz range as absorbers with thickness ranging from 3 to 5 mm, and a minimum reflection loss of  $-36$  dB was obtained at 2.6 GHz with an absorber thickness of 4 mm.

The complex permeability ( $\mu' - j\mu''$ ) and permittivity ( $\epsilon' - j\epsilon''$ ) of materials determine the reflection and attenuation characteristics of microwave absorbers. In addition, since metallic magnetic materials have large saturation magnetization,<sup>1,2</sup> and the Snoek's limit is at the higher frequency, their complex permeability values remain still high in such high frequency range.<sup>3</sup> However, the magnetization of these materials decreases owing to eddy current losses induced by electromagnetic wave. For this reason, it is better to use the smaller particles which are isolated by insulating materials such as epoxy resin. It is well-known that melt-spun technique is an excellent way to produce homogeneously microstructured materials with attractive properties for a variety of applications.<sup>4,5</sup> Many workers have investigated that fine  $\alpha$ -Fe microstructure can be produced from R-Fe compounds after hydrogenation-disproportionation reaction. For example, Sm<sub>2</sub>Fe<sub>17</sub> compounds heat-treated over 873 K in a H<sub>2</sub> atmosphere disproportionate into  $\alpha$ -Fe and SmH<sub>2</sub> phases.<sup>6</sup> Sugimoto et al. have reported good electromagnetic wave absorption properties of the  $\alpha$ -Fe/SmO composite derived from the Sm<sub>2</sub>Fe<sub>17</sub> ingots prepared by arc-melting technique.<sup>7,8</sup> In this paper, the nanocomposite materials of  $\alpha$ -Fe/Y<sub>2</sub>O<sub>3</sub> were prepared by melt-spun technique, and the electromagnetic wave absorption properties were characterized in a 0.05–20.05 GHz range.

Intermetallic compound ingots of Y<sub>2</sub>Fe<sub>17</sub> were first prepared by induction melting of high purity metals (>99.9% in purity) in Ar. Alloy ribbons with 1.5 mm width and 30  $\mu$ m thickness of Y<sub>2</sub>Fe<sub>17</sub> were prepared by a single-roller melt-spun apparatus at roll surface velocity of 20 m/s. After ball-milling, the powders with particle sizes below 3  $\mu$ m were heated at 873 K for 1 h in H<sub>2</sub> stream, followed by treating at 553 K for 2 h in O<sub>2</sub>. The resulting composite powders were characterized by XRD analysis. Epoxy resin composites were prepared from intimate mixtures of the composite powders with 20 wt% epoxy resin by pressing into cylindrical shaped compacts, followed by curing at 453 K for 30 min. They were cut into toroidal shaped samples of 7.00 mm outer diameter and 3.04 mm inner diameter.

The scattering parameters ( $S_{11}$ ,  $S_{21}$ ) of the toroidal shaped samples were measured on a Hewlett-Packard 8720B network analyzer. These parameters were used to determine the relative permeability ( $\mu_r$ ) and permittivity ( $\epsilon_r$ ) values. The reflection loss ( $RL$ ) curves were calculated from the relative permeability and permittivity at given frequency and absorber thickness with the

following equations:

$$Z_{in} = Z_0(\mu_r/\epsilon_r)^{1/2} \tanh\{j(2\pi fd/c)(\mu_r\epsilon_r)^{1/2}\} \quad (1)$$

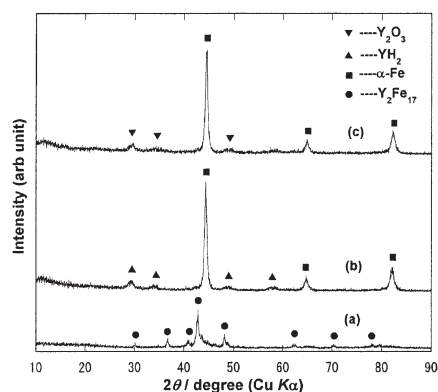
$$RL = 20 \log |(Z_{in} - Z_0)/(Z_{in} + Z_0)| \quad (2)$$

where  $f$  is the frequency of the microwave,  $d$  the thickness of an absorber,  $Z_0$  the impedance of air, and  $c$  the velocity of light.

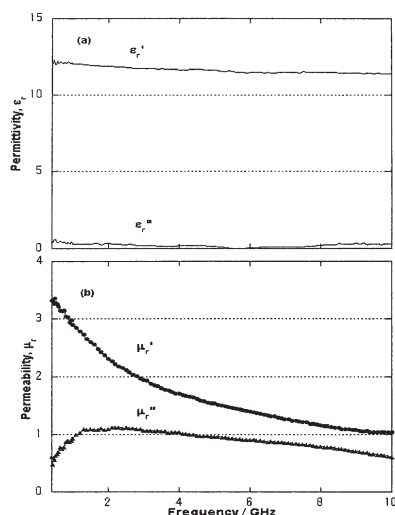
The electromagnetic wave absorption properties were determined from the frequency dependency of reflection loss ( $RL$ ).

Figure 1 shows the typical XRD patterns measured on the Y<sub>2</sub>Fe<sub>17</sub> powders: (a) as obtained, (b) after hydrogenating in H<sub>2</sub> at 873 K for 1 h, and (c) after oxidizing the sample (b) in O<sub>2</sub> at 553 K for 2 h. After hydrogenation, the resultant powder was disproportionated to  $\alpha$ -Fe and YH<sub>2</sub> phases. By the following oxidation, the peaks of Y<sub>2</sub>O<sub>3</sub> appeared (see Figure 1(c)). The grain sizes of Y<sub>2</sub>O<sub>3</sub> and  $\alpha$ -Fe were evaluated from the line broadening of the XRD peaks according to the Scherrer's formula, to be about 10 and 20 nm, respectively. In contrast with the  $\alpha$ -Fe/SmO composites,<sup>7</sup> the mean diameter of  $\alpha$ -Fe particles in  $\alpha$ -Fe/Y<sub>2</sub>O<sub>3</sub> composites (20 nm) is smaller than that of  $\alpha$ -Fe/SmO (30 nm) because of the melt-spun technique.

The frequency dependence of relative permittivity for resin composites of 80 wt%  $\alpha$ -Fe/Y<sub>2</sub>O<sub>3</sub> is shown in Figure 2(a). The real part ( $\epsilon_r'$ ) and imaginary part ( $\epsilon_r''$ ) of permittivity were almost constant in the 0.5–10 GHz range, in which the relative permittivity ( $\epsilon_r = \epsilon_r' - j\epsilon_r''$ ) showed almost constant ( $\epsilon_r' = 12$  and  $\epsilon_r'' = 0.6$ ). This indicates that the composites are of high resistivity. The resistivity value of  $\alpha$ -Fe/Y<sub>2</sub>O<sub>3</sub> composites is about 200  $\Omega$ m and ascribed to the fine  $\alpha$ -Fe and Y<sub>2</sub>O<sub>3</sub> nanoparticles, of which Y<sub>2</sub>O<sub>3</sub> plays a role as an insulator among  $\alpha$ -Fe particles.



**Figure 1.** X-ray diffraction patterns of the powders: (a) as obtained, (b) after hydrogenation–disproportionation at 873 K for 1 h in H<sub>2</sub>, and (c) after oxidation at 553 K for 2 h in O<sub>2</sub>.

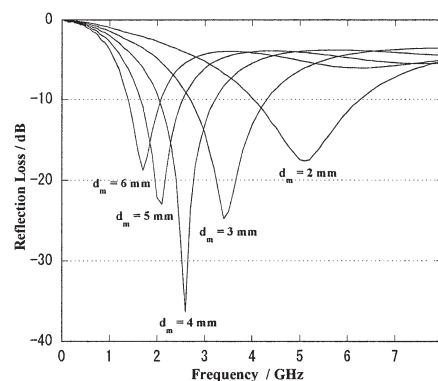


**Figure 2.** Frequency dependence of (a) relative permittivity  $\epsilon_r$ , and (b) relative permeability  $\mu_r$  for a resin composite with 80 wt%  $\alpha$ -Fe/ $Y_2O_3$  powder.

The real part  $\mu_r'$  and imaginary part  $\mu_r''$  of relative permeability are plotted as a function of frequency in Figure 2(b). The  $\mu_r'$  value declined with frequency from 3.4 to 1.03 in the 0.5–10 GHz range. However, the imaginary part of permeability  $\mu_r''$  first increased from 0.4 to 1.02 with frequency up to 2.7 GHz, and then decreased in the higher frequency range. The imaginary part of relative permeability ( $\mu_r''$ ) exhibited a peak over a wide frequency range 1.5–3.5 GHz. The maximum  $\mu_r''$  value of 1.02 was obtained at 2.7 GHz.

Figure 3 shows a typical relationship between reflection loss (RL) and frequency for the resin composites with 80 wt%  $\alpha$ -Fe/ $Y_2O_3$  powders. Firstly, the minimum reflection loss was found to move toward the lower frequency region with increasing the composite thickness. Secondly, the RL value of the resin composite below  $-20$  dB was recorded in the 2–3.5 GHz frequency range. In particular, a minimum RL value of  $-36$  dB was obtained at 2.6 GHz with a matching thickness ( $d_m$ ) of 4 mm, and the minimum  $d_m$  value of 3 mm was observed at 3.5 GHz.

Compared with the results reported by Sugimoto et al.,<sup>7</sup> the minimum RL value shifts to the higher frequency from 0.95 to 2.6 GHz with the thinner absorbers, and the electromagnetic wave absorption bandwidth, with  $RL < -20$  dB, is broadened from 0.57 to 1.5 GHz (see Figure 3). These peculiar frequency characteristics can be understood as follows: The  $\alpha$ -Fe particle size is very small (20 nm), and hence the electromagnetic wave absorption is ascribed to natural resonance of  $\alpha$ -Fe and the domain wall resonance is not effective. Y. D. Yao et al. have reported an enhancement of coercivity value ( $H_c$ ) from 100 to



**Figure 3.** Frequency dependence of reflection loss (RL) for resin composite with 80 wt%  $\alpha$ -Fe/ $Y_2O_3$  powder.

883 Oe on ultrafine  $\alpha$ -Fe (20 nm) powders.<sup>9</sup> This is due that for such ultrafine particles a large fraction of the surface atoms dominate their magnetic properties. The shift of natural resonance frequency could be attributed to the increase in  $H_c$  as suggested by Cho et al.<sup>10</sup>

In conclusion, the nanocomposite materials of  $\alpha$ -Fe/ $Y_2O_3$  are uniformly obtained by melt-spun technique after following hydrogenation–disproportionation and oxidation treatments. The good electromagnetic wave absorption properties are due to the low relative permittivity and high permeability value in the 2.0–3.5 GHz range.

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