Journal of Industrial and Engineering Chemistry 16 (2010) 437–440

Contents lists available at ScienceDirect

Journal of Industrial and Engineering Chemistry

journal homepage: www.elsevier.com/locate/jiec

Electromagnetic wave shielding effectiveness of $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder/epoxy composites

Hyun Bin Kim [a], Joon Pyo Jeun [a], Seong Min Hong [b], Phil Hyun Kang [a,*]

[a] Advanced Radiation Technology Institute, Korea Atomic Energy Research Institute, 1266, Sinjeong-dong, Jeongeup-si, Jeollabuk-do, Republic of Korea
[b] Department of Materials Science and Engineering, Chungnam National University, Daejeon, Republic of Korea

ARTICLE INFO

Article history:
Received 18 May 2009
Accepted 27 August 2009

Keywords:
Electromagnetic shielding
Magnetic powder
Permeability
Permittivity

ABSTRACT

Magnetic powders composed of $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ were prepared using a ball milling technique, and selected powders were annealed at 823 K in a vacuum. Scanning electron microscopy observations determined the shapes of the powders to be of a flake type. To test the electromagnetic wave absorption properties, $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ and epoxy composites were fabricated using an electron beam curing technique with powder/epoxy ratios of 20/80, 30/70, 40/60, and 50/50 by weight. The complex permeability and permittivity of the composites were measured using a network analyzer (0.5–8 GHz), and were used to calculate the refection losses as a function of the composite thickness and frequency. The band width of a reflection loss below −20 dB was 700 MHz from 4.23 GHz to 4.93 GHz at 3.8 mm thickness, attributed to the composite containing 50 wt% of powder. For the composite containing 50 wt% of annealed powder, the minimum reflection loss was observed near 8 GHz at 2.8 mm thickness.

1. Introduction

In the last decade, with the increasing use of high frequencies, there has been growing concern regarding the harmful effects of electromagnetic waves to humans and electrical devices [1]. In particular, the 1–5 GHz frequency range is used by mobile devices and wireless network systems, and in the near future, it is expected that the frequencies will shift to a higher range in order to increase the transfer rates. There are three kinds of electromagnetic wave absorption mechanisms, i.e., dielectric loss, conductive loss, and magnetic loss [2]. Among these mechanisms, however, magnetic materials are problematic for absorbing electromagnetic waves in the high frequency region due to an eddy current loss [3].

The electromagnetic wave absorption properties of materials are determined from the frequency dependency of the reflection loss (R.L.), which is calculated from the measured values of the permeability (μ_r) and the permittivity (ε_r) [4]. Thus, the permeability and the permittivity of materials in a high frequency region are dominant factors for absorbing microwaves. Nanocrystalline $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy, known as 'FINEMET', may be a good candidate for microwave absorption due to its permeability in the high frequency region. This alloy is composed of an ultrafine grain structure. These grains are composed of a bcc α-Fe solid solution, and have diameters of approximately 10 nm. This alloy exhibits excellent soft magnetic properties such as high permeability ($\mu_r \sim 10^5$ at 1 kHz), low saturation magnetostriction ($\lambda \sim 2 \times 10^{-6}$), relatively high saturation magnetization ($B \sim 1.2$ T), high resistivity ($\rho \sim 115\ \mu\Omega$ cm), and low structural anisotropy ($\langle K \rangle \sim 5$ J/m^3) [5].

In this study, microwave absorbing composites were fabricated with nanocrystalline $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder in epoxy. The experimental results and optimization of the dependency of the frequency and on the thickness of the composite are described below.

2. Experimental details

2.1. Preparation of the samples

Magnetic powders were prepared from $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy using a ball milling technique. Subsequently, selected powders were annealed at 823 K in a vacuum. The $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powders were then mixed with epoxy (diglycidyl ether of bisphenol A type) at different weight ratios of powders/epoxy: 20/80 (F1), 30/70 (F2), 40/60 (F3), 50/50 (F4), Annealed powders were also mixed with epoxy at a ratio of 50/50 (F5). A cationic initiator (triarylsulfonium hexafluroantimonate) was mixed into these samples at 3 wt%. Electron beam irradiation was carried out on each specimen using an electron beam accelerator at a dose rate of 10 kGy/scan. A 300 kGy electron beam was used to cure the epoxy resin in 150 mm × 150 mm steel molds with thicknesses of 2 mm. This electron beam curing

* Corresponding author.
E-mail address: phkang@kaeri.re.kr (P.H. Kang).

1226-086X/$ – see front matter © 2010 The Korean Society of Industrial and Engineering Chemistry. Published by Elsevier B.V. All rights reserved.
doi:10.1016/j.jiec.2009.08.010

technique was accomplished rapidly in several minutes, thus helping maintain the powder dispersion in the composite [6].

2.2. Analysis methods

The shapes of the powders were observed using a SEM (scanning electron microscope). The magnetic properties of the powders were measured with a VSM (vibrating sample magneto meter, Lakeshore, 7407). The reflection coefficients (S_{11}) and the transmission coefficient (S_{21}) of the microwaves were measured using a network analyzer (Agilent, E5071A) with a coaxial line. The toric sample dimensions were 3 mm inner diameter, 7 mm outer diameter, and 1 mm thickness. The complex permeability ($\mu_r = \mu_r' - j\mu_r''$) and the complex permittivity ($\varepsilon_r = \varepsilon_r' - \varepsilon_r''$) values were calculated from the measured S-parameters (S_{11} and S_{21}) using the Nicolson–Ross model [7].

3. Result and discussion

3.1. Microstructures of the powders

Images of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder are shown in Fig. 1. The flake type shapes of the powder particles are the result of the ball milling technique. The diameter of the powder particles is below 15 μm, and the thickness is approximately 300 nm. Flake type magnetic powders are favorable for the enhancing microwave absorbing ability [8].

3.2. Electromagnetic properties of the powders

It is well known that the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy has a nanocrystalline bcc α-Fe(Si) phase surrounded by an amorphous phase [5]. Its soft magnetic properties are explained by a random anisotropy model [9]. The magnetic properties of the powders are shown in Table 1. The coercivities of the powders are increased by the annealing effects to above the values reported for a film or core type $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ [10]. It seems that there is an influence from the rough surface of the powder, as shown in the SEM image. The saturation magnetizations of the composites increase with increasing mass fractions of the powder, which would affect the initial permeabilities of the composites shown in Fig. 2. Thus, the F4 specimen shows the highest permeability at 0.5 GHz, and the initial permeabilities decreased with a decreasing mass fraction of the powder in the composites.

The real parts of the permeability decreased with an increasing frequency and approached 1 at 7.67 GHz. The imaginary part of the permeability peaked at 4.5 GHz. On the other hand, when comparing composites with the same powder fraction, such as pristine powder (F4) vs. annealed powder (F5), the F5 sample showed a lower initial permeability in the real part, and an opposite trend in the imaginary part. The higher coercivity and magnetization seems to be caused by the annealing effects, and the increase in the remanence ratio improves the permeability properties at high frequencies.

The permeability of the composites that used a larger size $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ flake powder had a decreased imaginary part and the same real part in the high frequency regions [11]. It seems that the eddy current loss that occurred from the incidence of the electromagnetic waves was reduced due to the smaller magnetic powder size.

The real and imaginary parts of the permittivity are illustrated in Fig. 3. The real parts of the permittivity increased with an increasing fraction of the powder in the composites. Considering that the permittivity was derived from atomic and electronic polarization, it seems that these polarizations were augmented by increasing the powder fraction in the composites. There are also two peaks in the imaginary part, the first at 2.45 GHz and the second at 5.75 GHz.

Table 1
Magnetic properties of the powders.

Quantity	$Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ (pristine)	$Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ (annealed)
Magnetization, M_s	70.74 emu/g	109.31 emu/g
Remanence, M_r	8.95 emu/g	26.91 emu/g
M_r/M_s	12.65%	24.62%
Coercivity	31.92 Oe	83.40 Oe

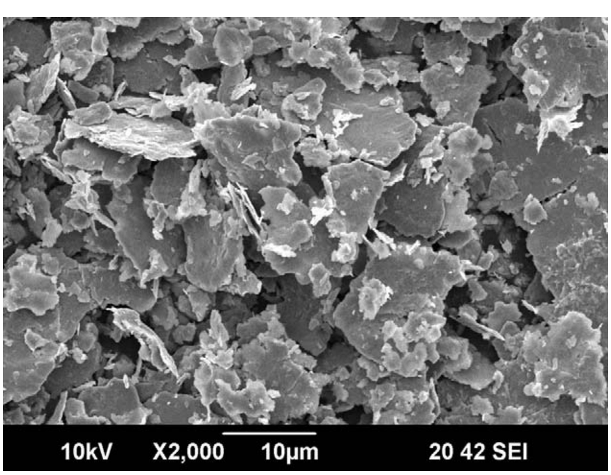

Fig. 1. SEM image of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powders.

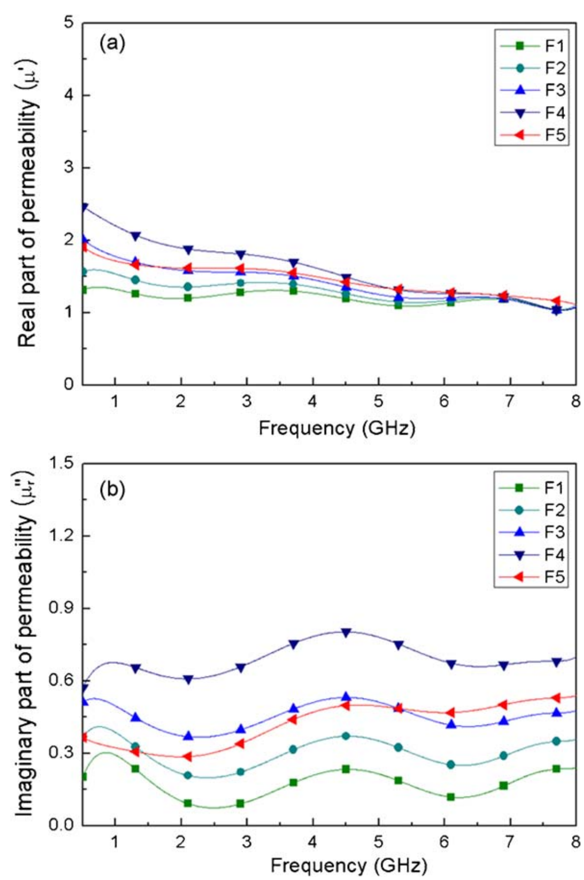

Fig. 2. The measured complex relative permeability. (a) Real part and (b) imaginary part.