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Synthesis of absorbing coating based on magnetorheological gel with controllable electromagnetic wave absorption properties

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Abstract

Owing to the increasingly complicated electromagnetic environment and changeable battleground, it is especially urgent to study electromagnetic wave absorbing coating with enhanced absorbing capacity and adaptive ability. Magnetorheological gel (MRG) is prepared by mixing magnetic particles with high viscosity polymer matrix. Under magnetic field, these magnetic particles move to form columnar or chains, which allows electromagnetic (EM) waves to penetrate and scatter along the chains. Accordingly, the MRG-based coating that absorbs electromagnetic waves was successfully synthesized. Moreover, flower-like carbonyl iron particles (FCIPs), as the filled magnetic particles, were prepared by in situ method. We studied the effect of magnetic field, particle concentration and matrix curing on electromagnetic (EM) absorbing properties of the MRG-based coating. With the purpose to test absorption performance of the MRG-based coating under different magnetic field intensity, a device that regulates the magnetic field is essential. Thus, we built the device that contains permanent magnet, frame, lifting table. From the results, magnetic field can make absorption peak frequency (APF) move toward high frequency. The amount of shifting to high frequency increases as the magnetic field increases. The magnetic-induced frequency offsets of four samples with different mass fractions (40 wt%, 50 wt%, 60 wt%, 70 wt%) are 2.88 GHz, 1.26 GHz, 1.52 GHz and 0.64 GHz from 0 to 300 mT, respectively. Meanwhile, a suitable magnetic field is beneficial for the coating to absorb more electromagnetic waves. This characteristic of MRG will make it have a wide application prospect in real-time electromagnetic protection and electromagnetic stealth.

Keywords: magnetorheological gels (MRGs), absorbing coating, microwave absorption, magnetic-induced frequency shifting

(Some figures may appear in colour only in the online journal)

1. Introduction

Wireless technology is widely used in military field or civil domain [1, 2], such as radar detection, locking and tracking, communication base station, electric rail transit, medical and scientific research equipment and various household appliances [3–6]. These devices generally operate at gigahertz (GHz) range [7, 8]. There is no doubt that excessive electromagnetic (EM) wave generated from these electronic devices bring electromagnetic radiation that has greatly threatened human health and disturbed various commercial or industrial equipment [9–14]. Worse yet, electronic equipment work in various frequency bands and radar detection technology has also been greatly upgraded, so electromagnetic environment (military and civilian) is becoming more and more complicated [8]. Absorbing materials can absorb unwanted electromagnetic wave energy...
impinged onto the surface and transform it into heat and/or other types of energy [1]. Most of the existing absorbing materials that absorb electromagnetic (EW) waves in a particular frequency band are unable to meet the requirements for multi-band or full coverage. While in some cases, especially in the military application of anti-radar camouflage and stealth, the frequency that needs to be absorbed is varied. This forces us to look for absorbing material with variable microwave-absorption performance.

Generally, magnetorheological (MR) materials contain magnetorheological elastomer (MRE), MR foam, magnetorheological gel (MRG), magnetorheological fluid (MRF) [15–20]. They are a type of smart hybrid material. They are all made of magnetic particles distributed in non-magnetic matrix [21–23]. To our best knowledge, the MR materials possess so excellent magnetic-control properties that MR materials exhibit great applications in the fields of noise reduction, vibration attenuation, smart sensing, etc. The same is true for electromagnetic protection and stealth, etc [24–26]. That’s because carbonyl iron particles (CIPs) in MR materials present high frequency of Snoek’s limit, high saturation magnetization, high relative permeability at radar wave frequency, and high Curie temperature [27–29]. Besides, CIPs possess both magnetic and dielectric properties, which is good for electromagnetic impedance matching [30]. All of these render CIP a promising microwave absorber. Meanwhile, the CIPs in MR materials can response to magnetic field (MF) to form chains that parallel to the direction of magnetic field [15, 31, 32]. The movement of the CIPs will result in the transformation of microstructure of MR materials [33]. Hence, the interaction between particles and matrix changes accordingly, as well as electromagnetic parameters (conductivity, permeability, permittivity and so on). While, the microwave-absorption capability of the MRG-base coating is decided by the complex permeability and permittivity at a given frequency and layer thickness [1, 30, 34]. Therefore, we can use the magnetic field to control the particles to get the desired absorbing performance. Min Dandan et al prepared the oriented FCI/EP composites. The measurement results showed that they obtained the higher permeability and modest permittivity of the composites after orientation in the frequency range of 2–18 GHz. The calculated absorption properties indicated that the orientation plays an important role in decreasing the absorber thickness and broadening the absorption bandwidths [7]. Chen Qianwang et al fabricated a composite with highly aligned array of Fe3O4/CNT coaxial nanofibres in (methyl methacrylate) matrix under low magnetic field. It was found that microwave absorption of the magnetically aligned composite at 8.5–12.5 GHz was evidently enhanced [35]. Yu miao et al studied MRGs’ absorbing properties and discussed the effect of magnetic field intensity and direction angle on the absorbing. Compared to 0 mT, the effective absorption bandwidth (below −20 dB) of 400 mT extended by 40%. The microwave-absorbing peaks moved towards a higher frequency of 10.46%. While, in the process of varying the magnetic field direction angle (0°−90°), the peak frequencies reduced by 13.75%, and the value of the minimum RL decreased by 45.78% (−25.866 dB for the 0° sample; −37.708 dB for the 60° sample) [36].

### Table 1. Composition of the MRG-based absorbing coatings.

<table>
<thead>
<tr>
<th>Grouping</th>
<th>Number</th>
<th>mCO/mMDI</th>
<th>Content of flower-like CIP, wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>The first group</td>
<td>1</td>
<td>10:1</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>10:1</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>10:1</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>10:1</td>
<td>70</td>
</tr>
<tr>
<td>The second group</td>
<td>5</td>
<td>20:1</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>15:1</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>20:3</td>
<td>60</td>
</tr>
</tbody>
</table>

From these works, in order to achieve desired absorbing performance, magnetic field drives the particles to move to change micro-internal structure of composites from isotropy to anisotropy [33]. While, the particles could no longer move when these materials solidified. The absorbing performance could not be further adjusted. Fortunately, like other MR materials, MRG is also made from CIPs and non-magnetic matrix, while its matrix is high viscosity polymer [19, 37, 38]. The CIPs are not easy to settle and matrix do not solidify. So the CIPs can move over and over. Microstructures of MRG can be regulated continuously, rapidly, and reversibly under magnetic field [39]. These inspired us to prepare desired MRG-based absorbing coating.

We did the following two jobs: one is to modify CIP (type EW) by iron nano-flakes to improve electromagnetic loss capacity. The flower-like CIPs prepared have both good electromagnetic wave loss ability and high saturation magnetization. Only in this way can we get a large regulation range of absorbing properties in a small magnetic field. The other is to prepare and test the MRG-based coatings. The absorption of the MRG-based coatings in different magnetic fields (from 0 to 300 mT) is tested by arcuate method. The magnetic field is provided by permanent magnet. After that, the effects of magnetic field, particle concentration and matrix curing on electromagnetic (EM) absorbing property of the MRG-based coatings were analyzed.

### 2. Experimental

#### 2.1. Materials

The raw materials required were listed as follows: diphenylmethane diisocyanate (MDI: 4,4- ≈ 50%, 2,4- ≈ 50%) was offered by Yantai Wanhua Polyurethanes Co. Ltd China, while castor oil (CO) was supplied by and Sinopharm Chemical Reagent Co. Ltd China. All chemical reagents were analytically pure and used as received.

CIP: soft magnetic carbonyl iron particles (CIP: type EW) were provided by BASF Corporation, Germany. The size is d50 = 6.5 μm. Sodium borohydride (NaBH4), Ferrous sulfate heptahydrate (FeSO4·7H2O), Polyvinylpyrrolidone (PVP), hydrochloric acid (HCl) and absolute ethyl alcohol were obtained from Kemiu (Tianjin, China).
2.2. Preparation of EW-type flower-like CIP

For the synthesis of flower-like CIPs, Fe$^{2+}$ is reduced into Fe nuclei with excessive sodium borohydride solution. These Fe nuclei gather into sheet structure on the surface of the raw CIPs. The details can be seen in our previous research [21].

2.3. Preparation of MRG-based absorbing coating

A series of MRG-based coatings were synthesized to discuss the magnetron absorption performance. The preparation process could be described as follows: the first step is to obtain PU matrix. Before the cross-linking reaction of CO and MDI, the water inside CO was evaporated completely in a drying oven. Then, the CO and MDI were dumped into a beaker to mix uniformly. The next step was to add the flower-like CIPs into the above mixture, following by stirring until the mixture appears well-distributed. After that, the mixture last was transferred to the drying oven for about 2 h and the vulcanization temperature was set at 80°C. During the whole curing process, the mixture was taken out every 15 min and was stirred for 2 min with the aim of particles uniform dispersion. Finally, the steady MRG-based coatings came into being after placed several days at room temperature. In this paper, seven samples were prepared through this method, and the specific composition of them were listed in table 1. The fabrication process is shown schematically in figure 1.

2.4. Establishment of magnetron absorbing coating testing device

Because MRG is viscoplastic fluid, arcuate method is selected instead of $T/R$ to measure the absorbing performance. With the
purpose to test absorption performance of the MRG-based coatings under different magnetic field intensity, a device that regulates the magnetic field is essential. Thus, based on arcuate test system, the device (figure 2) that contains permanent magnet, frame, lifting table was built. The device is supposed to provide variable magnetic field for these MRG-based coatings. The size of the permanent magnet is $210 \times 210 \times 30\,\text{mm}$. The lifting table can adjust the distance between the magnet and sample to vary the magnetic field intensity. To the best of our knowledge, calibrating the magnetic field should be done before testing. An acrylic cavity frame, whose cavity size is $210 \times 210 \times 2\,\text{mm}$, is placed on the top of the frame, and the cavity is unloaded with MRG absorbing coating during calibration. Then the PF-035 type digital tesla meter serves to measure the magnetic field at the bottom of the cavity. Record the position of the upper surface of the lifting table when the magnetic field at the bottom of the cavity frame is 0, 50, 100, 150, 200 and 300 mT.

2.5. Characterization

The morphology and size distribution of flower-like CIPs were captured under a JEOL JSM-7800F field-emission scanning electron microscope (FE-SEM) at an acceleration voltage of 30.0 kV. The magnetic performance ($M-H$ curve) of the flower-like CIPs and MRG-based coatings were characterized at room temperature using a vibrating sample magnetometer (VSM Lake Shore 7407). The viscosity of the MRG-based coatings was characterized by an advanced commercial rheometer (Modal: MCR301, Anton Paar). The rheometer works in the mode of rotational shear, and the test temperature is at $25\,^\circ\text{C}$. The test gap is 1 mm. The shear rate is $10\,\text{s}^{-1}$. Scanning range of magnetic field is 0–1 T. The scanning electron microscope (SEM) images of MRG were recorded after gold sputtering treated. However, it could not exert magnetic field on SEM in the detection process, that is, it is impossible to observe the particle distribution of MRG-based absorbing coating under magnetic field. For this reason, put an appropriate amount of MRG-based absorbing coating between two glass slides. The distribution of particles in the MRG absorbing coating before and after the application of the magnetic field were further observed by the optical digital microscope (KEYENCE, VHX-600E). The absorption characteristics of these MRG-based coatings in the range of 2–18 GHz were measured using bow method with a vector network analyzer. Using the reflection loss $RL$ ($RL = 10\log\Gamma_p \,\text{dB}$) to express the performance of absorbing electromagnetic waves. Here, $\Gamma_p$ is power reflectivity of absorbing materials.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{image}
\caption{(a) The microstructure image in SEM without magnetic field; (b) The photo of optical digital microscope without magnetic field; (c), (d) Typical microstructure of absorbing coatings under a magnetic field: (c) The original image; (d) The image after image segmentation.}
\end{figure}
3. Results and discussion

3.1. Characterization of flower-like CIP

From figure 3, there are plenty of nano flakes evenly surrounded on the surface of CIP. Iron nano flakes greatly change its surface into globular flowers. The flower-like CIP (type EW) is a kind of soft magnetic particle with narrow hysteresis loop, small enclosing area, high permeability, easy magnetization and demagnetization. The value of \( M_r \) reaches 193.41 emu g\(^{-1}\) which is nearly 25 emu g\(^{-1}\) higher than that of flower-like CIP (type CN). Relatively high magnetization produces greater force between particles and particles, adjacent chains will merge to form a longer and thicker columnar structure, resulting in highly shape anisotropy of microstructure of the MRG-based coating [40]. This is favor to get a large regulation range of absorbing property.

3.2. Characterization of MRG coatings

Figure 4(a) presents SEM image of MRG-based coating without magnetic field. It is plain to see the particles in the polyurethane matrix are relatively evenly distributed with little aggregation. With the aid of optical digital microscope, the distribution of particles in the absence of magnetic field is also shown in figure 4(b). The highlight is absorber particles, while yellow background is polyurethane matrix. The distribution of particles in optical digital microscope is similar to SEM image. Once a uniform magnetic field is applied across the slide on the optical digital microscope, the micro-morphology of the MRG-based absorbing coating is shown in figure 4(c). At the same time, image analysis technology is to distinguish the absorber particles from the polyurethane matrix (figure 4(d)). White is the absorber particle, and black represents polyurethane substrate. From these two pictures, the absorbing particles redistribute from uniformity into chains or columnar structure under a magnetic field. Compared with the distribution in the zero field, the microstructure changes from isotropic distribution to anisotropic chains. The ‘phase transition’ phenomenon that is called magnetron characteristic appears. The dramatic change of MRG-based coating under magnetic field is the basis for achieving real-time and reversible magnetically controlled electromagnetic wave absorption.

3.3. Microwave absorption properties

3.3.1. Magnetron absorbing properties of MRG-based coating with flower-like CIPs of 40 wt%, 50 wt%, 60 wt% and 70 wt%.

The magnetized flower-like CIPs can be simplified as magnetic dipoles whose magnetic dipole moments are consistent with the applied magnetic field [41, 42]. There

![Figure 5. Reflection losses (RL) curves of MRG absorbing coating with different flower-like CIPs (the quality of CO and MDI is 10:1): (a) 40 wt%; (b) 50 wt%; (c) 60 wt%; (d) 70 wt%.](image-url)
are interaction force between the magnetic dipoles [24, 43]. The flower-like CIPs can be driven by interaction force to form chains or columnar structure, resulting in shape anisotropy of microstructure of the MRG-based coating [33, 40]. The appearance of these anisotropic chains indicates that the particles have been rearranged. The interaction between the particles and the particles or matrix will change, which undoubtedly changes the physical properties (conductivity, complex permittivity, complex permeability) of the MRG-based coating itself. Generally, microwave absorption properties were mainly associated with impedance matching and loss factor [27, 44, 45]. The impedance matching ensures that electromagnetic waves can enter absorbing materials, while loss factor $\alpha$ represents loss capability. The equations are as follows [27, 46]:

$$R = \frac{Z_{\text{in}} - Z_0}{Z_{\text{in}} + Z_0}$$  \hspace{1cm} (1)

$$Z_{\text{in}} = \sqrt{\frac{\mu_0 H_x}{\varepsilon_0 \varepsilon_r}}$$  \hspace{1cm} (2)

$$Z_0 = \sqrt{\frac{\mu_0}{\varepsilon_0}}$$  \hspace{1cm} (3)

$$\alpha = \frac{\sqrt{2} \pi f}{c} \sqrt{(\mu'' \varepsilon'' - \mu' \varepsilon') + \sqrt{(\mu'' \varepsilon'' - \mu' \varepsilon')^2 + (\mu' \varepsilon'' + \mu'' \varepsilon')^2}}$$  \hspace{1cm} (4)

where $Z_0$ is the intrinsic impedance of free space, $Z_{\text{in}}$ is the input impedance of the absorber, $\varepsilon_r$ ($\varepsilon_r = \varepsilon' - j\varepsilon''$) is the complex permittivity of MRG-based coating; $\varepsilon_0$ is the complex permittivity of free space. $\mu_r$ ($\mu_r = \mu' - j\mu''$) is the complex permeability of MRG-based coating; $\mu_0$ is the complex permeability of free space.

Essentially, the impedance matching and loss factor depend on complex permittivity and permeability. This will ultimately affect the reflection loss RL. These test results confirm that the magnetic field has a great influence on the microwave absorption performance (figures 5, 6). From the minimum RL value, the samples 1, 2 and 3 have the similar trend, with the increase of the magnetic field, the minimum RL values decrease first and then rise. According to Debye theory, the imaginary permittivity can be defined as follows [7, 47]:

$$\varepsilon'' = (\varepsilon_s - \varepsilon_\infty)/(1 + \omega^2 \tau^2) + \sigma/\omega\varepsilon_0,$$  \hspace{1cm} (5)

where $\varepsilon_s$ is the static permittivity; $\varepsilon_\infty$ is the relative dielectric permittivity at the high frequency limit; $\omega$ is the angular frequency; $\tau$ is the polarization relaxation time; and $\sigma$ is the electrical conductivity. In the MRG-based coating, electrical conductivity $\sigma$ is a highly significant factor that influences the

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Figure 6. MRG absorbing coating with different flower-like CIPs: (a) Optimal reflection loss RL and (b) absorption peak frequency vary with the magnetic field; (c) magnetic hysteresis loop; (d) viscosity varies with the magnetic field (the quality of CO and MDI is 10:1).
imaginary part of permittivity $\varepsilon''$. When the magnetic field is applied, isolated particles gather into chains. Furthermore, with the increase of magnetic field, there will build up much more chains and the growing interaction between particles and small chains make the main chain grow thicker. It is reasonable for chains formed by CIPs to have more opportunities to establish conductive paths at the same mass fraction, thus achieving enhanced electrical conductivity compared to the evenly distributed composites $[40]$. Therefore, the improved electrical conductivity mainly results in the enhancement of the imaginary permittivity of the MRG-based coating. A high $\varepsilon''$ value brings high loss factor, which is in favor of dissipation of electromagnetic waves. As a result, the value of reflection loss RL decreases.

It was proved that the magnetic moments lie preferentially in the easy planes; the anisotropy chains formed by the flower-like CIPs is ascribed to easy magnetization and result in the enhancement of permeability $[7, 48, 49]$. In summary, the appropriate magnetic field makes complex permittivity and permeability increase. It is helpful to improve the impedance matching and loss ability of absorbing materials, enhance microwave absorbing intensity. CIPs are magnetic loss materials, whose complex permeability is higher than the complex permittivity. Nevertheless, excessive magnetic fields makes impedance matching worse, which is not conducive to absorbing electromagnetic waves. As for sample 4, due to high particles’ content, the viscosity is much higher than the other samples. The particles are difficult to move. The number and size of chains are inferior to those of other samples when the magnetic field is same. In other words, sample 4 need a larger magnetic field to form conductive channels. At 0–300 mT, the complex permittivity and permeability increases slowly. It is likely to avoid the impedance matching deterioration. Thus, the RL of sample 4 keeps decrease at 0–300 mT.

The absorption peak frequency (APF) shifts according to the actual situation to absorb the target frequency bands or frequency points. It is an important aspect of intelligent controllability. Magnetic field can make APF move toward high frequency. The amount of shifting to high frequency increases as the magnetic field increases. The microwave absorption induced by natural ferromagnetic resonance of magnetically anisotropic composite is strongly affected by the applied external magnetic field and the internal anisotropy $H_a$. During the chaining process, the magnetic easy axes of the chains aligned mainly parallel to the surface of the MRG-based coating, which increases the internal anisotropy field evidently $[35]$. So the natural resonance frequency $f_r$ ($f_r = \gamma H_a/2\pi$) moves to high frequency. Moreover, the magnetic-induced frequency offsets of the four samples

Figure 7. Reflection losses (RL) curves of MRG absorbing coating with different matrix solidification degree (the content of flower CIP is 60 wt%): (a) 20:1; (b) 15:1; (c) 10:1; (d) 20:3.

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were 2.88 GHz (40 wt%: 10.32–13.2 GHz), 1.26 GHz (50 wt%: 10.1–11.36 GHz), 1.52 GHz (60 wt%: 8.24–9.76 GHz), and 0.64 GHz (70 wt%: 7.76–8.4 GHz), respectively. The general trend is that the offset of APF decreases with the particle content. This is mainly due to the different viscosity and magnetization caused by flower CIP content. The moving velocity of CIPs in a magnetic field is as follows [50]:

\[ v = \frac{\mu_0 M_s a^2 \phi}{18 \eta} \]

where \( a \) denotes the radius of the microparticles. The gradient of the magnetic field and the volume fraction of the microparticles are denoted by \( \delta \) and \( \varphi \), respectively. \( \eta \) denotes the viscosity of the sample. The movement speed of the particles affects the formation of the chains. Under the low content, the viscosity and saturation magnetization are low (figures 6(c), (d)). The particles gradually form a chain structure under the magnetic field that always cause the movement of APF. Nevertheless, at the high content, viscosity is relatively high, which will impede the movement of particles. Thus, the APF does not change much. The viscosity of sample 4 is much higher than that of the other three samples. Therefore, the magnetic-induced offset frequency of sample 4 is the smallest.

### 3.3.2. Magnetron absorbing properties of MRG-based coating with curing degree

It is known from the above analysis that the viscosity has a great influence on the absorption properties, and the curing of the polyurethane matrix is closely connected the viscosity. Therefore, the MRG absorbing coatings with different matrix curing are prepared by adjusting the mass ratio of CO and MDI. From figures 7, 8, the minimum RL value varies with the magnetic field. Like the first group of samples, the peak moves to high frequency along with magnetic field. It can be seen from figures 7 and 8(a), the minimum RL value is not very regular. Theoretically, if the flower-like CIP content is the same, the difference of minimum RL value is not large. However, the minimum RL value of sample 6 is obviously lower than that of other samples. The reason is that there is a thickness error of 0.2 mm in the acrylic cavity used to hold the MRG coating, so that the minimum RL value is deviated. Fortunately, the change trend of these samples with magnetic field has little relationship with the thickness of the coating.

The APF of all the samples shifts faster before 100 mT and changes slowly afterwards. Magnetic particles can respond quickly to magnetic fields. Once the magnetic field applied, the particles are arranged along the magnetic field. The microstructure of magnetorheological coating has a great change from homogeneous distribution to anisotropic structure. This brings great frequency shift. Continue to increase magnetic field, the movement of chain particles has been restricted, and the microstructure changes are not significant. The frequency changes are relatively gentle. The frequency offsets are 1.36 GHz, 1.52 GHz, 1.52 GHz and 1.4 GHz respectively for the four samples whose mass ratios of CO and MDI are 20:1, 15:1, 10:1 and 20:3. The magnitude of
magnetic-induced frequency offset is similar, which indicates that when the content of CIP is the same, the saturated magnetic field is the same, simply changing the curing degree of the matrix has little effect on the frequency offsets. But in practical application, the change of curing will affect the density, adhesion and packaging of the coating, so it is necessary to select the best one according to the need.

4. Conclusion

In order to achieve real-time and reversible control of absorbing performance, the intelligent EW absorption of MRG coating has been explored preliminarily in this paper. Microwave absorption of magnetically anisotropic composite are strongly affected by the applied external magnetic field. The appropriate magnetic field is helpful to improve the impedance matching and loss ability of MRG-based coating, enhance microwave absorbing intensity. What’s more, during the reorientation process, the particles aligned mainly parallel to the surface of the sample, which increases the internal anisotropy field evidently. So the natural resonance frequency \( f_r \) (\( f_r = \frac{\nu H_s}{2\pi} \)) move toward high frequency. This suggests that MRGs are magnetic-field-sensitive electromagnetic wave-absorbing material and they can adjust the microwave-absorption peak as needed. At the same time, suitable particle concentration and matrix curing can bring large magnetic-induced frequency range. Consequently, this paper offers a promising strategy to design novel intelligent absorbing coating based on MRG for electromagnetic protection and antiradar camouflage. What’s more, MRG absorbing coating reported here expected to be useful in future investigations on the controllability of MRG electromagnetic properties.

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