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Analysis of the Permeability Spectra of Spinel Ferrite Composites Using Mixing Rules

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Abstract—Magnetic permeability spectra of composite materials containing spinel ferrites (Ni-Zn or Mn-Zn types) have been studied using a permeability dispersion formula by the numerical analysis. The variation of d.c. permeability and the frequency dispersion parameters due to domain wall and gyromagnetic spin resonances with particle content were evaluated using two mixing rules, Coherent Model Approximation (CMA) and Maxwell-Garnet Approximation (MGA) assuming the isotropic particle shape. The variation of d.c. permeability with particle content for spinel ferrite composite can be predicted by the CMA and MGA using the permeability value of embedded ferrite. The dc susceptibility and resonance frequency of domain wall and spin components estimated by the numerical analysis can also be qualitatively described by the CMA and MGA. The type of frequency dispersion, relaxation or resonance, for domain wall and spin components can be analyzed by the estimated damping factors.

I. INTRODUCTION

In electromagnetic compatibility and immunity (EMC and EMI), the suppression of undesired reflection and scattering waves, as well as reduction of currents on conducting surfaces, is one of the main issues. Various kinds of electromagnetic wave absorbers (EM-absorbers) or shielding materials are used for solving EMC/EMI problems. EM-absorbers or shielding structures employ composite materials with desirable electromagnetic, mechanical, and thermal properties [1-5]. The complex permeability and permittivity spectra, as well as the electrical conductivity of the magnetic, dielectric, and metallic materials are the important characteristics in the designing of

EM-absorbers or shielding materials. Hence adequate analysis and the prediction of material parameters of composite structure are required.

Ferrites and composite materials on their basis have been widely used for EMC/EMI devices. High frequency permeability spectra of these materials have been the subject of considerable interest [3, 6-8]. Permeability spectra of ferrites contain three components: domain wall resonance, the rotational relaxation of magnetization, and the gyromagnetic spin resonance [9-11]. In the ferrite granular composite materials, to describe frequency dispersion of permeability and its variation with content of inclusions, the demagnetizing effect in the embedded ferrite particles should be taken into account [11-13]. Demagnetization effect depends not only on the shape of particles, but also on mutual disposition/alignment of inclusions. Various mixing rules, *e.g.*, Bruggeman's effective medium Approximation (EMA) [14], the Maxwell-Garnet Approximation (MGA) [15], or Coherent Model Approximation (CMA) [16] have been considered to explain the variation of effective permeability and permeability dispersion parameters [6, 7, 17].

The objective of this study is to provide an analytical description of complex permeability spectra of granular composite materials containing spinel ferrites (Ni-Zn and Mn-Zn) over the microwave frequency range. Herein, a permeability dispersion formula, which includes the domain wall and gyromagnetic spin resonance contributions, is proposed, and frequency dispersion parameters of measured

permeability spectra are fitted numerically fit to this formula. Variation of the low-frequency permeability and the frequency dispersion parameters with volume fraction of ferrite inclusions will be discussed using the MGA and CMA mixing rules.

II. SAMPLE PREPARATION AND MEASUREMENTS

Commercially available sintered Ni-Zn ($\text{Ni}_{0.24}\text{Zn}_{0.65}\text{Fe}_{2.04}\text{O}_4$) and Mn-Zn ($\text{Mn}_{0.53}\text{Zn}_{0.41}\text{Fe}_{2.06}\text{O}_4$) ferrites were used for metal granular composite materials. Ferrite particles were prepared by mechanical grinding of sintered cores and particle size was controlled below 45 μm .

An SEM photograph of Ni-Zn ferrite particles and the size distribution of Ni-Zn and Mn-Zn ferrite particles are shown in Fig. 1. The mean particle diameters estimated by the log-normal distribution function are 3.24 μm and 2.51 μm for Ni-

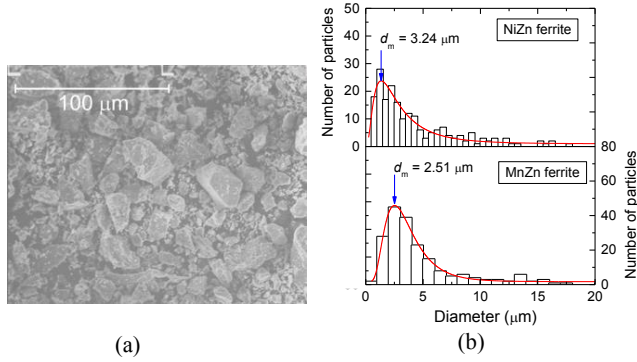


Fig. 1. SEM photograph of Ni-Zn ferrite particles (a) and distribution of particle diameters for Mn-Zn and Ni-Zn ferrite (b).

Zn and Mn-Zn ferrite particles, respectively.

Granular composite materials were prepared by mixing ferrite powders with polyphenylene sulphide (PPS) resin powder, melting the resin at 300°C and pressing the mixture at the pressure of 31.83 MPa in the cooling process down to the room temperature. The obtained samples were cut into a toroidal form with the inner diameter of 3.01 mm, and the outer diameter of 6.99 mm. The thickness of the samples of about 1 mm was controlled to avoid the dimensional resonance of the electromagnetic wave in the coaxial line. The particle content was estimated using the density values of fillers, PPS resin, and the granular composites.

Relative complex permeability $\mu_r = \mu_r' - j\mu_r''$ spectra in the frequency range from 100 kHz to 40 MHz were obtained using an impedance analyzer (HP4194A) by measuring the input impedance of samples loaded in a coaxial line [18]. Over the frequency range from 10 MHz to 6 GHz, the high-frequency complex permeability μ_r was measured by the standard 7/3-mm coaxial line techniques using Agilent E5071C network analyzer [19, 20].

III. RESULTS AND DISCUSSION

A. Coherent Model and Maxwell-Garnet Model

In this study, two mixing rules, CMA and MGA, are used to analyze the variation of magnetic permeability and related

permeability dispersion parameters with particle content (volume fraction) for spinel ferrite granular composite materials.

Coherent Model Approximation (CMA) was first introduced for the analysis of polycrystalline ferrites, in which crystal grains are surrounded by the non-magnetic grain boundary layer [16]. In this model, the complex permeability of a composite is scaled by the ratio between grain diameter D and the thickness of non-magnetic layer δ . This model can also be applied to the magnetic granular composite materials, so that the embedded particle diameter and the distance between particles correspond to D and δ [7, 21]. By the magnetic circuit calculation, the following formulas of relative permeability μ_c of the granular composite can be derived:

$$\mu_c = \frac{\mu_B(D+\delta)}{D+\mu_B\delta} \text{ and } \varphi = \left(\frac{D}{D+\delta}\right)^3, \quad (1)$$

where μ_B is the relative bulk permeability of the embedded ferrite particles, and φ is the volume fraction of ferrite in the composite. From (1), the relative permeability of the composite μ_c can be re-written as

$$\mu_c = \frac{\mu_B}{(1-\mu_B)\varphi^{\frac{1}{3}} + \mu_B}. \quad (2)$$

Since the relative magnetic susceptibility is defined by $\chi_c = \mu_c - 1$, the χ_c of the composite can be represented by

$$\chi_c = \frac{\chi_B\varphi^{\frac{1}{3}}}{1+(1-\varphi^{\frac{1}{3}})\chi_B}, \quad (3)$$

where χ_B is the magnetic susceptibility of the ferrite particles.

Another important mixing rule, the Maxwell-Garnet Approximation (MGA), is based on the Clausius-Mossotti relation for dielectric composites [15, 22]. This mixing rule can be applied for the magnetic permeability of ferrite composite materials as well, assuming that quasistatic and low comparatively ferrite contents conditions are satisfied [6]. Relative permeability of composite materials can be described as

$$\frac{\mu_c - 1}{\mu_c + 2} = \varphi \frac{\mu_B - 1}{\mu_B + 2}. \quad (4)$$

This relation yields the following mixing formulas of permeability and susceptibility:

$$\mu_c = \frac{\mu_B(1+2\varphi)+2(1-\varphi)}{\mu_B(1-\varphi)+(2+\varphi)}, \quad (5)$$

$$\chi_c = \frac{\chi_B\varphi}{1+\frac{1-\varphi}{3}\chi_B}. \quad (6)$$

Magnetic permeability of composite materials can be calculated as a function of volume fraction φ from (2) and (5). Since the bulk permeability μ_B depends on frequency over a wide frequency range, it determines the frequency dispersion of permeability for a composite.

B. Permeability Dispersion Formula and Dispersion Parameters of Spinel Ferrites and Their Composites

In the polycrystalline ferrites, two types of resonance formulas related to the domain wall (DW) vibration and

gyromagnetic spin rotation (further denoted by just “spin”) can be applied to explain the frequency dispersion of permeability. In this study, the permeability dispersion is described using Lorentz and Landau-Lifshitz-Gilbert equations [11],

$$\mu_r = 1 + \chi_d + \chi_s \quad (7)$$

$$= 1 + \frac{\omega_d^2 \chi_{d0}}{\omega_d^2 - \omega^2 + j\beta\omega} + \frac{(\omega_s + j\alpha\omega)\omega_s \chi_{s0}}{(\omega_s + j\alpha\omega)^2 - \omega^2},$$

where χ_d and χ_s are the magnetic susceptibilities for DW and spin motions, $\omega_d = 2\pi f_d$ and $\omega_s = 2\pi f_s$ are the resonance angular frequencies, χ_{d0} and χ_{s0} are the static magnetic susceptibilities of each component, α and β are their damping factors, and $\omega = 2\pi f$ is the angular frequency of the applied electromagnetic field.

In the vicinity of the spin resonance frequency, the third term of (7) can be approximated by the Lorentz type dispersion formula assuming $\alpha \ll 1$ [6], and then (7) can be rewritten by the two-term Lorentzian dispersion formula,

$$\mu_r = 1 + \frac{\chi_{d0}}{1 - \left(\frac{\omega}{\omega_d}\right)^2 + j\beta_c \frac{\omega}{\omega_d}} + \frac{\chi_{s0}}{1 - \left(\frac{\omega}{\omega_s}\right)^2 + j\alpha_c \frac{\omega}{\omega_s}}, \quad (8)$$

where $\beta_c = \beta/\omega_d$ and $\alpha_c = \alpha/\omega_s$.

On the other hand, if the damping factor α and β are very large, the DW and spin resonance components can be presented in the two-term Debye type relaxation form,

$$\mu_r = 1 + \frac{\chi_{d0}}{1 + j\frac{\omega}{\omega_{d_rel}}} + \frac{\chi_{s0}}{1 + j\frac{\omega}{\omega_{s_rel}}}, \quad (9)$$

where ω_{d_rel} and ω_{s_rel} are the relaxation frequency of DW and spin components, respectively.

Combining the permeability dispersion formula with the corresponding mixing rules, the d.c. susceptibility χ_0 , resonance and relaxation frequencies (ω_{res} and ω_{rel}) of composite materials can be calculated. In the case of the CMA, the formulas are

$$\chi_0 = \frac{\chi_{B0}\varphi^{\frac{1}{3}}}{1 - \chi_{B0}(1 - \varphi^{\frac{1}{3}})}, \quad (10)$$

$$\omega_{res} = \omega_{resB} \sqrt{1 + \chi_{B0}(1 - \varphi^{\frac{1}{3}})}, \quad (11)$$

$$\omega_{rel} = \omega_{relB} \left(1 + \chi_{B0}(1 - \varphi^{\frac{1}{3}})\right). \quad (12)$$

In the case of the MGA, the formulas are

$$\chi_0 = \frac{\chi_{B0}\varphi}{1 - \chi_{B0}\frac{1-\varphi}{3}}, \quad (13)$$

$$\omega_{res} = \omega_{resB} \sqrt{1 + \chi_{B0}\frac{1-\varphi}{3}}, \quad (14)$$

$$\omega_{rel} = \omega_{relB} \left(1 + \chi_{B0}\frac{1-\varphi}{3}\right), \quad (15)$$

where ω_{resB} , ω_{relB} , and χ_{B0} are the bulk ferrite parameters. As is seen from comparing (11) and (12), or (14) and (15), the dependence of ω_{res} on the volume fraction φ is different from that of ω_{rel} . Therefore, the permeability dispersion type (resonance or relaxation) for composite materials can be

evaluated from the variation of ω_d or ω_s with φ . Further, the damping factor of permeability dispersion in composite materials can also be examined from the CMA and MGA using the relations: $\beta_c = \beta/\omega_{res}$ and $\alpha_c = \alpha/\omega_{res}$ or $\beta_c = \beta/\omega_{rel}$ and $\alpha_c = \alpha/\omega_{rel}$.

C. Complex Permeability Spectra of Ni-Zn and Mn-Zn Ferrite Granular Composite Materials

The measured complex permeability spectra of Ni-Zn ferrite and Mn-Zn ferrite composite materials at several particle

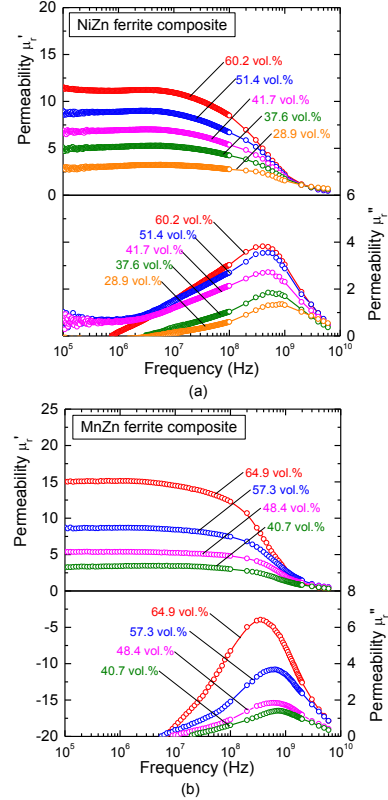


Fig. 2. Complex permeability spectra of Ni-Zn (a) and Mn-Zn (b) ferrite granular composite materials.

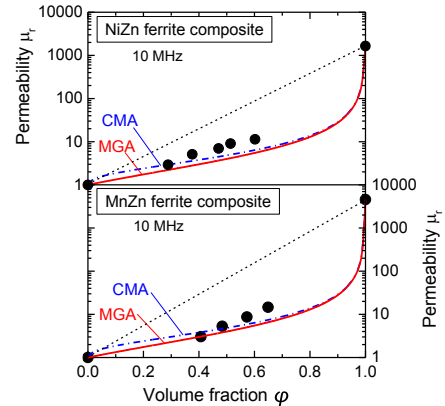


Fig. 3. Absolute permeability at 10 MHz as a function of volume fraction. Blue and red lines indicate the calculation curves by CMA and MGA, respectively.

contents are shown in Fig. 2. Sintered bulk ferrites have the relative permeability values of $\mu_r = 4550$ for Mn-Zn ferrite and 1650 for Ni-Zn ferrite at 10 kHz. Since the μ_r value becomes 10 to 15 at 60 vol.% content, a large decrease of μ_r is observed in the composite structure. In both composites, the real part of relative permeability μ_r' shows frequency dispersion starting from several MHz. The imaginary part μ_r'' has a broad maximum in the frequency range of a few hundred megahertz. The low-frequency μ_r' decreases with the decreasing particle content. Simultaneously, the peak of μ_r'' slightly shifts to the higher frequencies with decreasing particle content.

The low-frequency permeability dependences on the volume fraction ϕ of ferrite for the two spinel ferrite composites are shown in Fig.3. For both composites, the value of μ_r at 10 MHz is assumed to represent the d.c. permeability value. Blue dashed-dotted and red solid lines indicate the theoretical curves calculated from (2) and (5) using the abovementioned bulk ferrite $\mu_r = \mu_B$ values. Fairly good agreement between experimental and theoretical values is obtained. Hence the variation of d.c. permeability with volume fraction can be described by the CMA and MGA mixing rules for the spinel ferrite composite materials. The CMA predicts a slightly higher permeability value than the MGA in the low-volume fraction range. In both mixing rules, the demagnetizing effect is taken into account in the different manner. The MGA contains the Lorentz local field concept, while the CMA is derived from the magnetic circuit calculation through Ampère's integral.

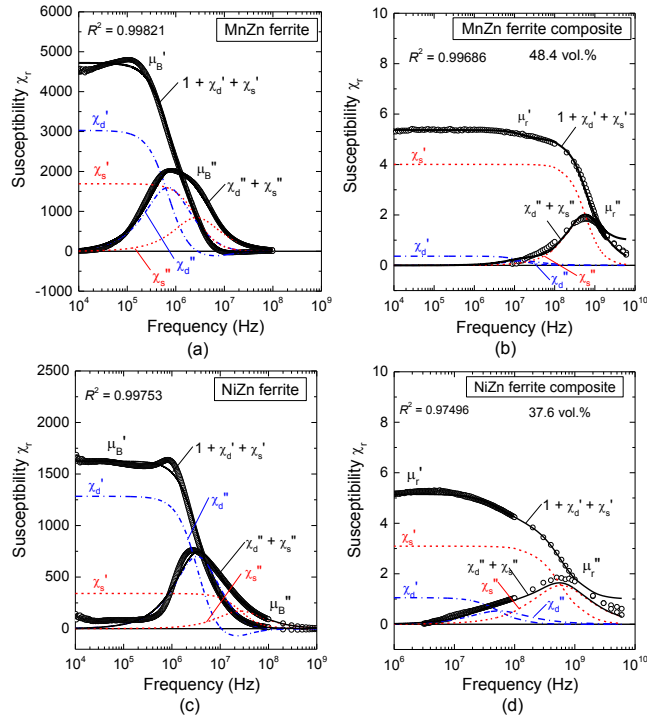


Fig. 4. Complex permeability spectra of MnZn ferrite (a), MnZn ferrite composite (48.4 vol.%) (b), NiZn ferrite (c) and NiZn ferrite composite (37.6 Vol.%) (d). Blue and red lines indicates the calculation curves for DW and spin components, respectively. Black lines are permeability dispersion ones.

Accordingly, the d.c., or low frequency permeability of spinel ferrite can be predicted by these mixing rules. It should be noted that as Fig.1 shows, the spinel ferrite inclusions are of the arbitrary crumb-like shape, so the demagnetization factors should be approximately 1/3 in each direction; they are arbitrary dispersed, no special alignment. If the particles were flakes or spheroids, then the MGA in the form as in this study will be not applicable; modified Bruggeman Asymmetric Rule or other approximations should be applied [23].

D. Numerical Analysis of Permeability Spectra of Spinel Ferrite Composites

The d.c. susceptibilities χ_{d0} and χ_{s0} , resonance frequencies f_d and f_s , and damping factors β and α of DW and spin resonances of spinel ferrites were evaluated by the non-linear least mean square fitting of the measured data to the real part of (7). The results of fitting are shown in Fig.4 for bulk Mn-Zn ferrite; 48.4 vol.% Mn-Zn ferrite composite; bulk Ni-Zn ferrite; and 37.6 vol.% Ni-Zn ferrite composite. The blue dash-dotted lines indicate the calculated real and imaginary parts of DW susceptibility χ_d' and χ_d'' using the determined dispersion parameters, respectively. The red dashed lines correspond to those of spin susceptibility χ_s' and χ_s'' . Black solid lines exhibit the calculated permeability dispersion curves. From this result, it may be concluded that the large low-frequency permeability of sintered spinel ferrites is mainly originated by the domain wall vibration. On the other hand, the spin contribution is dominant in the spinel ferrite composites.

E. Variation of Permeability Dispersion Parameters with Volume Fraction in Spinel Ferrite Composites

The variations of the d.c. susceptibility, resonance frequency, and damping factor with particle content for DW and spin components of permeability spectra were analyzed using the CMA and MGA. Fig. 5 shows the resonance frequency and the d.c. susceptibility of the DW and spin components as a function of volume fraction ϕ for Mn-Zn ferrite composite. Solid circles indicate the experimental values. Blue and red solid lines indicate the theoretical curves calculated from (10) and (11) by CMA, and (13) and (14) by MGA, respectively. Fig.6 shows the same analysis data for Ni-Zn ferrite composites. From Fig.5 (b) and Fig.6 (b), the estimated d.c. susceptibility of DW and spin components has fairly good agreement with theoretical curves for both ferrite composites. Then the variation of the d.c. susceptibility with ϕ can be described by the two mixing rules for the DW and spin contributions. However, the discrepancy between experimental and theoretical values in the d.c. susceptibility of the domain wall component, corresponding to the Mn-Zn ferrite composite, is relatively large.

From Fig.5 (a) and Fig.6 (a), the variation of the resonance frequency of spin component f_s with volume fraction can also be described by the CMA and MGA rules. On the other hand, the volume fraction dependence of the DW resonance frequency f_d indicates the relaxation type permeability dispersion in the Mn-Zn ferrite composite; in the Ni-Zn ferrite composite, the experimental data of f_d is located between the

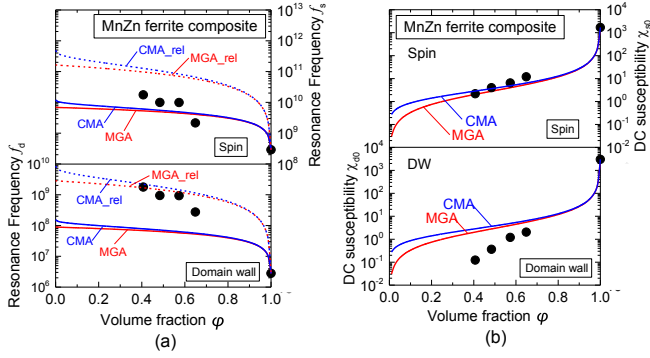


Fig. 5. DW and spin resonance frequencies (a), d.c. susceptibility of DW and spin components (b) for the MnZn ferrite composite as a function of volume fraction ϕ . Blue and red lines are the calculation curves from CMA and MGA.

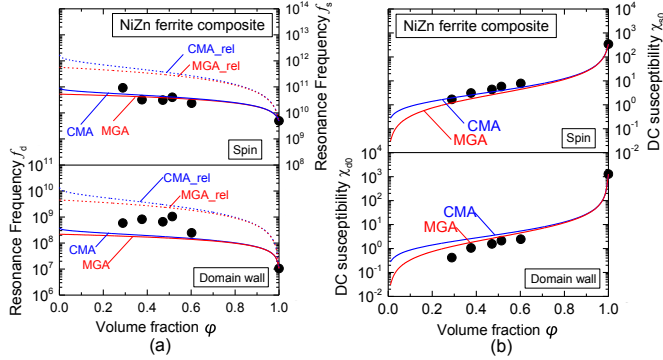


Fig. 6. DW and spin resonance frequencies (a), d.c. susceptibility of DW and spin components (b) for the NiZn ferrite composite as a function of volume fraction ϕ . Blue and red lines are the calculation curves from CMA and MGA.

theoretical curve of resonance and that of relaxation. Hence, it is considered that the permeability dispersion of the domain wall component is the relaxation type, and that of spin component is the resonance one.

The variations of the normalized damping factors β/f_d and α/f_s with volume fraction are shown in Fig.7 for Mn-Zn and Ni-Zn ferrites. Blue solid and red dashed lines are the calculation curves from the CMA and MGA. Both mixing rules can quantitatively predict the variation of the spin damping with volume fraction ϕ . However, for the DW damping variation, a large discrepancy was observed in both spinel ferrites. This may be caused by the difference of the magnetic

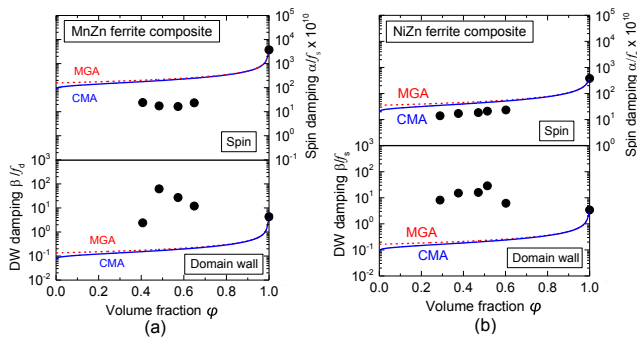


Fig. 7. Normalized damping factors of DW β/f_d and spin α/f_s as a function of volume fraction for Mn-Zn ferrite (a) and Ni-Zn ferrite (b). Blue and red lines are the calculation curves from CMA and MGA.

domain structure between bulk ferrite and embedded ferrite particles due to demagnetization in ferrite inclusions. Since the domain wall resonance is sensitive to the microscopic structure of magnetic domains, the prediction of the DW components, especially damping factor, from those of the bulk ferrite is considered to be difficult.

It should be noted that the two mixing models in this study, CMA and MGA, assume the isotropic particles in the composite structure; the effect of the shape anisotropy of particles or the effect of the distribution of particle size are not taken into account. Hence the mixing theories considering the effect of particle shape or particle size distribution on the permeability dispersion [24, 25] may have to be employed for the quantitative analysis of the dispersion parameters.

F. Effect of Damping for Permeability Dispersion Shift

From the numerical analysis, two kinds of complex susceptibility spectra corresponding to the domain wall and spin resonances can be obtained; the imaginary part of susceptibility χ'' has a maximum at a certain frequency. This maximum frequency is related to the resonance frequency of each component but generally does not coincide with the resonance frequency due to the damping effect. The maximum angular frequency of domain wall and spin resonances, $\omega_{d,max}$ and $\omega_{s,max}$ can be derived from the imaginary part of (7) as a function of damping factors by the equation $d\chi''/d\omega = 0$. The following formulae can be obtained for the domain wall and spin components [11]. For the domain wall damping, this is

$$\frac{\omega_{d,max}}{\omega_d} = \frac{1}{6} \sqrt{12 - 6\left(\frac{\beta}{\omega_d}\right)^2 + 6\sqrt{16 - 4\left(\frac{\beta}{\omega_d}\right)^2 + \left(\frac{\beta}{\omega_d}\right)^4}}, \quad (15)$$

and for the spin damping, the formula is

$$\frac{\omega_{s,max}}{\omega_s} = \frac{1}{\sqrt{\alpha^2 + 1}}. \quad (16)$$

The relationship between the normalized maximum angular frequencies ($\omega_{d,max}/\omega_d$ and $\omega_{s,max}/\omega_s$) and damping factors

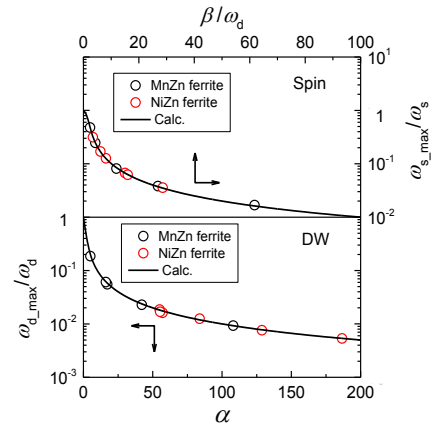


Fig. 8. Normalized maximum angular frequency for domain wall resonance $\omega_{d,max}/\omega_d$ and spin resonance $\omega_{s,max}/\omega_s$ as a function of damping factor β/ω_d and α . Solid lines indicate the theoretical curves from Eqs. (15) and (16).

(β/α_d and α) is shown in Fig. 8. Open circles indicate the normalized maximum angular frequencies determined by the numerical analysis of experimental data. The solid curves are the theoretical variation of the maximum frequency with damping factor calculated by (15) and (16). Fig. 8 shows that the experimental and calculated values agree well. Hence the proper damping factors of permeability dispersion in spinel ferrite composite materials can be obtained by the numerical fitting for each composite material. Further, the effect of damping on the permeability dispersion shift can be demonstrated by the numerical analysis of complex permeability spectra.

IV. CONCLUSION

Magnetic permeability spectra of spinel ferrite composite materials have been studied by the numerical analysis and the two mixing theory (Coherent Model Approximation and Maxwell Garnett Approximation). The variation of the d.c. permeability with volume fraction can be described by the two mixing theories for both Mn-Zn and Ni-Zn ferrites. The d.c. susceptibility, resonance frequency, and damping factor of domain wall and gyromagnetic spin resonances were estimated by the numerical analysis of experimental data. The variation of the permeability dispersion parameters except for the domain wall damping factor with particle content can be predicted by the two mixing rules. The discrepancy between theory and experiment for the domain wall damping may be attributed to the difference of the magnetic domain structure between bulk ferrite and embedded ferrite particle. The effect of damping on the resonance frequency shift can be demonstrated by the numerical analysis.

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