RESEARCH ARTICLE

Through Plane Networked Graphene Oxide/Polyester Hybrid Thermal Interface Material for Heat Management Applications

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ABSTRACT
The role of electronic devices in our lives is increasing rapidly, with more research focusing on miniaturization, creating more demand for thermal interface materials (TIM). Grease-based TIM presently available have good thermal conductivity values, but issues such as contamination, pump-out, and an additional curing step are observed. Fibrous textile substrates are soft and flexible, making them suitable for occupying the asperities between the heat sink and heat-producing devices. However, they are insulating in nature and can be made conductive using conductive fillers such as graphene oxide (GO). In this article, a networked through-plane thermally conductive TIM using the cutting waste of polyester and GO was fabricated. The methodology involved functionalizing the PET substrate and studying its interaction with GO. A networked GO/PET, (N-GOPET) hybrid TIM was fabricated from waste PET with good through-plane heat conduction performance, softness, and cuttability as a promising replacement for grease-based TIM.

1. Introduction

Electronic devices play a significant role in our lives and are likely to play an even greater role in the future, given their wide range of applications in every sector [1, 2]. The failure factor of an electronic device increases exponentially with an increase in temperature; hence, the thermal management process is vital to ensure its proper functioning and lifecycle [3, 4]. There are various methods to remove heat from the devices, including using cooling pipes, thermoelectric coolers and heat sink [5]. Among these methods, a combination of a heat sink and cooling fan is the most effective and facile method for heat management applications [1, 6]. The heat from the device is removed by attaching a heat sink; however, due to the solid contact, air pockets exist, this inhibits heat transfer since air is a poor conductor of heat [7, 8]. Thermal interface materials (TIM) fill the air gaps and reduce the interfacial resistance between the heat sink and the heat-producing device, ensuring high heat dissipation to the atmosphere. The continuous rising interest in miniaturization, yet powerful devices are creating more demand for efficient and high conductive TIMs.

The most common and widely used TIM are grease-based, which have issues related to contamination, pump-out, uniform spreading, curing requirement and maintenance [1, 9]. A considerable amount of research in the electronic packaging industries is focused on developing pad-based thermal interface materials using carbon-based fillers such as graphene, graphene oxide, carbon nanotubes (CNTs), carbon fibers (CF) with excellent in-plane and through-plane thermal conductivity [1, 7, 9]. The through-plane network is vital to remove the heat from the device to the heat sink, whereas the in-plane thermal conductivity is important to remove the hot spots [10–12]. The investigation reported in previous studies is good from academic prospects; however, it has little practical applicability owing to
the complexity of the process which limits its large-scale manufacturing. These processes include the alignment of conductive fillers in the vertical direction, which requires an additional step of a magnetic field, electric field, chemical vapor deposition (CVD), or freeze-drying techniques [1, 13, 14]. Among carbon-based fillers, graphene derivatives have emerged as the rising star for fabricating TIM, considering its outstanding thermal conductivity, low coefficient of thermal expansion (CTE), low contact resistance, high surface area, and excellent mechanical properties [15]. However, it has certain limitations such as electrical conductivity, poor aqueous dispersibility and agglomeration. Among different graphene derivatives, graphene oxide is widely used as a filler due to its insulating properties, aqueous dispersibility and surface functional groups, which help to improve interfacial bonding between the filler and the matrix [16–18].

Textile substrates are flexible and soft and are suitable to fill the gap between the heat sink and heat-producing components. However, textile fibers mostly made up of cotton and polyester, are thermally insulating, making them unsuitable for the purpose. Coating textile-based materials with highly conductive materials such as graphene seem to be a promising method to make it conductive, as reported by many previous works [19, 20]. A summary of textile based TIM is given in Table 1. Concerns related to textile waste are also rising, which can be separated into two categories: pre-consumer waste and post-consumer waste. The pre-consumer waste is generally the cutting waste (leftover fabric after giving structure through cutting) from the garment industries, contributing to around 25% of textile waste [21]. Most of the post-consumer fabrics are recycled since they have a stable structure. Polyester is the most popular textile material, accounting for more than 80% of synthetic textiles with personal and industrial applications. Polyester is a non-biodegradable textile which causes a significant environmental burden [22, 23]. A thermal interface material combining the cutting waste of non-biodegradable polyester nonwoven fabrics with graphene oxide would be very beneficial from a sustainability point of view. These materials can be replaced and used repetitively, minimizing pre-consumer waste from the textile industry.

In this article, a hybrid TIM using a combination of PET waste fabric and GO with a through-plane conductive network was fabricated using a facile hot press machine, and coating GO using a conventional brush coating technique. The fabricated TIM was evaluated using a self-made chip stack heat transfer arrangement for heat management performance. The crystal structure, functional groups, and morphology were evaluated using XRD, FTIR, and SEM to confirm the bonding between GO and PET and through-plane GO conductive network. The hybrid pad-based TIM fabricated in this study can be used for replacing grease-based TIM for commercial applications.

2. Experimental

2.1 Materials

All chemicals, including sodium hydroxide (NaOH), 98% sulfuric acid (H$_2$SO$_4$), hydrochloric acid (HCl), potassium permanganate (KMnO$_4$), sodium nitrate (NaNO$_3$), 60% hydrogen peroxide (H$_2$O$_2$) and graphite flakes were purchased from Merck assay. Cutting waste of PET fabrics was procured from the fabric cutting workshop and was shaped into 0.15 cm width fabric 3 mm thick strips using a surgical blade. The fabrics were cleaned and alkali functionalized according to the protocol followed in our previous study [16]. Commercially available TIM (Arctic silver 5 3.5 G and HY510) grease was purchased from local departmental stores.

2.2 Methods

Graphene oxide was prepared using a modified Hummer’s method following the same procedure given in our previous study [17]. To fabricate hybrid GO/PET TIM, the cleaned fabric strips were brush coated with 3 mg l$^{-1}$ GO solution followed by drying in an oven at 80°C for three cycles, after which the coated fabric was rolled up and pressed using a hot-pressing machine for 5 mins at 100°C as
represented schematically in Figure 1 to form a conductive network in the through-plane direction. The PET fabrics were first treated with alkali before coating with GO and were labeled as AFPET for alkali functionalized PET, and after GO coating was named as AFPETGO. The reason for selecting GO instead of rGO is to avoid the problems associated with the electrical conductivity, such as flicker noise and short circuits. After hot pressing, the top and bottom layer was again coated with GO solution for three cycles. The GO solution was sonicated for 10 mins before coating for homogenous dispersion and interlayer separation. The sample was named N-GOPET for networked graphene oxide-coated polyester. For comparison, the fabric strip was also dip-coated and hot-pressed and named D-GOPET for dispersed graphene oxide polyester. All the samples were weighed before and after coating in order to calculate the percentage uptake given by Eq. (1).

\[
\text{\% uptake} = \frac{\text{final weight} - \text{initial weight}}{\text{initial weight}} \times 100
\] (1)

2.3 Characterization techniques

The functional groups of the GO synthesized and the interaction between polyester substrate were analyzed via Fourier-transform infrared (FTIR) spectroscopy using a Spectrum one FTIR spectroscopy (Perkin Elmer). A transmittance mode was adopted to generate the FTIR spectra that were recorded over the wavenumber range of 4000–550 cm\(^{-1}\). The morphological properties of the hybrid composite were investigated via scanning electron microscopy (Hitachi 3000). The thermal conductivity of the steel plate was analyzed using the Hot Disk Thermal Constant Analyzer model (TPS 2500).

Thermal performance of the hybrid materials fabricated and two grease-based commercially available TIM (Arctic silver 5 3.5 G and HY510 grease available at a local market) for comparison was checked using a self-made experimental setup resembling a chip-stack electronic configuration as shown in Figure 2. The experimental setup includes a thermal scanner; thermocouples were used with microcontroller programming for temperature measurement. The setup was made using two 1.5 cm diameter cylindrical stainless-steel blocks on which a thermocouple was attached, which was programmed to take three temperature readings per second. The thermal conductivity of the stainless-steel blocks was evaluated using Hot Disk Thermal Constant Analyzer and was found to be around 15.81 Wm\(^{-1}\)K\(^{-1}\) which was much higher than the expected values of the TIM samples. During the testing, the bottom metal block was heated at 90°C using a hot plate and was allowed for equilibrium to reach, after which the TIM materials and top metal block were placed. Downward pressure was applied on the top block using a 0.8 kg metal block insulated at the contact surface using a 1-cm-thick glass wool to prevent heat conduction from the top plate to metal weight. The constant heat maintained at 90°C was continuously supplied to the bottom block throughout the test and room
temperature, and humidity was strictly maintained at 27°C and 40%, respectively. The top and bottom plate temperature profile was obtained until equilibrium was reached to evaluate the thermal performance in practical sense.

3. Results and discussion

The synthesis of GO was confirmed using XRD analysis as shown in Figure 3(a). The peak at 2θ = 10.2° corresponds to the interlayer distance of 0.86 nm which is much higher than that of graphite (0.34 nm). This might be due to the presence of hydroxy, carboxyl, carbonyl and epoxy functional groups [24]. The presence of other broad peaks at 2θ = 18°, 29° and 42° are due to the incomplete oxidation of graphite sheets and the short range of graphene-like sheets [25, 26]. The synthesized GO and alkali functionalized PET strips with GO coating (AFPETGO) were further characterized for functional group analysis as given in Figure 3(b).

The FTIR spectrum of GO shows the successful introduction of oxygen functional groups with various oxygen functional peaks. The broad peak at 3414 cm\(^{-1}\) can be attributed to the stretching vibrations of the hydroxyl groups (O–H) arising due to the moisture. The peak at 1722 cm\(^{-1}\) is the result of carboxy and carbonyl functional groups (C=O). The peak at 1626 cm\(^{-1}\) can be attributed to the stretching mode of aromatic unoxidized graphite (C=C). The peak at 1398 cm\(^{-1}\) is the result of the

![Figure 2](image2.png)  
**Figure 2.** Schematic of the chip stack electronic configuration for evaluating heat management performance.

![Figure 3](image3.png)  
**Figure 3.** (a) XRD pattern of GO (b) FTIR spectrum of AFPET, AFPETGO and GO.
deformation vibration of tertiary C–OH groups. The peak at 1039 cm\(^{-1}\) belongs to the stretching vibration of C–O functional groups (alkoxy and carboxyl functional groups). These results are in agreement with previous works on graphene oxide synthesis [27]. The alkali treatment is used to improve the adhesion between GO and PET substrate as well as nanoparticle uptake as reported in our previous work [16]. The FTIR spectrum of AFPET showed a strong peak at 1714 cm\(^{-1}\) which can be assigned to the carboxyl functional group (C=O), whereas the broad peak at 3,450 cm\(^{-1}\) is due to the hydroxyl functional groups (O–H). The peak at 1410 and 1341 cm\(^{-1}\) corresponds to the aromatic and carboxylic ester functional (C–O–C) groups, respectively. The peak at 873 cm\(^{-1}\) arises due to the hydrogen atoms of the benzene ring. The peak at 700 cm\(^{-1}\) is a result of the C–H vibration of the aromatic ring [28, 29]. The FTIR spectrum after GO coating showed significant differences with disappearance and changes in various peak intensities. The peak disappearance at 1,722 cm\(^{-1}\) (C = O) and 3,414 cm\(^{-1}\) (O–H) can result from ester bond formation between GO and PET. The ester bond formation can be understood based on bond polarity differences between the hydroxy and carboxy functional groups. These results agree with previous works that indicated that surface carboxylic groups play a key role in bonding with coating materials [28, 30]. Besides this, the peaks in the GO spectrum such as peak at 1625 and 1396 cm\(^{-1}\) disappeared, whereas there was a significant reduction in the peak intensity at 700 cm\(^{-1}\) indicating strong interaction of GO with AFPET fabric [31].

The networked graphene oxide/polyester (N-GOPET) hybrid TIM was fabricated by the procedure given in Section 2.2. The graphene oxide uptake for both N-GOPET and dispersed graphene oxide/polyester (D-GOPET) was maintained to be the same (40 wt%). In the real system, it is vital to have a through-plane conductive network to transfer the heat from the heat-producing device to the heat sink and from the heat sink to the ambient atmosphere. The performance of TIM depends on various factors, including contact mechanics, tribology, rheology, heat transport, and percolation which is much difficult to evaluate in a practical sense [9, 32]. Hence, a much easier and more practical way to assess the TIM performance is by imitating a real-time system and plotting a graph of temperature difference vs time which will give an idea of whether the TIM materials can bring thermal equilibrium to stop the device from overheating. For that, a self-made thermocouple-based chip-stack setup was made using stainless steel blocks and a heat plate, as described in Section 2. The most common test to evaluate the TIM performance is to install it in real-time computer system and check the temperature of the heat sink and chipset using thermal infrared cameras [33]. Recently, chip stack structure has become more famous considering the simplicity of the setup with fairly accurate measurements to give an idea about the real-time TIM performance [34]. Generally, a heat conduction setup is made up of two copper columns between which TIM was sandwiched, as reported by Song et al. [35]. The heating coils were used to increase the temperature to 100°C and the temperature was measured using a thermocouple attached to the recorder. The temperature difference between the two plates was measured to determine the thermal performance. A similar type of setup was reported, but thermal infrared cameras were used instead of a thermocouple to determine the temperature of the top and bottom plates in a study by Zahid et al. [36].

The currently available methods to determine the thermal conductivity of hybrid materials are not suitable since the presently available machines give bulk conductivity values, whereas a hybrid material has different conductivity values in different directions and particularly for a material to be used in TIM is designed to have a good thermal conductivity in through-plane direction. This setup helps to determine the heat transport properties from the bottom plate to top plate, which is a measure of heat transport in the through plane direction which is vital to reduce the device temperature. Comparing the real-time thermal performance of N-GOPET TIM with that of the commercially available TIM can give an estimate of the thermal conductivity and TIM performance rather than measuring bulk thermal conductivity [36]. Hence, the top and bottom plate temperature profiles were taken using a thermocouple and plotted vs time for N-GOPET, D-GOPET, pure polyester fabric, arctic silver, and thermal grease as given in Figure 4(a). It can be seen from the graph that the bottom block temperature decreases, whereas the top steel block increases, which is due to heat transfer between the bottom block to top block. TIM was sandwiched between the two blocks; hence, heat is transferred through the TIM,
Figure 4. (a) Temperature profiles of top and bottom plate for various TIM. (b) Temperature difference vs time plot for various TIM.

so TIM’s performance can be evaluated. Arctic silver (\(k = 9.4 \text{ Wm}^{-1}\text{K}^{-1}\)) was performing best compared to other materials in bringing the equilibrium temperature within a noticeably short period of time. The N-GOPET was performing similar to the commercial thermal grease (\(k = 1.9 \text{ Wm}^{-1}\text{K}^{-1}\)).

The top and bottom plate temperature difference is expected to be lower for TIM with excellent thermal conductivity. Hence, the temperature difference (\(\Delta T\)) vs time was also plotted for clarity in the results obtained, as shown in Figure 4(b). The highest temperature difference was found in that of pure polyester, followed by D-PETGO, indicating its inability to transfer heat. Although the GO loading was the same between the N-GOPET and D-GOPET, there was marginal difference between the heat conduction performance, indicating the coating method’s influence, which helps to form a conductive network in the through-plane direction. The crystal structure plays a major role in the heat transfer mechanism. The more organized crystal structure in the vertical direction leads to effective vibration and hence transport of phonons leading to excellent heat transport in the through-plane direction [1]. This result is consistent with many previous studies that reported the influence of filler orientation in forming the conductive network to ensure effective heat conduction [37, 38].

The temperature difference and time to reach equilibrium were the same for grease and N-GOPET TIM. Artic silver has outperformed all other TIM with the lowest temperature difference within just 50 seconds. The time needed to reach the equilibrium for thermal grease and N-GOPET was almost 100 seconds, whereas D-GOPET and pure polyester took almost 220 and 300 seconds, respectively. The testing was performed for longer duration; however, after equilibrium, there was a marginal difference of \(\pm 2\text{°C}\). The results shown in Figure 4, using a chip-stack setup, demonstrate that the N-GOPET has conductive networks in the through-plane direction.

The SEM images for N-GOPET, D-GOPET and polyester fabrics were taken in order to confirm the morphology and adhesion of GO particles, as given in Figure 5. A curved network (highlighted in yellow) of graphene coating is visible in the enlarged SEM image of Figure 5 (ai). The curved network is visible due to the presence of a through-plane circular network created through the fabrication process described in Section 2. To further evaluate the direction of flow, the samples vertical section was taken and mounted for the SEM imaging as shown in Figure 5 (aii). The presence of a through-plane 3-D network can be seen in the vertical direction of heat flow in the through-plane direction, indicated by a red arrow. The vertical image was further magnified, and a yellow line was drawn to define the GO network and fibers. The 3-D GO network is responsible for efficient flow rate from the bottom steel plate to the top steel plate since it offers uniform networked transport of phonons as compared to random fibers present in the D-GOPET sample. The TIM fabricated by dispersing GO in the substrate (D-GOPET sample) does not have conductive networks; however, the GO flakes are clearly visible dispersed in the substrate as shown in
Figure 5(b). Morphology of polyester fibers without any fillers; hence, no conductive network can be observed in Figure 5(c), which aligns with the thermal conduction results obtained in Figure 4. The reason for the difference in the heat transfer performance between D-GOPET and N-GOPET could be due to the fact that the heat transfer becomes extremely slow due to the matrix interaction since the non-crystalline polymer structure makes thermal conduction difficult as the vibration is disordered compared to the crystalline structure in which there is rapid movement of phonons through ordered vibrations [13, 32]. The presence of a conductive network in the through-plane direction in the N-GOPET sample causes rapid heat transfer with minimal contact with the substrate leading to better heat management performance [8, 12, 39, 40].

The fundamental reason for using TIM is to fill in the asperities that exist between the two solid contacts to ensure efficient heat conduction between the heat sink and heat-producing devices. Hence, it is essential that the fabricated TIM shows flexibility and softness [9]. Figure 6(a) demonstrates the flexibility of N-GOPET TIM, which is obvious considering the use of the fibrous textile substrate. The fabricated TIM should also be able to form different shapes to ensure its usability in devices with different shapes and sizes. The cuttability of N-GOPET TIM into different shapes is demonstrated in Figure 6(b), indicating its usability in devices of different shapes and sizes. The results suggest that an N-GOPET-based TIM can be a potential replacement for commercially available grease-based TIM overcoming its limitations in terms of performance and reusability.

4. Conclusion

A sustainable networked graphene oxide/polyester-based N-GOPET hybrid thermal interface material was fabricated using the cutting waste of nonwoven polyester and graphene oxide. The heat
management performance was evaluated and compared with commercially available TIM using a chip stack setup, and it showed similar performance to that of the grease-based TIM widely used. The networked structure (N-GOPET) showed significant improvement for transferring the heat in the through-plane direction compared with non-networked TIM (D-GOPET). The N-GOPET TIM is a pad-based material with softness, flexibility, cuttability, and good heat transfer performance and hence is a possible replacement for grease-based TIM.

**Acknowledgements**

We are very grateful to the Malaysian Ministry of Higher Education for awarding us a Fundamental Research Grant Scheme (FRGS/1/2021/TK0/USM/01/4) and Universiti Sains Malaysia for making this study possible.

**Disclosure statement**

No potential conflict of interest was reported by the author(s).

**Funding**

This work was supported by the Malaysian Ministry of Higher Education [FRGS/1/2021/TK0/USM/01/4].

**Declaration of interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors declare the following financial interests/personal relationships, which may be considered as potential competing interests.

**Author’s contribution**

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Junaid Khan and Mariatti Jaafar. The first draft of the manuscript was written by Junaid Khan and the corresponding author Mariatti Jaafar commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Conceptualization: Junaid Khan and Mariatti Jaafar; Methodology: Junaid Khan and Mariatti Jaafar; Formal analysis and investigation: Junaid Khan Writing – original draft preparation: Junaid Khan; Writing – review and editing: Mariatti Jaafar; Funding acquisition: Mariatti Jaafar; Supervision: Mariatti Jaafar.

**Data availability statement**

The data that support the findings of this study are available upon reasonable request from the authors.

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**Table 1. Summary of textile-based thermal interface materials and their thermal conductivity**

| Sr. No. | Substrate                        | Filler                   | Thermal conductivity (W m\(^{-1}\)K\(^{-1}\)) | Reference |
|--------|----------------------------------|--------------------------|-----------------------------------------------|-----------|
| 1      | Recycled cotton                  | Graphene Nanoplatelets   | 5.5                                           | [36]      |
| 2      | Bulgyeong traditional paper      | Graphene                | 6.78                                          | [41]      |
| 3      | Wool paper                       | Graphene flakes         | 5.87 (in plane)                              | [41]      |
| 4      | Aqua satin                       | Graphene flakes         | 4.35 (in plane)                              | [41]      |
| 5      | Merit paper                      | Graphene flakes         | 4.8 (in plane)                               | [41]      |
| 6      | New craft board                  | Graphene flakes         | 5.71 (in plane)                              | [41]      |
| 7      | Dearye traditional paper         | Graphene flakes         | 6.32 (in plane)                              | [41]      |
| 8      | Polymeric fabric                 | GO and rGO             | 1204                                          | [34]      |
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