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Enhanced electromagnetic wave absorption properties of Fe nanowires in gigaherz range

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Fe nanowires with 70–200 nm in diameter and 20–50 μm in length were synthesized by a chemical vapor deposition method for electromagnetic wave absorption application. The frequency dependences of relative permittivity (ε_r) and permeability (μ_r) were strongly dependent on the diameter of Fe wires. Compared with micrometer wires or flakelike samples, nanowires exhibited a magnetic resonance (μ_r'') peak in the range of 1–18 GHz, suggesting that nanowires have significant effect for reducing the eddy current loss, therefore, the resin compacts of 29 vol % Fe nanowires with thicknesses of 1.3–4.0 mm provided good electromagnetic wave absorption performances in the range of 5.6–18 GHz. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2775804]

The rapid development of wireless communications and high frequency circuit devices in gigahertz range calls for the study of electromagnetic (EM) wave absorbing materials. Among the candidates for EM wave absorbers, soft metallic magnets are particularly interesting[1–4] because they have large saturation magnetization values and their Snoek's limit is at a high frequency level.[5] Therefore, it is possible to make thin absorbers from metallic magnetic materials in gigahertz range.[6] However, metallic magnetic materials have high electric conductivity, which makes the high frequency permeability decreased drastically due to the eddy current loss induced by EM waves.[7] Metallic magnetic powders with submicrometer particle size dispersed in nonconductive matrix, such as resin or rubber, are favorable to EM wave absorbing materials. Fine metallic magnetic powders (e.g., Fe–Si–Al alloy, carbonyl iron, Fe, Co, or Ni and their permalloys) have been widely studied as effective EM wave absorption materials.[3,8–11] As well known, it is difficult to prepare submicrometer powders by simply mechanical grinding (MG) metallic magnets because the fracturing and cold welding of powders are repeated during the MG process.[12,13] Therefore, nanocomposites such as Fe/SiO_2, Fe/ZnO, and α-Fe/SmO have been attracting great interests.[4,14,15] More recently, the effective EM wave absorption properties of Fe catalyst encapsulated within carbon nanotubes to form nanowires or nanoparticles have been reported.[16,17] The encapsulated Fe nanowires showed high anisotropy field, which resulted in magnetic resonance shifting to higher frequency, suggesting that it can be used as EM absorber in the high frequency range. However, the permeability values decreased due to the addition of nonmagnetic components into these nanocomposites. In this letter, we report the absorption properties of Fe nanowires synthesized by a chemical vapor deposition (CVD) without using template considering easy mass production.

Fe nanowires were prepared by thermally decomposing $Fe(CO)_5$ vapor at 523 K under a flow of Ar carrier gas at a rate of 150 SCCM (SCCM denotes cubic centimeter per minute at STP) in a CVD furnace. Alternatively, the furnace temperature was increased to above 673 K while the flow rate of Ar carrier gas was maintained to 150 SCCM to give Fe microwires. When the flow rate of Ar carrier gas was declined to 50 SCCM and the furnace temperature was kept at 523 K, Fe flakelike samples were obtained. The as-obtained samples were characterized by x-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM, JEOL JSM-6320 F). Epoxy resin composites were prepared by homogeneously mixing epoxy resin with Fe wires or flakelike samples and pressing into cylindrical-shaped compacts. These compacts were cured at 453 K for 30 min, and then cut into toroidal shaped samples (ϕ_{out}: 7.00 mm, ϕ_{in}: 3.04 mm). In the 0.05–20.05 GHz range, the relative permeability (μ_r) and permittivity (ε_r) values were measured on the toroidal shaped samples using a network analyzer (Agilent Technologies E8363A). The reflection loss (RL) curves were calculated from μ_r and ε_r at the given frequency and absorber thickness.[18]

Large-scale nanowires with 70–200 nm in diameter and 20–50 μm in length were prepared when the temperature of reaction chamber was retained to 523 K under Ar flow rate of 150 SCCM [Figs. 1(a) and 1(b)]. The diameter of wires enlarged with increasing the growth temperature. When temperature was increased to 673 and 823 K, the wires had diameters of 8–12 μm [Fig. 1(c)] and 15–20 μm, respec-

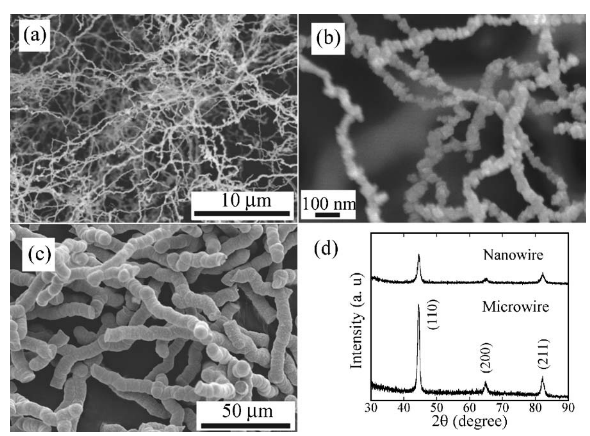

FIG. 1. FE-SEM images of [(a) and (b)] as-synthesized Fe nanowires, (c) microwires synthesized at 673 K under a flow of Ar carrier gas at a rate of 150 SCCM, and (d) XRD patterns of nanowires and microwires.

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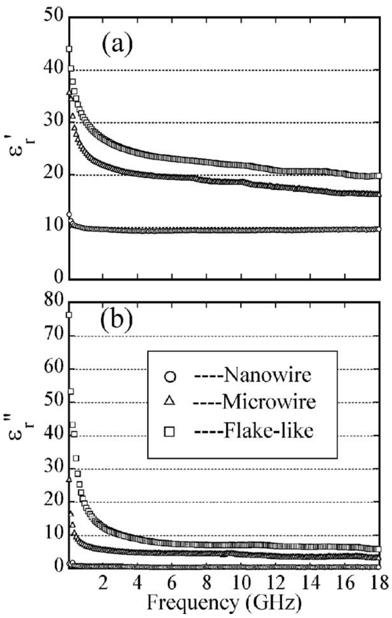

FIG. 2. Frequency dependences of (a) ε_r' and (b) ε_r'' for the resin composites with 29 vol % Fe nanowires, microwires (8–12 μm in diameter), and flakelike samples in the 0.05–18 GHz range.

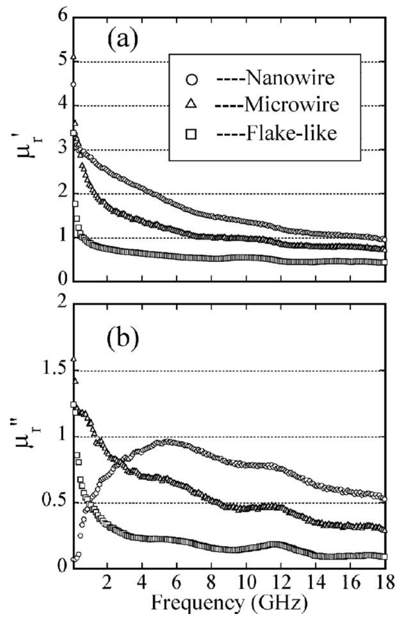

FIG. 3. Frequency dependences of (a) μ_r' and (d) μ_r'' for the resin composites with 29 vol % Fe nanowires, microwires (8–12 μm in diameter), and flakelike samples in the 0.05–18 GHz range.

tively. On the other hand, we found that the morphology of Fe was strongly dependent on the flow rate of Ar gas, which was related to the carrying quantity of $Fe(CO)_5$ into the reaction chamber. Decreasing the flow rate of Ar gas, the flakelike samples of Fe began to form. When the flow rate was decreased to 50 SCCM at 523 K, the final product mainly contained the irregular shaped flakelike samples with edge lengths of above 100 μm and thicknesses of 6–30 μm, accompanying with a small fraction (less than 1%) of nanowires. XRD data confirmed that the wires [Fig. 1(d)] and flakelike samples are α-Fe.[19]

Figure 2 show that the real part (ε_r') and the imaginary part (ε_r'') of relative permittivity for the resin composites with 29 vol % Fe nanowires were almost constant between 0.5 and 18 GHz, for which the relative permittivity ($\varepsilon_r=\varepsilon_r'-j\varepsilon_r''$) showed less variation ($\varepsilon_r'=\sim 10$ and $\varepsilon_r''=\sim 0.5$). However, for the resin composites with 29 vol % Fe microwires (8–12 μm in diameter), the ε_r' and ε_r'' values declined with increasing frequency from 35 and 26 to 16 and 3 in the 0.05–18 GHz, respectively. The similar dispersive behavior was also observed for the ε_r' and ε_r'' curves of the resin composites with 29 vol % Fe flakelike samples in the 0.05–18 GHz, in which the ε_r' and ε_r'' values decreased from 44 and 76 to 20 and 5.5, respectively. Comparing with the resin composites of microwires and flakelike samples, nanowire composites exhibited a lower permittivity level, which can be attributed to the decrease of space-charge polarization between Fe nanowires isolated by epoxy resin more efficiently after mixing with epoxy resin homogeneously. This result also agrees with the previous work, in which the permittivity of rubber composites containing Fe particles decreased with decrease in Fe particle size.[20] The real part of relative permeability (μ_r') gradually declined from 4.5 to 0.98 with increasing frequency in the 0.05–18 GHz range for the resin composites of Fe nanowires, as shown in Fig. 3(a). The imaginary part of the relative permeability (μ_r'') increased from 0.05 to 0.96 over a range of 0.05–5.6 GHz, and then decreased gradually in the higher frequency range [Fig. 3(b)]. The μ_r'' curve exhibited a peak in a broad frequency range (1–18 GHz). In comparison with Fe nanoparticles,[15] the magnetic resonance of Fe nanowires shifted to higher frequency, probably due to the large shape anisotropy field of nanowires. When the volume fraction of nanowires in the composites increased to 30 vol %, the ε_r' and ε_r'' values increased markedly to 15 and 0.9, respectively, which indicates lower electric resistivity, finally resulting in the decrease of μ_r' and μ_r''. For the resin composites with 29 vol % Fe microwires (8–12 μm in diameter), the μ_r' value decreased from 5.1 to 0.7 in the 0.05–18 GHz range. Furthermore, the μ_r'' value also declined from 1.6 to 0.3 with frequency, and no magnetic resonance peak was present in the 0.05–18 GHz range. When the resin composites contained 29 vol % Fe flakelike samples, the μ_r' and μ_r'' values decreased sharply from 3.4 and 1.25 to 0.4 and 0.1 in the 0.05–18 GHz range, respectively. The above results indicated that the dispersive behavior of permeability was determined mainly by the size rather than the morphology of Fe.

Figure 4(c) shows the typical relationship between RL and frequency for the resin composites with 29 vol % Fe nanowires. The RL values of less than −20 dB were obtained in the 5.6–18 GHz (G and X bands range) with absorber thicknesses of 1.3–4.0 mm, and a minimum RL value of −47 dB was observed at 9.4 GHz with a thickness of 2.0 mm. Compared with the resin composites of ferrites used as conventional EM wave absorption materials, the thicknesses of Fe nanowire absorbers decreased by about 30%–50% in the same high gigahertz region.[21,22] However, for the resin composites with 29 vol % Fe microwires (8–12 μm in diameter) or flakelike samples, they only gave weak absorption [Figs. 4(a) and 4(b)]. It is well known that the relative permittivity ($\varepsilon_r'-j\varepsilon_r''$) and permeability ($\mu_r'-j\mu_r''$) of materials determine the EM wave absorbing characteristics, such as frequency, thickness, and absorbing bandwidth. In this work, the measured permittivity values are of the same order of magnitude, and no dielectric response peak was observed in the 0.05–18 GHz range (Fig. 2). Therefore,

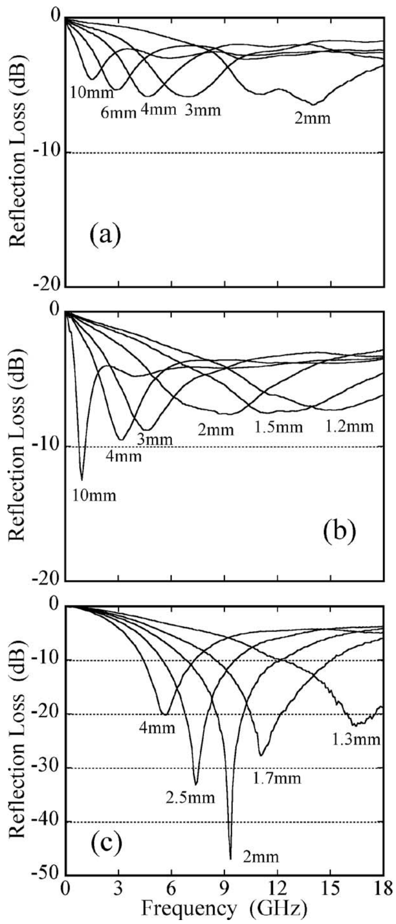

FIG. 4. Frequency dependences of the reflection loss (RL) for the resin composites of 29 vol % Fe: (a) flakelike samples, (b) microwires (8–12 μm in diameter), and (c) nanowires at different thicknesses in the 0.05–18 GHz range.

the EM wave absorbing characteristics strongly depend on the magnetic resonance of these composites. From the frequency dependence of relative permeability for the resin composites with Fe microwires (8–12 μm in diameter) or flakelike samples (Fig. 3), one can observe that the μ_r' and μ_r'' values drastically decline with frequency, and no magnetic resonance peak is present in the 0.05–18 GHz range. However, the μ_r' value gradually decreases with increasing frequency and one magnetic resonance peak can be observed in the 1.0–18 GHz range for the resin composites with Fe nanowires. The above experimental results suggested that the Fe wires with nanoscale diameter have significant effect for reducing the eddy current loss and possess a remarkable feature for EM wave absorption in the 1.0–18 GHz range. On the other hand, the Fe nanowires are isolated by the coating of epoxy resin, therefore, such composite morphology results in the weak internal interaction of Fe nanowires, which might be another key factor for the spectra of μ_r' and μ_r''.

In summary, large-scale Fe nanowires were prepared by a CVD method without using template. The resin composites with Fe nanowires showed good EM wave absorbing characteristics in the 5.6–18 GHz (G and X bands range). This study demonstrated the possible application of producing thin and light EM wave absorbers from Fe nanowires. By comparing nanowires with micrometer wires or flakelike samples, the different absorption properties mainly resulted from the size effect of Fe on the frequency dependences of relative permittivity and permeability on their resin composites.

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