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Thermal conductivity enhancement of nanofluids containing graphene nanosheets

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This paper envisages a mechanism of heat conduction behind the thermal conductivity enhancement observed in graphene nanofluids. Graphene nanofluids have been prepared, characterized, and their thermal conductivity was measured using the transient hot wire method. The enhancements in thermal conductivity are substantial even at lower concentrations and are not predicted by the classical Maxwell model. The enhancement also shows strong temperature dependence which is unlike its carbon predecessors, carbon nanotube (CNT) and graphene oxide nanofluids. It is also seen that the magnitude of enhancement is in-between CNT and metallic/metal oxide nanofluids. This could be an indication that the mechanism of heat conduction is a combination of percolation in CNT and Brownian motion and micro convection effects in metallic/metal oxide nanofluids, leading to a strong proposition of a hybrid model. © 2011 American Institute of Physics. [doi:10.1063/1.3650456]

INTRODUCTION

In the pursuit of improving thermal conductivity of liquids for meeting the increasing cooling requirements posed by emerging high heat flux devices, suspensions of nano sized particles in liquids, known as nanofluids, have emerged as potential candidates. What makes them different is the anomalous thermal conductivity enhancements reported in the literature. Nanofluids are a class of fluids having stable suspensions of nanoparticles, with sizes typically less than 100 nm. Generally nanofluids are classified into three metallic, metal oxides and carbon based nanofluids. They can also be distinguished based on the shapes like spherical, cylindrical, and sheet-like particles. So far, a large number of investigations have been performed on metallic and metal oxide nanofluids, where the particles are spherical and also on carbon nanotube (CNT) based nanofluids, where the particles are cylindrical. However, the decade long research in nanofluids has not come to a definitive conclusion to suggest a perfect replacement to the existing coolants. Graphene is a new entrant in nanofluids which has a two dimensional planar geometry. Graphene sheets have very high thermal conductivity, of the order of 5000 W/mK,¹ which is greater than that of CNT (>2000 W/mK).²

The history of nanofluids dates back to 1993 when Choi³ coined the term nanofluids and observed that nanofluids can conduct heat more efficiently than the conventional fluids. There has been number of research papers reporting the anomalous behavior of nanofluids with a few contradicting the same. Lee *et al.* (1999)⁴ and Wang *et al.* (1999)⁵ initiated the research reporting enhancements 20% for CuO in EG, 12% for CuO in water, and 10% for Al₂O₃ in

water. The real breakthrough was from the Argonne National Laboratory group with Eastman et al. $(2001)^6$ reporting 40% enhancement with only 0.3% copper particles of 10 nm size and Choi (1993)³ reporting a phenomenal 150% increase in thermal conductivity of Multiwall CNT-engine oil nanofluid with just 1% volume fraction of nanotubes. The finding of Das *et al.* in $(2003)^7$ showing strong temperature dependence of nanofluids with Al₂O₃ and CuO particles as used by Lee et al. (1999)⁴ was a significant milestone in nanofluid research which improved the scope of nanofluid as an alternative for existing coolants. This was later confirmed by Chon *et al.* $(2005)^8$ and Li *et al.* (2006).⁹ But the temperature effect on thermal conductivity enhancement of nanofluids was not observed in CNTs.¹⁰ Recently Yu et al. (2010)^{11,12} studied the thermal conductivity enhancement of graphene oxide (GO) nanofluids and found no temperature dependence of enhancement. There have been number of theories explaining the mechanism behind the anomalous behavior of nanofluids; the most accepted being Brownian motion, $^{13-18}_{13-18}$ percolation theory, $^{10,13-16}$ micro convection cell model, $^{13-18}$ liquid layering theory, ^{13–16,19,20} and ballistic heat transport.^{13–16,20} The recent bench marking exercise on nanofluids²¹ concluded that nanofluids do not exhibit anomalous thermal conductivity enhancements and follow the classical Maxwell model. However, the results are limited to the samples considered and not universal.

The shape and size of the nanoparticles play an important role in defining the physics of the heat conduction. It is observed from the above results that metal oxide and metallic nanofluids exhibit thermal conductivity enhancement, which increases with increase in concentration and temperature. The Brownian motion^{17,18} and micro convection model^{17,18} have been able to explain this trend successfully. Liquid layering theory^{22,23} explain that the nanoparticles act as insulators when their characteristic length is much bigger

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than the particle size due to interfacial resistance which decreases the effective thermal conductivity rather than increasing it. Ballistic heat transport²⁰ operates when the separation between the particles is very small of the order of 1-2 nm, where the phonons initiated in one particle can persist in fluids and get transported to other particles. The last two mechanisms (Liquid layering theory and Ballistic heat transport) were successful for explaining the increase in metallic nanoparticles at lower concentrations. However, these models could not explain the behavior of CNT, as there was no enhancement with temperature, which is an essential feature. But the geometry of CNT with high aspect ratio was conducive for chain formation. This led to the percolation model,¹⁰ which was able to explain the temperature independence and the anomalous enhancement. Similar trend has been observed in GO nanosheets.^{11,12} The flake structure of graphene oxides could also result in chain structure as in CNT. Since graphene has similar geometry, one would expect a similar trend. But the recent observation by Baby and Ramaprabhu (2010)²⁴ on graphene nanofluid showed a strange behavior of temperature dependent thermal conductivity enhancement unlike its carbon predecessors and a maximum of 64% enhancement which gave a great possibility to explore the suitability of graphene as a smart fluid and explain the physics of heat conduction in graphene nanofluids.

In this study, homogeneous and stable graphene nanofluids were prepared, and the effects of particle volume fraction and temperature on thermal conductivity were investigated. The nanofluid is well characterised by using transmission electron microscopy (TEM) images, ultravioletvisible (UV-vis) absorption spectrum, and dynamic light scattering (DLS) measurements. The enhancements are compared with other nanofluids, which reveal the shape dependent pattern of enhancement. In addition to the thermal conductivity measurements, a qualitative explanation of the results by taking hints from the existing knowledge, explaining the possible mechanism behind the thermal conductivity enhancement and temperature dependence, is put forward. This new picture sheds light on the apparent contradiction in the temperature dependence of thermal conductivity in GO and graphene based nanofluids.

GRAPHENE SYNTHESIS AND CHARACTERIZATION

Since it is difficult to utilize mechanically exfoliated graphene flakes for mass production of functional devices, there have been intense efforts to develop methods for synthesis of large-scale, high-quality graphene. There are many synthetic routes to prepare graphene, such as micromechanical cleavage, intercalation, solvo-thermal synthesis, and chemical vapour deposition. The chemical route to prepare graphene sheets in large quantities^{25,26} is followed here. The raw material for bulk production of graphene sheets by the chemical method is usually graphite powder, which is inexpensive. In addition, the chemical methods are versatile in terms of being well suited to chemical functionalization. Therefore, in this paper, the chemical method was applied to produce graphene nanosheets (GnS), which is well dispersed in water without any need of surfactants.

Graphene is prepared from natural graphite (sp² hybridized, $30\,\mu\text{m}$ nominal particle size) by oxidation-reduction process. The graphite sheet is first exfoliated and oxidized to GO using Hummer's method.²⁶ Later this oxide is reduced to water soluble graphene in three steps: (1) pre-reduction (2) sulfonation, and (3) post-reduction. Pre-reduction of GO with sodium borohydride at 80°C for 1h removes the majority of oxygen functionalities from GO. The stoichiometrically controlled sulfonation with the aryl diazonium salt of sulfanilic acid in an ice bath for 2h improves the water solubility with minimal impact on the unique properties of graphene. Post reduction with hydrazine (100°C for 24h) removes any remaining oxygen group present other than acid functionality. Thus, a graphene nanofluid of required volume concentration is obtained and it was observed that the sample is very much stable even after 6 months of preparation without addition of any surfactants. This can be explained by sedimentation ratio, from Stokes-Einstein theory (1897) [Eq. (1)], which is given by

$$\nu = \frac{2r_p^2 |\rho_p - \rho_m|g}{9\varepsilon_m},\tag{1}$$

where $\rho_{\rm m} =$ density of the medium, $\varepsilon_{\rm m} =$ viscosity of the fluid, $\rho_{\rm p} =$ density of the particle, and $r_p =$ radius of the nanoparticle.

Lower the value of sedimentation ratio, higher the stability of the suspension. In this case, the density of graphene is comparable to the density of water and the sedimentation ratio is close to zero. This results in the higher stability of the suspension.

The TEM images of graphene (Fig. 1) reveal that the sheets are interconnected and entangled. This shows that the graphene is likely to form networks, which might act as conduction paths. The DLS measurement indicates a trimodal distribution of size of graphene sheet (Fig. 2) showing the presence of graphene sheet lengths from 5 nm to 1500 nm. About 52% of the particles are in the size range close to 5 nm, about 34% near 1500 nm and the remaining in the range of 100 nm.

In order to ensure the purity of graphene nanofluids, UV-visible absorption spectrum analysis is carried out (Fig. 3). Absorption spectrum measurements are taken after

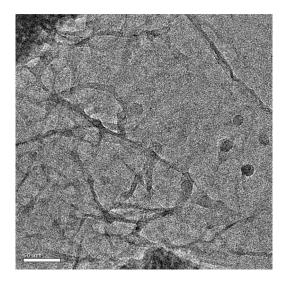


FIG. 1. TEM image of graphene flakes. Scale bar is 50 nm.

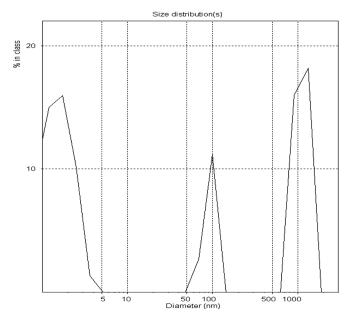


FIG. 2. DLS plot showing the size distribution of as-synthesized graphene dispersion.

the preparation of graphene nanofluids from GO. It was found that the absorption maximum of the sample was 269 nm. The value is in good agreement with the absorption spectrum data of graphene in the literature.²⁷ Also the nonexistence of absorption peak at 229 nm, which is that of GO, ensures the complete reduction of GO to graphene.

EXPERIMENTAL METHOD

Thermal conductivity was measured by the apparatus built on the principle of traditional transient hot-wire (THW) method in a temperature-controlled bath with operating limits $25 \,^{\circ}$ C to $60 \,^{\circ}$ C. In this method, a thin metallic wire is used as a line heat source as well as temperature sensor and forms an arm of a Wheatstone bridge as shown in Fig. 4. The wire is surrounded by the liquid whose thermal conductivity is to be measured. The wire is then heated by sending current through it. Higher the thermal conductivity of the surrounding liquid, lower will be the temperature rise of the wire. This principle is used to measure thermal conductivity of the liquid. The

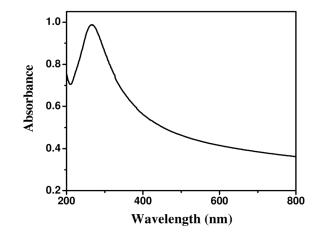


FIG. 3. UV-vis absorption spectrum of graphene.

experiment lasts for a maximum of 2-5 s and hence, this is a very fast method. Also, within such small time duration, natural convection of liquid does not set in.²⁸ In conjunction with an advanced electronic data acquisition system, the method gives very accurate measure of thermal conductivity. The experimental setup has been validated by measuring the thermal conductivity of de-ionized water and the error in measurement was within 1.2%. Further details of the experimental setup can be found in Patel *et al.* (2010).²⁹ The thermal conductivity measurements were carried out eight times for each sample of graphene nanofluid for temperatures between 30 °C and 50 °C in steps of 5 °C, which ensured the repeatability of the experiment. The average value of measured thermal conductivity is reported in the paper.

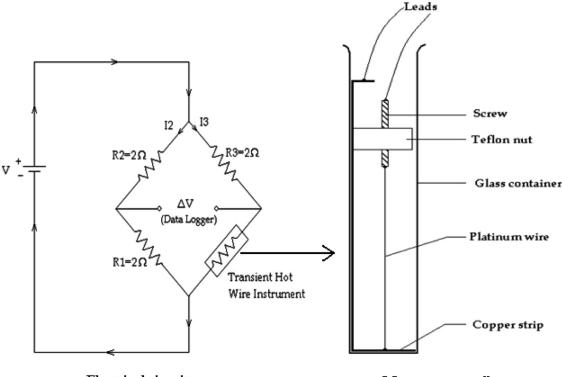
RESULTS AND DISCUSSION

The dependence of thermal conductivity of graphene nanofluids on temperature and concentration has been investigated thoroughly. The thermal conductivity of graphene nanofluids shows enhancement, which depends on concentration and temperature, unlike its carbon predecessors, GO and CNT nanofluids, where the thermal conductivity does not vary significantly with temperature. The importance of particle size is stressed in Das *et al.* (2003)⁷ where stochastic motion of nanoparticles is predicted and in Venkata Sastry *et al.* (2009)¹⁰ where aspect ratio of nanotubes are mentioned playing a role in forming chain like structure enhancing thermal conductivity.

Fig. 5 depicts the dependence of thermal conductivity of graphene nanofluids on concentration in the range 0.05-0.2 vol. % at different temperatures. It is observed that the thermal conductivity increases with increase in concentration of graphene, which is expected. The maximum enhancement obtained was 27% at 0.2% concentration.

Fig. 6 compares the enhancement of thermal conductivity with concentration of graphene with other nanofluids at same temperature. It is clear that the rate of increase of enhancement with concentration of graphene and CNT are comparable and is much superior to metallic and ceramic nanofluids. The anomalously high enhancement for CNT was explained by percolation model, proposed by Venkata Sastry et al. (2009).¹⁰ According to the model, CNTs were assumed to form long chain of interconnected networks which acted as conducting paths. The geometry of nanotubes with high aspect ratio is ideal to form such a network. The geometry of graphene, being planar, could also form such interconnected networks. This view is supported by the TEM image (Fig. 1). A comparable rate of enhancement is not necessarily an indicator of comparable percolation, as the heat conduction through the network is still a strong function of thermal conductivity. It could also be proposed that the extent of percolation is lower in case of Graphene, since its thermal conductivity is about 2.5 times that of CNT.

The thermal conductivity values were compared with effective medium theory by Maxwell,³⁰ where it is assumed that the particles are non-interacting. Fig. 7 shows the thermal conductivity values compared at different concentrations with the Maxwell theory. It is observed that at lower



Electrical circuit

Measurement cell

FIG. 4. Schematic of the transient hot wire equipment.

concentration, the Maxwell theory is able to predict the thermal behavior. However, as the concentration increases, it fails to do the same. This behavior of Graphene nanofluids is yet another indicator of the network model. The networks act as strong conducting paths for heat conduction and hence higher enhancements.

Another important observation from Fig. 5 is the temperature dependence. It is clear that the thermal conductivity enhancement of graphene nanofluids depends on temperature. The thermal conductivity of graphene nanofluids increases with temperature. The enhancement increased from about 10% to 27% with 20 $^{\circ}$ C rise in temperature at 0.2% concentration. This temperature-dependent behavior was not observed in CNT. This trend brings out the fact that the mechanism is not solely a percolation based one, as temperature has no role in the network formation.

Since GO nanofluids, recently investigated by Yu *et al.* (2010),^{11,12} show temperature independence of enhancement, the percolation model might fit in perfectly. However, due to similar shape of graphene and GO, one might expect the model to be applicable to graphene nanofluids as well. This will lead to the conclusion that temperature should have no effect on graphene nanofluid thermal conductivity. This apparent contradiction arises because of the different sizes of

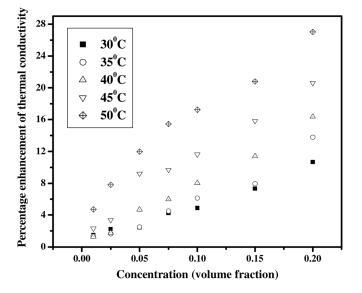


FIG. 5. Thermal conductivity enhancement of graphene nanofluids as a function of concentration.

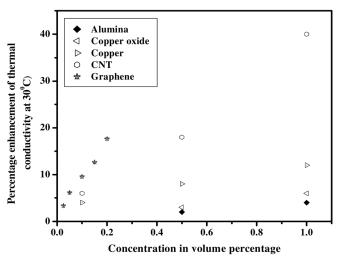


FIG. 6. Comparison of spherical (metals and metal oxide), rod (CNT), and flat sheet (graphene) nanofluids in their thermal conductivity enhancement.

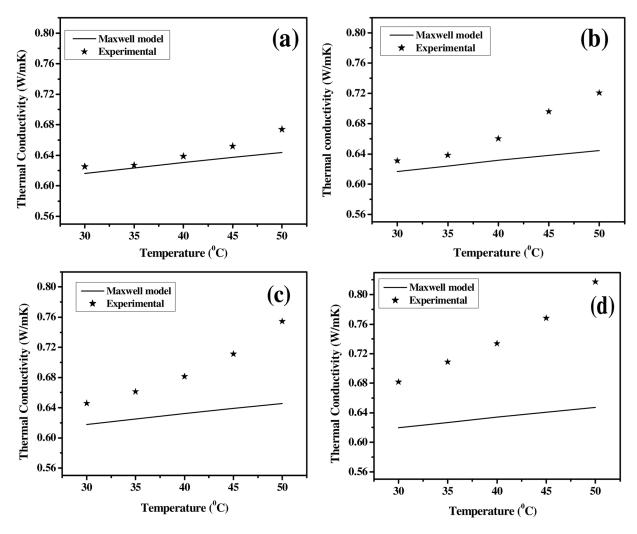


FIG. 7. Comparison of experimental data with Maxwell model prediction at different concentrations: (a) 0.01%, (b) 0.05%, (c) 0.1%, and (d) 0.2%.

particles and concentrations. Yu *et al.* $(2010)^{11,12}$ have studied the GO nanofluids with sheet sizes ranging from 1 to 3 μ m and concentrations from 1% to 5%. But in the present investigation, sheet sizes vary from as low as 5 nm to 1500 nm and concentrations from 0.01% to 0.2%. It shows that the particle size distribution and the concentration play an important role in the network formation. It is an intuitive guess that the larger sheets will have more probability of forming a network and with concentration as high as 1%, GO nanofluids prepared by Yu *et al.* (2010)^{11,12} should be more interconnected than in the present study.

The temperature dependence of nanofluids was earlier observed in metallic and ceramic nanofluids.^{7–9} The effect of particle size may be attributed mainly to two reasons, viz., high specific surface area of the nanoparticles and Brownian motion. As the particle size reduces, the surface area per unit volume increases and the heat transfer being dependent on surface area, the effectiveness of the nanoparticles in transferring heat to the base liquid increases. Also, smaller the particle, higher is the Brownian motion velocity, which adds up to the contribution by the nanoparticles to the total heat transfer by continuously creating additional paths for heat flow in the fluid.

To add to the above discussion, the thermal conductivity enhancement of graphene has been plotted in Fig. 8 along with aluminium, CNT, and copper nanofluids at 0.1 vol. % at different temperatures from 30 to 50 °C. It is clear that CNT nanofluids show temperature independence. The CNT equivalent is a hypothetical line constructed to show the path graphene would have followed had the only mechanism of conductivity is percolation as in CNT. It appears that the enhancement with temperature is much higher for Graphene compared to metallic nanofluids. The micro convection model [Eq. (2)] proposed by Patel *et al.* $(2005)^{17}$ predict the temperature dependence of thermal conductivity of metallic nanofluids. For a given particle and volume fraction, the model predicts a linear behavior with the slope proportional to particle thermal conductivity. The metallic equivalent line is a hypothetical line constructed based on micro convection model, where the thermal conductivity of graphene is used in place of the thermal conductivity of metal. The thermal conductivity values should have fitted into this line had graphene's behavior been exactly similar to metallic nanofluids (Brownian motion dominated). But the data points falls below this line. Thus thermal conductivity of graphene nanofluids does not purely follow micro convection or percolation model but comes in between both the theories.

Micro convection model Patel *et al.*¹⁷ (2005)

$$\%Enhancement = \frac{k_p}{k_m} \left(1 + c \frac{u_p d_p}{a_m} \right) \frac{d_m}{d_p} \frac{\epsilon}{1 - \epsilon} \times 100 \quad (2)$$

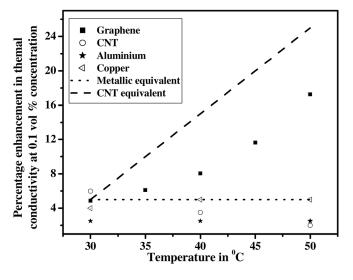


FIG. 8. Comparison with micro convection and Maxwell models.

where c = empirical constant, $d_m = \text{diameter of liquid particle (m)}$, $d_p = \text{particle diameter (m)}$, $k_m = \text{thermal conductivity}$ ity of medium, $k_p = \text{thermal conductivity}$ of particle, $r_p = \text{radius of the particle}$, $u_p = \text{particle velocity due to}$ Brownian motion (m/s), $\alpha_m = \text{thermal diffusivity (m^2/s)}$, and $\epsilon = \text{particle volume fraction}$.

Thus, it may be concluded that graphene thermal conductivity mechanism falls well between that of CNT (percolation) and Metallic (Brownian motion and convection). Hence, it may be postulated that the majority of larger sheets of graphene forms a network and the smaller ones contribute to Brownian motion effects. The experimental observations seem to qualitatively fit in this percolation-Brownian motion based hybrid theory.

From the above discussions it is clear that the presence of larger aspect ratio CNT leads to strong network chain structure, which acts as heat conduction paths giving anomalously higher thermal conductivity enhancements compared to the classical Maxwell model. GO nanofluids exhibit temperature independent behavior, which can be attributed to larger sized sheets $(1-3 \,\mu m)$ and volume concentration (1%-5%). These are strong signs of network formation and hence, GO can be clearly explained by percolation theory. But graphene nanofluids exhibit enhancement of thermal conductivity with both concentration and temperature. This can be attributed to different sized particles present in graphene nanofluids. The larger sized particles contribute to form network like chain structure following percolation theory. The smaller sized particles undergo random motion contributing Brownian motion. This Brownian motion induces micro convection causing thermal conductivity enhancement with the increase in temperature. This led to the strong proposition of a hybrid nature for heat conduction in graphene nanofluids involving micro convection and percolation phenomena.

CONCLUSIONS

Nanofluids containing GnS were prepared and their thermal conductivity was measured by transient hot wire method. The thermal conductivity is substantially enhanced even at lower concentration. As expected, the enhancement improves with increasing concentration. Thermal conductivity also showed temperature dependence, which were unlike the case in CNT and GO nanofluids. The possible mechanism could be a Brownian motion-percolation hybrid, the earlier one explaining the temperature dependence and the latter one explaining the magnitude of enhancement of thermal conductivity.

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