Synergistic Enhancement of Woven Copper Wires with Graphene Foams for High Thermal Conductivity of Carbon Fiber Laminated Composites

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Short Report

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Abstract

Enhancing thermal conductivity of carbon fiber laminated composites (CFRP) in out-of-plane directions without sacrificing mechanical properties is still challenging for fabrication of high-performance composites with structural and functional integration. In this work, a novel hybrid sandwich composite was fabricated by weaving copper wires through carbon fiber (CF) fabrics, laminating graphene foams (GrFs) onto surfaces, and infiltrating with epoxy via vacuum-assisted resin transfer molding technique. High-efficiency heat transfer pathways were constructed to greatly increase out-of-plane thermal conductivity of composites with maintaining CF continuity. Microstructure, electrical property, and thermal conduction of composites were experimentally measured and theoretically simulated. The hybrid sandwich composites exhibited much higher electrical and thermal conductivity than the CFRP, and their out-of-plane thermal conductivity was up to 1.097 W/m·K, increasing by 104% in comparison with that of CFRP. Such remarkable thermal enhancement is mainly attributed to high intrinsic conductivity of the copper wire and GrF, continuous heat transfer pathways, and synergistic effect of copper wire with GrF for rapid heat transfer and diffusion. The hybrid sandwich composites show great potential to be used as high-performance materials with structural and functional integration in the fields of aerospace and transportation.

1. Introduction

Developing advanced composites with high structural and functional integration have been an inevitable tendency to meet increasing requirements in the fields of the aerospace, transportation, and electronics industries [1–3]. Carbon fiber laminated composites (CFRP), as one of the most representative polymeric composites, have been widely used in many fields due to their high strength and light weight [4–8]. Notably, CFRP possess typically anisotropic characteristics due to high alignment of carbon fibers (CF) along in-plane direction of composites, and their thermal conductivity (λ) especially in the out-of-plane direction is rather low (only 0.4 W/m·K for 60 vol.% T700-CF/epoxy composites) [9], severely restricting their application as multi-functional composites in many fields. Such low out-of-plane thermal conductivity (λ⊥) mainly results from the existence of interlaminar resin layers with extremely low thermal conductivity of less than 0.2 W/m·K, greatly hindering heat transfer along through-thickness direction of laminated composites [10]. In that case, construction of high-efficiency heat transfer pathways becomes extremely important to improve out-of-plane thermal conductivity of laminated composites [11, 12].

Many strategies for improving thermal conductivity of CF-laminated composites have been developed in the past decades [13–18]. One of the most widely-used methods is to incorporate thermally-conductive fillers (e.g. copper powders [10, 19], boron nitride [20], carbon black [13], graphene [21–23], carbon nanotubes (CNT) [24], etc.) into resin matrix. But these conductive fillers are generally wrapped with thermally-insulating resin, and the resultant improvement of thermal conductivity is rather limited [25, 26]. Even increasing filler loadings for higher conductivity, the drastic increment in resin viscosity inevitably causes poor wettability and decreased mechanical properties of composites [25, 27]. Another approach is to decorate CF with thermally-conductive fillers (Cu, CNT, etc.) by electrophoretic deposition or in-situ
growth [28–32]. It is effective to improve the in-plane thermal conductivity ($\lambda_{||}$) rather than the $\lambda_{\perp}$ values of composites because of the low thickness of decoration layers (less than 500 nm) which cannot form long-distance heat transfer along through-thickness directions. Recently, z-pin technology has been rapidly developed by vertically inserting thermally-conductive pins into CF laminates to form continuous pathways for rapid heat transfer [33], and the $\lambda_{\perp}$ value of z-pinned composites was up to 8.80 W/m·K [34]. However, such enhanced thermal conductivity for the z-pinned composites is at the cost of severely destroying fiber continuity and sacrificing mechanical properties of composites [35].

Three-dimensional (3D) weaving technique has been utilized to construct 3D CF skeletons with maintaining fiber continuity for remarkable reinforcement in interlaminar strength of composites [36, 37]. Notably, the 3D weaving technique has still many strict requirements to fiber yarns, e.g. high flexibility, large stretchability, and good knittability [25]. For achieving high $\lambda_{\perp}$ values of composites, z-directional fibers (z-fiber) with high intrinsic thermal conductivity are indispensable. However, highly-conductive CFs like pitch-based CFs are extremely brittle and unavailable for 3D weaving [25]. By comparison, copper (Cu) wires possess a high $\lambda$ value of 398 W/m·K and satisfactory stretchability, and they can be easily woven as z-fibers through CF fabrics to improve $\lambda_{\perp}$ of laminated composites. It is also worth pointing out that, for anisotropic 3D woven composites, their thermal conductivity measured through flash method strongly depends on the thermal diffusivity ($\alpha$) which was experimentally measured and inversely proportional to the half-rise time ($t_{1/2}$) required to reach half $\Delta T_{\text{max}}$ [38]. Serious heat accumulation usually occurs on surfaces of 3D-woven composites because of the remarkable difference in thermal conductivity between surface resin and z-fibers, consequently resulting in uneven heat distribution and low thermal diffusivity [1, 39]. Fortunately, graphene foam (GrF) possesses high intrinsic thermal conductivity and porous structure, and it can be easily infiltrated with resin to form continuous high-efficiency heat transfer pathways throughout surface resin layers for rapid heat diffusion [40]. In that case, combination of the Cu wires as highly-conductive z-fibers with the graphene foam as high-efficiency heat diffuser would effectively diminish heat accumulation and accelerate heat diffusion for high thermal conductivity of composites, which has not been reported so far.

The motivation of this work is to fabricate high-performance composites with enhanced out-of-plane thermal conductivity. A hybrid sandwich composite was prepared by weaving Cu wires as z-fibers through CF fabrics, laminating graphene foams (GrFs) as heat diffuser onto surfaces, and infiltrating with epoxy via vacuum-assisted resin transfer molding (VARTM) technique. Microstructures, electrical conductivity, thermal conductivity of composites were investigated, and corresponding heat conduction was simulated using finite element analysis. The hybrid sandwich composites exhibited a high $\lambda_{\perp}$ value of 1.097 W/m·K, increasing by 104% in comparison with that of CFRP, which is mainly attributed to the high intrinsic conductivity of Cu wire and GrF, continuous heat transfer pathways, and synergistic effect of Cu wire with GrF for rapid heat transfer and diffusion.

2. Experimental section
2.1 Materials

Carbon fiber fabrics (T700-12k, plain weave) were provided by Toray Composite Materials America Inc. The fabrics had a single-fiber diameter of 7 µm, fabric thickness of 0.55 mm, and density of 1.8 g/cm$^3$. The axial and radial thermal conductivities for the carbon fibers were 9.38 and 0.98 W/m·K respectively. Epoxy resin (IN2) and curing agent (AT30 SLOW) were purchased from Easy Composites Ltd. Oxygen-free Cu wires with a diameter of 0.25 mm and thermal conductivity of 398 W/m·K were purchased from Dingsheng Metal Materials Company, China. Graphene foams (GrFs) were obtained by growing graphene onto nickel foam through chemical vapor deposition (CVD) and subsequently etching off nickel skeleton using a mixed solution of hydrochloric acid and ethanol \[41, 42\]. The graphene foams processed a porosity of 97% and apparent density of 6.26 mg/cm$^3$.

2.2 Fabrication of hybrid sandwich composites

In order to construct heat transfer pathways in the out-of-plane direction, Cu wires were woven manually through pores of laminated CF fabrics to obtain a Cu-CF hybrid structure with vertical alignment of Cu wires. Thereafter, graphene foams (GrFs) were laminated onto the upper and lower surfaces of the hybrid structure to form a GrF/Cu-CF/GrF sandwich structure for rapid heat diffusion. The sandwich structure was infiltrated with epoxy using vacuum-assisted resin transfer molding (VARTM) technique, and the hybrid sandwich composites were obtained after curing at room temperature for 24 h and post-curing at 60 °C for 6 h. For comparison, control samples of the CFRP and the Cu-CF hybrid composites were also prepared following the same procedures. Some detailed specification of the composites was listed in Table 1.

<table>
<thead>
<tr>
<th>Composites</th>
<th>Structure feature</th>
<th>Volume fraction (%)</th>
<th>Density (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CFRP</td>
<td>CF-laminated structure</td>
<td>51.2 48.8</td>
<td>1.509</td>
</tr>
<tr>
<td>Cu-CF hybrid composite</td>
<td>Cu-CF hybrid structure with vertical alignment of woven Cu wires</td>
<td>34.1 64.1 1.8</td>
<td>1.585</td>
</tr>
<tr>
<td>GrF/Cu-CF/GrF sandwich composite</td>
<td>Sandwich structure: Cu-CF hybrid structure as core, and GrF as surface layer</td>
<td>33.9 62.5 1.5 2.1</td>
<td>1.462</td>
</tr>
</tbody>
</table>

2.3 Characterization

Microstructures of composites were characterized by scanning electron microscopy (Thermofisher Scientific Verios G4 UC). Electrical conductivity of composites was measured at room temperature using SourceMeter (Keithley series 2400) in reference to ASTM D4496. Specimen surfaces were coated with
silver paste as electrodes to eliminate contact resistance, and volume electrical conductivity in in-plane and out-of-plane directions was measured and calculated according to the Eq. (1).

$$\sigma = \frac{S}{(R \times L)}$$

where $\sigma$, $R$, $S$, $L$ are volume electrical conductivity (S/m), electrical resistance (Ω), cross-sectional area of specimen (m$^2$), and length between electrodes (m), respectively.

Thermal conductivity of composites was evaluated through transient method using a laser flash system (LFA 467 Nanoflash, NETSCH, Germany) according to ASTM E1461. The thermal conductivity ($\lambda$) in in-plane and out-of-plane directions was calculated according to the following equations (2–3):

$$\lambda = \alpha \times \rho \times C_p$$

$$\alpha = 0.13879 \times L^2 / t_{1/2}$$

where $\alpha$, $\rho$, $C_p$, $L$, $t_{1/2}$ are thermal diffusivity (m$^2$/s), density (kg/m$^3$), specific heat capacity (J/kg·K), specimen thickness (m), and half-rise time (s), respectively. The $C_p$ values for the Ep, GrF/Ep, CF/Ep, Cu-CF hybrid, and GrF/Cu-CF/GrF sandwich composites were measured to be 1350, 1195, 1018, 1127, 1151 J/kg·K, respectively. The density of composites was measured using Densitometer (Mettler Toledo xs104) and shown in Table 1.

### 2.4 Theoretical calculation and finite element analysis

The in-plane and out-of-plane thermal conductivity of composites were theoretically calculated using parallel and series models based on the rule of mixtures, where the $\lambda$ values of components were approximately determined using the measured conductivity for theoretical calculation. Temperature distribution of composites during heating process was simulated using finite element analysis based of the following assumptions: (1) heat transfer between components was ideal without interfacial thermal resistance; (2) heat diffusion for the Cu wire, GrF, and epoxy was isotropic, while that for CF was anisotropic; (3) there was no heat exchange of composites with external environment; (4) boundary conditions for heat transfer in the in-plane and out-of-plane directions were same. The temperatures at cold and heat sides of composites were set to be 25 and 100 °C respectively, and the temperature distribution of composites at certain time was obtained through finite element analysis.

### 3. Result and discussion

#### 3.1 Microstructures of hybrid sandwich composites
Figure 1 shows the fabrication schematic and microstructures of the hybrid sandwich composites. It can be seen from Fig. 1a that Cu wires were woven vertically through pores of laminated CF fabrics to construct a Cu-CF hybrid structure with vertical alignment of Cu wire for rapid heat transfer in the out-of-plane direction. After that, thermally-conductive graphene foams (GrFs) prepared through CVD grown were laminated onto the upper and lower surfaces of the hybrid structure to fabricate a GrF/Cu-CF/GrF sandwich structure. Thereafter, the sandwich structure was infiltrated with epoxy using VARTM technique, and a GrF/Cu-CF/GrF sandwich composite was obtained after curing. In this hybrid sandwich composite, the woven Cu wires formed vertically-aligned heat transfer pathways for improvement of out-of-plane thermal conductivity, and they were vertically woven only through pores of fabrics without sacrificing CF continuity and mechanical properties of composites. Meanwhile, the GrFs laminated onto surfaces were beneficial to further increasing heat-transfer pathways and accelerating surface thermal diffusion for improved thermal conductivity of composites.

Figures 1b-e show microstructures and interfacial bonding between components of the hybrid sandwich composites. It can be seen from Fig. 1c that the highly-conductive GrF was well infiltrated with epoxy because of its high porosity of over 97% and interconnecting porous structure, beneficial to greatly improving thermal conductivity of epoxy [43]. In addition, it can be clearly observed from Fig. 1d that the porous GrF was deformed and compactly contacted with CF fabrics under vacuum pressure during VARTM process, forming interconnecting heat-transfer network for rapid heat diffusion between components. Moreover, satisfactory interfacial bonding between the GrF and Cu wires can be found from the fracture morphology shown in Fig. 1e, implying that heat can be rapidly transferred from the Cu wire to the GrF for diminishing heat accumulation and accelerating heat distribution throughout surfaces of composites. The compact contacts between the CF, GrF, and Cu wires are also beneficial to rapid heat transfer between components for improving thermal conductivity of composites.

### 3.2 Electrical conductivity of hybrid sandwich composites

Electrical conductivity of composites was measured and shown in Fig. 2. It can be seen that the sandwich composites exhibited the highest electrical conductivity in both in-plane and out-of-plane directions, followed by the Cu-CF hybrid composites and the CFRP, implying that the combination of Cu wire with GrF can greatly improve the electron transport of composites. For electron transport of composites in the in-plane direction, the conductive components of the CF, Cu wire, and GrF can be regarded to be arranged in a parallel mode. The Cu wires and GrF possess much higher intrinsic electrical conductivity than the CF, consequently resulting in remarkable enhancement in electrical conductivity for the sandwich composites. As for electron transport in the out-of-plane direction, the components including interlaminar resin layers were approximately arranged in a series mode, and the presence of electrically-insulating resin layers greatly hindered electron transport in the out-of-plane direction, showing a low electrical conductivity of only 1.53 S/m for the CFRP. After weaving Cu wires and laminating GrFs, continuous electron-transport pathways were constructed penetrating the interlaminar and surface resin layers, consequently resulting in rapid electron transport and high electrical conductivity of 25.46 S/m for
the sandwich composites (see Fig. 2b). Such remarkable enhancement in electrical conductivity is beneficial to rapid heat transfer of composites in the out-of-plane direction.

### 3.3 Thermal conductivity of hybrid sandwich composites

We measured and calculated thermal conductivity of composites using a laser flash system, and analyzed their heat diffusion behavior through finite element analysis as shown in Fig. 3. It is well known that the thermal conduction of composites is much more complicated than the electrical conduction, and it is closely associated with intrinsic thermal conductivity of components, electron and photon transport, heat transfer pathways, interface thermal resistance or scattering, and heat distribution [2]. It can be seen from Fig. 3a that, in the in-plane direction, the thermal conductivity of CFRP were gradually increased after weaving Cu wires and subsequently laminating GrFs, from 2.945 to 3.643 and 8.408 W/m·K respectively. Similar to the electron transport through laminated composites, the heat conduction in the in-plane direction can also be approximately simplified into heat transfer from individual components arranged in a parallel model [44]. In our work, the Cu wire and GrF possess much higher intrinsic thermal conductivity (398 and 1500 W/m·K respectively) than the CF with axial conductivity of 9.38 W/m·K, and they can form continuous high-efficiency heat-transfer pathways to greatly enhance thermal conductivity of composites even at low loadings of 1.5 vol.% Cu wire and 2.1 vol.% GrF. Such increased thermal conductivity of composites in the presence of Cu and GrF was also confirmed and consistent with theoretical calculation based on the rule of mixtures.

In comparison with their in-plane thermal conductivity, all the composites exhibited much lower thermal conductivity in the out-of-plane directions (Fig. 3b), showing typical anisotropic characteristics for the laminated composites. The out-of-plane conductivity of CFRP was as low as only 0.539 W/m·K, which is mainly attributed to the low radial thermal conductivity of CF (0.98 W/m·K) and the existence of interlaminar resin layers with extremely low conductivity of 0.2 W/m·K, severely hindering the heat transfer in the out-of-plane direction. After weaving Cu wire, continuous heat-transfer pathways were constructed in the out-of-plane direction by penetrating the interlaminar resin layers, and consequently the thermal conductivity of composites was improved to 0.609 W/m·K. After laminating GrFs onto the Cu-CF/Ep hybrid composites, the thermal conductivity of composites was further improved to 1.097 W/m·K, increasing by 80% and 104% in comparison with that of the hybrid composites and CFRP, which is mainly attributed to the increased heat-transfer pathways and resultant enhancement in thermal diffusion of surface resin layers. The thermal conductivity of composites in the out-of-plane direction was calculated using a series mode based on the rule of mixtures [45], and the calculated results show similar tendency to the measured conductivity of composites.

We further compared thermal diffusivity ($\alpha$) of the components and composites in unsteady heating conditions to evaluate heat transfer response of specimens to temperatures. It can be seen from Figs. 3c-d that the neat Ep showed the lowest thermal diffusivity of 0.13 mm$^2$/s among all specimens, indicating the poorest thermal diffusion due to amorphous structures of epoxy. After adding CF, Cu wire, and GrF, the thermal diffusivity was gradually increased due to the high thermal conductivity of these components.
(See Figs. 3c-d). Notably, the GrF/Ep with 2.5 vol.% GrF exhibited high thermal diffusivity of up to 0.849 mm$^2$/s in the out-of-plane direction, increasing by 553% in comparison with that for Ep. It means that the presence of GrF with high intrinsic conductivity and continuous heat transfer pathways can greatly enhance thermal diffusion of resin layers for high-efficiency heat transfer. In our work, the GrFs were laminated onto the Cu-CF hybrid structure, and it played an important role of heat diffuser in quickly transferring heat from Cu wires to surfaces of composites, greatly diminishing heat accumulation occurred between Cu wires and adjunct surface resin, and consequently accelerating heat diffusion and distribution throughout surface of composite. As a result, the hybrid sandwich composites exhibited the high-efficiency heat transfer, high thermal diffusivity, and high thermal conductivity due to the continuous heat-transfer pathways and synergistic effects of Cu wires with GrFs.

Heat transfer behavior of composites in the in-plane and out-of-plane directions was simulated using finite element analysis, and the corresponding temperature distribution at certain time was shown in Figs. 3e-m. It can be seen from Figs. 3h-j that, in the in-plain direction, the heat was slowly transferred along CFs in the CFRP due to the low thermal conductivity of CFs. After weaving Cu wires and laminating GrFs, continuous high-efficiency heat-transfer pathways were constructed to rapidly transfer heat along the Cu wires and GrFs, consequently resulting in extremely rapid heat diffusion of the sandwich composites shown in Fig. 3i. In the out-of-plane direction, the heat transfer for the CFRP was rather slow due to the existence of interlaminar resin layers (see Fig. 3k). Once the Cu wires were vertically woven through fabrics, the heat can be rapidly transferred along Cu wires in the out-of-plane direction (see Fig. 3l). It is notable that heat distribution in the Cu-CF hybrid composites was rather inhomogeneous with severe heat accumulation especially in the interfacial region between Cu wires and surface resin layer, consequently resulting in long time to reach thermal equilibrium (longer half-rise time) and low thermal diffusivity. When the GrF was laminated onto the hybrid composites, the accumulated heat can be quickly transferred to surfaces of composites, greatly accelerating heat diffusion of the sandwich composites (see Fig. 3m). Therefore, the hybrid sandwich composites exhibited rapid heat diffusion and high thermal conductivity, which is mainly attribute to the continuous heat-transfer pathways and the synergistic effects of Cu wires with GrFs.

### 3.4 Mechanism on heat transfer of hybrid sandwich composites

Heat conduction mechanism of the hybrid sandwich composites along in-plane and out-of-plane directions was illustrated in Fig. 4. Thermal conduction in solid materials is generally classified into electron and phonon transport, the former is dominant in electrically-conductive materials, while the latter in electrically-insulating materials and closely associated with structural integrity and lattice vibration [46, 47]. As shown in Fig. 4, in the in-plane direction, the heat conduction for the hybrid sandwich composites results from contributions of components (CF, GrF, and Cu wires) arranged in a parallel mode. The Cu wire and GrF possess higher thermal conductivity than the CF, and their presence can greatly improve the thermal conductivity of composites in the in-plane direction. In the out-of-plane direction, the vertically-woven Cu wires formed continuous heat transfer pathways from bottom to top, penetrating interlaminar
resin layers and greatly improving the heat transfer of composites. In the presence of GrF, much more thermally-conductive pathways were introduced to accelerate surface thermal diffusion of composites. As a result, the hybrid sandwich composites exhibited rapid heat transfer and high thermal conductivity, which is mainly attributed to the high intrinsic thermal conductivity of Cu and GrF, continuous heat-transfer pathways in out-of-plane direction, and synergetic effects of Cu wires with GrFs.

We further compared thermal conductivity of the hybrid sandwich composites with that reported in literature. It is worth pointing out that thermal conductivity for laminated composites is strongly dependent on the fiber type, fiber content, intrinsic conductivity of components, microstructure, and even fabrication technique. In that case, we chose the carbon fiber reinforced epoxy composites prepared using VARTM with similar initial $\lambda_{\perp}$ values (0.4–0.6 W/m·K) for performance comparison, and focused on the enhancement in out-of-plane conductivity before and after modifications. In our work, the initial $\lambda_{\perp}$ value for CFRP was 0.539 W/m·K, after weaving Cu wires (1.5 vol.%) and laminating GrFs (2.1 vol.%), the $\lambda_{\perp}$ value for the hybrid sandwich composites was up to 1.097 W/m·K, increasing by 104% in comparison with that of CFRP. The hybrid sandwich composites exhibited higher $\lambda_{\perp}$ value (1.097 W/m·K) than the CF/Ep composites modified with CF-mat (0.95 W/m·K) [48], graphene-silver nanowire (0.55 W/m·K) [49], CVD-CNT (0.629 W/m·K) [50], CNT (0.52 W/m·K) [51], MWCNT(0.73 W/m·K) [52], and graphene oxide (0.79 W/m·K) [53]. Such remarkable enhancement in out-of-plane direction is mainly attributed to the continuous heat-transfer pathways and the synergistic effect of Cu wires with GrFs. In our work, the heat transfer pathways were constructed through weaving Cu wires through fabric pores and laminating GrFs onto surface of hybrid composites, and the high continuity of CF fabrics were still well maintained to realize high-efficiency load transferring, showing high structural and functional integration for the hybrid sandwich composites.

4. Conclusion

In order to improve out-of-plane thermal conductivity of CF-laminated composites with maintaining CF continuity, a novel strategy of vertically-weaving Cu wires through CF fabric pores and laminating GrFs onto surfaces has been developed to construct high-efficiency continuous pathways for rapid heat transfer. The GrF/Cu-CF/GrF sandwich composites was prepared using VARTM technique, and their microstructure, electrical conductivity, and thermal conductivity in both in-plane and out-of-plane directions were investigated in detail. We found that the Cu wires and GrFs could greatly improve the thermal conductivity of CFRP, especially in the out-of-plane direction, increasing by 104% from 0.539 to 1.097 W/m·K. Such remarkable enhancement is mainly attribute to the continuous heat-transfer pathway and the synergetic effect of Cu wires with GrFs. Finite element analysis and heat conduction mechanism also revealed the high-efficiency heat transfer and diffusion. There is great potential for the hybrid sandwich composites to be utilized as high-performance composites with structural and functional integration.

Declarations
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Conflict of interest The authors declare no competing interests.

References


Figures
Figure 1

(a) Schematic on fabrication of the hybrid sandwich composites. (b-e) SEM images of fracture morphology of composites
Figure 2

Electrical conductivity of composites in (a) in-plane and (b) out-of-plane directions.
Figure 3

Thermal conductivity of composites in (a) in-plane and (b) out-of-plane directions, and thermal diffusivity of components in (c) in-plane and (d) out-of-plane directions. Finite element analysis models of composites with (e-g) various structures, and the corresponding temperature distribution in (h-j) in-plane and (k-m) out-of-plane directions at certain time during heating process.
Figure 4

Schematic illustration of heat transfer in the hybrid sandwich composites