Preparation and absorption performance of CNTs/PUR honeycomb composite absorbing material

Yankai Zhao 1, Song Bi *1, Genliang Hou 1, Zhaohui Liu 1, Hao Li 1, Yongzhi Song 1, Zhiling Hou 2

1 304 Teaching and Research Section, Rocket Force University of Engineering, Xi’an 710025.
2 College of Mathematics and Physics, Beijing University of Chemical Technology, Beijing, 100029, China.

*Corresponding Authors: Email: xiaozhu-youyou@163.com (B. S.)
Tel/Fax: +86 10 64433867

Abstract: In this paper, a carbon nanotubes/polyurethane resin (CNTs/PUR) honeycomb composite absorbing material was prepared, and the influence of the content of carbon nanotubes on the absorbing performance of single-layer and double-layer honeycomb composite absorbing materials was investigated. The mechanical properties of single-layer and double-layer materials are discussed. The honeycomb core and carbon nanotubes are compounded together by the impregnation method to form a CNTs/PUR honeycomb composite absorbing material. The microstructure shows that the carbon nanotubes are uniformly dispersed in the water-based polyurethane resin, and the impregnated layer and the honeycomb wall are well combined. The reflectivity of the material shows that as the content of carbon nanotubes increases, the absorption performance of the material first increases and then decreases; when the content of carbon nanotubes is 5.6%, the single-layer honeycomb composite absorbing material has the best absorption performance. The effective absorption bandwidth is 10.6 GHz (2~18GHz), and the maximum absorption strength is -24.5 dB; when the combination mode is 2-4, the double-layer honeycomb composite absorbing material has good absorption performance, and the maximum absorbing strength is -32.2 dB, the bandwidth is 13.7 GHz (2~18GHz).

1. Introduction

With the increasing of electromagnetic interference and electromagnetic radiation, absorbing materials have attracted more and more attention [1-3]. There are two types of absorbing materials: coating type and structure type. Compared with coated absorbing materials, structural absorbing materials have both bearing and absorbing in one, which is a research hotspot in the field of absorbing in the future [4-7]. The honeycomb sandwich structure absorbing material, as a typical structural absorbing material, has the characteristics of low density, high specific strength and high specific rigidity, and is widely used in the aerospace field [8-11]. The honeycomb sandwich structure absorbing material is composed of two upper and lower panels and an aramid honeycomb core in the middle. Generally, the research focus on the honeycomb sandwich structure absorbing material is focused on the honeycomb core structure. There are two ways to make the honeycomb core structure Wave-absorbing honeycomb core: coating method and foam filling method [12-15]. The coating method is to disperse various absorbents (carbon black, metal magnetic powder) into the polymer to form a slurry, and combine the honeycomb core and the absorbent
to form a wave-absorbing honeycomb core by dipping, or pass the absorbent through a spray gun Spray on the honeycomb core wall for compounding. Foam filling is to foam the absorbent and polymer, fill the formed foam into the honeycomb core to realize the composite, or fill the wave-transmitting material into the wave-absorbing honeycomb core to realize the composite of the material [16-19]. AA K et al. [20] coated the 20mm thick aramid honeycomb material with thermoplastic resin, which was filled with carbon powder as a lossy filler, and the results showed that the absorption bandwidth of 18GHz was displayed when the weight gain was 10%, and the maximum absorption strength was 7dB (80%). Pei-h Zhou [21] impregnated aramid paper frame with conductive carbon black (non-magnetic) and polyimide (PI) solution to prepare a wave-absorbing honeycomb composite material. The results showed that the thickness of the honeycomb composite impregnated layer was 5mm thick. When it is 0.2mm, the reflectance value of the entire 8-12 GHz frequency band is less than -10dB. HL et al. [22] prepared honeycomb cores with different pore sizes coated with epoxy resin and filled with conductive carbon black as detrimental fillers by dipping. The results show that the 8mm thick aramid honeycomb material, when the impregnated layer thickness is 25μm, in the frequency range of 5-18 GHz, the RL value is lower than -10dB. In the frequency range of 6~18GHz, the RL value can reach -15dB. Yan-f He et al. [23] used a spray method to prepare a radar absorbing material with a honeycomb sandwich structure coated with metal magnetic powder. When the weight of the honeycomb composite material under the thickness of 5mm is 65%, the frequency range is 2.6~18 GHz. The reflection loss is less than 5dB (70% power absorption). The above studies all used conductive carbon black as the absorbent to be combined with the honeycomb core, but did not study the case where the more difficult to disperse absorbent (carbon nanotubes, graphene) and the honeycomb core were combined together. Shenzhen Entron Advanced Materials Research Institute Co., Ltd. [24] disclosed a leaf-shaped nano-Fe$_3$O$_4$ filled honeycomb sandwich structure absorbing composite material, with an effective absorption bandwidth of 2.6 GHz (12.8GHz) at a thickness of 4mm, undefined 15.4GHz), the maximum absorption intensity at 14.2 GHz reaches 36dB. Ji Zhijiang et al. [25] prepared a gypsum-based absorbing material with a honeycomb structure by filling gypsum in a honeycomb structure impregnated with acetylene carbon black, and achieved an absorbing efficiency of less than -8 dB in the 2–18 GHz bandwidths. There are five absorption peaks in the rate curve, and the absorption peak near 2.2 GHz can reach -20.3 dB. Filling the honeycomb structure with foam or wave-consuming substances increases the weight of the honeycomb sandwich structure material to a certain extent, which is not in line with the development direction of light-weight wave-absorbing materials. In summary, this article uses acidified carbon nanotubes as absorbents, dispersed in water-based polyurethane resin to prepare a wave-absorbing slurry, and composites the honeycomb core and carbon nanotubes by dipping to form a honeycomb composite material. By adjusting the content of carbon nanotubes, the absorbing properties of single-layer and double-layer absorbing honeycomb cores were explored, and the absorbing mechanism was studied.

2. Experimental materials and methods

2.1 Materials
Carbon nanotubes (CNTs, provided by Suzhou First Element Nanotechnology Co., Ltd.), aramid honeycomb core (Nom-ex, 180mm×180mm×5 mm, side length 2.78mm) (provided by Wuhan Magnetoelectric Company), water-based polyurethane resin (NL-236, 30%, provided by Wuhan Magnetoelectric Company), water-based wetting and dispersing agent (SP-712, provided by Xi’an TEDA Chemical Glass), water-based defoamer (SP-852, provided by Xi’an TEDA Chemical Glass), deionized water.

2.2 Preparation of CNTs/ PUR honeycomb composite
As shown in Figure 1, a certain amount of acidified carbon nanotubes was added to 200g of water-based polyurethane resin, and then 600g of deionized water was added, and the glue was stirred in a water bath at 30°C. At this time, the color of the glue is dark green. Adding 10g of dispersant during the stirring process to make it evenly mixed. After 1 min, adding 10g of defoaming agent to remove bubbles.
generated by the stirring. After stirring for 30 minutes, taking out the glue solution and ultrasonically shake it for 30 minutes. At this time, the color of the glue solution was black. Continuing to stir the solution for 15 minutes and then took it out. At this time, the glue solution was the dipping glue solution.

Pouring the dipping glue into the dipping tank, and placed the aramid honeycomb in the dipping tank for 20 minutes, and moved it slightly left and right with tweezers to ensure complete dipping. Then took out the impregnated aramid honeycomb and placed it on the hollowed out stainless-steel net, dried it at room temperature for 1 min, put it in a drying box and raise to 100 °C, kept it for 10 min for pre-curing. During the pre-curing process, turned over once every 2.5 minutes to avoid the gradient distribution of the glue on the honeycomb wall. After turning over 4 times, the pre-curing was completed. Finally, put it in a drying oven and heat it up to 120°C, and keep it for 1 hour to complete the final curing. At this time, the honeycomb core was the honeycomb absorbing sample. If the weight gain standard was not reached, repeated the above operation. Four samples were prepared by adjusting the content of carbon nanotubes (3.8%, 4.7%, 5.6%, and 6.5%). Table 1 lists the weight of the honeycomb composite material, and Figure 2 shows a sample of the prepared honeycomb composite absorbing material.

![Fig. 1 Flow chart of preparation of absorbing honeycomb core](image)

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| Serial number | Absorbent composition | Height of honeycomb (mm) | Original weight of honeycomb (g) | Weight of honeycomb after impregnation (g) | Absorbent content (g) |
|---------------|-----------------------|--------------------------|-------------------------------|---------------------------------|---------------------|
| 1             | CNTs (8g)             | 5                        | 8.13                          | 16.0                            | 0.30                |
| 2             | CNTs (10g)            | 5                        | 8.13                          | 15.6                            | 0.39                |
| 3             | CNTs (12g)            | 5                        | 8.13                          | 15.7                            | 0.47                |
| 4             | CNTs (14g)            | 5                        | 8.13                          | 15.5                            | 0.55                |
Fig. 2 Samples of honeycomb composite material

2.3 Analysis and testing equipment
A scanning electron microscope (SEM) was used to characterize the honeycomb wall absorbing coating, and the instrument model was Hitachi S-4800. A universal testing machine was used to carry out a compression test on the honeycomb composite material, the model is WDW-10A. In addition, the arc-shaped frame method is used to test the reflectivity of the prepared wave-absorbing honeycomb, and the instrument model is CEYEAR 3672C.

3. Results and analysis

3.1 Morphology analysis
Figure 3 is SEM of the thickness of the impregnated layer. It can be seen that the surface of the impregnated layer is relatively smooth. The carbon nanotubes are wrapped so that the absorbent will not peel off due to the external forces. The thickness of the honeycomb wall is 100μm. The thickness of the impregnated layer of the four samples are 32μm, 37μm, 30μm and 37μm, which basically meets the requirements of light and thin honeycomb composite materials.

Figure. 4 is SEM of the impregnated layer of honeycomb composite material. It can be seen from the figure that the carbon nanotubes are tubular and uniformly distributed in the polyurethane resin. As the content of the absorbent increases, the carbon nanotubes in the polyurethane resin evolve gradually from a dotted dispersion state to a linear winding state. There are a few pores on the surface of the polyurethane resin, which is related to the introduction of air during the vigorous stirring of the slurry. The surface tension increases with the generation of pores, which makes the impregnated layer tightly adhere to the honeycomb core wall.
3.2 Analysis of Absorbing Performance

The design of the absorbing material is the process of matching the input impedance of the structure with the inherent impedance of the free space. According to the structural characteristics of the absorbing material, the electromagnetic wave energy incident on the absorbing material is partially absorbed, and the rest is reflected. The reflection is described by the following formula:

$$\Gamma = \frac{Z_{in} - Z_0}{Z_{in} + Z_0}$$

(1)

Where, \( \Gamma \) is the reflection coefficient, \( Z_{in} \) is the input impedance of the microwave absorber, and \( Z_0 \) is the inherent impedance of free space. Then, in the transmission line theory, the microwave absorber is regarded as an equivalent circuit, and the input impedance is calculated as follows:

$$Z_i = \eta_i Z_{i-1} + \eta_i \tan h \left( \gamma_i d_i \right)$$

$$\eta_i + Z_{i-1} \tan h \left( \gamma_i d_i \right)$$

(2)

\( Z_i \) is the input impedance of the i-th layer, \( \eta_i \) is the impedance of the i-th layer of intrinsic impedance, \( \gamma_i \) is the propagation constant of the i-th layer, and \( d_i \) is the thickness of the i-th layer.

The natural impedance \( \eta \) and the propagation constant \( \gamma_i \) are determined by the frequency-dependent characteristics of the material:

$$\eta = Z_0 \sqrt{\frac{\mu_i}{\varepsilon_i}}$$

(3)

$$\gamma_i = 2\pi j \frac{\sqrt{\varepsilon_i \mu_i}}{\lambda}$$

(4)

In formula (4), \( \varepsilon_i \) is the relative permittivity of the material, and \( \mu_i \) is the relative permeability of the material. Since the electromagnetic characteristic is a frequency-dependent value, the input impedance becomes a frequency-dependent value, which makes impedance matching challenging in a wide frequency range. Using transmission line theory, the input impedance of a structure composed of uniform materials in the direction of electromagnetic wave propagation is calculated. The honeycomb structure has a unique geometric shape and is an inhomogeneous medium whose inherent impedance varies with position. However, if the unique geometry of the honeycomb is much smaller than the wavelength, it can be approximated as a homogeneous medium.
3.2.1 Analysis of Absorbing Performance of Single-Layer Composite Materials

The absorbing parameters of the single-layer honeycomb sandwich absorbing material are analyzed. The obtained values are listed in Table 2. In order to facilitate observation, Figure 5 and Figure 6 are drawn to make the data more intuitive.

Table. 2 Absorbing parameters of single-layer honeycomb sandwich absorbing materials

| Serial number | Height of single honeycomb (mm) | Maximum peak frequency (GHz) | Peak value (dB) | Frequency band (GHz) (RL<-10dB) | Bandwidth (GHz) (RL<-10dB) |
|---------------|---------------------------------|------------------------------|----------------|---------------------------------|----------------------------|
| 1             | 5                               | 12.7                         | -10.5          | 11.4-13.9                       | 2.5                        |
| 2             | 5                               | 11.3                         | -14.0          | 8.7-14.4                        | 5.7                        |
| 3             | 5                               | 10.3                         | -24.5          | 6.6-17.2                        | 10.6                       |
| 4             | 5                               | 9.7                          | -18.3          | 6.4-14.7                        | 8.3                        |

Fig. 5 shows the reflectivity of a single-layer honeycomb composite material in the range of 2~18 GHz. It can be seen that as the content of carbon nanotubes increases, the effective absorption frequency band and absorption peak of the composite material gradually shift to the lower frequency band. When the carbon nanotube content is 3.8wt%, the absorption bandwidth of the composite material is only 2.5 GHz, and the maximum absorption intensity is -10.5dB. With the increase of carbon nanotube content, the samples of 4.7wt%, 5.6wt% and 6.5wt% respectively showed certain electromagnetic wave absorption capacity; the carbon nanotube content was 5.6wt%, the effective absorption bandwidth was 10.6 GHz, and the maximum absorption intensity reaches -24.5 dB; in comparison, when the content is 6.5wt%, the low-frequency absorption band is slightly broadened, and the high-frequency absorption band is slightly reduced. The effective absorption bandwidth is 8.3 GHz and the absorption intensity is -18.3 dB; all electromagnetic waves are absorbed in the Ku band; when the content is 3.8%, the electromagnetic wave absorption capacity is poor, the absorption bandwidth is only 5.6 GHz, and the maximum absorption intensity is -14 dB. From the above analysis, it can be seen that as the content of carbon nanotubes increases, the electromagnetic wave absorption capacity of the single-layer honeycomb composite material first increases and then decreases. On the one hand, the combination of the honeycomb structure and the adsorbent makes electromagnetic waves in the material multiple times scattering, and with the increase of CNTs concentration, a conductive path (permeation network) is formed, resulting in a higher resistance loss. On the other hand, the dense penetration network will lead to a higher dielectric constant, the conductivity of the sample will increase, and the skin depth will become shorter, so that microwaves can be reflected from the surface of the material. Therefore, when the content is 5.6wt%, its microwave absorbing performance is the best, not the composite material with 6.5wt% content. The composite material sample with a content of 5.6wt% showed a maximum absorption strength of -24.5dB, and an absorption bandwidth of 10.6GHz (2-18GHz).
Fig. 5 Reflectivity of single-layer honeycomb composite at different concentrations

Fig. 6 shows the absorption band diagram of a single-layer honeycomb composite material. It can be seen from the figure that as the content of carbon nanotubes increases, the effective absorption band first increases and then decreases, and the effective absorption band gradually shifts to the low-frequency direction. The difference in the absorption band corresponds to the absorption bandwidth in FIG. 5. When the content is 3.8wt%, its absorption frequency band is 11~14 GHz, when the content is 4.7%, its absorption frequency band is 8.8~14.4 GHz, when the content is 5.6wt%, its absorption frequency band is 6.6~17.2 GHz, when the content is 6.5%, the absorption frequency band is 6.3~14GHz.

3.2.2 Analysis of the absorbing performance of double-layer composite materials

Table. 3 shows the absorbing parameters of the double-layer honeycomb sandwich absorbing material. The double-layer structure consists of 6 samples, and each sample is superimposed as the concentration gradient increases. The values obtained are listed in Table 3. For the convenience of observation, Figures 7 and 8 are drawn to make the data more intuitive.
### Table 3: Absorbing parameters of double-layers honeycomb sandwich absorbing materials

| Serial number | Height of double honeycomb (mm) | Maximum peak frequency (GHz) | Peak value (dB) | Frequency band (GHz) (RL<10dB) | Bandwidth (GHz) (RL<10dB) |
|---------------|---------------------------------|------------------------------|----------------|-------------------------------|---------------------------|
| 1             | 10                              | 4.3, 11.4                    | -10.7, -12.0   | 3.6-5.6 8.5-13.8             | 7.3                       |
| 2             | 10                              | 4.4, 15.2                    | -11.8, -15.2   | 3.4-6.6 8.5-17.2             | 11.9                      |
| 3             | 10                              | 4.3, 12.5                    | -11.5, -14.1   | 3.5-17.0                      | 13.5                      |
| 4             | 10                              | 6.4, 16.4                    | -15.3, -27.0   | 4.7-9 13-18                   | 9.3                       |
| 5             | 10                              | 6.1, 16.5                    | -28.4, -32.2   | 4.3-18                        | 13.7                      |
| 6             | 10                              | 5.2, 15.4                    | -24.7, -23.5   | 3.6-8.3 12.0-17.6            | 10.3                      |

As shown in Fig. 7, it is the reflectivity map of the double-layer honeycomb composite material in the range of 2~18GHz. For convenience, the concentrations of the four adsorbents (3.8wt%, 4.7wt%, 5.6wt%, and 6.5wt%) are expressed as 1, 2, 3, and 4 respectively. The combinations of these six samples are called 1-2, 1-3, 1-4, 2-3, 2-4 and 3-4. The six curves in the figure are the reflection curves of the two-layer combination of six concentration gradients of composite materials. It can be seen that the absorption strength of the first three curves is not good, but as the concentration of carbon nanotubes at the bottom of the honeycomb material increases, the absorption bandwidth increases. When the combination of materials is 1-2, the absorption of the composite material The frequency band is 7.3GHz, but the absorption strength is not high, about -12dB. When the combined modes are 1-3 and 1-4, the absorption bandwidths increase to 9.3GHz and 13.7GHz (2~18GHz), but the absorption intensity is still not high, and the maximum absorption intensities are -15.5dB and -14.1dB (2~18GHz). The reason may be that the upper layer absorber content is small and the permeation network is not formed, resulting in low resistance loss. Although the concentration of the bottom layer increases, the formation of the percolation network enhances the dielectric properties of the material and reflects electromagnetic waves. At the same time, the superposition of the double-layer structure increases the absorption channel, so that electromagnetic waves are reflected in the material multiple times, expanding the absorption frequency band thereby. In addition, the electromagnetic wave resonates in the double-layer structure, and the absorption peak changes from a single peak to a double peak, and the double peak gradually shifts to the low frequency band. In the last three curves, as the concentration of upper and lower carbon nanotubes increases, the absorption intensity increases significantly, while the absorption bandwidth decreases with the increase of the upper carbon nanotube concentration. It can be observed that when the combined modes are 2-3, 2-4 and 3-4, the bandwidths are 9.3GHz, 13.7GHz and 10.3GHz (2~18GHz), and the maximum absorption intensities are -27dB, -32.2dB and -24.7dB (2~18GHz). Only when the upper layer material concentration is controlled within a proper range, can the wave be absorbed and transmitted, and the lower layer material concentration is controlled within the proper range, can it absorb waves to a greater extent and the material can have excellent absorption properties. Based on the above analysis, when the double-layer material is in the first three combinations, its absorbing performance first increases and then decreases. Similarly, the trends of the latter three combinations of double-layer materials are also the same. When the combination mode is 2-4, the material has good absorbing performance, the maximum absorbing strength is -32.2dB, and the bandwidth is 13.7GHz (2~18GHz).
Fig. 7 Reflectivity of double-layers honeycomb composite at different concentrations

Figure 8 shows the absorbing frequency band diagram of the double-layer honeycomb composite material. It can be seen in the figure that the effective absorbing bandwidth of the first three combinations of absorbing materials increases with the increase of the upper carbon nanotube content. When the combination is 1-2, 1-3, 1-4, the absorbing frequency bands are 3.6~5.6 GHz and 8.5-13.8 GHz, 3.4-6.6 GHz and 8.5-17.2 GHz, and 3.5-17.0 GHz, respectively. When the combination is 2-3, 2-4, 3-4, the absorbing frequency bands are 4.7~9 GHz and 13~18 GHz, 4.3~18 GHz, 3.6-8.3 GHz and 12.0~17.6 GHz, respectively. The difference in the absorbing frequency band corresponds to the absorbing bandwidth in Figure 7.

3.3 Analysis of the principle of wave absorption

The ability of absorbing materials to effectively absorb electromagnetic waves depends on two conditions: impedance matching and electromagnetic loss. According to the electromagnetic
transmission theory, when the electromagnetic wave is incident on the surface of the material, the loss formula of the electromagnetic wave is as follows:

$$R_L = 20\log \left| \frac{Z_{\text{in}} - Z_0}{Z_{\text{in}} + Z_0} \right|$$  \hspace{1cm} (5)

$$Z_{\text{in}} = \sqrt{\frac{\mu}{\varepsilon}} \tan g d$$ \hspace{1cm} (6)

Among them, $R_L$ is the reflectivity, $Z_{\text{in}}$ is the input impedance, which is related to the dielectric constant ($\varepsilon$), permeability ($\mu$) and thickness ($d$) of the material, $g$ is the propagation constant of electromagnetic waves, and $Z_0$ is the air impedance. When $Z_{\text{in}}=Z_0$, $R_L$ is 0, and the material can achieve full absorption of electromagnetic waves [31-35].

Fig. 9 shows a schematic diagram of single-layer CNTs/PUR honeycomb composite material absorbing waves. When electromagnetic waves are incident on the surface of the single-layer honeycomb composite material, because the absorber is a thin layer on the honeycomb wall, and the absorber is a dielectric material, the dielectric constant is much larger than the magnetic permeability, the impedance matching performance is relatively poor, a small part of the incident wave penetrates the absorbent and is lost, most of the incident wave is reflected to other honeycomb walls, the electromagnetic waves are repeatedly reflected on the honeycomb wall and the wave absorption performance of the material is finally realized. It can be seen that the honeycomb composite material is related to the lossy ability of the absorbent, and the resistance and lossy ability of the absorbent can be adjusted by adjusting the concentration of the absorbent, thereby improving the wave absorbing performance of the material.

Fig. 9 Schematic diagram of absorbing principle of single-layer CNTs/PUR honeycomb sandwich composite absorbing waves

Fig. 10 is a schematic diagram of the double-layer CNTs/PUR honeycomb composite material for absorbing waves. Compared with the single-layer honeycomb composite material, the double-layer honeycomb composite material has more channels for the incident wave to reflect on the honeycomb wall, which is conducive to the attenuation of electromagnetic waves. And absorption. The material between the two is usually glass fiber, and the two are bonded together by an adhesive. Compared with the single-layer structure, the double-layer structure design is more stable in a certain sense. At the same time, the glass fiber is lighter and meets the overall lightweight standard of the material. In short, the design of double-layer structure or multi-layer structure provides more possibilities for improving the absorbing performance of materials, which is the development trend of structural absorbing materials in the future.
4. Conclusion

In this paper, CNTs/PUR honeycomb composite materials are prepared. The absorbent is compounded with the honeycomb material to form a stable honeycomb composite material. As the content of the single-layer honeycomb composite absorbing material increases, its effective absorbing frequency band and absorption peak gradually move to the low-frequency band. The composite materials with an absorbent content of 4.7wt%, 5.6wt%, and 6.5wt% all exhibit certain electromagnetic absorption capacity, when the absorbent content is 5.6wt%, the absorption performance is the best, the absorption frequency band is 6.6~17.2 GHz, the effective absorption bandwidth is 10.6 GHz, and the absorption intensity reaches -24.49 dB. The effective absorption frequency band of the double-layer honeycomb composite absorbing material moves to the low frequency band with the increase of the absorbent content, the absorption peak changes from a single peak to a double peak, and the double peaks gradually move to the low frequency band. The combination mode is 2-4, the absorbing performance is the best, the absorbing frequency band is 4.3-18 GHz, the maximum absorbing intensity is -32.2dB, and the bandwidth is 13.7GHz (2~18GHz). Compared with the single-layer structure, the double-layer structure or the multi-layer structure design provides more channels for material absorbing, which is the development trend of structural absorbing materials in the future.

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