Fabrication and electromagnetic characteristics of microwave absorbers containing carbon nanofibers and magnetic metals

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ABSTRACT

The ultimate aim of this study is the development of microwave absorbers containing both dielectric and magnetic lossy materials. Carbon nanofibers (CNFs) were used as dielectric lossy materials and NiFe particles were used as magnetic lossy materials. Total twelve specimens for the three types such as dielectric, magnetic and mixed radar absorbing materials (RAMs) were fabricated. Their complex permittivities and permeabilities in the range of 2~18 GHz were measured using the transmission line technique. The parametric studies in the X-band (8.2~12.4 GHz) for reflection loss characteristics of each specimen to design the single-layered RAMs were performed. The mixed RAMs generally showed the improved absorbing characteristics with thinner matching thickness. One of the mixed RAMs, S09 with the thickness of 2.00 mm had the 10 dB absorbing bandwidth of 4.0 GHz in the X-band. The experimental results for selected specimens were in very good agreements with simulation ones in terms of the overall reflection loss characteristics and 10 dB absorbing bandwidth.

Keywords: radar absorbing materials (RAMs), carbon nanofibers (CNFs), NiFe particles, complex permittivity and permeability, reflection loss

1. INTRODUCTION

The absorption and the interference shielding of electromagnetic wave have been very important issues for commercial and military purposes. Electromagnetic interference (EMI) shielding problems are increasing in electronic and military communication owing to sensitive electronic devices and densely packed systems, so that the research on the EMI shielding has received more and more attention in recent years. On the other hand, the stealth technique is the most typical application of electromagnetic wave absorption technology. By reducing the detectability of aircrafts or warships, of which the radar cross section (RCS) is a measure, they could evade radar detection, which affects not only the mission success rate but also survival of them in the hostile territory. In facts, the stealth technique can be categorized into two methods. One is the shape optimization of the body so that incident electromagnetic wave can be scattered yielding minimum reflective wave. The other is the use of electromagnetic wave absorption materials and/or structures. In the early stage, many researchers mainly concentrated on the reduction of RCS and development of the radar absorbing materials (RAMs), but nowadays investigation on the radar absorbing structures (RASs) using fiber reinforced polymeric composite materials has received much attention. Generally, electromagnetic wave absorption characteristics of material depend on its dielectric properties (the permittivity), magnetic properties (the permeability), thickness and frequency range. [1-3].

There have been many studies on RAMs such as composites containing conductive fillers or magnetic materials. However, some problems still remain in those studies. The dielectric absorbers with dielectric lossy materials have the heavy matching thickness and narrow absorbing bandwidth. At the same time, the magnetic absorbers with magnetic

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lossy materials also have the heavy weight and poor characteristics in the GHz range due to their Snoek's limit [4]. The enhanced electromagnetic wave absorbers require lightness, thinness and wide-absorbing bandwidth. Recently, nano-composites containing both dielectric and magnetic lossy materials have been studied to solve these problems. Che et al. [5] fabricated the carbon nanotube/Fe composites and enhanced microwave absorption characteristics. Pan et al. [6] prepared the electroless Ni-P deposited strontium ferrite powders and measured the complex permittivity and permeability. These are the experimental studies to develop the new composite fillers and RAM. Saitoh et al. [7] fabricated composites mixed with dielectric titanium oxide powders and magnetic carbonyl iron powders. They designed the double-layered microwave absorbers with the broad-band absorbing characteristics.

This research has focused on the fabrication and performance evaluation of radar absorbing materials (RAMs) containing both dielectric and magnetic lossy materials. Carbon nanofiber (CNFs), conductive nano particles were used as dielectric lossy materials, and NiFe particles were used as magnetic lossy materials. Total twelve specimens with three types (dielectric, magnetic and mixed RAMs) were fabricated according to the weight of added fillers and compound ratio. Microstructures of the cross sections for mixed RAMs were observed to examine the dispersion state. The complex permittivities and permeabilities of specimens in the range of 2~18 GHz were measured using the transmission line technique. The parametric studies for reflection loss characteristics of each specimen to design the single-layered RAMs were conducted using the obtained electromagnetic data and numerical formula in a multi-layered medium. Finally, the experimental studies to evaluate the reflection loss characteristics of selected specimens were performed using the transmission line technique.

2. SELECTION OF LOSSY MATERIALS

Carbon nano particles such as carbon nanotubes (CNTs) and carbon nanofibers (CNFs) have been recently used as hopeful raw materials because of their outstanding physical and electrical properties. It is known that composites containing carbon nano particles may have various merits such as high tensile strength, elastic modulus, thermal conductivity and electrical conductivity according to fabrication processes and content ratios [8].

In this study, the CNFs (Showa Denko, Japan) were used to increase permittivity as dielectric lossy fillers. Its typical diameter and length are 100~200 nanometers and 10~20 micrometers. Fig. 1 shows a scanning electron microscopy (SEM) image of as-received CNFs.

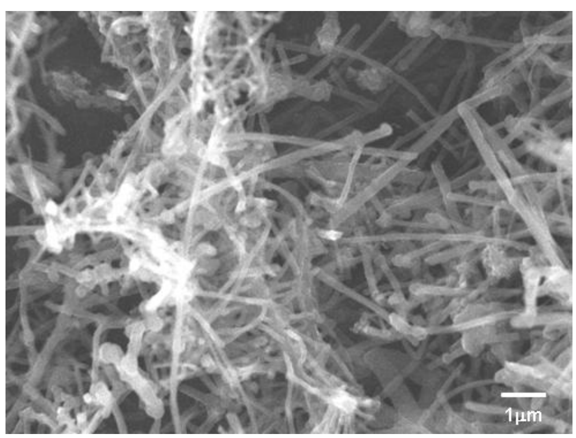


Fig. 1. SEM image of as-received CNFs.

Magnetic lossy materials are mainly divided into two kinds, ferrites and ferromagnetic materials. In the case of conventional ferrite materials, they have the good absorbing/shielding characteristics for electromagnetic wave in the resonance frequency range. However, it is known that they have the bad absorbing/shielding characteristics due to low Sneok's value in the high frequency range. Their added contents over 70~80 wt% to obtain regular permeability values are also important defects in terms of weight and efficiency. On the other hand, the ferromagnetic materials have high

Snoek's value in the high frequency range and large saturation magnetization comparing ferrites. They can maintain regular complex permeability values in the GHz frequency range and be applied to thin microwave absorbers. However, there is a serious defect, eddy current loss for using microwave absorbers. The metallic magnetic materials have the high electric conductivity so that the magnetic properties decrease rapidly with the frequency increase due to the eddy current loss induced by electromagnetic waves [4, 9]. Generally, the skin-depth of magnetic metallic particles in the GHz frequency range is known as about several micro meter. The solutions to suppress the eddy current loss are to use smaller particles than the skin-depth and to isolate them by the non-conductive materials such as polymer resin or rubber. Therefore, it is most effective to use the submicron sized ferromagnetic particles and fabricate the composites with polymer resin.

NiFe particles were used to increase permeability as magnetic lossy fillers in this study. They were fabricated by applying NiFe wire to the pulsed wire evaporation method. Fig. 2 shows a SEM image of as-received NiFe. Their sizes were distributed in the range from submicron to several micro meter. The micro-sized particles probably result from the local agglomeration phenomena. The composition of NiFe particles was analyzed as about 52 at% Ni and 48 at% Fe through the energy dispersive spectroscopy (EDS).

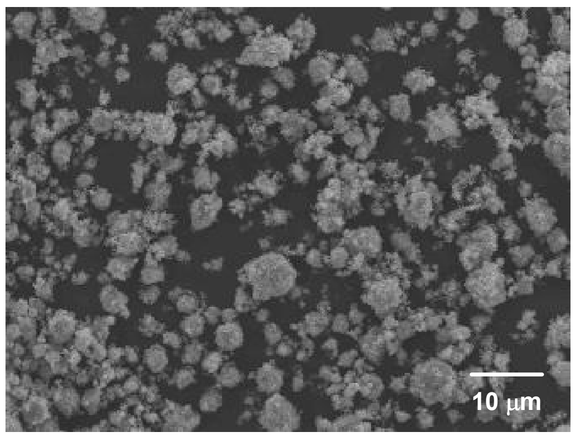


Fig. 2. SEM image of as-received NiFe particles.

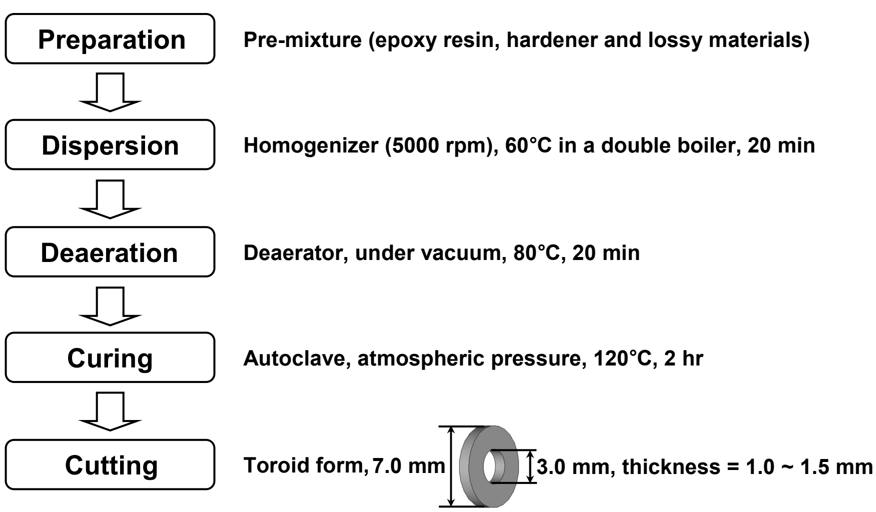


Fig. 3. Experimental procedure for fabrication of composites.

3. FABRICATION OF COMPOSITES AND MEASUREMENT OF ELECTROMAGNETIC CHARACTERISTICS

3.1 Fabrication of composites

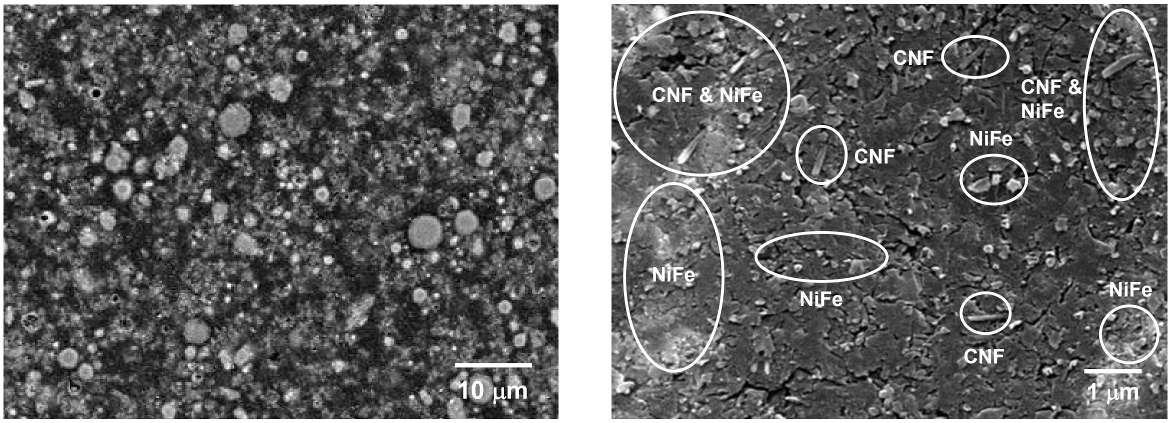
First, the pre-mixture containing epoxy resin, hardener and lossy materials was prepared. While maintaining 60°C in a double boiler, this mixture was sufficiently dispersed using the homogenizer with the speed of about 5000 rpm for 20 minutes. To remove the dissolved air or gas formed during mixing process, the deaeration process was conducted in a deaerator under vacuum for 20 minutes at 80°C. Then, the curing process was performed in an autoclave for 2 hours at 120°C and atmospheric pressure. Finally, the fabricated specimens were cut as the cylindrical toroid form (inner diameter: 3 mm, outer diameter: 7 mm, thickness: 1.0 ~ 1.5 mm) to measure the electromagnetic properties. Fig. 3 shows the experimental procedure for fabrication of composites.

Total twelve fabricated composites were classified into three kinds. The specimens of dielectric type (S01, S02 and S03) were composites containing only CNFs and the weight percentages of added CNFs were 0.5 wt%, 1.0 wt% and 2.0 wt%, respectively. The specimens of magnetic type (S04, S05 and S06) were composites containing only NiFe particles and the weight percentages of added NiFe particles were 10 wt%, 30 wt% and 50 wt%, respectively. The specimens of mixed type were composites containing both CNFs and NiFe particles and divided into two categories. In the first case, NiFe particles were added to the pre-mixture containing resin and the fixed CNFs of 1 wt%. The weight percentages of added NiFe particles were 10 wt%, 30 wt% and 50 wt% (S07, S08 and S09), respectively. In the second case, CNFs were added to the pre-mixture containing resin and the fixed NiFe particles of 30 wt%. The weight percentages of added CNFs were 0.5 wt%, 1.0 wt% and 2.0 wt% (S10, S11 and S12), respectively. The first experimental tests were conducted to observe the variation of material properties caused by the added magnetic fillers with respect to fixed dielectric lossy material. The second experimental tests were performed to observe the variation of material properties caused by the added dielectric fillers with respect to fixed magnetic lossy material. The type, notation and added weight percentages of fabricated specimens are provided in Table 1.

Table 1. Specifications for 12 fabricated specimens.

Type	Notation	Fillers	Wt%
Dielectric	S01	CNFs	0.5
	S02		1.0
	S03		2.0
Magnetic	S04	NiFe	10.0
	S05		30.0
	S06		50.0
Mixed	S07	NiFe to pre-mixture containing fixed 1.0 wt% CNFs	10.0
	S08		30.0
	S09		50.0
	S10	CNFs to pre-mixture containing fixed 30.0 wt% NiFe	0.5
	S11		1.0
	S12		2.0

Figs. 4 show the SEM images for cross section of S09 specimen. In the left image with 1,000 times magnification, NiFe particles were mainly observed due to the low magnification. However, in the right image with 10,000 times magnification, CNF and NiFe particles were well observed as the dispersed states.



Figs. 4. SEM images for cross section of S09 specimen.

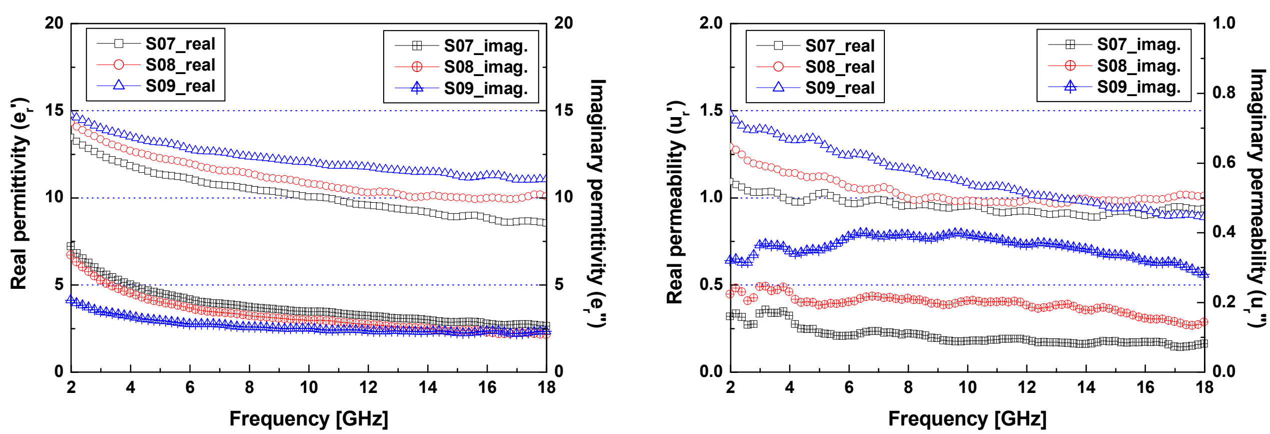
3.2 Measurement of electromagnetic characteristics

The transmission line technique was used to measure electromagnetic properties of composite specimens. Using a network analyzer (Agilent N5230A), S-parameters of fabricated specimens were measured in the frequency range from 2 to 18 GHz. The specimens for the measurement had the shape of cylindrical toroid as can be seen in Fig. 3. The complex permittivity and permeability of specimens were calculated using the magnitude and phase of measured S-parameters.

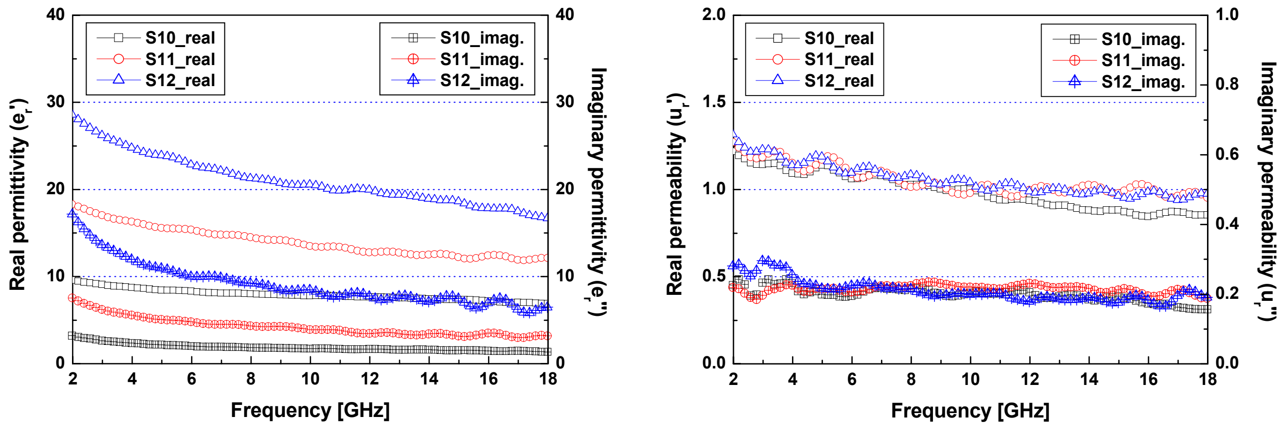
The complex permittivities and permeabilites of S01, S02 and S03 specimens were distributed in the range of ($10 < \varepsilon'_r < 17.5$, $1.4 < \varepsilon''_r < 11.5$) and ($\mu'_r \approx 1.0$, $\mu''_r \approx 0.0$), respectively. As the weight percentage of CNFs increased, both the real and imaginary parts of permittivity became higher. But the permeabilities were the nearly same regardless of added contents. These results show CNFs are the efficient dielectric lossy fillers with a little addition to the resin.

The complex permittivites and permeabilites of S04, S05 and S06 specimens were distributed in the range of ($2.9 < \varepsilon'_r < 6.1$, $0.1 < \varepsilon''_r < 0.4$) and ($\mu'_r < 1.5$, $\mu''_r < 0.4$), respectively. As the weight percentage of NiFe particles increased, both the permittivity and permeability became higher. The permittivities were relatively low compared with the results of CNFs. The real and imaginary parts of permeabilities in the GHz range were higher than those of air. These results show that NiFe particles were effective as the magnetic lossy fillers to improve the complex permeability.

Figs. 5 show the complex permittivities and permeabilites of S07, S08 and S09 specimens. As the weight percentage of NiFe particles increased, CNFs contents to total weight decreased comparatively so that the real parts of permittivities became higher and imaginary parts of permittivities became lower. These results mean that the content of CNFs is more dominant than that of NiFe in the case of the complex permittivity. The permeabilities had the similar tendencies as in the case of magnetic RAMs (S04, S05 and S06).



Figs. 5. Complex permittivities and permeabilites of S07, S08 and S09 specimens.



Figs. 6. Complex permittivities and permeabilities of S10, S11 and S12 specimens.

Figs. 6 show the complex permittivities and permeabilities of S10, S11 and S12 specimens. As the weight percentage of CNFs increased, the permittivities became higher and permeabilities had nearly constant values.

4. SIMULATION AND EXPERIMENTS FOR ELECTROMAGNETIC WAVE ABSORBERS

4.1 Principle and numerical formulas for electromagnetic wave absorber

Fig. 7 shows the single-layered electromagnetic wave absorber with thickness d. According to the transmission line theory, the input impedance at the surface of the absorber in Fig. 7 is

$$Z_{in} = Z_0 \frac{Z_d + Z_0 \tanh \gamma d}{Z_0 + Z_d \tanh \gamma d} \tag{1}$$

where the impedance of absorber material is $Z_0 = \sqrt{\frac{\mu}{\varepsilon}} = \sqrt{\frac{\mu_0 \mu_r}{\varepsilon_0 \varepsilon_r}}$ (ε_r is the complex relative permittivity) and the propagation constant γ is $j\omega\sqrt{\varepsilon\mu}$. Because the boundary back layer is the perfect electric conductor (PEC) in Fig. 7, the impedance at distance d, Z_d is zero. Thus the input impedance becomes $Z_{in} = Z_0 \tanh \gamma d$. The impedance of air is $Z_a = \sqrt{\frac{\mu_0}{\varepsilon_0}}$ and the impedance matching condition minimizing reflection of incident wave is $Z_{in} = Z_a$. The normalized wave impedance $\tilde{Z}$ for perpendicular incidence of plane wave is

$$\tilde{Z} = \frac{Z_{in}}{Z_a} = \sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh\left(j \frac{2\pi f d}{c} \sqrt{\mu_r \varepsilon_r}\right) \tag{2}$$

where ω is $2\pi f$ and the speed of light is $c = \frac{1}{\sqrt{\varepsilon_0 \mu_0}}$ [10, 11].

The condition minimizing reflection of incident plane wave is that $\tilde{Z}$ is equal to 1. This state is also called as the impedance matching condition or the quarter lamda (wavelength) matching condition.

A planar and inhomogeneous half-space with ε and μ varying as a function of z can be modeled by a multi-layered medium, where the electromagnetic property is piecewise constant in each region (Fig. 8). The generalized reflection coefficient $\tilde{R}_{i,i+1}$ at the interface between region i and i+1 considering whole structures can be written as

$$\widetilde{R}_{i,i+1} = R_{i,i+1} + \frac{T_{i,i+1}\widetilde{R}_{i+1,i+2}T_{i+1,i}e^{2ik_{i+1,z}(d_{i+1,}-d_i)}}{1 - R_{i+1,i}\widetilde{R}_{i+1,i+2}e^{2ik_{i+1,z}(d_{i+1,}-d_i)}} \quad (3)$$

where $R_{i,i+1}$ is the reflection coefficient between region i and region i+1 considering only reflection between two regions. $T_{i,i+1}$ is the transmission coefficient between region i and region i+1. $k_{i,z}$ is the complex propagation constant in region i. d_i is the distance to region i. The equation (3) can be simplified using $T_{ij} = 1 + R_{ij}$ and $R_{ij} = -R_{ji}$ as

$$\widetilde{R}_{i,i+1} = \frac{R_{i,i+1} + \widetilde{R}_{i+1,i+2}e^{2ik_{i+1,z}(d_{i+1,}-d_i)}}{1 + R_{i,i+1}\widetilde{R}_{i+1,i+2}e^{2ik_{i+1,z}(d_{i+1,}-d_i)}} \quad (4)$$

These equations are recursive relations that express $\widetilde{R}_{i,i+1}$ in terms of $\widetilde{R}_{i+1,i+2}$ [12]. The reflection loss [dB] in term of power or energy can be expressed as $RL \ [dB] = 20 \times \log_{10}(\widetilde{R}_{i,i+1})$.

In this study, the reflection loss characteristics for single-layered microwave absorbers were calculated using the above equations.

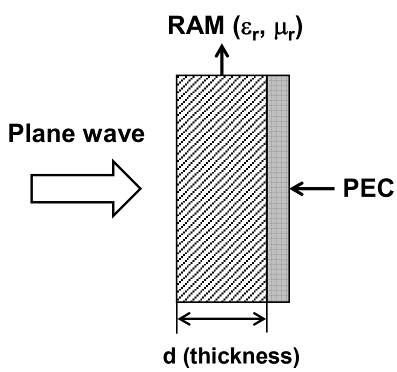


Fig. 7. Single-layered microwave absorber.

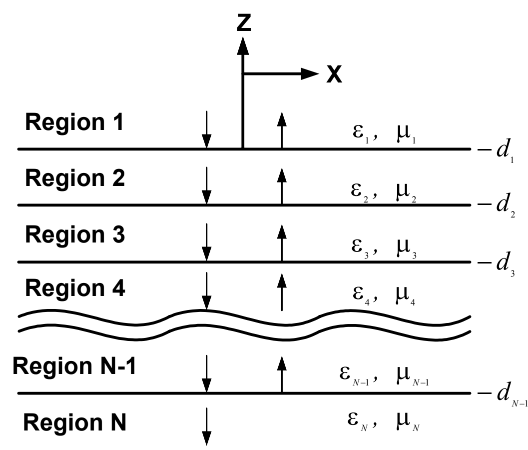


Fig. 8. Reflection and transmission in a multi-layered medium.

4.2 Simulation results for electromagnetic wave absorber

This section deals with the numerical simulation results of reflection loss characteristics for the single-layered microwave absorbers in the X-band (8.2~12.4 GHz) frequencies. First, the parametric studies to find the matching thickness and compare the absorbing capacity in the X-band frequencies were conducted using the obtained electromagnetic data and the above numerical formula. The reflection loss (RL) characteristics with the variation of thickness (0~20 mm) were calculated for each specimen using material data of the 10 GHz, the center of the X-band. And the reflection loss characteristics with the variation of frequency (2~18 GHz) were calculated for each specimen to evaluate the 10dB absorbing bandwidth. Note that the 10 dB absorbing bandwidth means the frequency bandwidth having reflection loss characteristics over 90 %. The matching thickness and reflection loss characteristics for each specimen in the X-band are provided in Table 2.

In the case of dielectric RAMs, S02 had the maximum reflection loss and 10dB absorbing bandwidth. The real and imaginary parts of complex permittivity for S01 ranged lower than those of S02 and S03, so that it had relatively had thick matching thickness and narrow absorbing capacity. S03 had the high real parts of complex permittivity enough to reduce the matching thickness. However, its material properties were not the optimal values among the dielectric RAMs. It had smaller reflection loss and 10 dB absorbing bandwidth than those of S02.

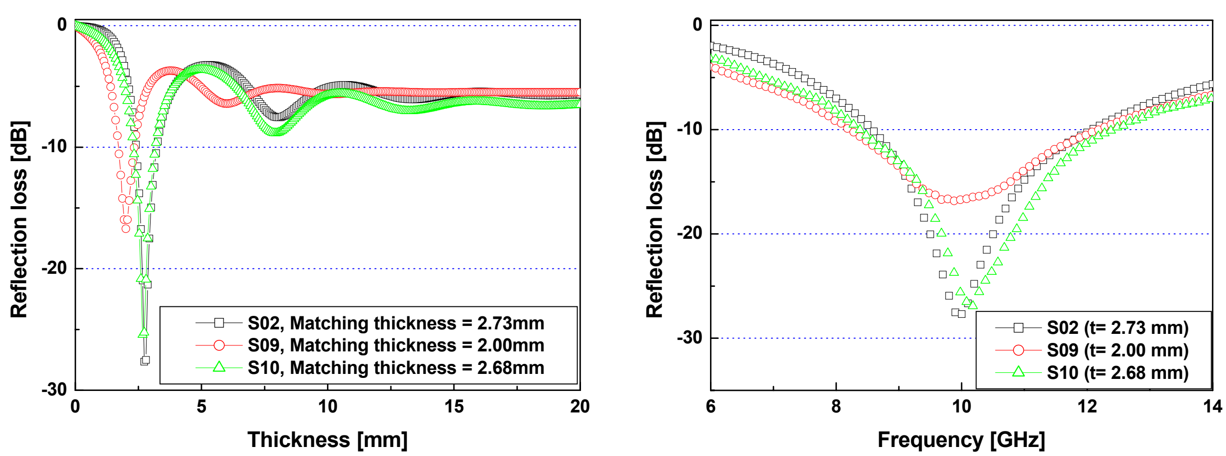
Table 2. Matching thickness and reflection loss characteristics for each specimen in the X-band.

Type	Specimen	Matching thickness [mm]	|Max. RL| [dB]	10 dB absorbing bandwidth [GHz]
Dielectric	S01	3.35	14.1	2.4 (9.0~11.4)
	S02	2.73	27.7	3.4 (8.6~12.0)
	S03	2.27	17.8	3.1 (8.5~11.6)
Magnetic	S04	13.9	7.6	0.0
	S05	11.2	32.0	1.1 (8.9~11.0)
	S06	2.80	14.3	3.3 (8.5~11.8)
Mixed	S07	2.47	24.6	3.6 (8.4~12.0)
	S08	2.27	17.1	3.5 (8.6~12.1)
	S09	2.00	16.7	4.0 (8.2~12.2)
	S10	2.68	25.3	4.0 (8.4~12.4)
	S11	2.07	15.0	3.2 (8.3~11.5)
	S12	1.60	9.4	0.0

In the case of magnetic RAMs, S04 and S05 had very thick matching thickness over 10 mm and narrow 10dB absorbing bandwidth due to their low complex permittivities (the real and imaginary parts had the values less than 4 and 0.3, respectively). S06 had relatively high complex permittivites and permeabilities (ε_r = 6.24 - j×0.36, μ_r = 1.10 - j×0.37 at the 10 GHz) and was the best having the 10dB absorbing bandwidth of 3.3 GHz with the thickness of 2.80 mm among magnetic RAMs. But, it had the low |Max RL| of 14.3 GHz to be applied to the microwave absorber.

In the case of mixed RAMs, the matching thickness and 10 dB absorbing bandwidth became thinner and wider. S07, S08, S09 and S10 had thin matching thickness less than 2.70 mm and broad 10 dB absorbing bandwidth over 3.5 GHz. S11 and S12 with very thin matching thicknesses had the decreasing tendencies in terms of the |Max RL| and 10 dB absorbing bandwidth.

Figs. 9 show the simulation results (reflection losses with thickness and frequency) in the X-band for three selected specimens (S02, S09 and S10) showing thin thickness and good absorbing capacity.

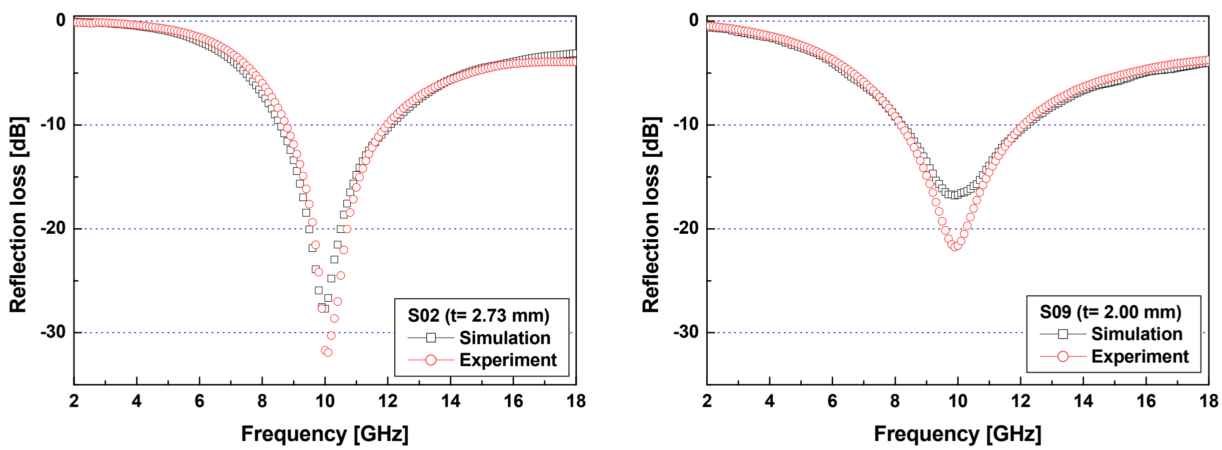


Figs. 9. Reflection loss characteristics with thickness and frequency for selected specimens in the X-band.

From above results, we could reach several concise conclusions to design the good single-layered microwave absorbers. First, the dielectric RAMs showed better absorbing properties when the real and imaginary parts of complex permittivity were ranged in 7.5~11.5 and 2.4~7.2, respectively. In addition, it was also found that CNFs were the very efficient fillers to enhance the complex permittivity with a small addition less than 2 wt%. Second, the magnetic RAMs relatively had low complex permittivities ($\varepsilon'_r < 6.5$, $\varepsilon''_r < 0.5$) so they had the thick matching thickness and narrow absorbing capacity. If the magnetic RAMs with higher complex permeability than those of S06 were to be applied to microwave absorbers, they probably could show the excellent absorbing properties. More specifically, a large addition over 50 wt% is necessary to improve the complex permeability for the magnetic fillers, NiFe particles. Finally, the mixed RAMs generally showed the enhanced absorbing characteristics with thinner matching thickness. In the case of specimens S07, S08, S09 and S10 with the proper combination for the complex permittivity and permeability, they are very effective mixed RAMs. On the other hand, S11 and S12 with relatively high complex permittivities proved to be disadvantageous to be applied to the microwave absorbers. This effect probably results from the additional reflective wave on the surface by the increase of dielectric loss tangent (conductive characteristics). Therefore, the control of the appropriate complex permittivity is essential to design and fabricate the improved mixed RAMs.

4.3 Experimental results for electromagnetic wave absorbers

The reflection loss characteristics for S02 and S09 in the X-band were measured using a network analyzer, Agilent N5230A. First, each specimen was fabricated according to the procedure provided in Fig. 4. Second, the fabricated specimens were cut as the designed thickness. Third, the back-surfaces of the specimens were coated with the silver paste. And the metal plates having the same form as specimens were stuck on the coated-surfaces of the specimens. Finally, the reflection loss (S11 [dB]) results of the specimens were measured. Figs. 10 show the simulation and experimental results for reflection loss of S02 and S09 specimens in the frequency range of 2~18 GHz. Experiment results were in very good agreements with simulation ones in terms of the overall reflection loss characteristics and 10 dB absorbing bandwidth. These good agreements means that the simulation techniques using the electromagnetic properties of materials and numerical formulas in a multi-layered medium are well set up.



Figs. 10. Simulation and experimental results for reflection loss of S02 and S09 specimens in the X-band.

5. CONCLUSIONS

This research has focused on the design, fabrication and performance evaluation of the single-layered microwave absorbers containing both dielectric and magnetic lossy materials. Carbon nanofibers (CNFs) were used as dielectric lossy materials and NiFe particles were used as magnetic lossy materials. Total twelve specimens with various weights of added fillers and compound ratios were fabricated. The regularly dispersed CNFs and NiFe particles were observed through the SEM images for the cross section of mixed type specimens. Their permittivity and permeability in the 2~18 GHz were measured using the transmission line technique. The parametric studies for reflection loss characteristics of each specimen to design the single-layered RAMs were performed using the theory of transmission and reflection in a multi-layered medium. The mixed RAMs generally had the improved absorbing characteristics with thinner matching thickness. In the X-band, one of the mixed RAMs, S09 with the matching thickness of 2.00 mm showed the 10 dB absorbing bandwidth of about 4.0 GHz (8.2~12.2). Another mixed RAM, S10 with the matching thickness of 2.68 mm had the 10 dB absorbing bandwidth of about 4.0 GHz (8.4~12.4). On the other hand, one of the dielectric RAMs, S02 with the maximum absorbing capacity showed the 10 dB absorbing bandwidth of about 3.4 GHz (8.6~12.0) with the matching thickness of 2.73 mm. Then, the experimental studies to evaluate the reflection loss characteristics of specimens S02 and S09 were performed in the range of 2~18 GHz using the transmission line technique. The experimental results were in very good agreements with simulation ones in terms of the overall reflection loss characteristics and 10 dB absorbing bandwidth. To design more improved RAMs with the broadband absorbing capacity, the further studies such as the multi-layered absorbers and optimal design are demanded.

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