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Stacked polypyrrole-coated non-woven fabric sheets for absorbing electromagnetic waves with extremely high frequencies

Yoshihiro Egami · Takashi Yamamoto · Kunio Suzuki · Tadashi Yasuhara · Eiji Higuchi · Hiroshi Inoue

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Abstract Conductive non-woven fabric sheets coated with polypyrrole nanoparticles and having resistivity from 1.5×10^5 to 3.0 Ω cm were prepared by immersing non-woven fabric sheets in solutions containing various concentrations of oxidizing agent and dopant and then exposing the sheets to pyrrole vapor. A stack of ten treated sheets could absorb more than 95% of electromagnetic waves in the frequency range of 75–110 GHz and more than 99% in the range of 85–105 GHz, irrespective of the resistivity of the sheet. This absorption was achieved even though the thickness of the stack was less than 10 mm. Furthermore, a conductive non-woven fabric sheet with resistivity of 7.5 Ω cm and thickness of only 0.5 mm could absorb about 90% of electromagnetic waves in the range of 75–110 GHz. These results clearly demonstrate that the stacked conductive non-woven fabric sheets prepared in this study are a new material that effectively absorbs electromagnetic waves with extremely high frequencies in the millimeter band.

Y. Egami · K. Suzuki · T. Yasuhara
Osaka Research Laboratory, Tayca Corporation, 3-47, 1-Chome, Funamachi, Taisho-ku 551-0022, Osaka, Japan

Y. Egami (✉) · E. Higuchi · H. Inoue
Department of Applied Chemistry, Graduate School of Engineering, Osaka Prefecture University, 1-1 Gakuen-cho, Sakai 599-8531, Osaka, Japan
e-mail: egami@tayca.co.jp

T. Yamamoto
Department of Communications Engineering, National Defense Academy, 10-20, 1-Chome, Hashirimizu, Yokosuka, Kanagawa 239-8686, Japan

Introduction

Information and communications technologies are rapidly developing, and electromagnetic waves with ultra high frequencies (0.3–3 GHz) and super high frequencies (3–30 GHz) are now used to transfer large amounts of information at high speed. Such electromagnetic waves have already been applied to systems such as cordless telephones (0.8–2 GHz), wireless local area networks (2.45–5.2 GHz), electronic toll collection (5.8 GHz), and wireless communication and information systems (60 GHz). In the near future, electromagnetic waves with extremely high frequencies over 60 GHz will be required for the spread of intelligent transportation systems (vehicle-mounted millimeter-wave radar: 76 GHz, etc.), security systems (millimeter-wave passive and active sensors for airport screening: 94 GHz), and ultra high speed communication systems (120 GHz, etc.) [1, 2]. However, to our knowledge, no electromagnetic absorbers have been developed that are effective in the extremely high frequency band. Moreover, the use of electromagnetic waves with such high frequencies may give rise to serious problems such as the malfunction of electronic equipment and information leakage. A solution to these potential problems is to develop new radar-absorbent materials (RAMs) that can effectively absorb broadband electromagnetic waves coming from various directions. Also, RAMs for use in vehicles and small spaces should be thin, lightweight, and flexible.

In general, RAMs are classified into three categories according to their mechanism of electromagnetic wave absorption: dielectric loss [3–9], magnetic loss [10, 11], and conductive loss [12, 13]. The radar absorption of RAMs depends on their composition, loading amount, and thickness. For instance, pyramidal urethane foam containing carbon powder and a rubber sheet containing carbonyl iron or ferrite are common commercial RAMs. The former is a typical dielectric loss material, while the latter is a typical magnetic loss material. But since the magnetic loss of carbonyl iron or ferrite can disappear at several GHz, the latter is a dielectric loss material based on the rubber sheet at the higher frequencies. The former can effectively absorb broadband electromagnetic waves with frequencies over 0.3 GHz, but it is thick (>100 mm). Meanwhile, the latter is relatively thin (<10 mm), but it is heavy and absorb electromagnetic waves in only a narrow frequency range. For example, the dielectric loss RAM used in electronic toll collection has an absorption maximum at 5.8 GHz [2].

Conductive polymers have been developed as alternatives to metals and carbon materials because they have not only high electrical conductivity but also advantageous mechanical properties including flexibility, toughness, malleability, and elasticity. Polypyrrole (PPY), a widely used conductive polymer, is prepared by various chemical and electrochemical methods. In practical applications, PPY is used in actuators [14], sensors [15], capacitors [16], and anti-corrosion materials [17, 18]. Moreover, since PPY is also a dielectric loss material [19, 20], it is used in heat generators [21], RAMs [8, 20, 22–24], and electromagnetic shields [8, 25]. However, to our knowledge, there are few reports on the application of PPY to RAMs that can absorb electromagnetic waves with extremely high frequencies over 75 GHz.

Recently, we successfully prepared polypyrrole nanoparticle-coated non-woven fabric sheets with various conductivity levels by exposing the non-woven fabric sheets, which had been coated with ammonium persulfate (APS) as an oxidizing agent and 1-butylammonium-2-naphthalenesulfonate (BANS) or 1-butylammonium-*p*-toluenesulfonate (BATS) as a dopant, to pyrrole vapor [26]. The conductivity of the conductive non-woven fabric sheets can be tuned by adjusting the amount of deposited PPY nanoparticles on the non-woven fabrics. In this study, we prepared stacks of various numbers of conductive non-woven fabric sheets with different resistivities, and evaluated their radar absorption in the extremely high frequency band over 75 GHz.

Experimental

Preparation of dopant solutions

1-Butylammonium-2-naphthalenesulfonate and BATS were used as dopants of the polypyrrole prepared in the present study because we have found experimentally that they are well-suited to forming uniform PPY nanoparticles with high conductivity. BANS and BATS were prepared as follows [26]. In brief, 1-butylamine (143 mL) was slowly added to a 1.57 M (=mol L^{-1}) aqueous solution of 2-naphthalenesulfonic acid while keeping the temperature below 20 °C to produce a 1.36 M BANS aqueous solution. A 1.36 M BATS aqueous solution was also prepared from 1-butylamine and *p*-toluenesulfonic acid by the same procedure.

Preparation of conductive non-woven fabric sheets

The oxidizing agent APS was dissolved in distilled water to produce APS solutions with various concentrations. 1.36 M BANS aq and 1.36 M BATS aq, as well as ethanol for improving the wettability of the fabrics, were added to the APS solution; the mixture was then stirred. The resultant solution is called Solution A. The concentration of ethanol was fixed at 5 M. The molar ratio of BANS to BATS, [BANS]/[BATS], and the molar ratio of both dopants to APS, ([BANS] + [BATS])/[APS]), were fixed to 1.

After immersing a non-woven fabric sheet (WO-ME150, Mitsubishi Paper Mills Limited, 50 × 50 cm^2) in Solution A for 10 min, the fabric sheet was pressed at 0.3 MPa with a nip roller (ϕ12 cm) and then dried for 10 min at 60 °C. The resultant fabric sheet is referred to as a pretreated fabric sheet. A laboratory dish of ϕ10 cm containing 20 mL of pyrrole was placed at the bottom of a closed acrylic box (100 × 100 × 100 cm^3). The pretreated fabric sheet was laid in the box filled with pyrrole vapor. After 10 min, the fabric sheet was removed from the box and dried for 10 min at 100 °C. The fabric sheet was washed thoroughly with distilled water and then dried for 10 min at 100 °C.

To measure radar absorption, the conductive non-woven fabric sheets were stacked on a 0.05-mm-thick Al foil, through which no electromagnetic waves could be transmitted.

Characterization of conductive non-woven fabric sheets

The morphology of the conductive non-woven fabric sheets was observed by scanning electron microscopy (Model S-900, Hitachi Co.) after being thinly coated with platinum using an ion sputtering instrument for 3 min to form an electrical contact. The sulfur content in the sheets was evaluated by X-ray fluorescence (XRF) (Model Axios, PANalytical Spectris Co., 40 kV, 60 mA). The resistivity of the sheets was measured with a resistivity meter (Hiresta UP Model MCP-HT450 and Loresta GP Model MCP-T610, Mitsubishi Chemical Analytech Co.).

The radar absorption was evaluated from the reflection loss, which was measured by the free space method using

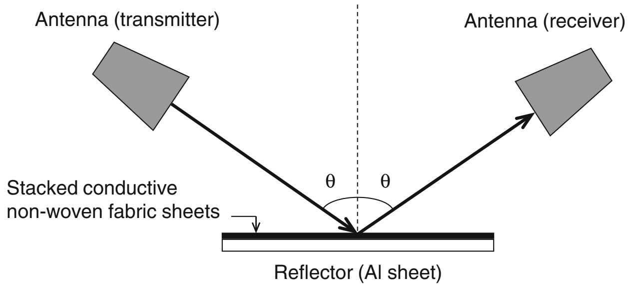

Fig. 1 Schematic illustration of setup for evaluating reflection loss by the free space method

two horn antennas [2, 27]. As shown in Fig. 1, electromagnetic waves with frequencies of 1–18 GHz or 75–110 GHz were emitted at an incident angle θ from a horn antenna (transmitter) and detected by another antenna (receiver) after being reflected at the same angle for various stack sizes of conductive non-woven fabric sheets on the Al sheet. The reflection loss (RL) is given in percent and decibels by the following equations.

$$\mathrm{RL}\ [\%] = 100\,(1 - S_r/S_s) \quad (1)$$

$$\mathrm{RL}\ [\mathrm{dB}] = 10\ \log\ (S_r/S_s) \quad (2)$$

Here, S_s and S_r represent the intensity of the electromagnetic waves emitted from the transmitter and detected by the receiver, respectively. Since S_r is less than or equal to S_s, the reflection loss in decibels is zero or negative. A smaller value of S_r indicates smaller reflection loss, which signifies higher radar absorption.

The exact radar absorbing behavior of electromagnetic waves by RAMs depends on their dielectric and magnetic properties, which are represented by complex permittivity ($\varepsilon_r = \varepsilon' - j\varepsilon''$) and complex permeability ($\mu_r = \mu' - j\mu''$), respectively [2]. In general, the ε_r and μ_r are calculated from the following equations;

$$\varepsilon_r = \varepsilon' - j\varepsilon'' = -j\lambda_0\gamma_r/(2\pi Z_c) \quad (3)$$

$$\mu_r = \mu' - j\mu'' = -j\lambda_0 Z_c\gamma_r/(2\pi) \quad (4)$$

Here, Z_c is the characteristic impedance, γ_r is the propagation constant of an RAM [2, 28]. The Z_c and γ_r are calculated from the following equations;

$$Z_c = \pm[(1-S_{11}^2 + S_{21}^2)^2 - 4S_{21}^2]^{1/2}/(1-S_{11})^2 - S_{21}^2,\quad \mathrm{Re}Z_c \geq 0 \quad (5)$$

$$\gamma_r = -(1/d)[\log\,[1-S_{11}^2 + S_{21}^2 - \{(1-S_{11})^2 - S_{21}^2\}Z_c]/2S_{21} + 2n\pi] \quad (6)$$

Here, n is the integer, S_{11} and S_{21} are reflection coefficient and transmission coefficient of the RAM, respectively. The S_{11} and S_{21} are measured by the coaxial waveguide method [2, 28] in the frequency range of 1–18 GHz with a Willtron Co. 37269A vector network analyzer. In this study, stacked conductive non-woven fabric sheets and a rubber sheet (thickness: 2.4 or 0.7 mm) containing carbonyl iron (30 wt%) and titanium slug (20 wt%) were used as a specimen.

The input impedance at the air interface (Z_{input}) is defined by the following equations [2].

$$Z_{\mathrm{input}} = Z_0(\mu_r/\varepsilon_r - \sin^2\theta)^{1/2}\tanh(\gamma_r t) \quad (7)$$

$$\gamma_r = [j\omega(\mu_r\varepsilon_r - \sin^2\theta)^{1/2}]/c \quad (8)$$

Here, θ is the incident angle in Fig. 1, Z_0 is $(\mu_0/\varepsilon_0)^{1/2} = 377\ \Omega$, t is the thickness of the material, ω is the angular frequency, c is the light speed. The permittivity of free space (vacuum or air) is $\varepsilon_0 = 8.854 \times 10^{-12}\ \mathrm{F\ m^{-1}}$, and the permeability of free space (vacuum or air) is $\mu_0 = 4\pi \times 10^{-7}\ \mathrm{V\ s\ (A\ m)^{-1}}$. In the impedance matching condition ($Z_{\mathrm{input}} = Z_0$), transmitted electromagnetic waves are completely absorbed by the RAM. The impedance matching condition is determined by the combination of six parameters ε', ε'', μ', μ'', t, and ω [27].

Results and discussion

Figure 2 shows SEM images of a conductive non-woven fabric sheet with resistivity of 70 Ω cm. In the higher magnification images (Fig. 2c, d), polypyrrole nanoparticles of about 10 nm in size are spread uniformly on the fiber with high surface coverage.

Figure 3 shows the logarithm of resistivity (R) of a conductive non-woven fabric sheet as a function of APS concentration. When the APS concentration was varied from 0.015 to 0.66 M, the R value decreased monotonically from 1.5×10^5 to 3.0 Ω cm. This clearly indicates that the R value can be tuned by adjusting the APS concentration. Such a decrease in R can be ascribed to the amount of deposited PPY nanoparticles being increased by the acceleration of the oxidative polymerization of pyrrole.

For conductive non-woven fabric sheets, the sulfur content as a function of the APS concentration was evaluated by XRF (Fig. 4). Since both BANS and BATS contain sulfur, the sulfur content should increase with the amount of deposited PPY nanoparticles or with the amount of dopant incorporated into the deposited PPY nanoparticles. The sulfur content increased with the APS concentration and as the R value decreased (Figs. 3, 4), clearly indicating that the decrease in the R value is attributable to increased deposition of PPY nanoparticles.

Figure 5 shows the real part (ε') and the imaginary part (ε'') of complex permittivity and the real part (μ') and the imaginary part (μ'') of complex permeability for free space for two RAMs in the frequency range of 1–18 GHz. The thickness of these two RAMs was 2.4 mm for a rubber

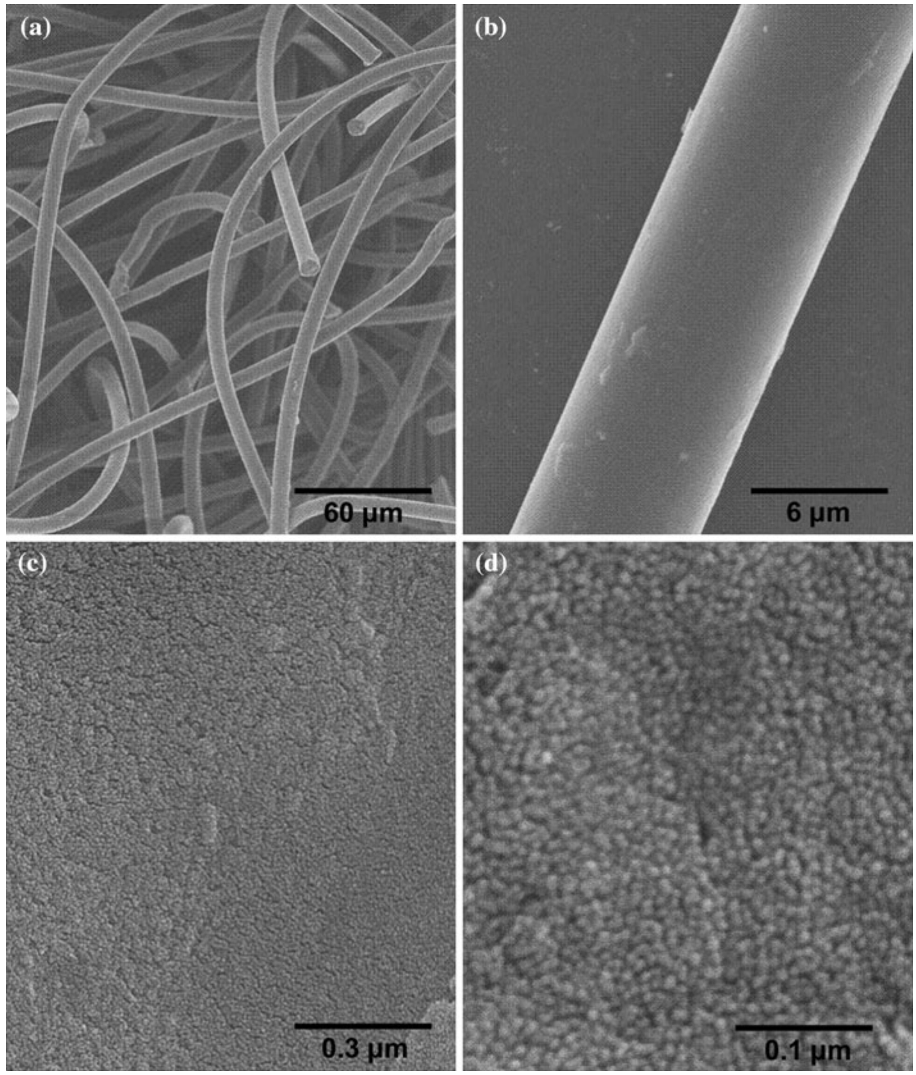

Fig. 2 SEM images of a conductive non-woven fabric sheet with resistivity of 70 Ω cm. Magnification: **a** ×500, **b** ×2,000, **c** ×100,000, and **d** ×300,000

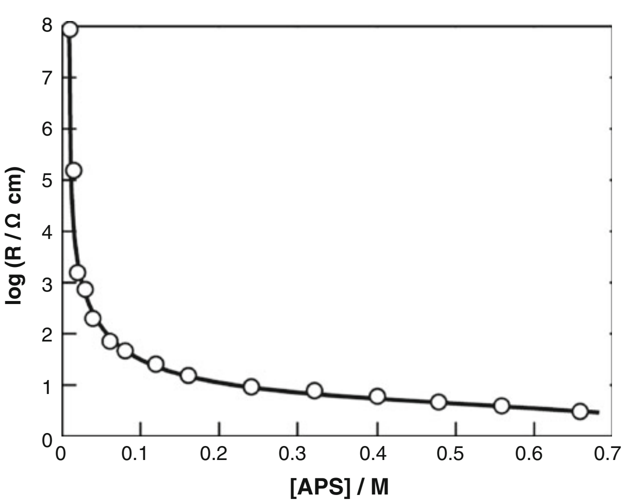

Fig. 3 Logarithm of resistivity of a conductive non-woven fabric sheet as a function of APS concentration. [BANS]/[BATS] = 1. ([BANS] + [BATS])/[APS] = 1. [Ethanol] = 5 M

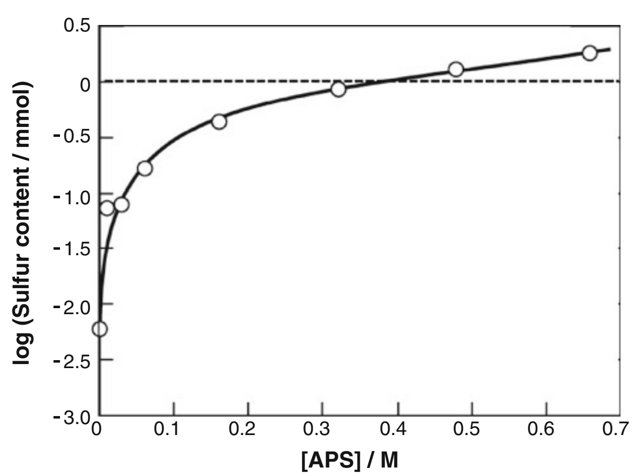

Fig. 4 Logarithm of the sulfur content in a conductive non-woven fabric sheet as a function of APS concentration. [BANS]/[BATS] = 1. ([BANS] + [BATS])/[APS] = 1. [Ethanol] = 5 M

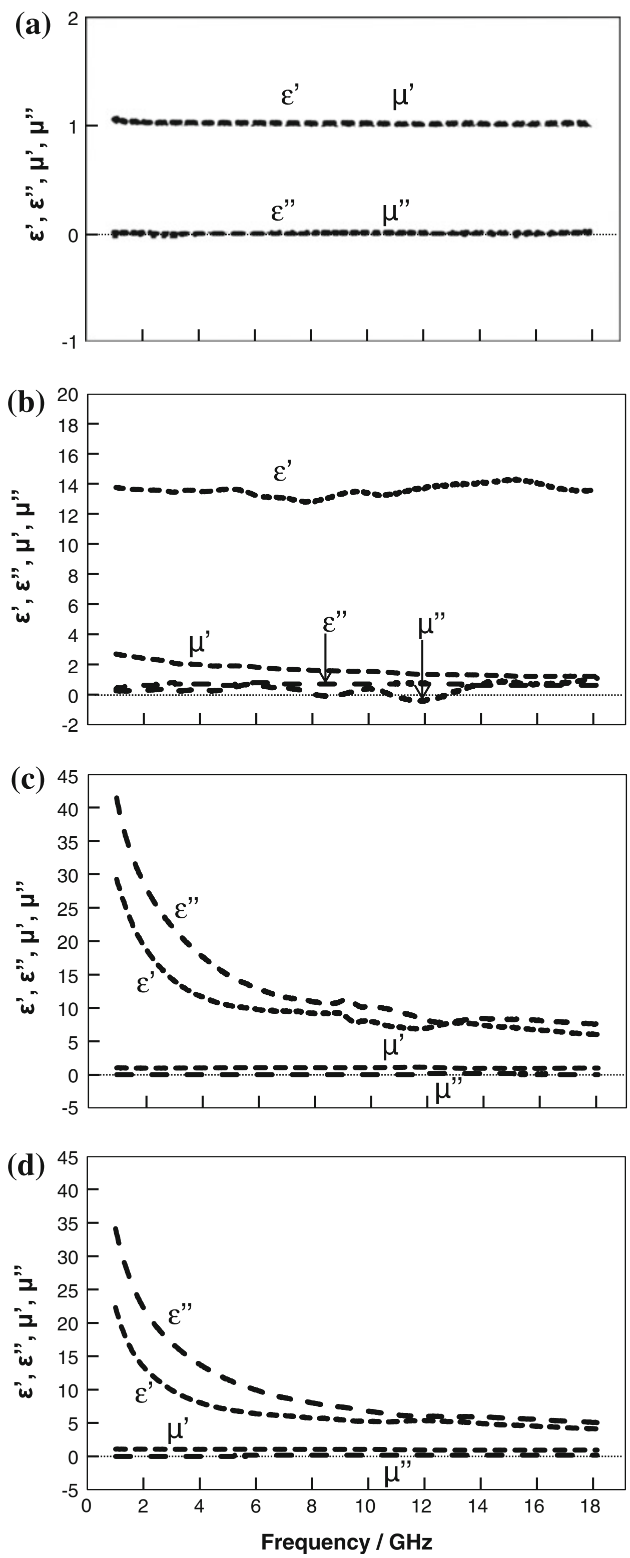

Fig. 5 Complex permittivity and complex permeability in the frequency range of 1–18 GHz for **a** free space, **b** a rubber sheet (thickness: 2.4 mm), **c** a stack of ten conductive non-woven fabric sheets (7.5 Ω cm/sheet), and **d** a stack of ten conductive non-woven fabric sheets (70 Ω cm/sheet)

sheet and 7.0 mm for a stack of ten conductive non-woven fabric sheets. For the rubber sheet, ε', ε'', μ', and μ'' were almost constant over the entire frequency range (Fig. 5b) similar to those in free space, suggesting that ε_r and μ_r were also constant irrespective of frequency. According to Eqs. 7 and 8, Z_{input} depends on ω because t, μ_r, and ε_r of the rubber sheet are all constant. In this case, there is a specific frequency at which Z_{input} is closest to Z_0. At this specific frequency, the rubber sheet exhibits its maximum radar absorption (i.e., the most negative reflection loss in decibels). Figure 6a shows the reflection loss, in percent and in decibels, as a function of frequency for the rubber sheet. The rubber sheet clearly has a specific frequency where a maximum is found, suggesting that the rubber sheet is not suitable to be a broadband RAM because it cannot effectively cover a wide frequency range.

For the stack of ten conductive non-woven fabric sheets, μ' and μ''were constant over the entire frequency range, whereas ε' and ε''monotonously decreased with increasing frequency (Fig. 5c, d), indicating that the RAM had dielectric dispersion. Such dielectric dispersion behavior has also been reported for RAMs such as graphite and other conductive polymers [6, 29, 30]. Compared with the existing RAMs, the stack of ten conductive non-woven fabric sheets exhibited high ε''. This may be ascribed to the high frequency dielectric loss of PPY nanoparticles deposited on the fabric. A higher ε'' corresponds to higher dielectric loss and greater conversion of electromagnetic energy to heat energy [30]. Therefore, the stack of ten conductive non-woven fabric sheets can provide improved radar absorption.

Figure 6b shows that stack of ten conductive non-woven fabric sheets does not have a minimum reflection loss in decibels or a maximum reflection loss in percent, thus signifying that the conductive non-woven fabric sheet can absorb electromagnetic waves over a wide frequency range. Similar behavior has also been observed for other RAMs composed of conductive polymer-coated fabrics [12, 22, 23], although the frequency range was lower in these previous cases.

Figure 7 shows reflection loss in the frequency range of 75–110 GHz for a stack of ten pristine non-woven fabric sheets and various stack sizes of the conductive non-woven fabric sheets (70 Ω cm/sheet). As can be seen from Eq. 2, when 90% (resp. 99%) of the electromagnetic waves are absorbed by a RAM, the reflection loss is −10 dB (resp. −20 dB). RAMs for practical use should have reflection loss better than −10 dB (i.e., >90% absorption) in order to prevent interference from broadband electromagnetic interference. The stack of ten pristine non-woven fabric sheets and stacks of one or two conductive non-woven fabric sheets have reflection losses worse than −7 dB in

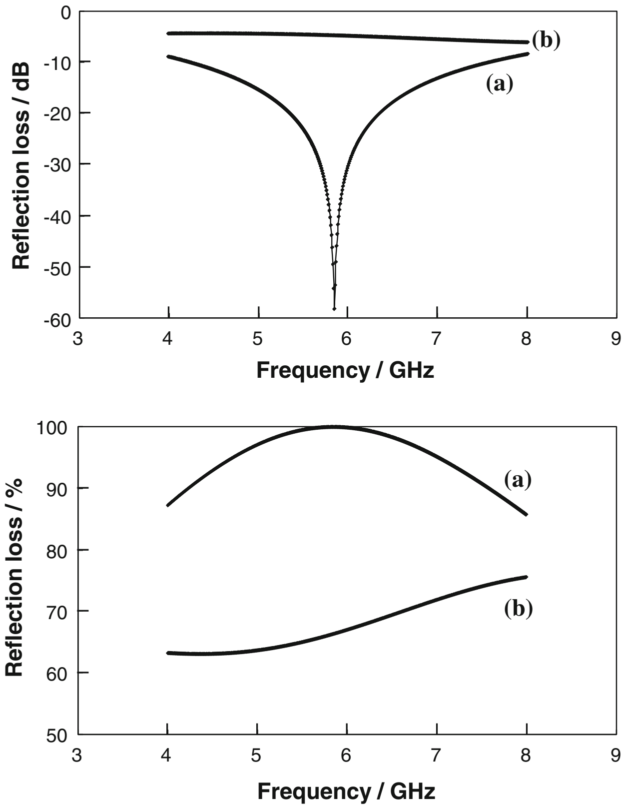

Fig. 6 Reflection loss in percent and in decibels in the frequency range of 4–8 GHz for (*a*) a rubber sheet (thickness: 2.4 mm) and (*b*) a stack of ten conductive non-woven fabric sheets (70 Ω cm/sheet). The incident angle of electromagnetic waves was 30°

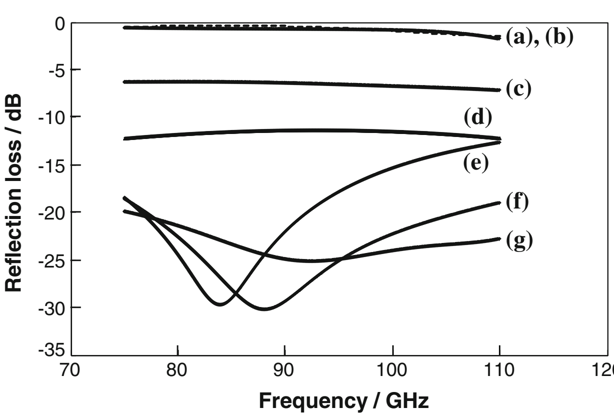

Fig. 7 Reflection losses in the frequency range of 75–110 GHz for (*a*) stack of ten pristine non-woven fabric sheets and stacks of (*b*) 1, (*c*) 2, (*d*) 3, (*e*) 5, (*f*) 7, and (*g*) 10 conductive non-woven fabric sheets (70 Ω cm/sheet). The incident angle of electromagnetic waves was 30°

the frequency range of 75–110 GHz (Fig. 7), indicating that they are not effective as RAMs. For a stack of three or more conductive non-woven fabric sheets, reflection loss was better than −10 dB, indicating that the stacks of conductive non-woven fabric sheets could serve as effective broadband RAMs. In particular, the stack of ten conductive non-woven fabric sheets had reflection loss better than -20 dB, absorbing more than 99% of the electromagnetic waves. To our knowledge, this is the first demonstration of a RAM that could effectively absorb electromagnetic waves in such the high frequency range.

Figure 8 shows reflection losses in the frequency range of 75–110 GHz for a stack of 10 pristine non-woven fabric sheets and for various stack sizes of the conductive non-woven fabric sheets (7.5 Ω cm/sheet). In this case, even the single conductive non-woven fabric sheet had reflection losses of −10 dB or better over almost the entire frequency range, indicating that it could serve as an effective RAM.

For the conductive non-woven fabric sheets with different resistivities, in Fig. 9, the average reflection loss calculated from the data in Figs. 7 and 8 is plotted against

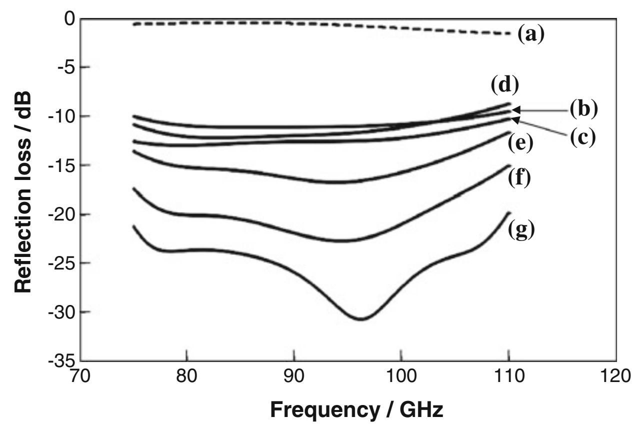

Fig. 8 Reflection loss in the frequency range of 75–110 GHz for stacks of (*a*) ten pristine non-woven fabric sheets and (*b*) 1, (*c*) 2, (*d*) 3, (*e*) 5, (*f*) 7, and (*g*) 10 conductive non-woven fabric sheets (7.5 Ω cm/sheet). The incident angle of the electromagnetic waves was 30°

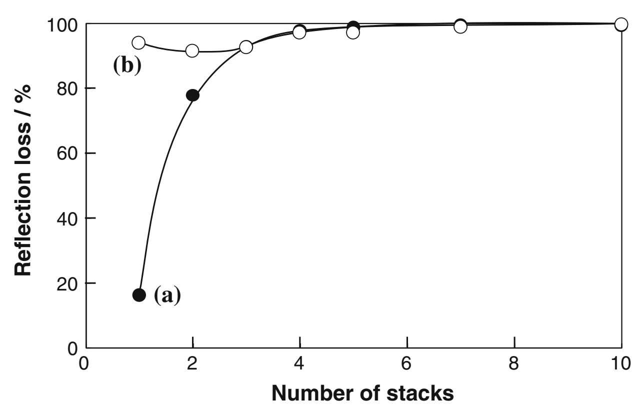

Fig. 9 Average reflection loss as a function of the stack size for conductive non-woven fabric sheets with resistivity of (*a*) 70 Ω cm/sheet or (*b*) 7.5 Ω cm/sheet

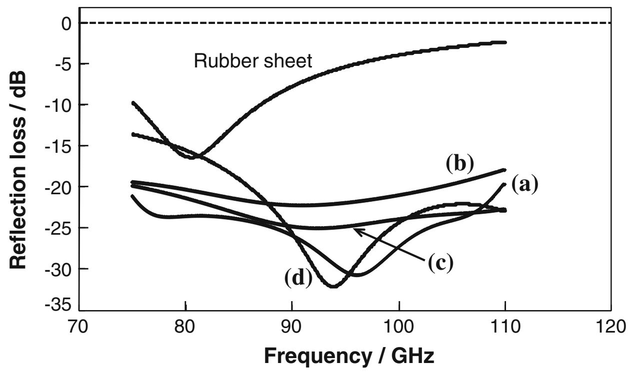

Fig. 10 Reflection losses in the frequency range of 75–110 GHz for a stack of ten conductive non-woven fabric sheets (*a*–*d*) and a rubber sheet (thickness: 0.7 mm). The resistivity of a conductive non-woven fabric sheet was (*a*) 7.5 Ω cm, (*b*) 15 Ω cm, (*c*) 70 Ω cm, and (*d*) 700 Ω cm. The incident angle was 30°

the stack size. The conductive non-woven fabric sheet with the lower resistivity had an average reflection loss greater than 90% even for a single layer. For sheets with higher resistivity, at least three sheets were required to surpass 90% absorption. Thus, in terms of radar absorption, a single sheet with low resistivity is comparable to three sheets with high resistivity. For the same level of performance, the lower resistivity sheet can be used to make a thinner, lighter RAM. These results strongly suggest that not only dielectric loss but also conductive loss contributes substantially to the radar absorption in the frequency range of 75–110 GHz.

Figure 10 shows the reflection loss in the frequency range of 75–110 GHz for a stack of ten conductive non-woven fabric sheets with various resistivities and for the rubber sheet. In the case of the rubber sheet, ε', ε'', μ', and μ'' are constant (Fig. 5b). In addition, since $t = 0.7$ mm for the rubber sheet, ω of the impedance matching condition can be determined, suggesting that the rubber sheet has a minimum reflection loss in decibels. In fact, Fig. 10 shows that the rubber sheet has its minimum reflection loss in decibels (i.e., maximum radar absorption) at about 80 GHz. All stacks of ten conductive non-woven fabric sheets have reflection losses better than −13 dB (>95% absorption) in the wide frequency range of 75–110 GHz, irrespective of resistivity. In particular, in the frequency range of 90–100 GHz, all stacks have reflection losses better than −20 dB (more than 99% absorption). Therefore, a stack of ten conductive non-woven fabric sheets can serve as an effective RAM for electromagnetic waves with extremely high frequency.

Figures 11 and 12 show reflection loss in the frequency range of 75–110 GHz for stacks of 10 conductive non-woven fabric sheets (7.5 or 70 Ω cm/sheet), and for a rubber sheet (thickness: 0.7 mm) when the incident angle of electromagnetic waves was 15°, 30°, and 45°. The best

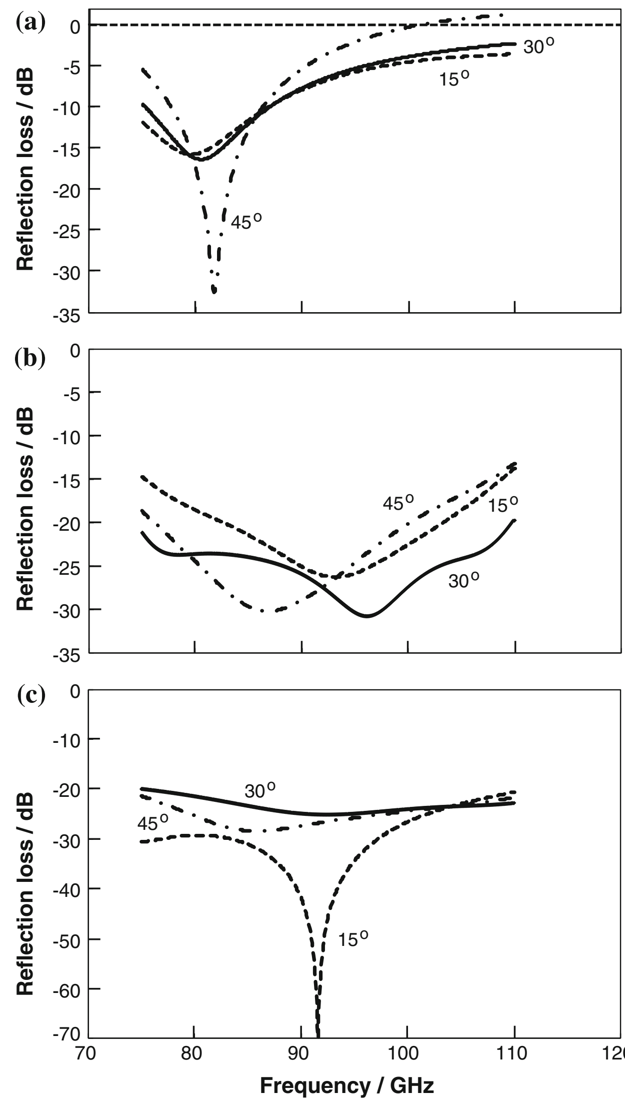

Fig. 11 Reflection losses in the frequency range of 75–110 GHz for **a** a rubber sheet (thickness: 0.7 mm), and stacks of conductive non-woven fabric sheet with resistivity of **b** 7.5 Ω cm/sheet and **c** 70 Ω cm/sheet. Each stack contained ten sheets. The incident angles were 15°, 30°, and 45°

reflection loss for the rubber sheet was −30 dB (maximum absorption: 99.9%) at 82 GHz at an incident angle of 45°, but reflection loss markedly worsened on both sides of 82 GHz (Figs. 11a, 12a), indicating that radar absorption had weakened. Moreover, the rubber sheet also exhibited its best reflection loss (maximum absorption) at a specific frequency for the incident angles of 15° and 30°, which indicates that the rubber sheet is a narrowband RAM. On the other hand, the stacked conductive non-woven fabric sheets (7.5 Ω cm/sheet) had reflection losses better than −13 dB (>95% absorption) over the entire frequency range, irrespective of incident angle (Figs. 11b, 12b). Furthermore, the stack of conductive non-woven fabric sheets (70 Ω cm/sheet) had reflection loss better than

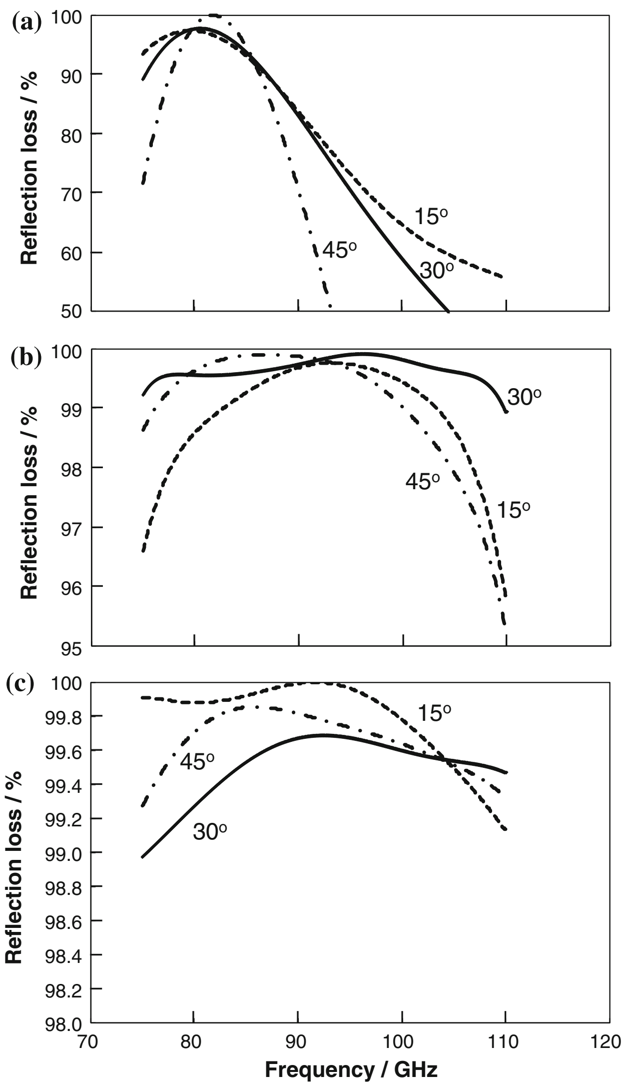

Fig. 12 Reflection losses in the frequency range of 75–110 GHz for **a** a rubber sheet (thickness: 0.7 mm), and stacks of conductive non-woven fabric sheet with resistivity of **b** 7.5 Ω cm/sheet and **c** 70 Ω cm/sheet. Each stack contained ten sheets. The incident angles were 15°, 30°, and 45°

−20 dB (>99% absorption) over the entire frequency range, regardless of incident angle (Figs. 11c, 12c). At an incident angle of 15° in particular, the stack exhibited a reflection loss of −70 dB at 92 GHz. These results clearly indicate that stacked conductive non-woven fabric sheets, with both high and low resistivities, are excellent broadband RAMs that can effectively absorb electromagnetic waves irrespective of incident angle. For commercial millimeter-wave absorbers, there is a trade-off relationship between the thickness of the absorbers and reflection loss. For instance, absorbers with reflection losses better than −10 dB (>90% absorption) are thicker than 5 mm. On the other hand, in this study, even one sheet of the conductive non-woven fabric sheet (Fig. 8b) have reflection loss better than −10 dB although its thickness is thinner than 0.5 mm, clearly indicating that the conductive non-woven fabric sheets are better than any other commercial millimeter-wave absorbers.

Conclusion

The findings obtained in this study are summarized as follows.

1) Non-woven fabric sheets coated with PPY nanoparticles were prepared by exposing non-woven fabric sheets containing APS as an oxidizing agent and BANS and BATS as a dopant to pyrrole vapor. The resistivity of the prepared conductive non-woven fabric sheets decreased from 1.5 × 10^5 to 3.0 Ω cm as the APS concentration was increased.
2) A stack of conductive non-woven fabric sheets (7.5 or 70 Ω cm/sheet) had constant μ' and μ'' values in the frequency range of 1–18 GHz, but ε' and ε'' values that varied with frequency, indicating that the RAM showed dielectric dispersion, in contrast to free space and a rubber sheet.
3) Reflection loss improved (i.e., radar absorption increased) as the stack size of conductive non-woven fabric sheets was increased.
4) A single conductive non-woven fabric sheet with resistivity of 7.5 Ω cm and a stack of least three non-woven fabric sheets (70 Ω cm/sheet) exhibited average reflection loss greater than 90% in the frequency range of 75–110 GHz, indicating that the lower resistivity sheet was superior.
5) Stacks of a conductive non-woven fabric sheet with resistivity of 7.5 or 70 Ω cm are excellent broadband RAMs that can effectively absorb electromagnetic waves, irrespective of incident angle.

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