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Microwave-Absorbing Performance and Mechanical Properties of Poly(vinyl chloride)/Acrylonitrile–Butadiene Rubber Thermoplastic Elastomers Filled with Multiwalled Carbon Nanotubes and Silicon Carbide

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ABSTRACT: Poly(vinyl chloride) (PVC)/acrylonitrile–butadiene rubber (NBR) were mixed with multiwalled carbon nanotubes (MWCNTs) and silicon carbide (SiC) to prepare microwave-absorbing composites. The complex permittivity, direct-current (dc) conductivity, microwave-absorbing performance, morphology, and mechanical properties of the composites were studied. The real and imaginary parts of the permittivity of the composites increased with increasing MWCNT content. The premixing of the MWCNTs with PVC was more beneficial to the dispersion of MWCNTs; this led to a higher dc conductivity and permittivity and better microwave-absorbing performance than the premixing of MWCNTs with NBR for the PVC/NBR/MWCNT composites. The PVC/NBR/MWCNT composites had a minimum reflection loss (RL_{min}) of −49.5 dB at the optimum thickness of 1.96 mm. The efficient microwave absorption of the PVC/NBR/MWCNT composites was due to a high dielectric loss and moderate permittivity. The incorporation of SiC into the PVC/NBR/MWCNT composites increased the real and imaginary parts of permittivity of the composites. When the SiC content was 70 phr, RL_{min} decreased to −34.9 dB at a thickness of 3 mm. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 130: 345–351, 2013

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INTRODUCTION

Microwave-absorbing materials (MAMs) have drawn great attention in recent years for their applications in protecting human beings from microwave radiation and electromagnetic interference in wireless communication. MAMs should have light weights, wide absorbing frequency ranges, and good mechanical properties. Polymers filled with carbon nanotubes (CNTs) show excellent microwave-absorbing performance and can be used for electromagnetic interference shielding.[1–9] The microwave-absorbing performance of epoxy/multiwalled carbon nanotubes (MWCNTs) was strongly affected by the diameter of the MWCNTs at 9.968 and 8.43 GHz.[1,2] The microwave-absorbing performance of the MWCNT composites can be enhanced by MWCNT modification.[3–7] $La(NO_3)_3$-doped MWCNT/poly(vinyl chloride) (PVC) composites exhibited better microwave-absorbing performance than the MWCNT/PVC composites, and their minimum reflection loss (RL_{min}) reached −25 dB around 13.5 GHz.[7]

Among ceramic-absorbing materials, silicon carbide (SiC) has a high thermal stability and a wide absorbing frequency range but a low absorbing efficiency. Because the absorbance ability of SiC is not satisfactory as a single absorbent, it is generally used as an auxiliary absorbent or after surface treatments to enhance the absorption capacity.[8] The microwave-absorbing performance of the MWCNT/SiC composites were closely related to the SiC spheres, which were composed of nanocrystals; the interfaces between the MWCNTs and SiC spheres; and the grain boundaries between the SiC nanocrystals.[9,10] The MWCNT/SiC composites reached a RL_{min} values of −38.7 dB around 12.9 GHz.[10] The addition of a certain mass of SiC reduced the percolation threshold and improved the microwave absorption of the carbon black/epoxy resin composites.[8]

The microwave-absorbing performance of polymer composites also depends on the dispersion of fillers in the matrix.[11] However, a few studies have been concerned with the effect of the dispersion of fillers into a two-phase thermoplastic elastomer (TPE) on the microwave-absorbing performance. PVC/acrylonitrile–butadiene rubber (NBR) TPE prepared by dynamic vulcanization has good chemical resistance, weather resistance, processability, elasticity, and oil resistance. The addition of certain fillers into PVC/NBR TPE can give it thermal and electrical conductivity and high strength.[12–14] Therefore, a PVC/NBR TPE filled with MWCNT and SiC should be promising for microwave absorbance with a great potential for practical application.

In this study, a PVC/NBR TPE was filled with MWCNTs and ultrafine SiC. The dependence of the microwave-absorbing performance on the dispersion of the MWCNTs in the PVC/NBR TPE was explored, and the direct-current (dc) conductivity, microwave characteristics, and mechanical properties of the TPE were studied.

EXPERIMENTAL

Materials

NBR (N241), with a 29% acrylonitrile content, was made by Japan Synthetic Rubber Co., Ltd. (Japan) and had a Mooney viscosity ($ML_{1+4}^{100^\circ C}$) of 56. The PVC compound was prepared by the mixture of PVC (DP 1300), diisodecyl phthalate (DIDP), and an organic tin stabilizer (TM-181) in our laboratory. MWCNT (NC7000) was produced by Nanocyl Co., Ltd. (Belgium) and had an average diameter of about 9.5 nm, a length of more than 1.5 μm, and a purity of higher than 90%. SiC (particle size = 0.5–0.7 μm) and dioctyl sebacate (DOS) were supplied by Aladdin Chemistry Co., Ltd. (China). Sulfur, zinc oxide, and stearic acid were purchased from Sinopharm Chemical Reagent Co., Ltd. (China). 2,2′-Dibenzothiazoledisulfide was industrial grade.

Composite Preparation

Two different processing methods were used to study the filler effects on the microwave-absorbing performance and mechanical properties of the PVC/NBR/MWCNT composites.

The formulation of the composites included the PVC compound (55.5 phr), an NBR master batch (44.5 phr), a variable amount of MWCNTs (0–8 phr), and a variable amount of SiC (0–70 phr). The PVC compound consisted of 35.9 phr PVC, 17.9 phr diisodecyl phthalate, and 1.7 phr stabilizer. The NBR master batch consisted of 35.9 phr NBR, 5.4 phr DOS, 0.5 phr sulfur, 0.5 phr 2,2′-dibenzothiazoledisulfide, 1.8 phr zinc oxide, and 0.4 phr stearic acid.

Method 1: Premixing of the MWCNTs with PVC.. The NBR compound was prepared with NBR, DOS, and other vulcanizers on a two-roll mill at room temperature. The PVC compound was premixed with different contents of MWCNTs in a Haake internal mixer at 150°C for 8 min at a rotor speed of 50 rpm. The PVC and NBR compounds were dynamically vulcanized on the two-roll mill at 150°C for 10 min. For the PVC/NBR/MWCNT/SiC composites, SiC was added to the composites together with MWCNTs.

Method 2: Premixing of the MWCNTs with NBR.. The NBR compounds were prepared with NBR, DOS, other vulcanizers, and different contents of MWCNTs on the two-roll mill at room temperature. Then, PVC and the NBR compounds were dynamically vulcanized on the two-roll mill at 150°C for 10 min.

Characterization

The dispersion of the MWCNTs was studied with transmission electron microscopy (TEM; JEM-2100 JEOL, Ltd., Japan). Composite samples were ultracryomicrotomed on an Ultracut UC6 (Leica, Germany). For microwave measurement, a cylindrical toroidal specimen with an outer diameter of 7.0 mm, an inner diameter of 3.0 mm, and a thickness of 4.0 mm was set in a coaxial line. The microwave scattering parameters (Ss; S_{ij}, i, j = 1, 2) were measured with a vector network analyzer (Agilent Technologies, E5071C) in the frequency range 2–18 GHz. Then, the complex permittivity (ϵ_r) was calculated from S according to the Nicolson-Ross-Weir (NRW) method.[15,16] Because the calculation result showed that the composites were not magnetic [complex permeability (μ_r) = $\mu' - i\mu'' \approx 1$], only ϵ_r ($\epsilon_r = \epsilon' - i\epsilon''$) was plotted and discussed. The volume conductivity of 1 mm thick specimens of the PVC/NBR composites were measured with an EST121 high-resistance meter according to ASTM D 257. Tensile tests were performed on a universal testing machine (Shenzhen SANS Material Test Instrument Co., Ltd.) at 23°C and a crosshead speed of 500 mm/min. The dumbbell-shaped samples were 75 mm in length, 2 mm in thickness, and 4 mm in width.

RESULTS AND DISCUSSION

Morphology

TEM observation was carried out to investigate whether the dispersion of the MWCNTs due to filler dispersion could significantly affect the electromagnetic characteristics and microwave-absorbing performance. For the 35.9/35.9/6 PVC/NBR/MWCNT composite with premixing with PVC, as shown in Figure 1(a), the MWCNTs were uniformly dispersed, even though a small amount of MWCNT aggregates (dark dots) still existed. Figure 1(b) shows the TEM image of the 35.9/35.9/6 PVC/NBR/MWCNT composite with premixing with NBR. The white regions suggest the absence of MWCNTs. A better dispersion of the MWCNTs was observed in Figure 1(a) compared to Figure 1(b). This analysis showed that the premixing of the MWCNTs with PVC was more beneficial to the dispersion of the MWCNTs than the premixing of the MWCNTs with NBR for the PVC/NBR/MWCNT composites.

dc Conductivity

In Figure 2, dc conductivity of the PVC/NBR/MWCNT composites with premixing with PVC increased from 1.64×10^{-11} to 7.61×10^{-6} S/cm as the MWCNT content was increased from 0 to 8 phr. The dc conductivity of the PVC/NBR/MWCNT composites with premixing with NBR was lower than that of the PVC/NBR/MWCNT composites with premixing with PVC by several orders of magnitude at MWCNT contents from 4 to 6 phr; this was in agreement with the previous morphological analysis. The dc conductivity of the PVC/NBR/MWCNT composites should have been related to the dispersion of MWCNTs in the composite. The good dispersion of CNTs favored the formation of a conductive pathway with a small concentration and a reduction of the percolation threshold.[17,18] The dispersion of MWCNTs in the composites with premixing with PVC was better than that of MWCNTs in the composites with premixing with NBR; this resulted in the composites with premixing with PVC having a lower percolation threshold. The percolation

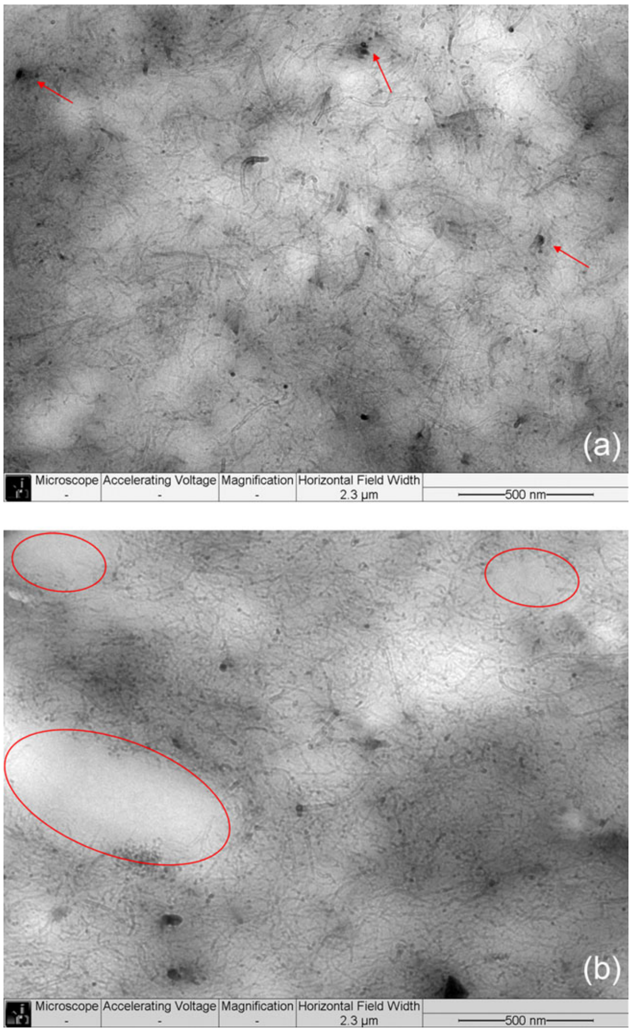

Figure 1. TEM images of the 35.9/35.9/6 PVC/NBR/MWCNT composite premixed with (a) PVC and (b) NBR. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

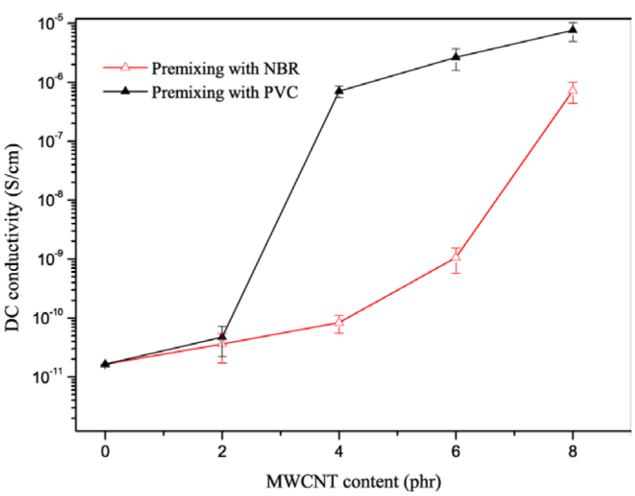

Figure 2. dc conductivity of the PVC/NBR/MWCNT composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

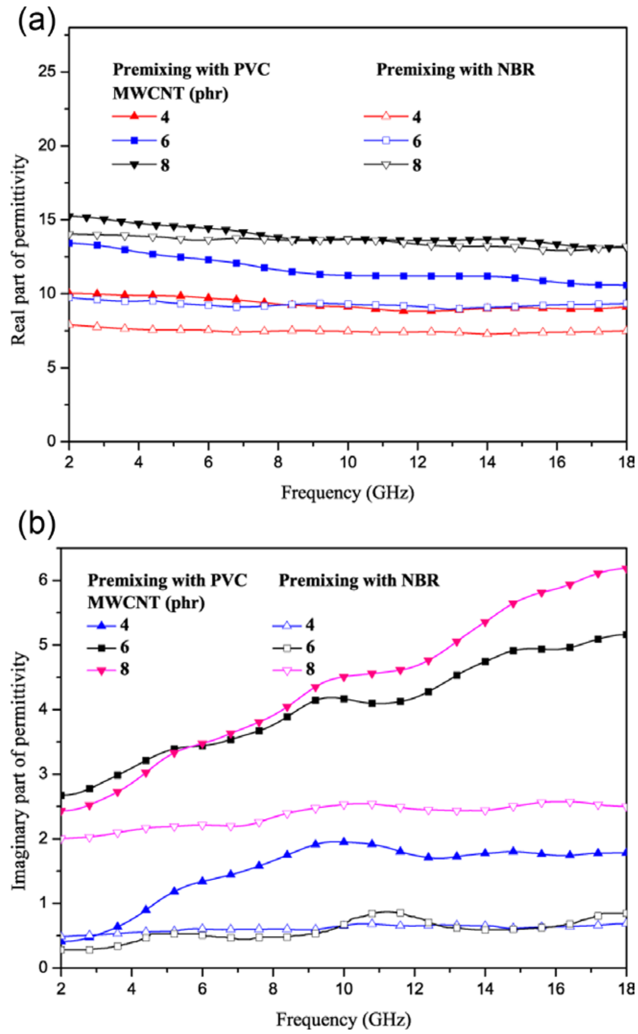

Figure 3. (a) Real and (b) imaginary parts of ϵ_r of the PVC/NBR/MWCNT composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

thresholds of the composites with premixing with PVC and the composites with premixing with NBR were 2–4 and 6–8 phr, respectively.

ϵ_r

As reported in the literature,[19] the real part of ϵ_r describes the dielectric relaxation and space charge polarization induced by the interaction of electromagnetic waves with boundary charges, and the imaginary part of ϵ_r describes the movement of free electrons. The real and imaginary parts of ϵ_r of the PVC/NBR/MWCNT composites increased with increasing MWCNT content in the frequency range 2–18 GHz (Figure 3). This was due to the conductive behavior of the MWCNTs in the PVC/NBR compounds. For the PVC/NBR/MWCNT composites with premixing with PVC, the real part of the permittivity increased from 9.3 to 13.8, and the imaginary part increased from 2.3 to 3.9 at 8 GHz with the MWCNT content increasing from 4 to 8 phr. Compared with the PVC/NBR/MWCNT composites with premixing with PVC, the PVC/NBR/MWCNT composites with premixing with NBR had lower real and imaginary parts of the

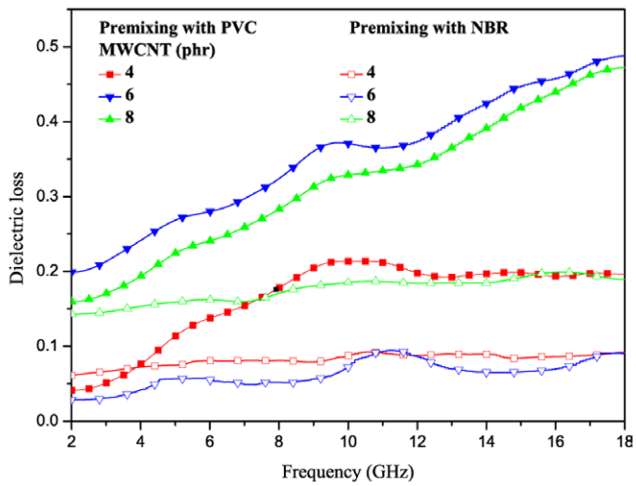

Figure 4. Dielectric loss of the PVC/NBR/MWCNT composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

permittivity at the same MWCNT content. This was probably because the dispersion of the MWCNTs in the PVC/NBR/MWCNT composites with premixing with PVC was better than that in the PVC/NBR/MWCNT composites with premixing with NBR; this led to a higher electrical conduction and interfacial polarization.

Figure 4 shows the dielectric loss of the PVC/NBR/MWCNT composites in the frequency range of 2–18 GHz. For a conductive filler/polymer composite, its dielectric loss mainly consists of the loss of electrical conduction and the loss of interfacial polarization.[20] The PVC/NBR/MWCNT composites with premixing with PVC had a higher dielectric loss than the PVC/NBR/MWCNT composites with premixing with NBR at the same MWCNT content. This may have been due to the higher electric conduction of the PVC/NBR/MWCNT composites with premixing with PVC.

Microwave-Absorbing Performance

The reflection loss (RL) of a metal-backed single absorbing layer was calculated according to transmission line theory:

$$\mathrm{RL(dB)} = 20\lg\left|\frac{z_{\mathrm{in}}-1}{z_{\mathrm{in}}+1}\right| \quad (1)$$

where Z_{in} is the normalized input impedance of the absorbing layer and is related to the characteristics of the material by the following equation:

$$Z_{\mathrm{in}} = \sqrt{\frac{\mu_r}{\varepsilon_r}}\tanh\left(j\frac{2\pi f d}{c}\sqrt{\mu_r\varepsilon_r}\right) \quad (2)$$

where d is the thickness of the absorbing layer, f is the frequency of the electromagnetic waves, and c is the velocity of the electromagnetic waves *in vacuo*.

The intrinsic properties of a MAM (ϵ_r and μ_r) and the extrinsic parameters (thickness of MAM and working frequency) could affect its microwave-absorbing performance. Therefore, we calculated RL of the absorbing layer at different thicknesses from 0 to 4 mm to determine the optimum thickness of the absorbing layer. The $\mathrm{RL_{min}}$ and the absorbing bandwidth are important parameters in evaluating the absorbing performance. Table I and Figure 5 show the microwave-absorbing performance and RL curves, respectively, of the PVC/NBR/MWCNT composites.

The PVC/NBR/MWCNT composites with premixing with PVC had a wider absorbing bandwidth and lower $\mathrm{RL_{min}}$ than the PVC/NBR/MWCNT composites with premixing with NBR at the same MWCNT content. For example, at the optimum thickness, $\mathrm{RL_{min}}$ of the 35.9/35.9/6 PVC/NBR/MWCNT composite with premixing with PVC was −49.5 dB, and the −10 dB absorbing bandwidth ($\mathrm{BW_{10}}$) was 3.0 GHz, but $\mathrm{RL_{min}}$ of the 35.9/35.9/6 PVC/NBR/MWCNT composite with MWCNT premixing with NBR was only −4.2 dB. In our study, there may have been two factors that mainly affected the microwave-absorbing performance, that is, the dielectric loss and the impedance matching condition. The dielectric loss is mainly a function of the imaginary part of permittivity, whereas the matching condition is to be studied with the microwave wavelength taken into account.[21] When the absorbing layer thickness satisfies eq. (3), the incident and reflected waves from the absorbing layer surface are out of phase, and this leads to little reflection at the air–absorber interface:

$$d = \frac{n}{4}\lambda_m = \frac{n}{4}\frac{\lambda_0}{\sqrt{|\mu_r||\varepsilon_r|}}\,(n = 1,3,5\ldots) \quad (3)$$

Table I. Microwave-Absorbing Performance of the PVC/NBR/MWCNT Composites at Different Thicknesses

		Optimum thickness			2 mm			3 mm		
	MWCNT (phr)	$\mathrm{RL_{min}}$ (dB)	f_m (GHz)	$\mathrm{BW_{10}}$ (GHz)	$\mathrm{RL_{min}}$ (dB)	f_m (GHz)	$\mathrm{BW_{10}}$ (GHz)	$\mathrm{RL_{min}}$ (dB)	f_m (GHz)	$\mathrm{BW_{10}}$ (GHz)
Premixing with PVC	4	−10.2	9.8	0.5	−8.9	13.1	0	−9.3	8.8	0
	6	−49.5	11.6	3.0	−44.8	11.4	3.0	−22.1	7.5	1.9
	8	−47.8	11.1	2.6	−40.4	10.3	2.4	−17.8	6.8	1.4
Premixing with NBR	4	−3.6	12.5	0	−3.5	14.2	0	−3.4	3.2	0
	6	−4.2	11.2	0	−3.1	12.7	0	−2.6	8.5	0
	8	−13.0	15.6	1.4	−10.9	10.4	0.8	−9.0	6.9	0

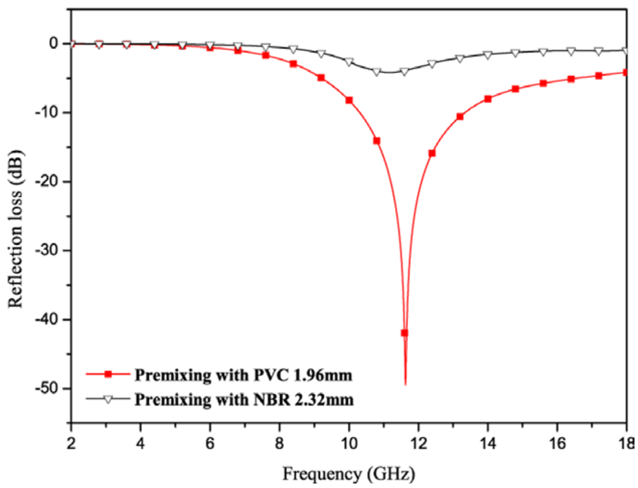

Figure 5. RL curves of the 35.9/35.9/6 PVC/NBR/MWCNT composites at optimal thickness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

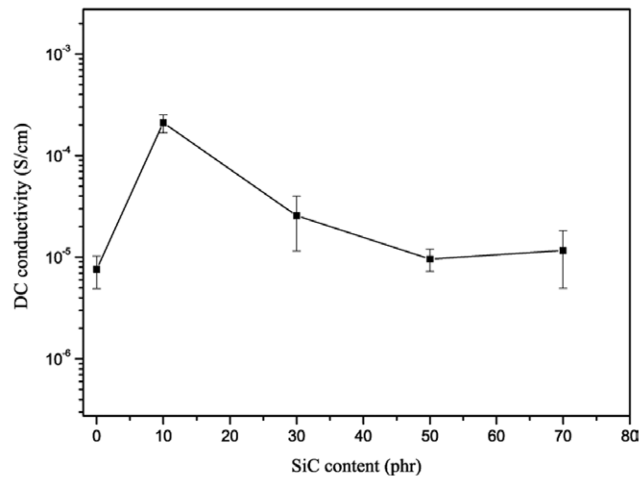

Figure 6. dc conductivity of the PVC/NBR/MWCNT/SiC composites.

where λ_m is the propagating wavelength in a material and λ_0 is the free-space wavelength. With increasing thickness of the absorbing layer, the matching wavelength of the incident wave would increase, which means that the matching frequency moves to low frequencies.[19]

As discussed previously, the PVC/NBR/MWCNT composites with premixing with PVC had a higher dielectric loss than the PVC/NBR/MWCNT composites with premixing with NBR at the same MWCNT content, and this led to their better microwave-absorbing performance.

Mechanical Properties

As shown in Table II, the tensile strength of the PVC/NBR/MWCNT composites with premixing with PVC increased from 6.5 to 10.6 MPa, and the stress at 100% extension consistently increased from 2.3 to 9.0 MPa when the MWCNT content increased from 0 to 8 phr. Compared with those of the PVC/NBR/MWCNT composites with premixing with PVC, the tensile strength and elongation at break of the PVC/NBR/MWCNT composites with premixing with NBR were higher, but the stress at 100% extension was lower, and this showed that premixing with NBR was more beneficial to the reinforcement of PVC/NBR composites than premixing with PVC. This was attributed to the deformation behavior of TPE.[22] The effect of the premixing filler with rubber was smaller than that of the premixing filler with plastic on the yield of plastic.

Effect of SiC on the Microwave-Absorbing Performance and Mechanical Properties

As a semiconductive material, SiC can reduce the percolation threshold of filler/polymer composites.[8] As shown in Figure 6, the conductivity of the PVC/NBR/MWCNT/SiC composite increased from 7.61 × 10^{-6} to 2.10 × 10^{-4} S/cm with SiC contents increasing from 0 to 10 phr. The conductivity of the PVC/NBR/MWCNT/SiC composites remained almost unchanged, whereas the content of SiC ranged from 30 to 70 phr compared with that of the PVC/NBR/MWCNT composite. This was probably because a mass of SiC would have diluted the concentration of MWCNT.

As shown in Figure 7, the incorporation of SiC into the PVC/NBR/MWCNT composites led to significant increases in both the real and imaginary parts of the permittivity. The real and imaginary parts of the permittivity of the 35.9/35.9/8/50 PVC/NBR/MWCNT/SiC composite increased to 20.6 and 7.0, respectively, at 8 GHz. The addition of SiC into the PVC/NBR/MWCNT composite also increased its dielectric loss (Figure 8). The dielectric loss of the 35.9/35.9/8/30 PVC/NBR/MWCNT/SiC composites was the highest and ranged from 0.49 to 0.64. This was because new interfaces introduced by the addition of SiC could lead to strong interfacial polarization.

Table II. Mechanical Properties of the PVC/NBR/MWCNT Composites

	MWCNT (phr)	Tensile strength (MPa)	Elongation at break (%)	Stress at 100% extension (MPa)
Premixing with PVC	0	6.5 ± 0.4	387 ± 26	2.3 ± 0.0
	4	8.5 ± 0.2	343 ± 13	5.9 ± 0.2
	6	9.2 ± 0.2	261 ± 20	7.1 ± 0.2
	8	10.6 ± 0.2	218 ± 8	9.0 ± 0.2
Premixing with NBR	4	10.5 ± 0.2	401 ± 16	5.7 ± 0.3
	6	10.6 ± 0.1	303 ± 26	6.9 ± 0.2
	8	11.3 ± 0.2	311 ± 11	7.2 ± 0.2

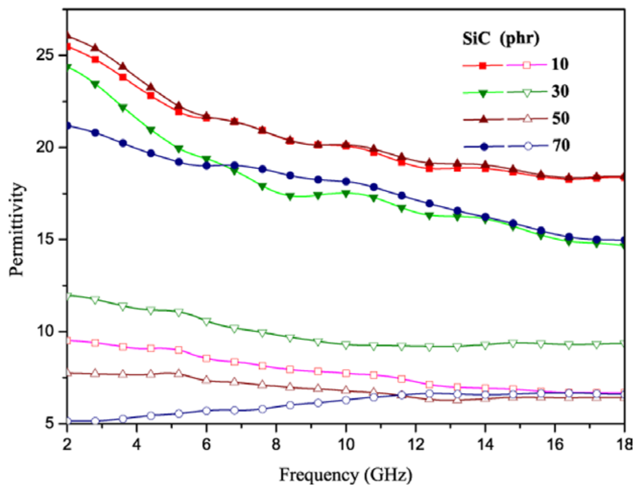

Figure 7. ϵ_r values of the PVC/NBR/MWCNT/SiC composites. The solid symbols indicate the real permittivity, and the hollow symbols indicate the imaginary permittivity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

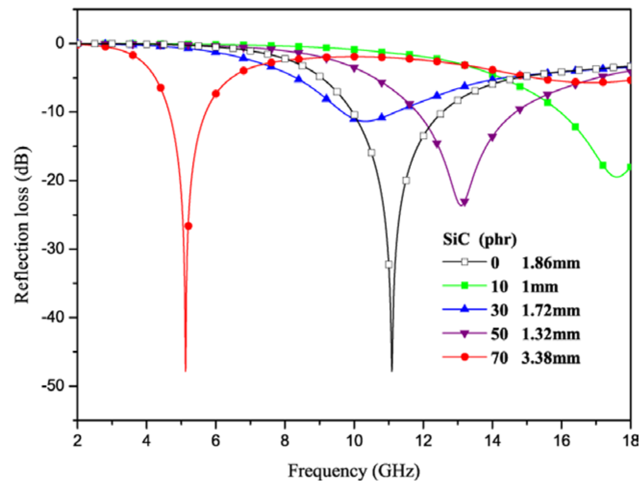

Figure 9. RL curves of the PVC/NBR/MWCNT/SiC composites at optimum thickness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

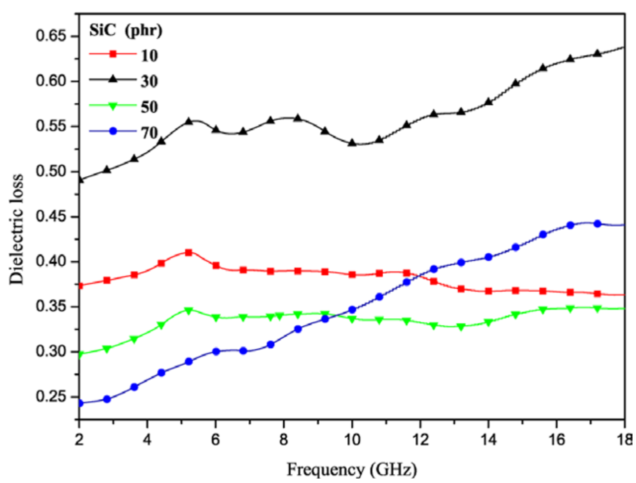

Figure 8. Dielectric loss values of the PVC/NBR/MWCNT/SiC composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table III and Figure 9 show the microwave-absorbing performance and RL curves of the PVC/NBR/MWCNT/SiC composites, respectively. Compared with the 35.9/35.9/8 PVC/NBR/MWCNT composite, the absorbing peaks of the 35.9/35.9/8/10 and 35.9/35.9/8/50 PVC/NBR/MWCNT/SiC composites moved to high frequency; this indicated better absorbing performance of the composites at high frequency. The absorbing peaks of the 35.9/35.9/8/30 and 35.9/35.9/8/70 PVC/NBR/MWCNT/SiC composites moved to low frequency. The 35.9/35.9/8/70 PVC/NBR/MWCNT/SiC composite had a lower RL_{min} (−39.4 dB) than the PVC/NBR/MWCNT composites (−17.8 dB) at a thickness of 3 mm; this indicated a better microwave-absorbing performance.

Although the dielectric losses of the PVC/NBR/MWCNT/SiC composites with SiC contents ranging from 10 to 50 phr were higher than that of the PVC/NBR/MWCNT composites, the RL_{min} values of the PVC/NBR/MWCNT/SiC composites were higher and the BW_{10} values were narrower. This unexpected phenomenon may have been due to the high permittivity of the PVC/NBR/MWCNT/SiC composites. The higher the permittivity was, the lower the wave impedance was, and this led to an impedance mismatch at the air–material interface; thus, few electromagnetic waves could penetrate into the absorbing layer and be dissipated. In other words, a reasonable permittivity of the PVC/NBR composites favored microwave absorption.

Table III. Microwave-Absorbing Performance of the PVC/NBR/MWCNT/SiC Composites at Different Thicknesses

	Optimum thickness			2 mm			3 mm		
SiC content (phr)	RL_{min} (dB)	f_m (GHz)	BW_{10} (GHz)	RL_{min} (dB)	f_m (GHz)	BW_{10} (GHz)	RL_{min} (dB)	f_m (GHz)	BW_{10} (GHz)
0	−47.8	11.1	2.6	−40.4	10.3	2.4	−17.8	6.8	1.4
10	−19.5	17.6	2.0	−15.7	8.3	1.8	−13.8	5.3	1.0
30	−11.3	10.3	1.5	−10.9	9.0	1.0	−10.0	5.6	0
50	−23.6	13.1	2.9	−19.9	8.3	1.8	−18.0	5.3	1.1
70	−47.8	5.1	1.1	−24.3	8.8	1.9	−34.9	5.8	1.2

Table IV. Mechanical Properties of the PVC/NBR/MWCNT/SiC Composites

SiC content (phr)	Tensile strength (MPa)	Elongation at break (%)	Stress at 100% extension (MPa)
0	10.6 ± 0.2	218 ± 8	9.0 ± 0.2
10	10.6 ± 0.2	271 ± 10	7.9 ± 0.3
30	11.2 ± 0.1	262 ± 14	8.9 ± 0.3
50	9.4 ± 0.4	205 ± 8	8.1 ± 0.5
70	10.6 ± 0.2	166 ± 11	9.9 ± 0.3

As shown in Table IV, the tensile strength of the PVC/NBR/MWCNT/SiC composites almost remained unchanged, whereas the elongation at break decreased from 218 to 166% with SiC contents increasing from 0 to 70 phr.

CONCLUSIONS

The microwave-absorbing performance of the PVC/NBR/MWCNT composites with premixing with PVC was better than that of the PVC/NBR/MWCNT composites with premixing with NBR at the same MWCNT content; this was attributed to the higher conductivity of the PVC/NBR/MWCNT composites with premixing PVC. The 35.9/35.9/6 PVC/NBR/MWCNT composite had the widest BW_{10} of 3.0 GHz and the lowest RL_{min} of −49.5 dB at the optimum thickness of 1.96 mm. The addition of SiC changed the position of the absorbing peak. The 35.9/35.9/8/70 PVC/NBR/MWCNT/SiC composite had a lower RL_{min} than the 35.9/35.9/8 PVC/NBR/MWCNT composite at a thickness of 3 mm, and the former had an absorbing peak at a lower frequency. The addition of SiC increased the real and imaginary parts of the permittivity of PVC/NBR/MWCNT, and this may have led to an impedance mismatch at the air–material interface and not favored the microwave-absorbing performance. The tensile strength and stress at 100% extension of the PVC/NBR composites increased with the addition of MWCNT and SiC, whereas the elongation at break decreased. The prepared composites have potential for microwave-absorption applications.

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