Microwave absorption properties of carbon fibre containing nonwovens

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A variety of nonwoven containing different weight percentages of pitch-based carbon fibre has been developed using through-air thermal bonding process or the spray bonding process and studied for its microwave absorbing capacity in the microwave frequency range 8 - 18GHz. Microwave reflectivity of the nonwoven is found closely related to the carbon fibre content. Electromagnetic parameters of the component fibres in the nonwoven have also been studied. Variation in microwave absorbing capacity of the nonwoven with the carbon fibre content is expounded according to the relationship between the electromagnetic parameters and the reflection coefficient of the nonwoven, as well as the relationship between the electromagnetic parameters and the attenuation constant of the nonwoven. The carbon fibre containing nonwoven has great potential in the military application as the radar camouflage material or in the non-defense application as the electromagnetic shielding material.

Keywords: Carbon fibre, Microwave absorption, Nonwoven, Permeability, Permittivity, Polyester, Reflectivity

IPC Code: Int. Cl.8 D04H

1 Introduction

With the development of radar reconnaissance technology, military applications require high performance camouflage materials with light weight and at low cost over a broad frequency band. This can be achieved by many ways, one of which is by designing and optimizing textiles reinforced by microwave absorbing fillers since it is easier to manufacture textiles in large scale and at a lower cost than most other camouflage materials. Furthermore, it is also easy to process textiles by various means to endow them with multiple camouflage properties.

Nonwoven textiles having variable thicknesses can be more easily manufactured in comparison to other textiles. It is found that only those materials which have enough thickness exhibit high microwave absorption capacity. There have been some reports published on the microwave absorption properties of the nonwoven. Lopes et al.1 reported the microwave properties of nonwovens impregnated with conductive carbon black at the X-band (8.2-12.4 GHz). It has been found that the absorption effectiveness of the nonwoven depends on the filler concentration. The nonwoven containing 33wt% of carbon black shows the best absorption. This property is expounded on the basis of the theory1 whether the conductive filler loading is below or above the threshold of conductivity. However, the impregnated nonwoven shows poor mechanical properties, air permeability and handling, which limits its application in adverse environment. The nonwoven fabricated with viscose-based carbon fibre and polypropylene fibre was investigated by Zhang et al.2. They found that the microwave reflection and the transmission properties of the nonwoven vary with the change of carbon fibre content in the nonwoven. It is also reported that when the carbon fibre content in the nonwoven increases from 5.1g/m² to 19.8g/m², the reflected microwave by the nonwoven increases and the transmitted wave decreases. By comparing the reflected wave of the nonwoven with that of a certain camouflage net material, it is concluded that a part of the electromagnetic wave is absorbed by the carbon fibre.
However, they did not report the optimum carbon fibre content at which the microwave absorption capacity of the nonwoven reaches the maximum. Also, their research was limited within the frequency range 9 - 10 GHz. The viscose-based carbon fibre was found to be expensive and unfavorable for the mass production and application of the nonwoven.

In order to study, in depth, the relationship between the carbon fibre content in the nonwoven and the microwave absorption properties in a broad frequency range, and to develop applicable nonwovens for military application or electromagnetic shielding, a variety of nonwoven has been fabricated with the pitch-based carbon fibre (PCF), heat-bondable bicomponent fibre and polyester fibre. The PCF was used as it is much economical as compared to viscose-based carbon fibre.

2 Materials and Methods

2.1 Materials

Pitch-based carbon fibre (PCF) was procured from the Osaka Gas Chemicals Co., Ltd, Osaka, Japan; the heat-bondable bicomponent fibre 4080 (skin of the fibre is constituted by polyethylene which melts at the temperature > 130°C and the core is polyester) from Ningbo Dacheng New Materials Co., Ltd, Ningbo, China; and polyester fibre from Yizheng Chemical Fibre Co., Ltd, Yizheng, China. The specifications of the fibres are given in Table 1. Microemulsion of amino-modified polyorganosiloxane was prepared as per the procedure reported by Wang et al.

2.2 Methods

2.2.1 Fabrication of Nonwoven

Nonwovens were prepared using blow room and carding line set-up of Zhengan Textile Machinery Co., Ltd. Firstly, the PCF, the bicomponent fibre 4080 and the polyester fibre were blended according to weight percentages as given in Table 2. Then the samples were sent to the FA022-8 multi-bin mixer and repeatedly processed to achieve proper blending. The above samples were passed through the carding process on the W1202 carding machine followed by the lapping process on the W1251-260 cross-lapping machine and then processed into fibre webs. Finally, the fibre webs with different weight percentages of the PCF were processed in the JXXP-260 thermobonding machine. The heat-bondable fibres in fibre webs were melted by using the hot air at 160°C and bonded with other fibres together, forming fibre webs and then the nonwovens. Area weight of the finished nonwoven was 308 g/m² and the material was processed at a speed of 6 m/min.

Contents of the heat-bondable fibre were not enough for the fibre webs with PCF contents of 90% and 100% to form firm nonwovens, hence the spray bonding process was adopted to fabricate nonwovens using the WBG 981 spray bonding product line (Changzhou Haofeng Machinery Co., Ltd). The bonding agent was acrylic ester emulsion containing 10 wt % of acrylic ester. On the specialized product line, 200 g/m² of bonding agent was sprayed to each side of the fibre web under high pressure. The fibre web was then squeezed to remove the excessive bonding agent and was processed on the JXXP-260 thermobonding machine, where water in the binding agent was evaporated by the effect of hot air at 130°C and the fibre web was formed into nonwovens at the pressure of about 6.5 g/cm². Area weight of the finished nonwovens was 320 g/m², thickness was 10 mm and speed of the production line was 4 m/min.

Table 1 — Specification of the component fibres in the nonwoven

<table>
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<tr>
<th>Fibre</th>
<th>Linear density</th>
<th>Density</th>
<th>Length</th>
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<tr>
<td>PCF</td>
<td>0.22</td>
<td>1.65</td>
<td>50</td>
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<tr>
<td>Bicomponent</td>
<td>0.167</td>
<td>1.14</td>
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</tr>
<tr>
<td>Polyester</td>
<td>0.167</td>
<td>1.38</td>
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Table 2 — Weight percentages of component fibres in nonwoven

<table>
<thead>
<tr>
<th>Specimens</th>
<th>PCF</th>
<th>Bicomponent fibre 4080</th>
<th>Polyester fibre</th>
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<tr>
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<td>80</td>
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<tr>
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2.2.2 Surface Insulation of PCF

The PCF (pitch-based carbon fibre) is a conductive material. If there are enough PCFs contacting with each other in the nonwoven, the conductive networks may form, thus imparting the high conductivity to the nonwoven. According to the theory\(^4\) about the interaction of a conductor with the electromagnetic field, when the electromagnetic wave impinges onto the surface of a conductor, the reflection coefficient \((R)\) is given as follows:

\[
R = \frac{\sigma + \sqrt{\sigma^2 - 2\omega \varepsilon_0 \varepsilon''}}{\sigma - \sqrt{\sigma^2 - 2\omega \varepsilon_0 \varepsilon''}} = 1 - 2 \frac{2\omega \varepsilon_0 \varepsilon''}{\sigma} \ldots (1)
\]

where \(\sigma\) is the conductivity of the conductor; \(\omega\), the frequency of the electromagnetic wave; and \(\varepsilon_0\), the permittivity of the free space (\(\varepsilon_0=8.854\times10^{-14} \text{ F/cm}\)).

\(\varepsilon''\) can be inferred from Eq. (1) that the higher the conductivity, the higher is the reflection coefficient of the conductor. High reflection coefficient indicates that much electromagnetic energy is reflected back to the space rather than entering into the conductor and being absorbed. Hence, the surface of the PCF shall be insulated to avoid the formation of conductive networks.

Polyorganosiloxane has low glass transition temperature, soft molecule segment and low surface tension. It tends to form thin membrane on the surface of fibres reducing the surface friction. Since the volume resistivity of polyorganosiloxane is low, the thin polyorganosiloxane membrane on the fibre surface can increase the contact resistance between fibres.\(^3\) Amino-modified polyorganosiloxane solution was used to insulate carbon fibres in the nonwoven in this study.

Amino-modified polyorganosiloxane was prepared by means of bulk polymerization. It was then emulsified into microemulsion with nonionic surfactant and was diluted with water to the concentration of 7%. Microemulsion was favourable for the amino-modified polyorganosiloxane to penetrate throughout the collection of fibres and to cover them thoroughly.

Nonwovens with different PCF contents were soaked in the microemulsion of amino-modified polyorganosiloxane for 50 min, and then dehydrated in a spin dryer for 5 min followed by baking in an oven at 120°C for 40 min. The dried nonwovens were then re-soaked in the microemulsion of amino-modified polyorganosiloxane for 50 min. The soaking-baking-soaking cycle was repeated for 3 times. After the cycle was complete, the nonwovens were baked in the oven for the last time and the baking temperature was raised to 180°C. The samples were clamped between two parallel lattices in the course of baking, and the distance between the lattices was adjusted at 10 mm to ensure that the thickness of nonwovens was 10 mm after the baking process. The last baking process was continued for 30 min till most of the remaining surfactant on the fibre surface decomposed off so as to reduce the moisture absorbency of the fibre and to keep high resistance between the fibres. The volume resistivity of nonwovens was measured according to the method reported by Smits.\(^5\) The resistivity of all the nonwovens processed should be above \(8\times10^3 \Omega \cdot \text{cm}\) and those with resistivity under this threshold were reprocessed following the above procedures until they met the requirement. Thickness of all the nonwovens after the process was 10mm and compressional recovery of the nonwovens was between 80% and 85% as the carbon fibre content changed from 0% to 100% on the basis of the method as described earlier.\(^6\) The processed nonwovens were cut to specimens of 180mm (L) \times180mm (W) \times10mm (D) in size for the measurement.

2.2.3 Measurement of Electromagnetic Parameter

Since it was difficult to test electromagnetic parameters of fibres, these parameters of the component fibres in the nonwoven were comparatively investigated by blending the fibres with epoxy to form homogeneous and isotropic composites and comparing them with the electromagnetic parameters of the composites.

It can be inferred from the theory by Wang et al.\(^7\) that the electromagnetic parameters (\(\mu'\) and \(\mu''\), the real and imaginary parts of permeability; and \(\varepsilon'\) and \(\varepsilon''\), the real and imaginary parts of permittivity respectively) of the composite at certain frequency \(\omega\) are the contribution by electromagnetic parameters of each component in the composite on the basis of their respective volume fraction in the composite. Electromagnetic parameters of different materials have the same percentage of contribution to those of the composite when their volume fractions in the composite are equal.

Electromagnetic parameters of composites were analyzed by an Agilent E8363A vector network analyzer on the basis of the method reported by Weir.\(^8\)
By measuring the scattering parameters of the network, the electromagnetic parameters of samples were calculated using the principle as reported by Weir.  

To compare the electromagnetic parameters of different fibres in the nonwoven, epoxy was selected to constitute composite with the fibres. Electromagnetic parameters of epoxy were measured and the results are given in Fig. 1. The curves show that the real part of the permeability ($\mu'$) of epoxy is close to 1 and the imaginary part ($\mu''$) is around 0, so the magnetic loss tangent ($\tan\delta_m = \mu''/\mu'$, a parameter representing the magnetic loss capability of the material) of epoxy is about 0, i.e. epoxy is the non-magnetic material. 

PCF, polyester fibre and the bicomponent fibre 4080 were cut to the segments of 10 mm in length for fabricating composites. Each type of the fibre segment was blended with epoxy respectively at the same volume fraction (7.5% by volume in the fibre/epoxy composite). The fibre/epoxy composite was repeatedly churned with a metal needle before it was cured until the fibre segments were evenly distributed in the epoxy like random coils. Then, the composite was filled into the slot of the sample holder for the measurement after being cured.

Electromagnetic parameters of the fibre/epoxy composite depend on the electromagnetic parameters of its constituents on the basis of their respective volume fractions in the composite. Since volume fractions of different fibres in their respective composites are equal, the differences among electromagnetic parameters and electromagnetic absorption capacities of these fibres can be disclosed by comparing electromagnetic parameters of fibre/epoxy composites.

2.2.4 Measurement of Reflectivity

Reflectivity of radar absorbing materials, as reported earlier, is defined as “when a plane electromagnetic wave with certain frequency and polarization vertically impinges on the surface of a plane target, the ratio of the backscattered power from the material placed on a metal plate (a perfect reflector of electromagnetic waves with the same surface shape and area as the material) to that from the metal plate after the material has been removed”, as shown below:

$$\Gamma_m(\omega) = \frac{P_a(\omega)}{P_m(\omega)}$$

where $\Gamma_m(\omega)$ is the reflectivity of the material; $P_a(\omega)$ & $P_m(\omega)$, the backscattered power of the electromagnetic wave with certain frequency $\omega$ reflected from the material to be studied and from the metal plate respectively.

Reflectivity of the nonwoven in the frequency range 8-18 GHz was tested with the RCS measurement system reported by Vinoy et al. The test was performed in a microwave anechoic chamber. There was a low permittivity target support column made of polyfoam in the chamber. Position of the target should be aligned with a laser beam to ensure that the incident microwave would be reflected back along the original path. Firstly, a metal plate of the size 180mm (L) ×180mm (W) ×4mm (D) was placed as the target and the power of the reflected wave from the metal plate was measured. A piece of nonwoven of the size 180mm (L) ×180mm (W) ×10mm (D) was then stuck to the metal plate and
placed as the target. The power of the reflected wave was measured. The reflectivity was then calculated using the Eq. (2). A network analyzer HP8757D was used in the system for measuring the reflection power. This method of measurement has the advantage of simulating the real conditions of a plane wave.

There is no transmission of the electromagnetic wave since the metal plate behind the material reflects any wave penetrating the material. It can be seen from Eq. (2) that the lower the reflectivity, the higher is the electromagnetic absorption capacity of the material and vice versa. So, the value of reflectivity indicates the electromagnetic absorption capacity of plane materials. When the unit of reflectivity is expressed in dB, the equation for reflectivity is expressed as follows:

\[ 10 \log_{10} \Gamma_m(\omega) = 10 \log_{10} \Gamma_\infty(\omega) - 10 \log_{10} \Gamma_m(\omega) \quad \ldots(3) \]

3 Results and Discussion

3.1 Electromagnetic Parameters of Fibre

It is observed that the PCF, polyester fibre and bicomponent fibre 4080 are nonmagnetic materials and, as discussed in section 2.2.3, the nonwovens comprising them are also non-magnetic materials. Figure 3 shows the real parts of permittivity (\(\varepsilon'\)) of composites studied. Figure 4 gives the dielectric loss tangent (\(\tan \delta = \varepsilon''/\varepsilon'\), a parameter representing the dielectric loss capability of the material) of the composites. It can be observed from the figures that the real part of permittivity and the dielectric loss tangent of the PCF/epoxy composite are much higher than those of the other composites. Dielectric loss tangent of the PCF/epoxy composite ranges between 1.0 and 2.0, while that of the polyester fibre/epoxy and bicomponent fibre/epoxy are close to 0, indicating that the PCF is a powerful microwave absorbing agent while polyester fibre and the bicomponent fibre 4080 are not.

3.2 Reflectivity of Nonwoven

Figure 5 shows the relationship between microwave reflectivity of nonwovens at the frequency of 12GHz and the content of PCF in nonwovens. Figure 6 shows the reflectivity of nonwovens with different PCF contents in the whole frequency range from 8 GHz to 18GHz. The results indicate that the reflectivity of the nonwoven varies with the PCF content in the nonwoven. Reflectivity of the nonwoven is quite high when the PCF content is 0%. As the PCF content increases from 0% the reflectivity sharply declines and reaches the minimum at 2% PCF content. After the PCF content exceeds 2%, the reflectivity sharply increases with the further increase in the PCF content. When the PCF content is above 20%, the trend of the increase in reflectivity with the rise of the PCF content slows down, and such a trend lasts until the PCF content increases in 100%.
It can be concluded from above results that the nonwoven with PCF content around 2% by weight and resistivity above $8 \times 10^3 \, \Omega \cdot \text{cm}$ is fit for the anti-radar camouflage of ground objects since the reflectivity of most of the terrain backgrounds is around 10 dB.

### 3.3 Explanation on the Variation in Reflectivity of Nonwoven with PCF Content

The prerequisite for the material to absorb microwave is that the incident microwave enters the material as much as possible, rather than being reflected from the surface of the material. The reflection coefficient $R$ and the transmission coefficient $T$ of the material are used to indicate the extent to which the microwave enters the material. The higher the absolute value of $R$ and the lower the value of $T$, the more microwave is reflected from the surface of the material and the higher is the reflectivity measured, and vice versa. The reflection coefficient $R$ at the interface between the free space and the material and the transmission coefficient $T$ can be expressed as follows:

$$ R = \frac{\eta_1 - \eta_0}{\eta_1 + \eta_0} = \frac{2\eta_0}{\eta_1 + \eta_0} \quad |R| \leq 1 \quad \ldots (4) $$

$$ T = 1 + R = 2 \frac{\eta_1}{\eta_1 + \eta_0} = 2 - \frac{2\eta_0}{\eta_1 + \eta_0} \quad \ldots (5) $$

where $\eta_1$ is the wave impedance of the material; and $\eta_0$, the wave impedance of free space ($\eta_0 \approx 377 \, \Omega$).

$$ \eta = \sqrt{\frac{\mu}{\varepsilon}} = \sqrt{\frac{\mu \varepsilon - j \mu \varepsilon}{\varepsilon \varepsilon - je \varepsilon}} \quad \ldots (6) $$

where $\varepsilon'$ and $\varepsilon''$ are the real and the imaginary parts of permittivity of the material respectively; and $\mu'$ and $\mu''$, the real and imaginary parts of the permeability respectively. Attenuation constant $\alpha$ indicates the absorption of microwave by per unit length of the material. It can be expressed as follows:

$$ \alpha = \frac{\omega}{\sqrt{2c}} \sqrt{\eta_1 \varepsilon - j \mu \varepsilon + \sqrt{(\mu \varepsilon + \mu \varepsilon)(\varepsilon \varepsilon + \varepsilon \varepsilon)}} \quad \ldots (7) $$

where $\omega$ is the angular frequency of the incident microwave; and $c$, the speed of light in free space.

For nonmagnetic materials ($\mu' = 1$ and $\mu'' = 0$), Eqs (6) and (7) are simplified as follows:

$$ \eta_i = \sqrt{\frac{\mu}{\varepsilon}} = \sqrt{\frac{\mu \varepsilon - j \mu \varepsilon}{\varepsilon \varepsilon - je \varepsilon}} \quad \ldots (8) $$

$$ \alpha = \frac{\omega}{\sqrt{2c}} \sqrt{\varepsilon + \sqrt{(\varepsilon \varepsilon + \varepsilon \varepsilon)}} $$

$$ = \frac{\omega}{\sqrt{2c}} \sqrt{\varepsilon + \sqrt{(\varepsilon \varepsilon + \varepsilon \varepsilon)}} \quad \ldots (9) $$

It can be inferred from Eqs (4)-(9) that $R$, $T$ and $\alpha$ of the nonwoven are all determined by $\varepsilon'$ and $\varepsilon''$ of the nonwoven. According to Eq. (8), the higher the value of $\varepsilon$ of the nonwoven, the lower is the value of $\eta_1$, and hence the higher the value of $|R|$ and the lower the value of $T$. It can also be inferred from Eq. (9) that the higher the value of $\varepsilon'$ and $\varepsilon''/\varepsilon'$, the higher is the value of $\alpha$.

Permittivity of the nonwoven studied is determined by permittivity of the component fibres including PCF, polyester fibre and bicomponent fibre 4080. Variation in contents of different fibres in the nonwoven inevitably induces the variation in $R$, $T$ and $\alpha$ of the nonwoven which is also shown by the curves given in Fig. 5.

It can be inferred from the discussion in section 2.2.3 that the higher the content of a certain fibre in the nonwoven, the greater is the contribution to electromagnetic parameters of the nonwoven. When the content of PCF in the nonwoven is 0%, the permittivity of the nonwoven is determined by polyester fibre, the bicomponent fibre 4080 and air on the basis of their respective volume fraction in the
nonwoven, since air also constitutes a part in the volume of the nonwoven and electromagnetic parameters of air are: \( \varepsilon^\prime=1, \mu^\prime=1, \varepsilon^\prime\prime=0 \) and \( \mu^\prime\prime=0 \). According to Figs 3 and 4, the values of \( \varepsilon^\prime \) and \( \varepsilon^\prime\prime/\varepsilon^\prime \) of polyester fibre and the bicomponent fibre 4080 are quite low. Hence, the values of \(|R|\) and \( \alpha \) of the nonwoven are also low and that of \( T \) is high. Consequently, it is quite easy for the microwave to penetrate the nonwoven without much loss. The microwave penetrating through the nonwoven and being reflected back by the metal plate behind the nonwoven is picked up by the receiving antenna, resulting in the high reflectivity. As the PCF content increases from 0%, the contribution to the permittivity of the nonwoven due to the PCF increases, and hence the values of \( \varepsilon^\prime \) and \( \varepsilon^\prime\prime/\varepsilon^\prime \) of the nonwoven also increase. According to above discussion, two trends are developed in this process. The first one is that the value of \(|R|\) of the nonwoven increases and that of \( T \) declines, which means that the microwave reflected at the incident surface of the nonwoven increases. On the other hand, the value of \( \alpha \) of the nonwoven increases in this process, thereby increasing the microwave absorbing capacity of the nonwoven, and this is the other trend. The two trends are developed when the PCF content is increased in the nonwoven. The former trend is unfavorable for the improvement of the microwave absorption of the nonwoven, while the latter one is good for the microwave absorption. However, at the initial stage as the PCF content increases from 0%, the values of \( \varepsilon^\prime \) and \( \varepsilon^\prime\prime/\varepsilon^\prime \) of the nonwoven are quite low, and hence the value of \(|R|\) is also low and that of \( T \) is high. Thus, most of the incident microwave can penetrate into the nonwoven. The latter trend is dominant in this process since the value of \( \alpha \) is in rapid development with the increase in \( \varepsilon^\prime \) and \( \varepsilon^\prime\prime/\varepsilon^\prime \), making the microwave absorbing capability to sharply increase. So, the general trend at this stage is that the reflectivity of the nonwoven sharply declines with the increase in PCF content.

When the PCF content exceeds 2%, the situation is reversed. Since the PCF content is high enough for the nonwoven to reflect much of the incident microwave at the incident interface, the further increment of \( \alpha \) no longer increases the microwave absorption of the nonwoven. On the other hand, the further increase in PCF content results in the increase in the value of \(|R|\) and the decrease in the value of \( T \). So, the general trend is that the reflectivity increases with the increment of the PCF content. The PCF content of 2% is the optimum for the nonwoven to exhibit the best microwave absorption capability. When the PCF content is above 20%, the value of \(|R|\) is so high (close to 1) that most of the incident microwave is reflected at the surface of the nonwoven and hence the trend that the reflectivity increases with the increment of PCF content slows down; such a trend remains unchanged until the PCF content is 100%.

4 Conclusions

4.1 Microwave absorbing capacity is found to be closely related to the PCF content in the nonwoven.

4.2 When the thickness of the nonwoven is 10 mm, the PCF content of around 2wt% and the resistivity of \( > 8\times10^3 \Omega \cdot \text{cm} \) are the optimum values for the nonwoven to give the maximum microwave absorbing capacity.

4.3 Test results of electromagnetic parameters of the component fibres in the nonwoven disclose the principle why the microwave absorption of the material varies with the change in PCF content. It is the variations in the reflection coefficient and the attenuation constant of the nonwoven induced by the variation of the PCF content that result in the variation in reflectivity.

4.4 The PCF is commercially available at a low price. Manufacturing process of the PCF-containing nonwoven is simple and economical. The PCF-containing nonwoven has excellent microwave absorbing capacity and weatherability, thereby showing the great potential in the military application as the radar camouflage material and in the non-defense application as the electromagnetic shielding material.

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References


