Research Article

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Lightweight and hydrophobic Ni/GO/PVA composite aerogels for ultrahigh performance electromagnetic interference shielding

https://doi.org/10.1515/ntrev-2022-0088
received December 1, 2021; accepted February 15, 2022

Abstract: Lightweight and high-performance electromagnetic interference (EMI) shielding materials are urgently required to solve increasingly serious radiation pollution. However, traditional lightweight EMI shielding materials usually show low EMI shielding performance, poor mechanical properties, and environmental stability, which greatly limit their practical applications. Herein Ni foam/graphene oxide/polyvinyl alcohol (Ni/GO/PVA) composite aerogels were successfully prepared by a freeze-drying method. The Ni/GO/PVA composite aerogels possessed low density (189 mg cm$^{-3}$) and high compression strength (172.2 kPa) and modulus (5.5 MPa). The Ni/GO/PVA composite aerogel was hydrophobic, and their contact angle can reach 145.2°. The hydrophobic modification improved the environmental stability of the composite aerogels. Moreover, the Ni/GO/PVA composite aerogels exhibited excellent EMI shielding performance. Their maximum EMI shielding effectiveness (SE) can reach 87 dB at the thickness of 2.0 mm. When the thickness is only 1.0 mm, the EMI SE can still reach 60 dB. The electromagnetic energy absorption and attenuation mechanisms of Ni/GO/PVA composite aerogels include multiple reflection and scattering, dielectric loss, and magnetic loss. This work provides a promising approach for the design and preparation of the lightweight EMI shielding materials with superior EMI SE, which may be applied in various fields such as aircrafts, spacecrafts, drones, and robotics.

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Keywords: electromagnetic interference shielding, aerogel, graphene oxide, Ni foam

1 Introduction

With the rapid development of communication technology, electronic devices have been widely used in our daily life [1–4]. When these devices transmit or receive electrical signals, electromagnetic waves are inevitably generated [5]. Electromagnetic radiation not only affects the reliability and lifetime of precision equipment, but also threatens human health [6–8]. Undoubtedly, the development of high-performance electromagnetic interference (EMI) shielding materials is one of the effective strategies to solve the above problems [9,10]. EMI shielding materials are very important in equipment with special-needs, which are widely used in communications, electronics, aerospace, military, navigation, medical, and many other fields. Traditional EMI shielding materials such as metals, have excellent EMI shielding properties, but the intrinsic high density and poor corrosion resistance limit their practical applications [11–13].
Recently, carbon-based materials such as carbon nanotubes (CNTs), carbon nanofibers, graphene, or their hybrids, have become popular alternatives for EMI shielding applications because of their lightweight, chemical stability, flexibility, and good mechanical properties \[1,14–23\]. For example, Wei et al. \[24\] synthesized a freestanding highly aligned laminated graphene film by a scalable scanning centrifugal casting method, which showed an EMI shielding effectiveness (SE) of 93 dB at a thickness of ≈100 µm. Jia et al. \[25\] fabricated a freestanding and ultrathin graphene oxide/silver nanowire (GO/Ag) film by a vacuum-assisted self-assembly method, which had an EMI SE of 62 dB at a thickness of merely 8 µm. Rani et al. \[26\] prepared a polyvinyl alcohol/chitosan/graphite oxide/nickel oxide (PVA/CS/GO/NiO) nanocomposite film using solution casting technique, and the EMI SE value was 12 dB for nanocomposite film containing 3.0/30 wt% of GO/NiO. Although some achievements have been realized in carbon-based EMI shielding materials, the densities of the reported materials are still not low enough for EMI shielding materials in aerospace industry. Exploring the simple and efficient methods to prepare lightweight EMI shielding materials with superior EMI SE value is still a challenge.

Porous materials such as aerogels, foams, and sponges are ideal candidates for lightweight EMI shielding materials due to their unique 3D skeleton structure and ultralow density \[27–29\]. More importantly, the porous structures are conducive to multiple internal reflection and absorption of electromagnetic waves \[30\]. Li et al. \[31\] fabricated a graphene aerogel with multilayer structure via mechanical compression, which showed high electrical conductivity (181.8 S m\(^{-1}\)) and EMI shielding performance (43 dB at thickness of 2.5 mm). Yu et al. \[32\] fabricated an anisotropic polyimide/graphene composite aerogel by unidirectional freezing and freezing drying, which exhibited EMI SE of 26–28 dB at thickness of 2.5 mm when the graphene content was 13 wt%. Yang et al. \[33\] prepared a 3D copper nanowires-thermally annealed graphene aerogel (CuNWS-TAGA) framework by freeze-drying followed by thermal annealing, which showed the maximum EMI SE value of 47 dB at thickness of 3.0 mm, ascribed to perfect 3D CuNWS-TAGA conductive network structures. Wang et al. \[34\] constructed a CNT/graphene/polyimide foam with stable compressibility and perfect conductive networks. The resultant composite foam exhibited an average EMI SE of 28 dB at thickness of 2.0 mm. However, the EMI shielding performance of these porous materials is relatively poor, especially at small thickness (≤2.0 mm), which cannot meet the increasingly demanding application requirements. It remains a challenge to maximize the EMI shielding performance at low density and small thickness through the rational design of the composition and structure.

From graphical abstract, we successfully designed and fabricated Ni/GO/PVA aerogels by freeze-drying process, which possess lightweight, hydrophobicity, high compression strength, and superior EMI shielding performance. The GO in aerogel was dispersed in PVA, which greatly increased the heterogeneous contact area and enhanced the interfacial polarization effect. The porous structure makes the electromagnetic wave be reflected and scattered many times, which increases the propagation distance of the electromagnetic wave. Combined with Ni foam with high conductivity and permeability, reflecting the large number of incoming electromagnetic

\[Figure 1: \text{Preparation process of Ni/GO/PVA aerogels.}\]
waves, the synergistic effect of dielectric and magnetic loss improves the EMI SE of the materials. In addition, the hydrogen bonding between GO and PVA improves the mechanical properties of the material. The surface energy of the material after hydrophobic treatment is low, and the droplets can roll on the surface, which realizes the self-cleaning function and improves the corrosion resistance. This design and fabrication strategy provides high-potential for the practical application of EMI shielding materials.

2 Materials and specimens

2.1 Materials

GO was purchased from Tangshan JianHua Technology Development Co., Ltd. PVA (MW: ~195,000) was purchased from Beijing Yili Fine Chemicals Co., Ltd. Ni foam (density: 8 mg cm\(^{-3}\)) was obtained from Kunshan Guangsheng jia-neng Materials Co., Ltd. \(1 \text{H},1\text{H},2\text{H},2\text{H}\)-Perfluorodecyltriethoxysilane (FAS-17, >96% purity) was purchased from Shanghai Macklin Biochemical Co., Ltd.

2.2 Preparation of Ni/GO/PVA composite aerogels

Figure 1 shows the preparation process of Ni/GO/PVA aerogels. Four Ni/GO/PVA aerogels with GO content were prepared, and the mass of GO accounted for 5, 10, 15, and 20% of GO and PVA mass, and were named Ni/GO/PVA-5, Ni/GO/PVA-10, Ni/GO/PVA-15, and Ni/GO/PVA-20, respectively. First, taking Ni/GO/PVA-15 as an example, 0.18 g GO was added into 50 mL of deionized water, after ultrasonic stirring, 1.00 g PVA was added into GO dispersion, and stirred in 80°C water bath for 1 h, PVA is completely dissolved and GO/PVA dispersion is obtained, which is left room temperature for cooling. Then, an appropriate size of Ni foam was into the mold; the GO/PVA dispersion was slowly poured into the mold until the Ni foam is not on the surface. It was put into a vacuum oven to remove bubbles. Then, the mold was placed in liquid nitrogen environment, and the GO/PVA dispersion was completely frozen. The mold was put into the freeze dryer and aerated at −50°C for 48 h to get Ni/GO/PVA aerogel. Without adding GO, Ni/PVA aerogels were prepared by the same method. 2.50 g FAS-17 was dispersed in 50 mL of ethanol, and the aerogels were extracted for 10 min and removed at 135°C for 30 min. The hydrophobic modified Ni/GO/PVA aerogels were obtained.

2.3 Characterization

The morphology and microstructure were observed with a scanning electron microscope (SEM) (HITACHI SU8010). The mechanical properties of the aerogels were tested using an electronic universal testing machine (SHIMADZU AGS-X) equipped with a 2-kN load cell at a loading rate of 5 mm min\(^{-1}\) in compression mode. The aerogels were cut into 10 mm \(\times\) 10 mm \(\times\) 10 mm, and each sample was tested 5 times under the same condition. The contact angles of the aerogels were measured with a contact angle goniometer (Dataphysics OCA 25) using deionized water droplets of 5 µL, at 5 different positions on the aerogel. The density of the aerogels was calculated from measured geometries and mass of the samples. The EMI shielding properties of the Ni/GO/PVA-5, Ni/GO/PVA-10, Ni/GO/PVA-15, and Ni/GO/PVA-20 aerogels were measured in a frequency range of 8.2–12.4 GHz (X-band) at room temperature by a

Figure 2: (a) Photograph of Ni/GO/PVA-20 aerogel; (b) histogram of aerogel density.
vector network analyzer (Ceyear 3672b) using the waveguide method. The samples were cut into 22.86 mm × 10.16 mm × 2.0 mm (length × width × thickness) sizes to satisfy the waveguide holders. The power coefficients of reflection (R), transmission (T), and absorption (A) were calculated from the measured scattering parameters ($S_{11}$ and $S_{21}$). The total EMI SE ($SE_T$) can be evaluated based on the following equations [35,36]:

$$R = |S_{11}|^2,$$
$$T = |S_{21}|^2,$$
$$SE_R = -\log_{10}(1 - R),$$
$$SE_A = -\log_{10}\left(\frac{T}{1 - R}\right),$$
$$SE_T = SE_R + SE_A.$$

### 3 Results and discussion

#### 3.1 Morphology and structure of Ni/GO/PVA composite aerogels

Figure 2(a) shows the porous structure of Ni/GO/PVA composite aerogels. Ni/GO/PVA aerogels are placed on bamboo leaves without bending the leaves, indicating that Ni/GO/PVA aerogels have very low density. Figure 2(b) compares the density of Ni/GO/PVA aerogels with different GO contents. It can be seen that the density of aerogels increases with the increase in GO content, and the density of Ni/GO/PVA-20 is 189 mg cm$^{-3}$.

Figure 3(a and b) shows the SEM images of PVA aerogel at different magnification rates. From Figure 3(a), we can see that PVA is interconnected from each other. After freezing and drying, a lot of pore structures are formed from PVA thin-walled honeycomb like structure. The porous structure increases the specific surface area of the aerogels. In addition, the local direction of the pore structure is consistent, which reflects the growth direction of ice crystal during freezing. Figure 3(b) is an enlarged PVA thin wall. More pore structures can be observed in PVA thin wall, which further expands the specific surface area of the aerogels. Figure 3(c and d) shows the SEM images of GO/PVA aerogel (20% mass fraction of GO) at different magnification rates. Figure 3(c) shows that GO is homogeneously embedded in PVA, and the discontinuity of GO causes the aerogels not to have a complete conductive network. Therefore, the conductivity of the aerogels should be further modified. Figure 3(d) is a magnification map of the GO slice. Observed at high magnification, the GO lamella is composed of only 1–2 layers, indicating that GO can be dispersed uniformly without agglomeration in PVA, which greatly enlarges the specific surface area of GO, and is more conducive to electromagnetic wave contact with GO and multiple reflections inside the aerogel.

![Figure 3](image-url)

**Figure 3:** SEM photographs (a and b) PVA aerogels; (c and d) GO/PVA aerogels.
Figure 4: SEM photographs: (a and b) Ni foam; (c and d) Ni/PVA aerogels; (e and f) Ni/GO/PVA-20 aerogels.

Figure 5: Compression properties of Ni foam, Ni/PVA, and Ni/GO/PVA-20 (a) compression stress–strain curves; (b) modulus and maximum stress.
Figure 4(a and b) shows the SEM photographs of Ni foam with different magnifications. The high conductivity and continuous skeleton structure of Ni foam ensure excellent conductivity of the composite aerogels. In addition, magnetic Ni foam can give good magnetic loss to the composite aerogels, and further reduce the electromagnetic energy [37,38]. A large number of pores between the Ni foam frameworks facilitate the smooth flow of GO/PVA solution into the interior, while the high porosity of the Ni foam greatly reduces the density of the material. Figure 4(c and d) shows the SEM photographs of the Ni/PVA aerogels. Porous structure of PVA fills the pores of Ni foam and improves the compressive properties of the material. PVA adhered well to the nickel skeleton (Figure 4(d)), indicating that the combination of PVA and Ni foam is good, which ensures the stability of the material. Figure 4(e and f) are the SEM photographs of the Ni/GO/PVA aerogels. GO/PVA is evenly and tightly packed in the pores of Ni foam, providing support force for Ni skeleton, thereby enhancing the strength of the material. From Figure 4(f), it can be seen that PVA can be tightly coated on the surface of GO and Ni foam, showing a good combination.

3.2 Mechanical properties of Ni/GO/PVA composite aerogels

Figure 5(a) shows the compression properties of the Ni foam, Ni/PVA, and Ni/GO/PVA-20. The compressive stress–strain curves of the Ni foam can be divided into three stages: linear deformation, collapse, and densification stage. When the strain is in the range of 0–5%, the stress increases linearly with the strain, and the deformation is mainly elastic deformation. As the load increases, the weak area inside the Ni foam begins to collapse and cracks occur, and the deformation produced in this form is unrecoverable. When strain exceeds 70%, the deformation is too large. Most pores in the Ni foam are completely compacted, and the material enters the densification stage. The stress increases sharply with the increase in strain. Ni/PVA and Ni/GO/PVA-20 also have three deformation stages, and compared with Ni foam, the slope of the linear deformation stage is larger, and the stress at the end of linear deformation is greater. In addition, because of the presence of PVA and GO in the pores, Ni/PVA and
Ni/GO/PVA-20 begin to densify at a smaller strain. As shown in Figure 5(b), the relationship for the modulus and the stress at the end of the linear deformation stage is: Ni/GO/PVA-20 > Ni/PVA > Ni foam. PVA is filled with Ni foam pores, which provides a certain support force for the compression. The GO of Ni/GO/PVA-20 is distributed in PVA, and the hydrogen bond between GO and PVA ensures higher strength and modulus.

3.3 Hydrophobicity of Ni/GO/PVA composite aerogels

A large number of porous structures exist in Ni/GO/PVA-20 aerogels, and a large number of hydroxyl groups on PVA make the materials hydrophilic. As shown in Figure 6(a), the droplet drops on the surface of the Ni/GO/PVA-20 aerogel without hydrophobic treatment, and the droplet is absorbed by the material after 5 s. Hydrophobic Ni/GO/PVA-20 aerogels exhibit strong hydrophobicity. Figure 6(b) shows that when droplets of different colors are dropped on the surface of the material, the beads can remain on the surface of the material with a complete spherical shape and are not absorbed by the material. The average contact angle of Ni/GO/PVA-20 aerogels is 145.2° (Figure 6(c)). Then, the droplet drag experiment is carried out on the material, as shown in Figure 6(d). First, the material is moved upward to contact the droplet, and then slowly moved down the material. It is found that the droplet is dragged and deformed between the needle tube and the material. Due to the excellent hydrophobicity of the material, the droplet is not dragged to the surface of the material, but remained on the needle tube. The rolling experiment of water drops is shown in Figure 6(e) and the material is tilted to 17°. Drops of water from above can roll down smoothly and quickly. The droplet drag experiment and rolling experiment show that the material has excellent hydrophobicity. The hydrophobicity of Ni/GO/PVA-20 aerogels is attributed to
two points. First, the surface energy of FAS-17 coating on the aerogel is low, and the affinity between the material and water is reduced. Second, the micro porous structure of aerogels formed micron roughness on the surface, reducing the liquid-solid contact area, thus increasing the hydrophobicity of the materials. Ni/GO/PVA-20 aerogels with hydrophobic treatment have special applications for waterproofing and corrosion resistance. Meanwhile, water droplets can roll on the surface of the material, remove the adsorbed dust and pollutants, and achieve self-cleaning function.

3.4 EMI shielding performance of Ni/GO/PVA composite aerogels

Figure 7(a) shows the EMI shielding performance of Ni/GO/PVA aerogels with different GO contents. The $\text{SE}_T$ of Ni/GO/PVA-5 decreases with the increase in the frequency, for 8.2 GHz, the $\text{SE}_T$ is 39 dB, while for 12.4 GHz, the $\text{SE}_T$ is 26 dB, and the entire X band can shield 99% of the electromagnetic waves. The EMI shielding performance of Ni/GO/PVA aerogels can be obviously improved by increasing the GO content, and the EMI shielding performance of Ni/GO/PVA-20 is 87 dB, corresponding to an ultrahigh EMI shielding efficiency of $\sim 99.9999998\%$. The EMI shielding performance consists of the reflection of electromagnetic wave from the interface between the sample and air and the absorption of electromagnetic wave by the sample. Figure 7(b) shows the average values of $\text{SE}_T$, $\text{SE}_R$, $\text{SE}_A$ of Ni/GO/PVA in X-band. The $\text{SE}_R$ of Ni/GO/PVA-5 is greater than that of $\text{SE}_A$, indicating that the EMI shielding of Ni/GO/PVA is mainly reflected at low GO content. With the increase in GO content, the $\text{SE}_R$ of Ni/GO/PVA-10, Ni/GO/PVA-15, and Ni/GO/PVA-20 is less than that of $\text{SE}_A$, which shows that the EMI shielding performance of Ni/GO/PVA aerogels with high GO content is mainly the absorption. With the increase in graphene content, Ni/GO/PVA has no obvious effect on the reflection ability of the electromagnetic wave, but the absorption ability is obviously improved. The reflectivity depends on the impedance matching at the interface between the sample and free space. The impedance match is related to the electromagnetic parameters of the sample. The Ni foam in Ni/GO/PVA forms a complete three-dimensional conductive network. The change in GO has no obvious effect on the conductivity. Therefore, $\text{SE}_R$ is not affected by the change in the GO content. Electromagnetic waves produce reflection in the pores of Ni surface and GO surface. GO distributes unevenly in the pores of Ni foam. Multiple reflections greatly increase the propagation distance of electromagnetic waves. In addition, GO increases a large number of heterogeneous interfaces, and the ability of interface polarization loss increases. The increase in GO content enhances the ability of the electromagnetic wave attenuation, thus improving the EMI shielding ability of Ni/GO/PVA.

Figure 7(c) shows the EMI shielding performance of Ni/GO/PVA-20 aerogels with different thicknesses. The thickness of the aerogels increases and the EMI shielding performance is enhanced. From 1.0 mm to 2.0 mm, the EMI shielding performance is improved from 60 to 87 dB. Figure 7(d) shows the average values of $\text{SE}_T$, $\text{SE}_R$, and $\text{SE}_A$ in X-band for different thicknesses of Ni/GO/PVA-20. The reflection of electromagnetic waves of the samples varies little with thickness. The enhancement of EMI shielding performance of thick samples is due to the enhanced absorption of electromagnetic waves. With the increase in thickness, the propagation time of electromagnetic waves in the samples is longer, the number of GO sheets is more, and the reflection time of electromagnetic waves is increased. The propagation distance of the electromagnetic waves in the sample is larger, the electromagnetic waves are more likely to be attenuated, and the $\text{SE}_A$ increases. In addition, the thickness of the sample increased, the thickness of Ni foam increased, the electromagnetic wave propagation distance increased, and the magnetic loss capacity increased. Table 1 compares the EMI shielding properties of related materials in recent years. Ni/GO/PVA-20 aerogels exhibit excellent EMI shielding properties at small thickness.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Thickness (mm)</th>
<th>EMI SE (dB)</th>
<th>Refs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon nanotube/graphene/polyimide foam</td>
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<td>28</td>
<td>[35]</td>
</tr>
<tr>
<td>Cu nanowires/graphene aerogel</td>
<td>3.0</td>
<td>47</td>
<td>[34]</td>
</tr>
<tr>
<td>rGO aerogel</td>
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<td>40</td>
<td>[40]</td>
</tr>
<tr>
<td>Graphene aerogel</td>
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<td>43</td>
<td>[32]</td>
</tr>
<tr>
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<td>[29]</td>
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<tr>
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</table>
3.5 Analysis of EMI shielding mechanism

The EMI shielding mechanism of Ni/GO/PVA aerogels is divided into two parts: reflection and absorption of electromagnetic waves. As shown in Figure 8, when the electromagnetic waves reach the surface of the sample, the high conductivity of the three-dimensional conductive network of Ni foam makes the interface between air and sample impedance mismatch, and a part of the electromagnetic waves are reflected [40]. There are a lot of pores in the Ni foam. GO flakes and PVA distribute irregularly in the pores, on the one hand, preventing the electromagnetic wave spilling from the pores. On the other hand, the structure inside the sample causes the electromagnetic waves to reflect and scatter on the surface of the Ni hole wall and GO surface, increasing the electromagnetic wave propagation path, and the electromagnetic waves have a greater probability to be attenuated [41,42]. The absorption mechanism of the electromagnetic wave is divided into dielectric loss and magnetic loss. First, the high conductivity of the continuous conductive network of Ni foam conduces to fast electron transfer, and aerogel has strong conductance loss. Second, there are GO and PVA in the pores of the Ni foam, which make the samples have a large number of heterogeneous interfaces (Ni-GO, Ni-PVA, and GO-PVA). Under the action of electromagnetic waves, free charges gather at the interface, causing strong interfacial polarization loss. Finally, strong magnetic nickel can cause strong magnetic loss, and further dissipate electromagnetic energy through natural resonance, exchange resonance, and eddy current [37]. In general, the synergistic effect of the dielectric loss and magnetic loss and the porous structure of the aerogels make the material possess excellent EMI shielding performance.

4 Conclusion

Ni/GO/PVA composite aerogels with disordered GO, porous structure, and lightweight were successfully prepared by freeze-drying process. The results show that the compression process can be divided into linear deformation, collapse, and densification stage. The hydrogen bonding between GO and PVA and the binding force between PVA and Ni foam increase the modulus of the Ni/GO/PVA linear deformation stage. Ni/GO/PVA aerogels have waterproof properties, while water droplets can roll on the surface of the material, remove the adsorbed dust and pollutants, and achieve self-cleaning function. Moreover, with the increase in the GO content, the overall EMI shielding performance is improved. Ni/GO/PVA-20 has excellent EMI shielding performance and it can reach 87 dB at the thickness of 2.0 mm. The EMI shielding performance decreases with the decrease in thickness. The EMI shielding performance of Ni/GO/PVA-20 with thickness of 1.0 mm and 1.5 mm is 60 dB and 70 dB, respectively. The porous structure of the disordered GO and aerogels increase the propagation distance of electromagnetic waves. The dielectric loss, magnetic loss, and multiple reflection and scattering are the main mechanisms of electromagnetic energy attenuation.

Acknowledgments: The authors acknowledge the financial supports from Excellent Young Scientist Foundation of NSFC (No. 11522216); National Natural Science Foundation of China (No. 11872087); Beijing Municipal Natural Science Foundation (No. 182033); Aeronautical Science Foundation of China (No. 2016ZF51054); the 111 Project (No. B14009); Project of the Science and Technology Commission of Military Commission (No. 17-163-12-ZT-004-002-01); Foundation of Shock and Vibration of Engineering Materials and Structures Key Laboratory of Sichuan Province (No. 18kfgk01); Foundation of State Key Laboratory for Strength and Vibration of Mechanical Structures (No. SV2019-KF-32). Foundation of State Key Laboratory of Explosion Science and Technology of Beijing Institute of Technology (No. KFJJ21-06M).

Funding information: Excellent Young Scientist Foundation of NSFC (No. 11522216); National Natural Science Foundation of China (No. 11872087); Beijing Municipal Natural Science Foundation (No. 182033); Aeronautical Science Foundation of China (No. 2016ZF51054); the 111 Project (No. B14009); Project of the Science and Technology Commission of Military Commission (No. 17-163-12-ZT-004-002-01); Foundation of Shock and Vibration of Engineering Materials and Structures Key Laboratory of Sichuan Province (No. 18kfgk01); Foundation of State Key Laboratory for Strength
Acknowledgments: This work was supported by the National Natural Science Foundation of China (No. 51875158 and U20A2077) and the Key Research and Development Program of Shaanxi Province (No. 2020JFFG-A01-01). We also acknowledge the financial support from the National Key Research and Development Program of China (No. 2018YFA0208800) and the National Natural Science Foundation of China (No. 12072061). The authors would like to thank Professor Jie Wu for his valuable discussions and suggestions.

Conflict of interest: The authors declare no potential conflicts of interest.

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