Modified electrical and microwave absorption properties of silver nanowires grown on graphene nanoplatelets

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Abstract
Crystalline silver nanowires (AgNWs), with lengths up to few micrometres and diameters of nearly 100 nm have been effectively synthesized on graphene nanoplatelets (GNPs) by a modified polyol method. The possible mechanism for the development of silver nanowires is investigated. The GNPs/AgNWs nano-hybrid nano-materials were characterized and explored by x-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The GNPs/AgNWs nanohybrid materials were then added in E-glass/epoxy matrix in different weight percentage to create composites. These composites were characterized and investigated for electrical characteristics and microwave absorption measurements. The GNPs/AgNWs nano-hybrid epoxy composite has shown a prominent increase in conductivity compared to that of the composites prepared with GNPs alone in the epoxy matrix. The GNPs/AgNWs nanohybrid composite with a thickness of 1.2 mm exhibited a minimum Return Loss (RL) of $-6.2 \text{ dB}$ at 17 GHz whereas GNPs reinforced composite showed minimum RL of $-5.7 \text{ dB}$ even at 2.0 mm composite thickness. The enhancement of properties in composites is explained based on the structures of the GNPs/AgNWs nano-hybrid in the epoxy matrix.

1. Introduction

One-dimensional nanostructures such as nanowires, nanorods and nanotubes, have been extensively studied for their prospective applications in the field of nanodevices, nanosensors and nanocircuits because of their outstanding electronic and mechanical properties [1–4]. Researchers are showing their curiosity in microwave absorbing materials (MAMs) from many decades because of their potential commercial and military application [5]. MAMs are generally well-known for their potential of suppressing microwave radiations and to shield/reduce the electromagnetic interference (EMI) issues [6, 7]. An excellent MAM should be lightweight that can also additionally cover a wide range of frequency. To hand out these purpose polymer composites have been widely utilized with various type of dielectric [8, 9] and magnetic [10, 11] nano-fillers. Numerous microwave absorption researches have been documented with carbon nanotube-polymer nano-composites [12–14] while only a few studies are present for polymer nano-composites of graphene.

Graphene can be used as a superb microwave absorbing material when incorporated in polymers [15, 16] because of its high surface area ($\sim 2600 \text{ m}^2 \text{ g}^{-1}$) and sheet-like structure. The small stacks of graphene sheets with a thickness up to a few tens of nanometers, known as graphene nanoplatelets (GNPs), are also utilized by many researchers as reinforcements in polymer composites [17, 18]. It has been experienced that GNPs dissipated in a polymeric matrix contributes as nano-filler with a high aspect ratio, as well a micro-filler with a large area, because of their specific morphology. However, strong van der Waals forces among the GNPs induces the irregular dispersion and as a result, the composite characteristics fall away from expectations connected with the...
pledge of independently dissipated nano-fillers [19]. Uniform distribution of the nano-fillers induces the development of suitable networks for electrical and strain properties in the composite materials may facilitate the unusual potential of GNP for producing advanced materials [20]. Hence, uniform and stable dispersion of the nano-fillers in the polymer matrix facing serious challenges in this area.

Special measures are needed to enhance the dispersion and solubility of GNP within the polymer matrix, which are the dominant factors limiting the application. One possible way to improve the dispersion is the incorporation of secondary nano-filler in polymer matrix along with GNP; the presence of secondary nano-filler can improve the dispersion [21, 22]. The one-dimensional nano-filler like nanorods, nanotubes, and nanowires play a significant role when used as secondary nanofillers both in dispersion and the formation of the conductive network [23–26]. He et al [23] achieved a conductive network of graphene by adding silver (Ag) nanowires along with graphene in the polymer matrix. Woo et al [27] developed a robust network when incorporated silver nanowires (AgNWs) with single-wall CNTs. Researchers have been preparing graphene-silver nanowires composite for improved electrical and thermal properties [24], high capacitance [25] and for ammonia gas sensing properties [26]. Work on silver graphene-silver nanowires composites acting as transparent electrodes [28, 29] and as flexible conductors [30] have also been reported recently. Hybrid nanostructures prepared with graphene and metal wires is the key step towards the development of multifunctional materials with synergistic properties. These recent reports showed that there is a great potential in such multifunctional nano-composite for application in the electronics and aerospace industry.

The objective of the presented work is to synthesize GNP-Ag nanowires hybrids and use them as reinforcement in epoxy/E glass fiber composites for enhanced electrical and microwave absorption properties. The content of Ag is kept less than the GNP for obtaining lightweight structures.

2. Materials and methods

The GNP were bought from ACS Materials. The thickness of the platelets is 10 nm, the diameter is 5 μm and purity >95%. AgNO₃, CuCl₂, Ethylene Glycol (EG) and polyvinylpyrrolidone (PVP, Mw ≈ 40000) were obtained from Sigma Aldrich. All chemicals were used without further purification.

2.1. Synthesis of graphene-Ag nanowires (AgNWs) hybrid material

Graphene-Ag nanowires hybrids were prepared through a customized polyol technique. The plan of Xia et al [31] reported for the preparation of silver nanowires was modified for this purpose. The detail of the synthesis is as follows. The solution of CuCl₂·2H₂O (6 mg/2 ml) in EG was mixed to preheat EG and then heated for fifteen (15) min. In the later stage, 1.0 g of AgNO₃ and 200 mg of graphene nanoplatelets powder were supplied to 12 ml ethylene glycol individually and sonicated for a uniform solution in EG. Afterwards, GNP and AgNO₃ solutions were mixed in each other and sonicated for half-hour. The solution of PVP and GNP-AgNO₃ mixture (8 g/EG 24 ml) were mixed altogether in dropwise manner to the CuCl₂ solution. Following one hour of reaction time at 160 °C, the solution colour turns from black to grey which is the signal of AgNWs creation [32]. The AgNWs solution was cooled down to room temperature and the required GNP/AgNWs were attained by centrifugation following washing numerous times through de-ionized water and acetone. The composed substance was dried out initially on a hot plate and later through vacuum at 100 °C.

2.2. Fabrication of GNP/AgNWs reinforced epoxy nanocomposites

For the preparation of GNP/AgNWs reinforced epoxy hybrid composite, the prepared nanomaterial was ultrasonically dispersed in epoxy 5052; the addition of nano-filler in the epoxy matrix was changed from 1.0 to 2.6 wt%. After the sonication process of about 2 h, the composite mixture was degassed for 10 min to eliminate air bubbles. In the next step, the curative was added to the nanomaterial-filled epoxy through mechanical mixing. The resultant mixture of the GNPs/AgNWs was coated on the sheets of glass fiber by hand layup system followed by vacuum compression. The synthesized nano-composites were then cured for 4 about hours at 100 °C. The thickness of synthesized nano-composites was about 1.3 mm. Five nano-composite samples with variable nano-hybrid loadings were prepared (table 1 explains the sample coding). The nano-composites were cured first for 24 h at room temperature and later cured at 100 °C for 4 h.

The surface microstructure of the hybrid nano-composites was examined by Scanning Electron Microscopy (SEM, JED 2300) [33]. The direct current conductivity of the coated specimens was computed by a two-probe technique with a Keithley 2401 source and measurement unit in a linear geometry. Rectangular plates in the dimensions of 50 × 25 mm² were prepared.
3. Results and discussions

3.1. XRD analysis
XRD graph of as-synthesized GNPs/AgNWs hybrids is exhibited in figure 1. The diffraction pattern identifies the material as a mixture of Ag and graphene. The four major peaks corresponded to (002), (111), (200), and (220) crystallographic planes of Ag crystal, which indicate the formation of AgNWs [5, 34, 35]. The peak at 2θ = 27° shows the (002) plane of the graphite structure [36, 37].

3.2. Microstructure studies
The Scanning Electron Microscope (SEM) morphologies of GNPs/AgNWs hybrid is shown in figures 2(a)–(b). The development of high-density AgNWs on the graphene sheets is obviously noticeable. The morphology image exhibits that the length of the nanowire is in the range of 4 to 10 μm and their mean diameter is ∼100 nm. A few nanometer size graphene sheet is also noticeable in the SEM image. Growth of several particles of ∼200 nm size additionally can be witnessed on the graphene sheet. The EDS spectrum helped to determine the atomic percentage of Ag in GNPs/AgNWs hybrid. EDS measurements (figure 3) shows that for a synthesis procedure described above, the ratio of silver to carbon is 20 to 80.

3.3. The formation mechanism of Ag nanowires on GNPs
In the initial step, elevated temperature (160 °C) is crucial for the conversion of EG to glycolaldehyde. EG acts as a reducing agent for silver. The silver nuclei, formed after the reduction of Ag⁺ ions with glycolaldehyde, are preferentially grown on the edges of GNP sheets. It is because; the edge is a defective surface and facilitates the growth of nuclei [38]. Silver nanoparticles then have a huge capacity to adsorb at the edges of GNPs, which take part in the growth of substrate for the development of silver nanostructures. Van der Waal forces due to graphene sheets also facilitate Ag growth on GNPs surface. The Chloride ions from CuCl₂ facilitate the high-yield of the thermodynamically stable multiply twinned Ag seeds. The structural or crystallographic defects of twinned particles offer favourable sites for oxidation in the solution and these twinned seeds behave as reasonable seeds for one-dimensional growth in next steps [39]. Keeping the solution temperature constant at 160 °C finally provides small Ag nanoparticles with polyhedral shapes in EG solution. As the reaction continued and enough amount of precursor (i.e. AgNO₃) is also present in the solution, the small Ag nanoparticles lost their stability in the solution and started to dissolve and lead to the growth of bigger ones. These Ag nanoparticles

| Sample code | Concentration of GNP/AgNWs (wt%) | Electrical conductivity (S/cm) |
|-------------|---------------------------------|------------------------------|
| GNP/AgNWs-1.0 | 1.0 | 0.0204 |
| GNP/AgNWs-1.4 | 1.4 | 0.178 |
| GNP/AgNWs-1.8 | 1.8 | 0.00688 |
| GNP/AgNWs-2.2 | 2.2 | 0.0333 |
| GNP/AgNWs-2.6 | 2.6 | 3.333 |
| GNP/AgNWs-3.0 | 3.0 | 3.8 |

Figure 1. The XRD pattern of GNPs/AgNWs hybrid.
were fully dispersed due to the existence of PVP in the solution that might be chemically adsorbed on the surfaces of Ag by O–Ag bonding [40]. As chemical reactivity is different for different faces of face-centred cubic structured metal nanoparticles [41], the facets with higher surface energy have a high capacity to adsorb PVP as compared to the others. In case of silver, the PVP was bounded preferentially on then this inhibited the growth along direction [42, 43]. In this situation, the growth/development rate of end facets of twinned nanoparticles is remarkably greater than their etching rate, with the intention that fast and selective one-dimensional growth of nanoparticles occurs resulting in the formation of nanowires [42]. The schematic illustration of Ag nanowire formation on GNPs is represented in figure 4.

3.4. DC conductivity measurement

The electrical conductivity of the GNP and GNPs/AgNWs filled epoxy nano-composites as a function of the filler substance is shown in figures 5(a), (b), respectively. The percolation thresholds for GNP and GNPs/AgNWs are perceptibly noticed. Percolation thresholds for GNP and GNPs/AgNWs nanofillers in epoxy composites were observed at 3.0 and 2.5 wt% respectively. The percolation threshold value is recognized to be mainly dependent on the conductive filler’s microstructures. At the value of 2.5 wt% concentration, the GNPs/AgNWs epoxy nano-composites exhibit an electrical conductivity of value $2.1 \times 10^{-5} \text{Sm}^{-1}$, which is a rise of about an order of magnitude over the conductivity of the GNP nano-composites. The GNP epoxy nano-composites show superior performance with $4.3 \times 10^{-3} \text{Sm}^{-1}$ electrical conductivity value. The intrinsically high conductivity and large aspect ratios of GNP and AgNWs permit them to effortlessly create electrical pathways within the matrix.

It is generally supposed that the conductivity of filled conductive polymeric systems originates from the development of a conductive network by the fillers in the matrix [44]. In the (GNPs)$_{1-x}$(AgNWs)$_x$ hybrid
system, the conductive network was developed at a minor filler concentration of 2.5 wt% than that for the GNP filled systems (3.0 wt%). This can be described by the development of conductive pathways more proficiently when combining single-dimensional AgNWs with two-dimensional GNPs. The bridging role of CNTs has been observed in literature for GNP which facilitates the transportation of electrons among them [45]. The existence of conducting network can be explored by SEM image (figure 7). The SEM micrograph is showing the morphology of hybrid composites.

The AgNWs facilitates the dispersion and diffusion of GNP within the epoxy matrix by acting as small bridges connecting many GNPs together. It also helps to prevent aggregation of GNP sheets by incorporating themselves among GNP sheets during the growth process.

Figure 6 is a schematic showing the presence of a conducting network among GNP sheets and grown AgNWs on the surface of GNP sheets in an epoxy matrix. GNP alone could not form a conducting path easily at lower filler content.

Figure 7 shows the SEM image of the 3.0 wt% loading of GNP/AgNWs composite. Image shows that the AgNWs are well dispersed among GNP nano-sheets. The Ag particles visible in the image are formed during the synthesis of AgNWs.

The silver nanostructure improved the exfoliation as well as the dispersion of GNPs. The SEM images also propose that the thickness of GNPs is enlarged owing to the coating of the epoxy matrix.

![Figure 4. Schematic of GNP/AgNWs formation.](image)

![Figure 5. Surface electrical resistivity of the (a) GNP, and (b) GNPs/AgNWs nano-composites as a function of nano-filler loading.](image)
3.5. Microwave absorption

The microwave absorbing characteristics in terms of Return Loss (RL) in dB of the nano-composites were observed in the Ku band (11–17 GHz). The measurements GNPs reinforced and GNPs/AgNWs nano-hybrid reinforced epoxy composite samples are shown in figures 8(a), (b).

The selected concentrations for RL measurements are 2.0 wt% and 3.0 wt% which are around the percolation threshold for GNP/AgNWs nano-hybrids and for GNPs. It can be seen from figure 8(a) that for GNPs as filler in the epoxy composite, the magnitude of RL ($-5.7$ dB at 11 GHz) is larger for concentration of 2.0 wt%, while the magnitude of RL has been decreased with 3.0 wt% ($-1.5$ dB) i.e. to the percolation threshold value of GNPs. The RL of GNP/Ag NWs nanohybrids as fillers in figure 8(b) shows that the magnitude of RL is higher for 3.0 wt% than 2.0 wt%. The max value of RL for GNP/Ag NWs composite sample is $-6.2$ dB at 17 GHz. It is observed for both types of fillers that the magnitude of RL is lower at the percolation point. It is due to the rise in conductivity to a point where the composites act as reflecting medium instead of absorbing the microwaves. The reflection of electromagnetic also depends on conductivity in term of skin depth [46, 47].

Owing to have higher electrical conductivity, the microwaves will be unable to penetrate the material, where they could be absorbed. The higher RL for GNP/Ag NWs nano-hybrid composite in comparison to GNPs filled composite is due to interfacial polarization [48]. In our system, GNPs are playing the role of conductive medium. Because of the insulating matrix, in the applied field, mobile charge carriers in material migrate but their motion is constrained at the surfaces or grain boundaries. The accretion of charge carriers at the boundaries/ interfaces among GNPs and the host matrix developed polarization which is called interfacial or space charge polarization. By incorporating GNP/AgNWs nanohybrids, the number of interfaces associated with epoxy/GNP, GNP/Ag NWs/epoxy, and GNP/Ag NWs/epoxy has been increased. This enhances the interfacial polarization or now extra electric dipoles are built up in the structure to soak up electromagnetic energy. Thus RL for composites prepared by incorporation of GNP/AgNWs nanohybrids increases in comparison to GNPs composites. The other important point is that owing to the existence of AgNWs on the GNPs’s surface, dispersion of GNPs is relatively extra homogeneous within the matrix and AgNWs may also assist in the intercalation of GNP sheets. Therefore, a thin insulating coating of epoxy is mixed with the GNPs to develop a
nano-scale structure within the composites, that can entirely realize the properties of GNPs (i.e., large specific surface area) and consequence in a huge interfacial area in the nanocomposite between GNPs and polymer, which in turn supplies several sites for interfacial polarization.

4. Conclusions

A facile method for the growth of GNPs/AgNWs nano-hybrid was successfully developed. The AgNWs were well dispersed on the surface of GNP sheets, which possess large surface area per volume. The electrical measurements of the composite samples prepared with the incorporation of GNP/AgNWs nano-hybrids exhibit excellent conductivity as compared to pristine GNPs incorporated composites. The composite exhibited improved electromagnetic wave absorption properties with GNP/AgNWs nano-hybrids in comparison to the composite prepared with the incorporation of individual GNPs and AgNWs.

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Figure 8. Return loss (RL) data of (a) GNPs, and (b) GNP/AgNWs.
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