

Self-healing graphene oxide-based composite for electromagnetic interference shielding

Hyeon Jun Sim^a, Duck Weon Lee^a, Hyunsoo Kim^a, Yongwoo Jang^a, Geoffrey M. Spinks^b, Sanjeev Gambhir^b, David L. Officer^b, Gordon G. Wallace^b, Seon Jeong Kim^{a,*}

^a Center for Self-Powered Actuation, Department of Biomedical Engineering, Hanyang University, Seoul, 04763, South Korea

^b Intelligent Polymer Research Institute, ARC Centre of Excellence for Electro materials Science, AIIM Facility, Innovation Campus, University of Wollongong, North Wollongong, NSW, 2522, Australia

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ABSTRACT

The electromagnetic pollution issues have been arising from the fast-growing development for electronic devices. Hence, the demand for materials with high electromagnetic interference (EMI) shielding performance has increased. Here, we developed self-healable, flexible and printable graphene oxide/silver nanowire films and textiles with excellent EMI shielding performance. The maximum electromagnetic interference shielding effectiveness (EMI SE) of 92 dB was recorded for an 18 μm -thickness film. In addition, the specific EMI shielding effectiveness was 31 dB cm^3/g or 48,275 dB cm^2/g when normalized to film thickness. Both values are higher than reported EMI shielding products. The composite film and coated textile were tolerant of damage induced by cracking or scratching. Damaging the films by cracking reduced the electrical conductivity, mechanical properties, and the EMI SE was decreased from 72 dB to 56 dB at 8.2 GHz. After the healing process, the EMI SE was recovered to 71 dB and mechanical properties restored. The EMI SE of textile reached a maximum of 30 dB which is suitable to use as a commercial EMI shielding product. In addition, the textile exhibited high flexibility, and showed excellent mechanical stability with no change in performance after 1000 bending cycles.

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1. Introduction

Wearable electronic devices of various forms and for a variety of applications have been rapidly developing over the last ten years. These electronic devices could create significant electromagnetic interference (EMI) when the devices transmits, distributes or use electric energy [1–19]. The generated EMI could have a detrimental impact on the performance of nearby devices or the surrounding environment [20]. Of particular concern would be the impact of EMI on the performance of implanted biomedical devices, such as a pacemaker or pulse generator, which may cause a crucial problem to the wearer's health. Metal is a highly effective EMI shielding material, but the rigidity and weight of metal foils and fibers are not ideal for wearable applications or other areas such as aerospace, automobiles and portable electronics [1,3].

Recently, 2 dimensional (2D) electrical conductive materials including graphene and MXene have been reported for EMI

shielding products [1,2,7–11,21–24]. The EMI shielding performance depends on the electrical conductivity, dielectric constant and magnetic permeability, and all of these properties are influenced by the content of conductive filler, the filler aspect ratio and structure of composite shielding materials [14–19]. The 2D filler materials have significant advantage such as high electrical conductivity, excellent mechanical property, light weight, flexibility and large aspect ratio [1,9]. Highly flexible MXene films of nacre-like MXene-polymer composites have been shown to demonstrate a high EMI shielding effectiveness (SE) of 92 dB and specific EMI shielding effectiveness divided by thickness (SSE/t) of 30,830 dB cm^2/g [1]. Ultra-lightweight and highly conductive graphene/polymer foam composite have also been developed with a high EMI SE above 30 dB [3].

An enduring challenge with all EMI materials is to ensure mechanical robustness to avoid cracking or damage during use. Once an EMI shielding product has cracked, the EMI shielding performance is significantly decreased and it loses its usefulness. Wearable EMI shielding products will be exposed to various mechanical stimuli such as impact, crush, compression and abrasion and any of

* Corresponding author.

E-mail address: sjk@hanyang.ac.kr (S.J. Kim).

these actions could induce cracking damage to the EMI layer. Designers of EMI shields must choose a compromise between thick EMI shielding layers that resist damage and thin layers that are more flexible and lightweight.

Here, we report an alternative solution with EMI films that self-heal after damage. We describe flexible and printable graphene oxide/silver nanowire (GOSN) films and EMI shielding textile with excellent EMI shielding performance. Free-standing, flexible films and textiles were fabricated by a simple solution based casting or printing process with GOSN solution. The high electromagnetic interference shielding effectiveness (EMI SE) of 92 dB was recorded for an 18 μm -thick GOSN film. In addition, the specific EMI shielding effectiveness (SSE) and SSE/t were 31 $\text{dB cm}^3/\text{g}$ and 48,275 $\text{dB cm}^2/\text{g}$ which are higher than previously reported EMI shielding products. The damaged composite was perfectly self-healed in ambient condition by the action of moisture. After damage, the EMI SE value was decreased from 72 dB to 56 dB at 8.2 GHz. Healing restored the EMI SE value to 71 dB. The EMI shielding textile was fabricated by coating GOSN composite on to a cotton fabric. The EMI SE of the coated textile reaches a maximum of 30 dB which is suitable to use as a commercial EMI shielding product. The textile has high flexibility, and demonstrates excellent mechanical stability during 1000 bending cycles. In addition, the textile also showed the self-healing property and has high potential to apply EMI shielding cloth for wearable applications.

2. Experimental section

2.1. Fabrication of graphene oxide/silver nanowire composite

1 wt% graphene oxide dispersion with an average lateral particle size of 40 μm in water was prepared based on our previous study. The 1 wt% silver nanowire dispersed in ethanol solution was purchased (S27E-KNS7C4, Nanopyxis, Korea). The graphene oxide solution and silver nanowire solution was homogeneously mixed by stirrer (pc-420d, corning, USA) during 1 h. The ratio of the volume of each solution was varied from 20 wt% to 80 wt% of silver nanowire. The 1 ml mixed solution was poured into a rectangular Teflon mold (2.5 $\text{cm} \times 3.5 \text{ cm} \times 1 \text{ cm}$) and dried in ambient condition during 24 h.

2.2. Characterization

The morphology was analyzed by scanning electron microscopy (SEM-S4700 microscope, Hitachi, Japan). To make EMI shielding textile, the cotton textile was immersed in the mixed ink of 80 wt% content of silver nanowire. The textile was dried on the ambient condition during 24 h and EMI SE of textile was measured. The conductivity was measured by a four point probe sheet resistance measurement system (CMT-100s, Advanced Instrument Technology, USA). The electromagnetic interference shielding effectiveness (EMI SE) was measured using a vector network analyzer (8720D, Hewlett-Packard Company, USA). The self-healing performance was confirmed by the following process. Firstly, the graphene oxide/silver nanowire film of 5 μm thickness was fabricated and the EMI SE was measured. Next, the film was partially broken and the EMI SE was re-measured. To recover the damage, water was sprayed on to the damaged region and the film was dried in ambient condition for 1 h. Finally, the EMI SE of the healed film was measured.

3. Results and discussion

A schematic illustration representing the self-healable EMI shielding composite is shown in Fig. 1a. Based on our previously

described graphene oxide synthesis process [25], we first prepared an exfoliated graphene oxide solution. The graphene oxide particles have an average lateral size of 40 μm with a wrinkled appearance and contained functional groups such as hydroxyl, epoxide, ketone and carboxyl (Fig. S1). The graphene oxide solution and the silver nanowire solution were combined and stirred for 1 h. The average diameter of silver nanowire was about 37 nm and the length was above 10 μm (Figs. S2–3). The GOSN solution had sufficient viscosity to be successfully printed with brush, syringe and stencil. GOSN films were formed by casting and environmental evaporation of the solvent (Fig. 1b).

The dried GOSN films self-assembled into a multi-layered structure as shown in Fig. 1 c-d. The 2-dimensional graphene oxide sheets tend to arrange into layers and silver nanowires were sequentially stacked between layers due to their high affinity with the GO. The integrated network of GO and silver nanowires exhibited a high electrical conductivity of $2.9 \times 10^6 \text{ S/m}$. Moreover, the composite also showed a relatively low density (2.9 g/cm^3), which increases with weight of silver nanowire (Table S1).

The self-healing behavior of the GOSN films was demonstrated by introducing a micro-crack into a film and reversing this damage by exposing the damage site to water. The healing mechanism can be explained by reversible hydrogen bonding of graphene oxide [26]. When the water was sprayed on the crack point, GOSN were swollen since the functional group of graphene oxide has a high affinity with water. Swollen GOSN filled the micro-crack void and the subsequent water evaporation reformed the GOSN network within the void space and restored the conductive network across the damage zone.

As expected from the high electrical conductivity, the GOSN composite film also displays excellent EMI shielding performance. To confirm the EMI shielding performance of the GOSN composite, we measured the EMI shielding effectiveness (SE) as a function of the weight of silver nanowire in the composite (Fig. 2a). The composition of the composite films were altered by controlling the mixing ratios of the GO and silver nanowire solutions. This fabrication technique allowed free standing films to be prepared with silver nanowire mass fractions ranging from 20% to 80%. All composite films had a similar thickness of approximately 5 μm and the EMI SE was measured in the X-band. The EMI SE increased with the fraction of silver nanowire contained in the composite and the EMI SE in the 80 wt% composite film reached 72 dB at 8.2 GHz.

The phenomenon could be explained by Simon formula as below [27]:

$$SE = 50 + 10 \log \left(\frac{\sigma}{f} \right) + 1.7t \sqrt{\sigma f} \quad (1)$$

Where, σ [S/cm] is the electrical conductivity, f [MHz] is the frequency of electromagnetic wave and t [cm] is the thickness of shielding product.

According to equation (1), the crucial factors in determining EMI SE were electrical conductivity and film thickness. The electrical conductivity increased with silver nanowire from 10 S/m in the 20 wt% composite film to $2.9 \times 10^6 \text{ S/m}$ in the 80 wt% composite film (Fig. 2b). When the content of silver nanowire increases, the number of electrical pathways was enhanced, resulting in a reduced electrical resistance and increased film conductivity. The electrical conductivity of the composite film increased rapidly below 40% and the slope of conductivity increase decrease above 40%. Because the electrical network of the silver nanowire is not completely formed at the low concentration, it seems that the electrical conductivity is rapidly enhanced with silver nanowire content as a concept of the percolation threshold.

Experiment measurements of the EMI SE in X-band were similar

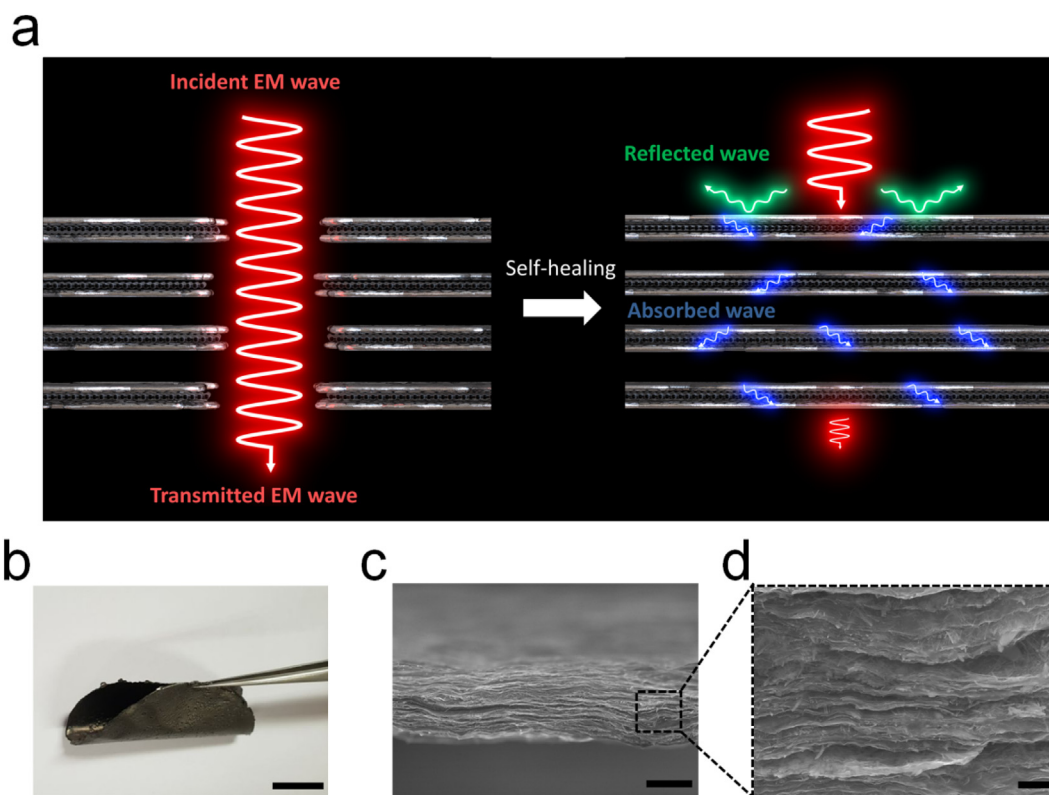


Fig. 1. (a) Schematic illustration of self-healable EMI shielding graphene oxide/silver nanowire composite. The graphene oxide/silver nanowire was swollen by hydrogen bonding of graphene oxide with water molecules after moisture was sprayed on the composite. The crack void was filled with swollen graphene oxide/silver nanowire and recovered after drying. Both the pristine and the healed composite had excellent EMI shielding performance. Electromagnetic wave strikes the surface of composite; some wave was reflected on the surface. After surface reflection, the remaining wave penetrates the interior of the composite, and the wave was absorbed by the composite. (b) The optical image of free-standing and flexible graphene oxide/silver nanowire film (scale bar: 2 cm). (c) SEM (scale bar: 2 μm) and (d) magnified SEM image (scale bar: 1 μm) of cross-sectional morphology of graphene oxide/silver nanowire composite.

to calculated result from equation (1) and we could predict that the composite retained excellent EMI SE over a broad frequency range (Fig. 2c and Fig. S4). In the films made from graphene oxide only with a thickness of 5 μm , the EMI SE was negligible when measured in the X-band (Fig. S5). The results indicated that the high EMI SE performance of the GOSN composites originated from their high electrical conductivity.

The EMI shielding mechanism of the composite films can be explained by the multi-layered structure. When the electromagnetic wave first strikes the surface of the composite, some waves were reflected through interactions with free electrons on the surface [17]. The remaining wave penetrates into the composite, with further interactions with free electrons causing a loss of electromagnetic energy [1]. The energy loss phenomenon was repeated at each layer through the thickness of the composite and electromagnetic wave energy was significantly decreasing as it passes through the film.

The contribution of absorption and reflection to the total EMI SE is important when shielding biomedical device since the reflected electromagnetic wave could affect nearby electronic devices. The contributions to the total EMI SE from absorption and reflection in the composite film at 8.2 GHz were determined and are shown in Fig. 2d and Table S2. The total EMI SE (SE_{total}) is the sum of absorption (SE_A), reflection (SE_R) and the multiple reflections (SE_M) in the EMI shielding. However, the SE_M is generally ignored when SE_{total} is above 15 dB [27]. It can be seen that, as the silver nanowire content in the composite films increased, both the SE_{total} and SE_A was enhanced. The contribution of absorption to the total EMI SE is enhanced in composites with a higher silver nanowire fraction. For

example, the contribution of absorption to the EMI SE in the 80 wt% composite is 99.2% and is much larger than that of reflection (0.8%). The high absorption ratio is a significant advantage when applied to biomedical and wearable device since the reflected electromagnetic wave was minimized.

Fig. 2e shows the EMI SE of the 80 wt% composite prepared with different film thicknesses. The EMI SE increased with film thickness and the maximum EMI SE of 92 dB was recorded for the thickest film at 18 μm . This shielding effectiveness is enough to block 99.99999994% of incident radiation with only 0.00000006% transmission.

Specific EMI shielding effectiveness (SSE) and SSE/t which SSE was divided to thickness were crucial since the EMI SE value was affected to structure, density and thickness of shielding product. The SSE of the 80 wt% composite was 31 $\text{dB cm}^3/\text{g}$ and good performance compared to similar thickness EMI shielding product. Especially, the SSE/t was 48,275 $\text{dB cm}^2/\text{g}$, which is several times higher than those (Fig. 2f and Table S3).

Commonly, EMI shielding product has been used as thick metal film, but inherent brittleness and heavy weight were limited for using in wearable device. To overcome the limitation, most research has focused on new shielding material. However, it is hard to satisfy the commercial EMI shielding requirement (above 30 dB) with thin thickness below 1 mm [10]. Our GOSN composite satisfy the several commercial requirement for EMI Shielding product such as high EMI SE (92 dB), low density (2.9 g/cm^3) compared to pure metal, thin-thickness (18 μm), high flexibility and simple fabrication process, therefore, composite film was suitable for EMI SE product.

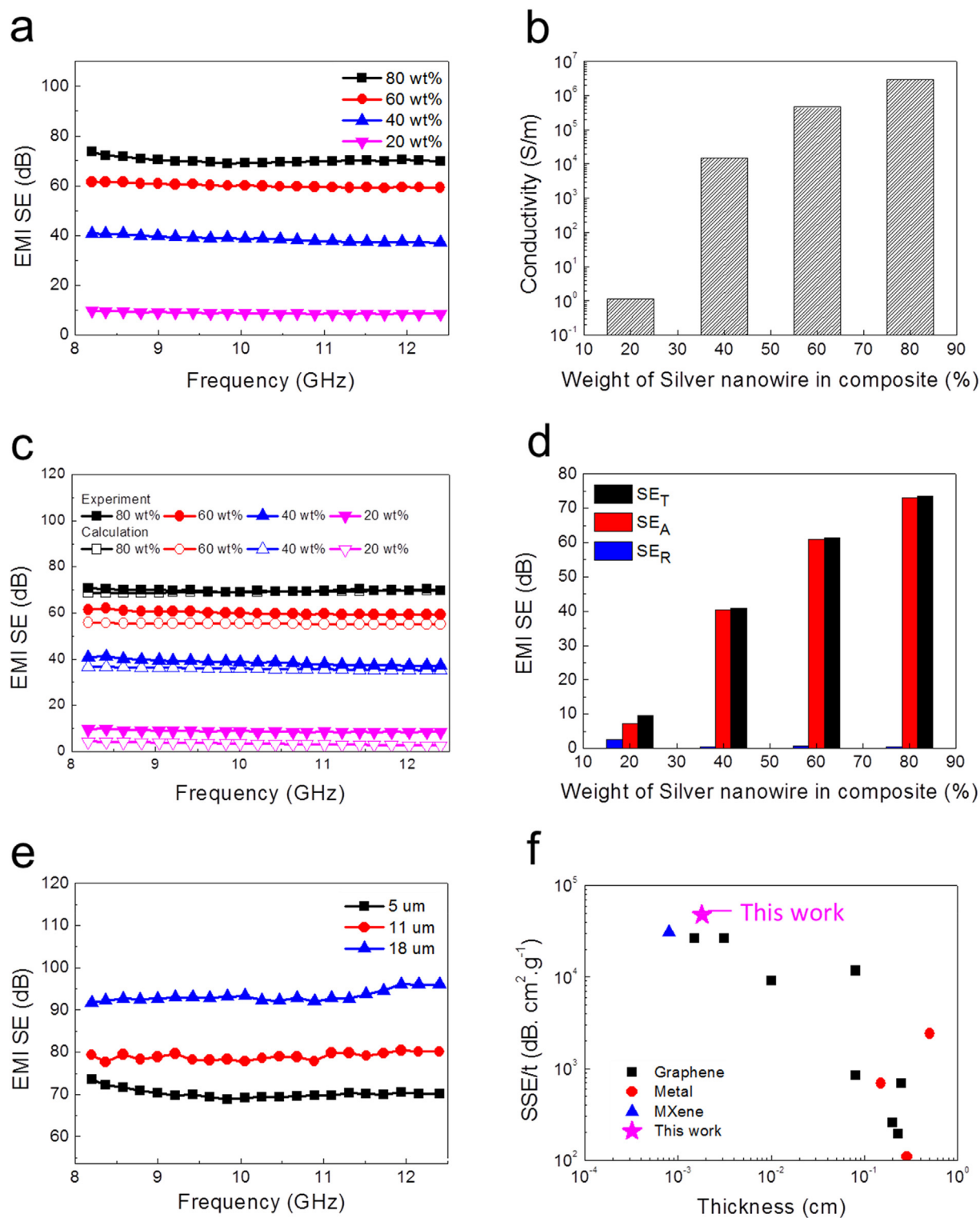


Fig. 2. The EMI shielding performance of graphene oxide/silver nanowire composite. (a) EMI SE of graphene oxide/silver nanowire composite with varying weight ratios of silver nanowire in the composite at a film thickness of about 5- μ m. (b) The dependence of electrical conductivity of graphene oxide/silver nanowire composite with the weight ratio of silver nanowire in the composite films. (c) The theoretical EMI SE values in the X-band is compared with experimentally calculated values. (d) Total EMI SE (SE_T), absorption (SE_A) and reflection (SE_R) with weight ratio of silver nanowire at 8.2 GHz. (e) EMI SE of the composite film with a silver nanowire content of 80 wt% and at different film thicknesses. (f) SSE/t vs. thickness value comparison of graphene oxide/silver nanowire composite with previously reported EMI shielding materials. (A colour version of this figure can be viewed online.)

The GOSN composite has the ability to recover cracking damage by exposure to water. To demonstrate this remarkable self-healing behavior, the EMI SE of the 80 wt% composite film with thickness of average 5 μm was measured in X-band before and after inducing damage. The SEM image of surface morphology of GOSN in the

initial state is shown in Fig. 3a. The silver nanowires form an interconnected network that effectively attenuates electromagnetic waves to a EMI SE of 72 dB (Fig. 3b). The composite film was then deformed to induce an internal crack (Fig. 3c). The presence of the micro-crack reduces the EMI SE significantly from 72 dB to 56 dB at

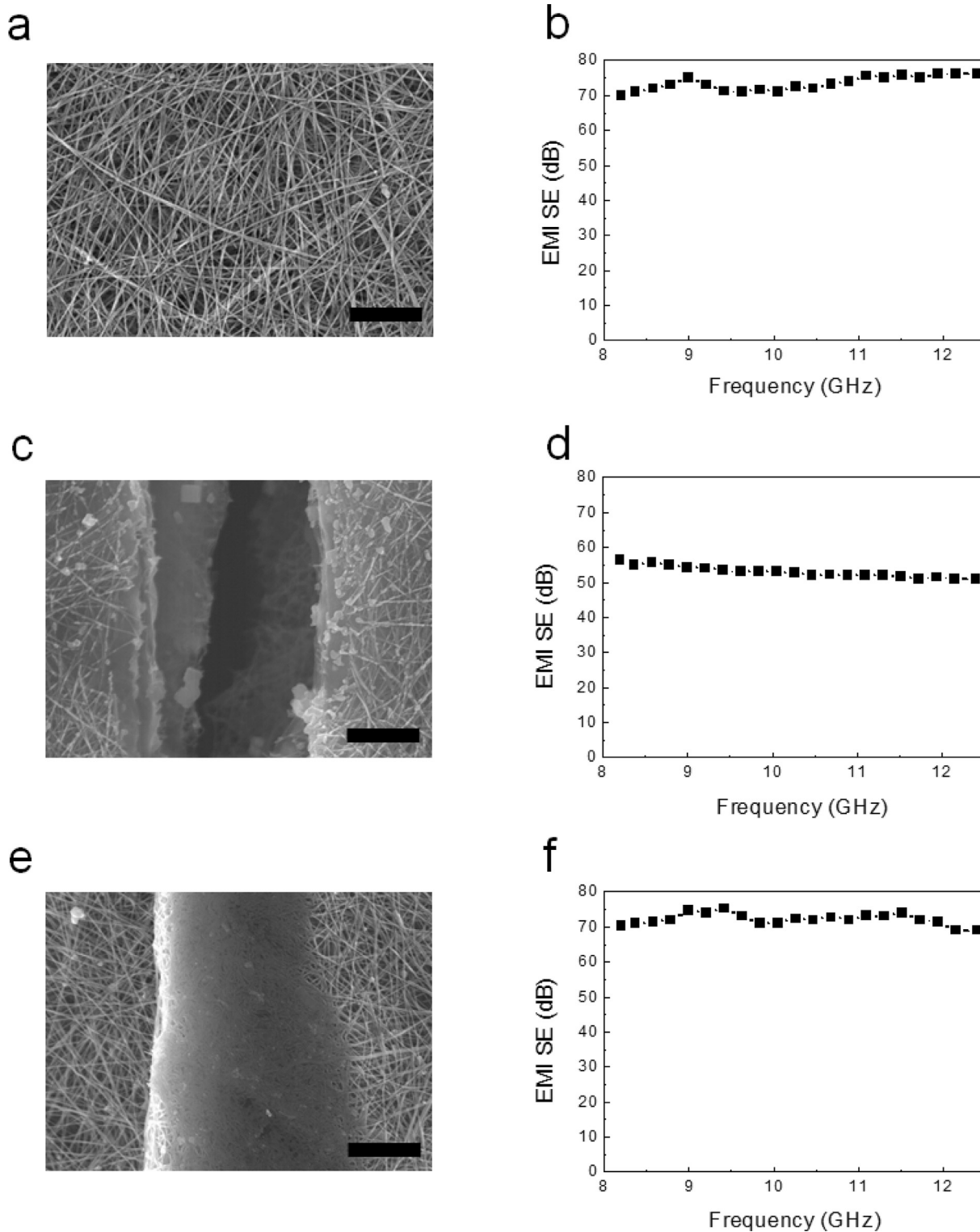


Fig. 3. The self-healing performance of graphene oxide/silver nanowire composite with a silver nanowire content of 80 wt% and a film thickness of 5 μm . (a) SEM image (scale bar: 2 μm) and (b) EMI SE of the original composite. (c) SEM image (scale bar: 2 μm) and (d) EMI SE of the damaged composite. (e) SEM image (scale bar: 2 μm) and (f) EMI SE of the healed composite.

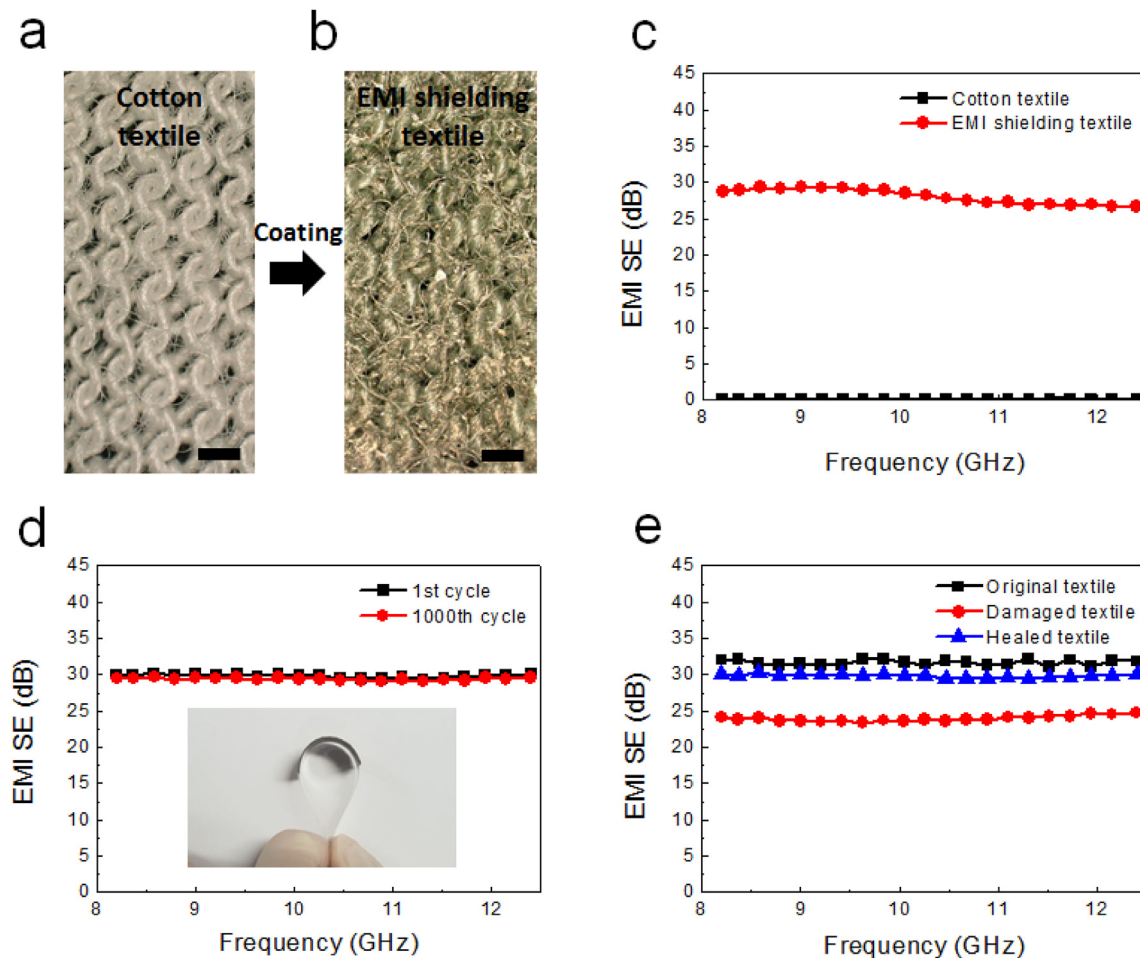


Fig. 4. The EMI shielding textile coated with graphene oxide/silver nanowire composite. The optical image of (a) uncoated cotton textile and (b) graphene oxide/silver nanowire composite coated cotton textile as EMI shielding textile (scale bar: 0.5 mm). (c) EMI SE of cotton textile and EMI shielding textile. (d) The EMI SE of EMI shielding textile with repeated bending cycles. Inset image show the bent EMI shielding textile. (e) EMI SE of the damaged and the healed EMI shielding textile. The EMI SE value was recovered after the healing process. (A colour version of this figure can be viewed online.)

8.2 GHz (Fig. 3d).

To recover the GOSN composite, the cracked region was sprayed with water and dried in the ambient environment condition for 1 h. The wetting causes swelling of the GOSN, and subsequent drying process fills the empty space of the micro-crack with GOSN composite (Fig. 3e and Fig. S6). As a result of the crack healing the EMI shielding performance was recovered from 56 dB to 71 dB at 8.2 GHz (Fig. 3f).

The cracking damage and wet/dry healing process also significantly influenced the electrical conductivity of the composite film. The electrical resistance increased from 15 Ω to 40 M Ω when the composite was damaged using a blade (Fig. S7). After spraying with moisture and air drying, the electrical resistance was recovered from 40 M Ω to 25 Ω . The electrical conductivity is a significant factor in determining the EMI shielding performance and the recovery of electrical conductivity supports the self-healing EMI performance. Although the electrical resistance was slightly increased with time, it still maintained a high conductivity after 7 days (Fig. S8). Finally, the healing of mechanical properties were also evaluated. The tensile strain at break and strength of the original composite were 1.6% and 4.1 MPa, respectively (Fig. S9). After healing, the tensile strain at break and strength of the healed composite were 1.5% and 3.9 MPa, respectively. The mechanical performance of healed composite was nearly fully recovered to the original composite values. The considerable recovery of electrical

and mechanical properties after substantial damage indicate the high potential to apply these new materials in real applications.

To develop more comfortable wearable devices, we investigated using the GOSN in a flexible EMI shielding textile, as shown in Fig. 4 a-b. The EMI shielding textile was simply fabricated by coating cotton textile with GOSN ink. The uncoated cotton textile, had a negligible EMI SE due to their low electrical conductivity (Fig. 4c). After conductive ink coating, however, the EMI SE reached 30 dB, which is suitable to be used in a commercial EMI shielding product. The EMI shielding textile has high flexibility and mechanical stability, as shown in Fig. 4d. Bending the EMI shielding textile about 150° had an insignificant affect on the EMI SE. After 1000 bending cycles, the EMI SE remained near the initial value without distortion. The EMI shielding textile also demonstrates the healing performance as shown in Fig. 4e. Scratching the EMI shielding textile decreased the EMI SE at 8.2 GHz from 32 dB to 24 dB. Exposing the textile to water and drying induced a healing process where the EMI SE recovered to 30 dB.

4. Conclusions

The GOSN composite was fabricated by a simple solution mixing and drying process. Free-standing, flexible and thin films having a multi-layered structure of graphene oxide and silver nanowire network. The electrical conductivity of composite films increased

with increasing silver nanowire content from 10 S/m at 20 wt% to 2.9×10^6 S/m at 80 wt%. The EMI SE also increased with the content of silver nanowire and with the film thickness. In the composite film with 80 wt% of silver nanowires, the highest EMI SE of 92 dB was recorded for an 18 μm -thick film and the SSE was 31 dB cm^3/g . Significantly, the SSE/t of 48,275 dB cm^2/g was several times higher than other EMI shielding products. Also, the EMI SE of composite films and textiles were successfully recovered through a wet/dry self-healing process. After the composite film was damaged by cracking, the EMI SE value was decreased from 72 dB to 56 dB at 8.2 GHz. After healing process, the EMI SE value was recovered to 71 dB. EMI shielding textile made using GOSN composite has high EMI SE of 30 dB which is suitable to use commercial EMI shielding product. The textile has high flexibility, and mechanical stability during 1000 bending cycles. In addition, the textile showed the self-healing property and has high potential to apply EMI cloth for wearable device, aerospace and military applications.

Competing interest

The authors declare no competing financial interest.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.carbon.2019.08.073>.

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