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Thermal Expansion of Graphene Composites

Shiren Wang,*[†] Madhava Tambraparni,[†] Jingjing Qiu,[‡] John Tipton,[§] and Derrick Dean[§]

[†]*Department of Industrial Engineering and* [‡]*Department of Chemical Engineering, Texas Tech University, Lubbock, Texas 79409, and* [§]*Department of Materials Science and Engineering, University of Alabama, Birmingham, Alabama 35294*

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ABSTRACT: Isolated graphene sheets were achieved by graphite intercalation and charge-induced exfoliation. The resultant graphene oxide sheets were incorporated into polymer composites and thermal expansion was investigated by a thermo-mechanical analyzer. The test results indicated that inclusion of graphene into composites resulted in low coefficients of thermal expansion (CTEs), and increasing graphene fraction reduced CTEs more significantly. The 5 wt % graphene oxide-based composite shows 31.7% reduction below the glass transition temperature. Preliminary measurement of thermal conductivity also indicated that graphene composites significantly improved the thermal conductivity of polymer matrix. Thermal conductivity of 5% graphene composites showed about 4-fold increment in comparison to the polymer matrix. This finding will provide a solid foundation for graphene-enabled thermal management in microelectronics.

Introduction

As electronic products continue to move toward miniaturization and high-performance, one of the most important challenges is significantly increasing power dissipation.[1,2] If it is not solved, the power dissipated in the form of heat will significantly slow down the development cycle time of new technology. Even a small increase in temperature reduces the device lifetime. A large rise in device temperature can lead to the fracture, delamination, melting, creep, electro-migration and even burning of packing materials.[3−5] Consequently, thermal interface materials are becoming an increasingly critical issue in the electronic device. Thermal interface materials are used to connect different thermal elements and ensure efficient transfers of heat. They can be inserted between a chip and a heat sink or between a heat sink and a heat spreader in the electronic device.[6]

Current thermal interface materials are mostly based on phase-change-materials or hybrid materials, including polymers, greases, or adhesives filled with thermal conductive particles like silver, zinc oxide, etc.[7−12] However, they are not able to meet the future requirements in thermal management due to their low thermal conductivity and other physical characteristics.[7−12]

Recently, graphene has attracted a great deal of attention. Graphene is a monolayer of carbon atoms, arranged in a honeycomb network or an unrolled single-walled carbon nanotube.[13−16] Graphene-filled polymers are considered to be very promising candidates for the high-performance thermal interface materials and expected to solve the challenge of increasing heat dissipation from electronic devices. Researchers at Columbia University have recently measured the mechanical properties of a single graphene layer by nanoindentation, indicating that Young's modulus is ∼1 TPa, and strength 130 GPa.[17] Others measured the thermal conductivity of a graphene layer, indicating its thermal conductivity to be in the range of ∼3080−5150 $W\cdot m^{-1}\cdot K^{-1}$.[18,19] This extremely high thermal conductivity is comparable to current carbon nanotubes.[20,21] Some computational results suggested that in-plane expansion, bond stretching, and bond bending effects in the graphene sheet cancel each other out, leading to a negative thermal expansion coefficient in the plane graphene sheets below the temperature of 470 K.[22,23] Due to its planar structure, thermal contraction in graphene sheets is more obvious than in other carbon structures, such as graphite, carbon nanotubes and diamond.[24,25] Recent studies suggest that the production costs for graphene in large quantities are much lower than for carbon nanotubes.[26,27] Currently, graphene sheets can be attained by graphite exfoliation, including mechanical cleavage of graphite,[16,28−31] chemical exfoliation of graphite,[32−40] thermal-induced exfoliation,[41,42] and direct synthesis, such as epitaxial growth,[43−47] and bottom-up organic synthesis.[48,49] Some attempts have been made to investigate the thermal conductivity of graphene-based polymer composites. Yu et al. used few-layer graphene sheets to explore epoxy polymer based thermal interface materials. They found that adding ∼25 vol % graphite nanoplatelets resulted in a thermal conductivity of 6.44$W\cdot m^{-1}\cdot K^{-1}$.[9,50] Ganguli et al. used silane to treat graphene oxide and dispersed the functionalized graphene into epoxy resin, and found that addition of 20 wt % graphene resulted in a thermal conductivity of 5.8 $W\cdot m^{-1}\cdot K^{-1}$.[51] Xie et al. theoretically studied the effect of graphene percolation on thermal conductivity, suggesting 2D graphene nanosheets more effectively enhance thermal conductivity than 1D carbon nanotubes.[52]

Since thermal interface materials are used to connect different thermal elements for thermal management, it is important to minimize thermal contact resistance for effective heat transfer. Thermal contact resistance mostly derives from the interface separation yielded by mismatched thermal expansion.[1,11] In addition, mismatched thermal expansion also results in thermal stress, which usually leads to the failure of electronic devices.[1,11] The thermal stress and disjointing thermal interface materials from a chip or a heat spreader will generate many local gaps. These local gaps will significantly increase the contact resistance and reduce the effective heat dissipation. Locally accumulated heat, if not distributed timely, will burn the device and lead to the device failure. Therefore, it is crucial to tune the CTE of this novel

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*Corresponding author. E-mail: shiren.wang@ttu.edu.

thermal interface material for desired thermal management. In this paper, the thermal expansion in graphene composites is investigated and the coefficients of thermal expansion (CTEs) are discussed.

Experimental Section

Graphene Preparation. Graphite (Nature flake graphite, sized at 45 μm, grade 230), was kindly provided by Asbury Carbons (Asbury, NJ). Sodium chlorate, nitric acid, and hydrochloric acid were purchased from Fisher Scientific Inc. Graphite was expanded to exfoliated graphene according to the modified Brodie's method. First, 10 g of graphite, 160 mL of nitric acid, and 85 g of sodium chlorate were mixed at room temperature. The mixture was kept for 24 h under stirring. Then it was washed with 5 × 200 mL of 5% hydrochloric acid and 7 × 1 L of distilled water. When the acidic and saline impurities were removed, the exfoliated graphene was achieved through sedimentation and finally dried at 60 °C. Graphene oxide (GO) was further exfoliated by ultrasonic processing and stabilized by ammonia according to the literature.[14] Introducing ammonia into solution will afford additional negative charge and help form a stable colloid. Filtration of exfoliated GO suspension resulted in solid samples of GO sheets. Volatile ammonia was removed when the film was placed into oven at 60 °C for 2 h.

Nanocomposite Preparation. Epoxy resin (brand Epon862) was provided by the Hexion Company. The resultant exfoliated GO was ground to paste in a small amount of acetone. A certain curing agent (based on the required curing ratio) was weighed and added to the GO paste. Subsequently, GO and curing agent were mixed in the acetone solvent under cup-horn ultrasonic processing at 30 W for $^1/_2$ h. The mixture of GO and curing agent was then transferred to the diluted epoxy resin, resulting in a new mixture which was processed with cup-horn ultrasonic processing at the power of 12 W for 3 h. Finally, the mixture was placed under high-speed shearing for 10 h. The resultant mixture of GO and epoxy resin were left in the vacuum system for 12 h to remove the residual solvent. The final mixture was cast into a metallic mold and cured under hot-press at 177 °C for 2.5 h. The subsequent postcure was conducted at 177 °C for another 2 h. The graphite-modified epoxy resin was prepared in the same procedure. Graphite and SWNT follow the same procedures used to prepare the composites. Filler fractions of 1% and 5% by weight, respectively, were prepared.

Structure and Property Characterization. The exfoliated GO was dispersed onto silicon wafer and characterized with atomic force microscope (AFM, Veeco Inc., Nanoscope III) at tapping model. Surface characteristics of GO was characterized by FT-IR. Thermal conductivity of composites was measured by TCI instrument(C-thermal Inc.) and thermal expansion behavior of nanocomposites was characterized by thermo mechanical analyzer (TMA Q400, TA Inc.). Nanocomposite morphology was further characterized by scanning electron microscope (SEM, Hitachi S-4300 VP).

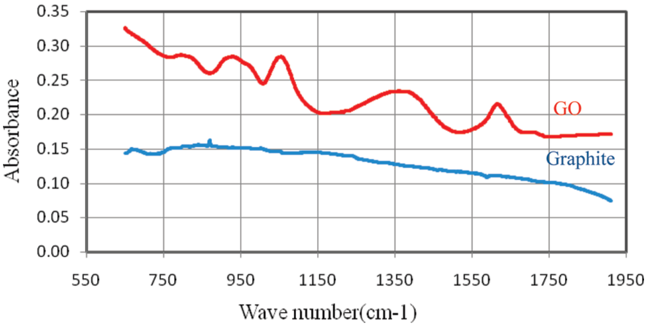

Figure 1. FT-IR of GO and graphite materials.

Results and Discussions

Graphite and GO materials were also characterized by FT-IR, and the results are shown in Figure 1. For the graphite spectrum, almost no vibration peak is observed except the vibration from carbon bonding. Several vibration peaks were observed in the GO spectrum. The absorption peaks at 801.6, 928.1, and 1054.7 cm^{-1} are contributed from epoxy group.[53−55] The vibration peak at 1360 cm^{-1} is contributed from C−OH stretching.[34,56] The sharp peak at 1615.3 cm^{-1} should be credited to the water absorption. These vibration peaks verified the successful attachment of epoxide and hydroxyl.

The GO were readily exfoliated by ultrasonic processing in water and stabilized by negative charge as described by the literature.[14] The electrostatic repulsion keeps GO isolated and results in a stable colloid. The aqueous solution of exfoliated graphene sheets was dropped onto silicon substrate and characterized by atomic force microscope (AFM). The thickness of suspended graphene sheets were measured by section analysis and the statistical results of layer thickness are shown in Figure 2. The measurement indicated most of GO sheets are less or around 1 nm thick, suggesting that monolayer graphene was achieved. This result agrees with the literature regarding monolayer graphene.[5,14,15,42] Generally, the thickness of single-layer graphene is 1 nm or less in AFM images due to the following reasons: attaching molecules on the graphene surface, imperfect interface

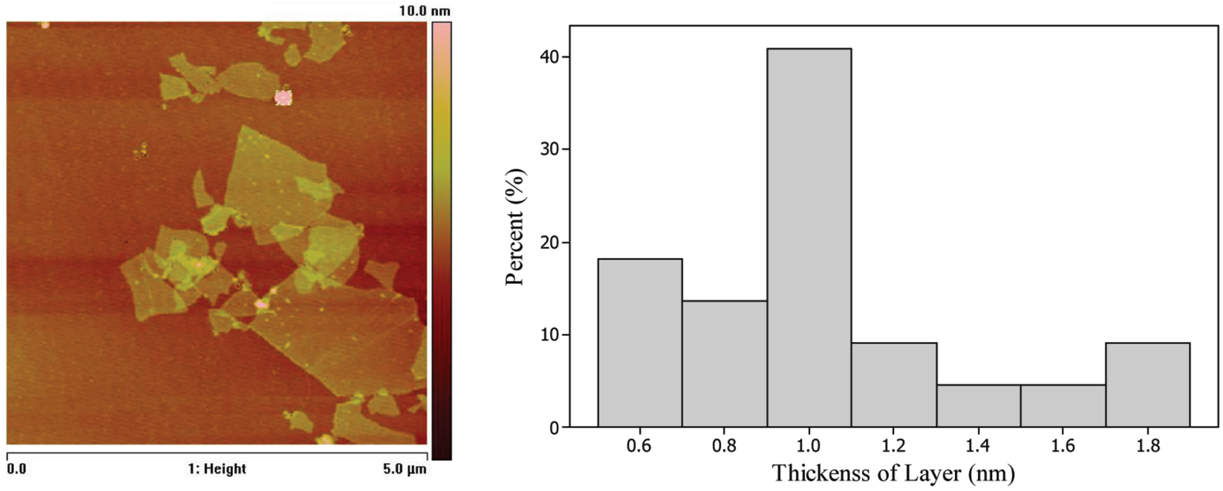

Figure 2. (a) Tapping-mode AFM image of exfoliated graphene. (b) Statistical results of GO sheet thickness.

between graphene and silicon substrate, and possible different attraction force between AFM probes as compared to the silicon substrate.[5,14,15,42] Even though some layers were more than 1 nm thick, their thickness is up to 2 nm, indicating they are in a state of two or three layers. Therefore, GO sheets were exfoliated to monolayer or few-layer state.

GO were dispersed into epoxy resin and molded into 1 in. × 1 in. squared sample with 5 mm thickness by a hot-press. The thermal conductivity of the composite samples was characterized by TCI instrument by using distill water as contact agent, and the results were given in Figure 3.

Epoxy resin shows very poor thermal conductivity, but inclusion of graphene sheets significantly changed this property. Incorporation of 1 wt % GO into epoxy resins showed similar effect on improving thermal conductivity as filling 1 wt % SWNT did. 5 wt % GO-filled epoxy resin showed a thermal conductivity close to 1 W/m·K, more than 4-fold increase in comparison to the neat resin. These results are consistent with the reported results.[9,50] In addition, it was reported that filling 20% GO can increase the thermal conductivity as high as 6.44 W/m·K.[9,50] These results suggested that graphene composite is a very promising thermal interface material for heat dissipation.

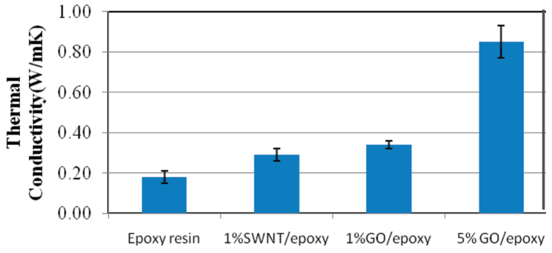

Figure 3. Thermal conductivity of the graphene composites.

The thermal expansions of the epoxy resin, graphite/epoxy, GO/epoxy, and SWNT/epoxy composites were tested by TMA Q400 at ramp rate 10 °C/min, and the results are shown in the Figure 4.

Obviously, these nanocomposites demonstrate different thermal expansion behavior since they show quite different CTEs. Three samples for each material were tested, and the CTEs and glass transition temperature (T_g) were summarized in the Table 1.

The neat epoxy resin shows the CTE around 8.2×10^{-5}/°C, while inclusion of graphite into epoxy resin did not show obvious reduction. This may stem from the fact that graphite has a positive CTE.[22-25] Incorporating 1 wt % GO or SWNTs into resins showed obvious effect on CTE reduction. This may accredit to their negative CTEs. Both computational and experimental researches suggested that CTEs of SWNT is = -1.5×10^{-6} /°C,[23,57-60] and integration of 1 wt % SWNTs into polymer resins results in obvious CTE decrease. For graphene, the computations results indicated that the in-plane CTE is also negative below 470 °C.[22,23] Therefore, graphene composites showed similar effect as SWNTs on bulk CTEs below T_g. When 5 wt % GO sheets were incorporated into epoxy resin, more significant reduction of CTEs below T_g was observed. The reduction is as high as 31.7%. At above glass transition temperature (T_g), CTEs of the composites showed slight variation in comparison to the neat resin, but large reduction was not observed.

The T_g was determined by the dramatic turn point on the thermal expansion curve. Inclusion of 1 wt % SWNT slightly reduced the T_g of epoxy resin, while some literature reported either a positive[61-63] or a negative effect[64-67] or no effect.[68]

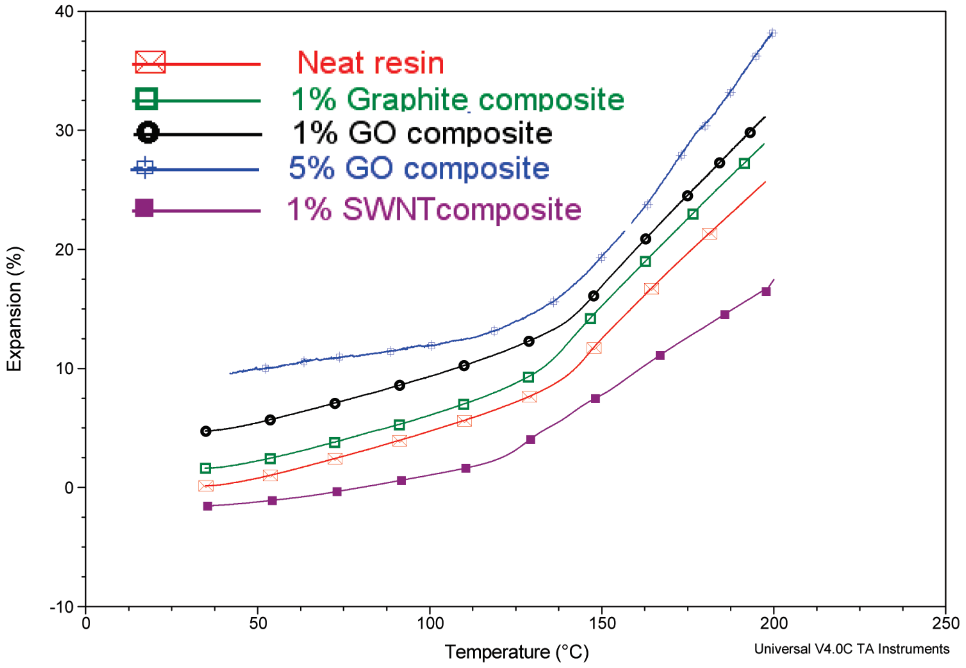

Figure 4. Thermal expansion of graphene composites.

Table 1. Summarized Results of Thermal Expansions of Nanocomposites

	CTE below T_g ($\times 10^{-5}$/°C)	CTE above T_g ($\times 10^{-5}$/°C)	T_g (°C)
epoxy resin	8.2 ± 0.2	28.2 ± 0.4	136.2 ± 1.8
1 wt % graphite/epoxy	7.7 ± 0.1	28.9 ± 0.6	135.3 ± 0.8
1 wt % GO/epoxy	7.2 ± 0.6	30.4 ± 2.5	140.0 ± 2.1
5 wt % GO/Epoxy	5.6 ± 0.7	31.1 ± 3.7	136.0 ± 1.9
1 wt % SWNT/epoxy	6.0 ± 0.6	28.1 ± 4.2	131.5 ± 3.5

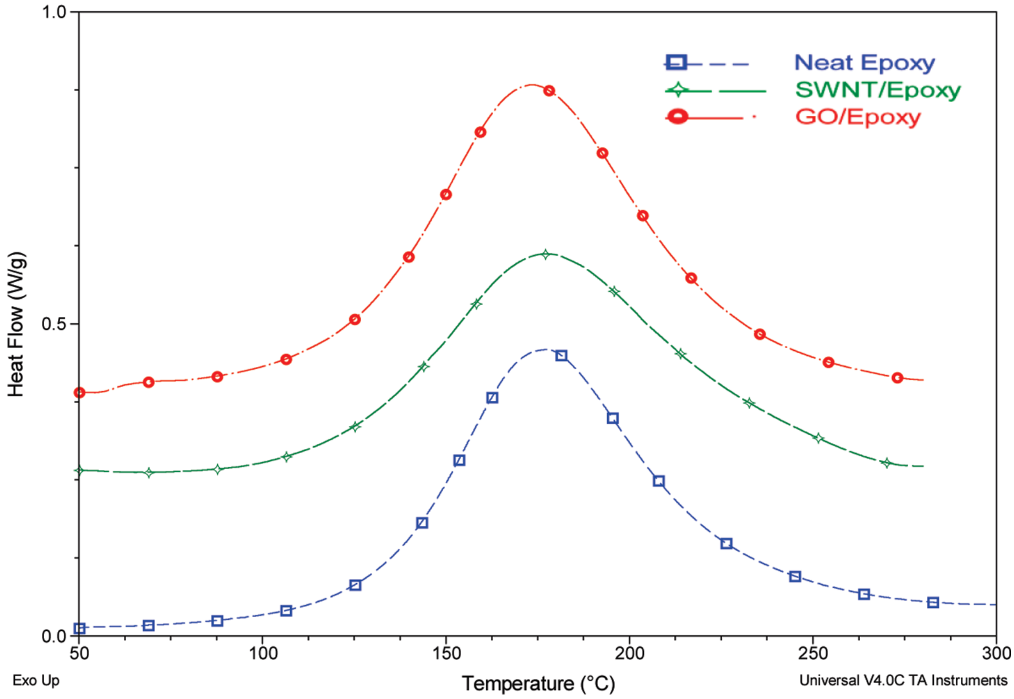

Figure 5. Ramping DSC curves of GO/epoxy system.

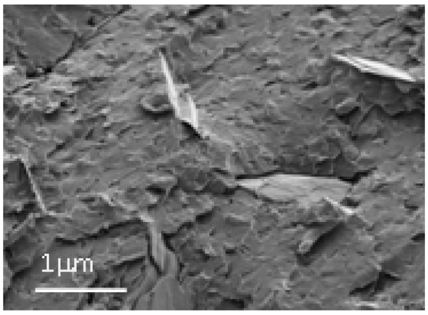

Figure 6. SEM image of the composite cross-section.

SWNT generally is at the scale of epoxy molecules and interference with epoxy curing reaction, resulting in low reaction conversion. This generally reduces the polymer cross-linkage and increase the polymer chain mobility. On the other hand, embedded nanotube may confine polymer chains and reduce the chain mobility. The balance of these two effects will influence the glass transition. If the compromise effects lead to positive effect on molecular chain mobility, the T_g will decrease. Otherwise, T_g will increase. On the other hand, graphene composites show a slight increase in the T_g, and this result is consistent with Ruoff's results on nature nanotechnology.[69] Inclusion of graphene sheets has similar effect on glass transition, but the epoxy and hydroxyl group of graphene oxide may participate in the curing reaction and make it more complicate. Further DSC tests were run to confirm this conjecture.

1 wt % SWNTs and GO-filled epoxy resin were scanned by differential scanning calorimetry(DSC) at 5 °C/min ramping, respectively, and the results are shown in Figure 5. The enthalpy of epoxy resin curing is 343 J/g. Incorporating SWNTs into epoxy resin significantly reduced the enthalpy to 321 J/g, a decrease of 6%. On the other hand, the curing enthalpy was almost not influenced when GO sheets were incorporated into epoxy resins since the curing enthalpy reached 346 J/g. A positive effect on mobility of polymer chain is not significant due to a tiny drop of curing conversion and this effect may be compromised by negative effect caused by nanomaterial confinement, hence, inclusion of 1 wt % SWNT fillers showed slightly reduction on the composite T_g. Inclusion 1 wt % GO sheets did not show negative effect on curing reaction and then there is no significant positive effect on polymer mobility. GO sheets generally show much stiffer modulus than polymer matrix, and lead to significant confinement on the polymer chains. Inclusion of GO into polymer matrix may cause significant effect on curing reaction due to the reaction between matrix and chemical groups on the graphene surface, the final effect on T_g will depend on the balance of two effects, influence on reaction conversion and molecular confinement.

GO-filled composite was characterized by SEM, as shown in Figure 6. Isolated graphene sheets were homogeneously dispersed in the polymer matrix and no big bundles were observed. In addition, high embedding and tight binding with polymer matrix indicate good interfacial bonding between graphene sheets and epoxy resins. Some graphene sheets are monolayer state while others are in few-layer state. This is consistent with previous AFM results of the suspended graphene. This further confirms that hydroxyl and epoxide on graphene surface have strong interaction with epoxy resins and significantly improve the interphase interfaces.

Conclusions

Graphite flaks were intercalated by modified Brodie's method, and then exfoliated into single or few-layer graphene oxide by ultrasonic processing. AFM characterization verified graphene has been exfoliated into single-layer or few-layer states. Subsequent graphene sheets were incorporated into polymer composites at 1 and 5 wt %, respectively. The experimental results indicated that graphene oxide-filled composites showed both improved thermal conductivity and low CTEs. Incorporation of graphene sheets did not show any negative effect on composite T_g. These results suggest that graphene composites can be tuned to low CTEs while maintaining high thermal-stability, and then graphene composites can stand up to a wider range of temperature. This finding will pave a way for graphene-enabled thermal management in microelectronics.

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