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ARTICLE *in* INTERNATIONAL JOURNAL OF HEAT AND MASS TRANSFER · JUNE 2012

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## Enhanced thermal conductivity of ethylene glycol with single-walled carbon nanotube inclusions

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### ARTICLE INFO

#### Article history:

Received 10 October 2011

Received in revised form 22 January 2012

Accepted 1 March 2012

Available online 23 April 2012

#### Keywords:

Nanofluids

Single-walled carbon nanotube

Thermal conductivity

### ABSTRACT

In the present work, we report measurements of the effective thermal conductivity of dispersions of single-walled carbon nanotube (SWNT) suspensions in ethylene glycol. The SWNTs were synthesized using the alcohol catalytic chemical vapour deposition method. Resonant Raman spectroscopy was employed to estimate the diameter distribution of the SWNTs based on the frequencies of the radial breathing mode peaks. The nanofluid was prepared by dispersing the nanotubes using a bile salt as the surfactant. Nanotube loading of up to 0.2 vol% was used. Thermal conductivity measurements were performed by the transient hot-wire technique. Good agreement, within an uncertainty of 2%, was found for published thermal conductivities of the pure fluids. The enhancement of thermal conductivity was found to increase with respect to nanotube loading. The maximum enhancement in thermal conductivity was found to be 14.8% at 0.2 vol% loading. The experimental results were compared with literature results in similar dispersion medium. Experimental results were compared with the Hamilton–Crosser model, the Lu–Lin model, Nan's effective medium theory and the Hashin–Shtrikman model. Effective medium theory seems to predict the thermal conductivity enhancement reasonably well compared to rest of the models. Networking of nanotubes to form a tri-dimensional structure was considered to be the reason for the thermal conductivity enhancement.

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## 1. Introduction

Conventional heat transfer fluids exhibit poor heat transfer performance due to their low thermal conductivities. Many research activities have been carried out in the past to improve the thermal properties of these fluids by seeding a small quantity of highly thermally conductive solid in it [1]. However, this has not been very successful because of settling of the solid particles due to their large size [1]. Recent developments in the field of nanoscience have led to the development of a new class of fluids termed “nanofluids” [2]. Over the past decade, nanofluids—colloidal dispersions prepared by uniformly dispersing nanostructures such as nanoparticles, nanotubes and nanofibers in conventional heat transfer fluids—have gained attraction worldwide because of their superior thermal performance [2–5]. Many research groups experimentally reported a substantial increase in the thermal conductivity [3] and the convective heat transfer coefficient [4] of such fluids.

Nanoparticles of various metals and metal oxides have been used for nanofluid production. These predominantly include copper, copper oxide, aluminum, aluminum oxide, zinc oxide, titanium dioxide, etc. [3,5]. More recently, a significant number of studies have made use of carbon-based nanostructures, namely graphite [6], fullerene [7], single-walled carbon nanotubes (SWNTs) [8], multi-walled carbon nanotubes (MWNTs) [9–20], nano diamond [21] and graphene [22,23] to prepare nanofluids. Recent studies reveal that carbon nanotubes have very high thermal conductivity [24–28], hence it is obvious to expect that the suspensions consisting of nanotubes/graphene sheets would show higher thermal conductivity enhancement compared to other nanoparticles.

The first experimental observation of thermal conductivity enhancement was reported by Choi and co-workers for the case of MWNTs dispersed in poly-( $\alpha$  olefin) oil [9]. They reported an enhancement of 160% at a nanotube loading of 1 vol%. This huge enhancement, however, could not be reproduced by subsequent studies [10–19]. Despite such a large discrepancy in the experimental results, all the results were found to be significantly higher than those predicted by analytical models. Therefore, more studies need to be performed to measure the effective thermal conductivity of nanotube suspensions in different base fluids.

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In the present work, we report the effective thermal conductivity of SWNT suspensions in ethylene glycol (EG). We also compare the measured effective thermal conductivity with existing analytical models to determine which model is in best agreement with the experimental observations.

## 2. Materials and methods

### 2.1. Material synthesis

SWNTs were synthesized by the catalytic chemical vapour deposition (CVD) technique using ethyl alcohol as the carbon source as described by Maruyama et al. [29]. Cobalt and iron catalysts supported with zeolite were employed. Amorphous carbon impurities and metal particle impurities were dramatically suppressed at the reaction temperature of 800 °C. The etching effect caused by the decomposed hydroxyl (—OH) radical, which removes carbon atoms with dangling bonds during the CVD process was reported as the reason for the high purity of the SWNTs [29]. A transmission electron microscopy (TEM) image of the 'as grown' SWNTs from ethanol feedstock is shown in Fig. 1.

### 2.2. Characterization by Resonant Raman Spectroscopy

Resonant Raman spectra (RRS) of the 'as grown' SWNT sample were acquired using a macro-Raman apparatus with a 50 cm single monochromator and a CCD detector. The excitation wavelength used was 488 nm with an argon ion laser.

A typical RRS obtained from the SWNT sample is shown in Fig. 2. The low frequency features (100–400  $\text{cm}^{-1}$ ) show the Radial Breathing Mode (RBM) peaks, the frequencies of which are dependent on the SWNT diameter. The G-band observed around 1592  $\text{cm}^{-1}$  is the characteristic feature of the graphitic layers, and corresponds to in-plane vibrations of the carbon atoms. In addition to the G-band and RBM, the D-band observed near 1350  $\text{cm}^{-1}$  arises from defects in the graphitic structure. A high ratio of the intensities of the G band and D-band ( $I_G/I_D$ ) reveal that the purity of the bulk sample is very high [30].

The expanded RBM signal [50] along with the diameter distribution is shown in the inset of Fig. 2. The RBM frequency ( $\omega_{\text{RBM}}$ )

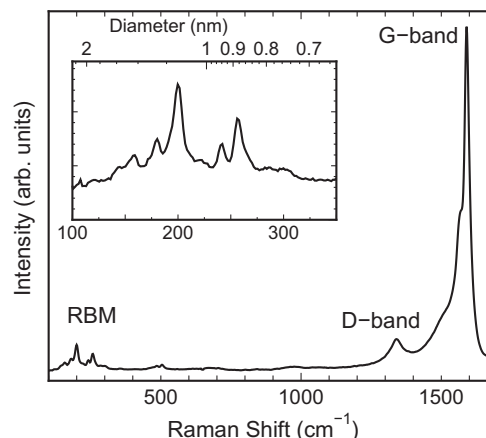


Fig. 2. Resonant Raman spectra of 'as grown' SWNTs from ethanol feedstock (excitation wavelength 488 nm). The Graphite-like band (G-band); Defect induced band (D-band) and the Radial Breathing Mode (RBM) are shown. An expanded view of the RBM signals along with the diameter distribution is shown in the inset.

is roughly proportional to the inverse of the nanotube diameter, following the equation  $\omega_{\text{RBM}} = A/d + B$ , where  $d$  (nm) is the diameter of the nanotube,  $A$  ( $\text{cm}^{-1} \text{ nm}$ ) and  $B$  ( $\text{cm}^{-1}$ ) are constants [30]. The parameters  $A$  and  $B$  are typically determined experimentally, and for the present case the commonly used values of 248 and 0 were used for  $A$  and  $B$  respectively [29–31]. From the expanded RBM signal shown in Fig. 2, the typical diameter distribution of the SWNT samples synthesized using ethanol feedstock was estimated to be 0.8–1.6 nm.

### 2.3. Nanofluid preparation

The highly hydrophobic nature of SWNTs makes it very difficult to disperse them in a liquid medium. An important prerequisite for the nanofluid is the preparation of a stable and homogenous suspension. Two popular techniques prevail in the open literature for creating stable dispersions. The first technique is to attach a hydrophilic functional group onto the nanotube sidewalls by strong acid treatment [10]. The other technique is to encapsulate the SWNTs using surfactants. Though acid treatment is frequently used to disentangle MWNTs, it may not be very suitable for SWNTs as it will heavily damage the sidewalls [32]. In our present work, we made use of a surfactant to prepare the nanofluid dispersion.

The commonly used surfactant in the nanofluid preparation are sodium dodecylbenzene sulphonate (SDBS) [14], Sodium dodecyl sulfate (SDS) [11], Gum Arabic [12], hexadecyltrimethylammonium bromide (CTAB) [33], and Octyl phenol ethoxylate (Triton-X100) [34]. Wenseleers et al. reported the potential of using sodium deoxycholate (DOC), a bile salt, as a dispersion agent for SWNTs [35]. For the present experiments, DOC was chosen as the surfactant to make SWNT/EG dispersions.

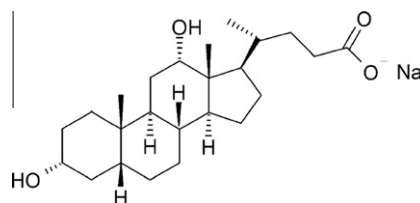


Fig. 3. Structure of the sodium deoxycholate surfactant.

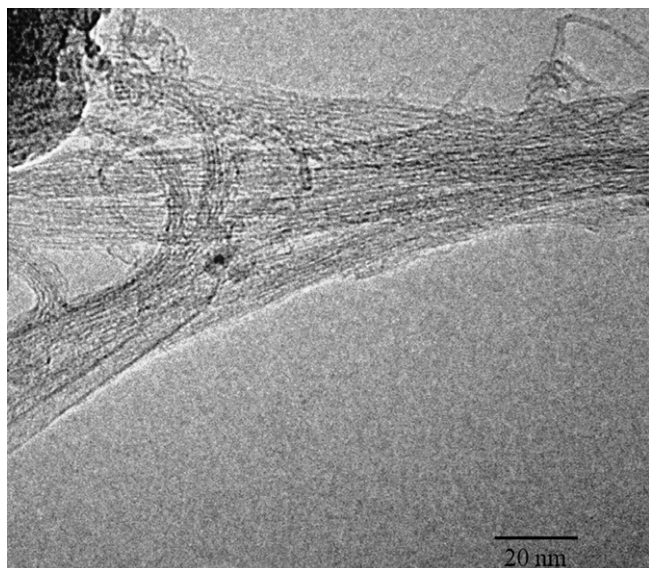


Fig. 1. TEM (200 kV) image of 'as grown' SWNTs from ethanol feedstock at 800 °C over a Fe/Co mixture embedded in zeolite (HSZ-390HUA). \*(It is recommended to use lower acceleration voltage such as 120 kV. For convenience; we have used 200 kV for our measurements).

The structure of DOC is shown in Fig. 3. DOC possesses a rigid structure consisting of a cholesterol group with dissimilar sides [36]. It consists of a steroid skeleton with a carboxylic acid side chain at one end and two hydroxyl groups on its steroid backbone. The two polar hydroxyl groups on the  $\alpha$ -face and methyl group on the other side facilitate strong adsorption of the DOC onto the surface of SWNTs [36,37].

The stable dispersions were prepared by adding the necessary loading of SWNTs in ethylene glycol and then 0.5 w/v % of the surfactant. The density of the SWNTs used for calculation purpose was  $1.6 \text{ g/cm}^3$  assuming a closely packed hexagonal structure and a diameter of 1 nm and a Van der Waals spacing of 0.34 nm. The dispersions were subjected to bath sonication for 90 min followed by tip sonication using an ultrasonic processor (Hielscher GmbH, UP-400S with H3/Micro Tip 3) for 120 min at a power flux level of  $368 \text{ W/cm}^2$  (80% amplification). The pH of the dispersion was 7. The length of the nanotubes after sonication from our Atomic Force Microscopy (AFM) measurements was found to be between 150–500 nm.

#### 2.4. Thermal conductivity measurement setup

In the present study we make use of the transient hot wire (THW) technique to measure the thermal conductivity of the nanofluids. A platinum (Pt) hot wire of  $10 \mu\text{m}$  diameter and 20 mm length is placed inside the test cell. The Pt wire acts as both the heater and an electrical resistance thermometer. This technique is based on the measurement of temperature and temporal response of the hot wire when it is subjected to an electrical pulse.

The relation between thermal conductivity  $k$  and measured temperature  $T$  can be summarized as follows [38]. Assuming a thin, infinite line heat source with constant heat output in an infinite medium, the energy equation can be written as

$$\frac{1}{\alpha} \frac{\partial T}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left( r \frac{\partial T}{\partial r} \right) \quad (1)$$

With initial condition and boundary conditions

$$T(t=0) = T_0 \quad (2)$$

$$\lim_{r \rightarrow 0} \left( r \frac{\partial T}{\partial r} \right) = \frac{q}{2\pi k} \quad \text{and} \quad \frac{\partial T}{\partial r} \bigg|_{r=\infty} = 0 \quad (3)$$

the analytical solution is of the form

$$T(r, t) = T_0 + \frac{q}{4\pi k} \left\{ -\gamma + \ln \left( \frac{4\alpha t}{r^2} \right) + \dots \right\} \quad (4)$$

Here,  $\alpha$  is the thermal diffusivity and  $\gamma = 0.5772$  is Euler's constant. If the temperature of the hot wire at times  $t_1$  and  $t_2$  were  $T_1$  and  $T_2$  respectively, then by eliminating the higher order terms in Eq. (4), the thermal conductivity of the fluids can be approximated in the following form:

$$k = \frac{q}{4\pi} \frac{\ln(t_1/t_2)}{T_1 - T_2} \quad (5)$$

During the experiments, the Pt wire is heated with the aid of a constant DC power supply. The temperature rise of the hot wire is determined from the change in resistance of the hot wire, which can be measured in time using a Wheatstone-bridge circuit. With known electric power supply and slope of the curve  $\ln(t)$  versus  $T$ , the thermal conductivity  $k$  is calculated using equation Eq. (5).

#### 2.5. Calibration of the THW setup and uncertainty analysis

To validate the THW setup, the thermal conductivity of pure deionized water and ethylene glycol were measured at different

temperatures. A comparison of the measured and reference thermal conductivity data is shown in Fig. 4. The upper line corresponds to the thermal conductivity of water according to the ASTM-D2717 standard [39]. The lower line corresponds to the thermal conductivity of EG reported by Di Guilio and Teja [40].

The measured results were in good agreement with the reference data reported in the literature. A detailed uncertainty analysis of the THW setup based on the method prescribed by [41, 42] was carried out, and the experimental uncertainty was found to be  $\pm 2\%$ .

### 3. Results and discussion

#### 3.1. Experiments

Thermal conductivities of the SWNT-dispersed EG samples were measured for different SWNT loadings using the THW setup. All the experiments were performed on the same day of sample preparation, and all values reported below are statistical averages of at least 20 measurements. Fig. 5 shows the thermal conductivity enhancement versus different SWNT loadings measured at room temperature. The thermal conductivity increased with increasing SWNT loading in a slightly nonlinear fashion. A maximum enhancement of 14.8% at a loading of 0.21 vol% was found in this study. A bare Pt hotwire is employed in this study. Such a bare hot wire is susceptible to leakage current which might affect the experimental results [43]. Hence to check the credibility of the results we performed additional measurements with an electrically insulated Tantalum (Ta) wire as reported by Alloush et al. [44]. For 0.21 vol% loading, measurements with Ta wire provided an enhancement of  $13.2 \pm 3.5\%$  which is consistent with Pt hot wire measurements.

The results of this study show thermal conductivity enhancements are less than those reported by Choi et al. [9] for poly-( $\alpha$  olefin) oil seeded with MWNTs. Xie et al. [10], Liu et al. [13] and Lamas et al. [18] reported enhancements of 12.7%, 12.4% and 11.5% respectively at 1 vol% loading. Ruan and Jacobi recently reported an enhancement of 9.3% at a volume fraction of 0.24% for EG/MWNT nanofluids [20]. Chen et al. reported an enhancement of 18% for nano-diamond-based nanofluids at 1 vol% [16]. Yu et al. reported an enhancement of 36% at for SWNT/EG nanofluids at 1 vol%

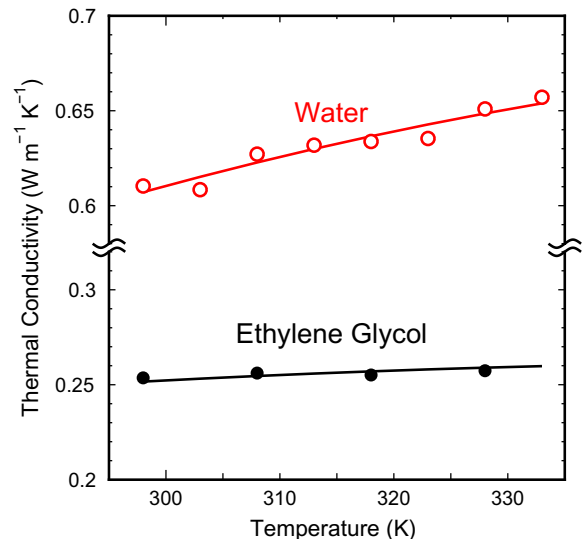


Fig. 4. Comparison of thermal conductivity values measured in this work with the reference literature. Open and filled circles correspond to the measured thermal conductivity of water (top) and EG (bottom) respectively. Solid lines correspond to the literature data.

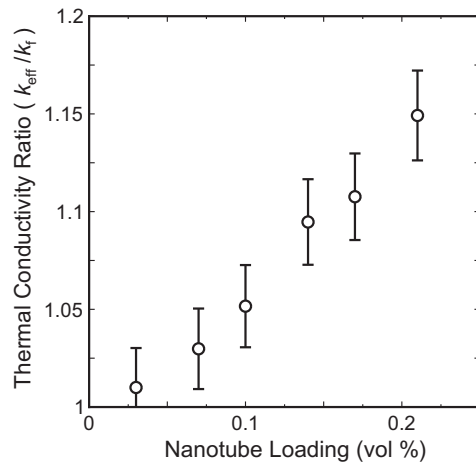


Fig. 5. Effective thermal conductivities of SWNT/EG dispersions for different concentrations at 298 K.

[21]. Baby and Ramaprabhu [23] developed graphene-based nanofluids and reported an enhancement of up to 7% at a very low loading of 0.05 vol%. Eastman et al. [45] reported an enhancement of 40% at 0.3 vol% for Copper/EG based nanofluids. In this work, such a high enhancement was not observed. The magnitude of effective thermal conductivity enhancement for EG-based nanofluids reported in this work is in consistent with the existing literature data, however at different nanotube concentrations. This difference could be attributed to the aspect ratio of the material used, purity level, and treatment method adopted to prepare the nanofluid dispersion.

### 3.2. Comparison with theoretical models

The experimental results are compared with the Hamilton–Crosser model (H–C) [46], the Lu–Lin model [47], Nan’s effective medium theory [48,49] and the Hashin–Shtrikman model (H–S) [50]. For the present model calculations, fluid thermal conductivity of  $0.252 \text{ W m}^{-1} \text{ K}^{-1}$ , an aspect ratio (length to diameter ratio) of 300, and interfacial resistance of  $10^{-8} \text{ m}^2 \text{ K W}^{-1}$  [51] were taken for calculations. The experimentally reported thermal conductivity of SWNTs vary from  $1750 \text{ W m}^{-1} \text{ K}^{-1}$  to  $6000 \text{ W m}^{-1} \text{ K}^{-1}$  [24–28]. Molecular dynamics simulations report a value ranging from  $250 \text{ W m}^{-1} \text{ K}^{-1}$  to  $6600 \text{ W m}^{-1} \text{ K}^{-1}$  at room temperature [52,53]. The present calculations were performed assuming a thermal conductivity of  $1750 \text{ W m}^{-1} \text{ K}^{-1}$  [24].

Figs. 6 and 7 show the comparison of the above models with the present experimental results. It can be seen from Fig. 6 that the Hamilton–Crosser model and Lu–Lin model completely underestimate the effective conductivity at all concentrations. Nan’s effective medium theory completely overestimates the effective conductivity when the interfacial thermal resistance or thermal boundary resistance (TBR) is not taken into account. However, when the influence of interfacial thermal resistance is taken into account, Nan’s model underestimates the effective conductivity but still performs much better than the H–C and Lu–Lin models. Hence, it can be said that the large thermal resistance across the interface plays a significant role in reducing the effective conductivity of the nanofluid. For the present experiments, interfacial thermal resistance was found to be  $2.4 \times 10^{-9} \text{ m}^2 \text{ K W}^{-1}$  by fitting the experimental data which is one order of magnitude lower than the one reported by Huxtable et al. from their transient absorption measurements of SDS encapsulated SWNT suspensions in octane fluid [51]. Such a low thermal resistance across the interface obtained from the experimental data fit is clearly surprising. It is also

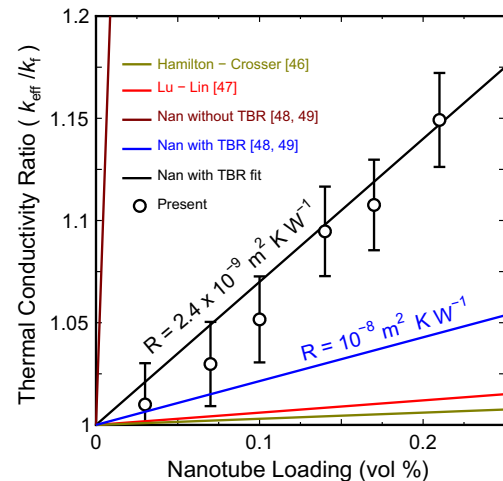


Fig. 6. Effective thermal conductivity of SWNT/EG dispersions and comparison with theoretical models [45–48].

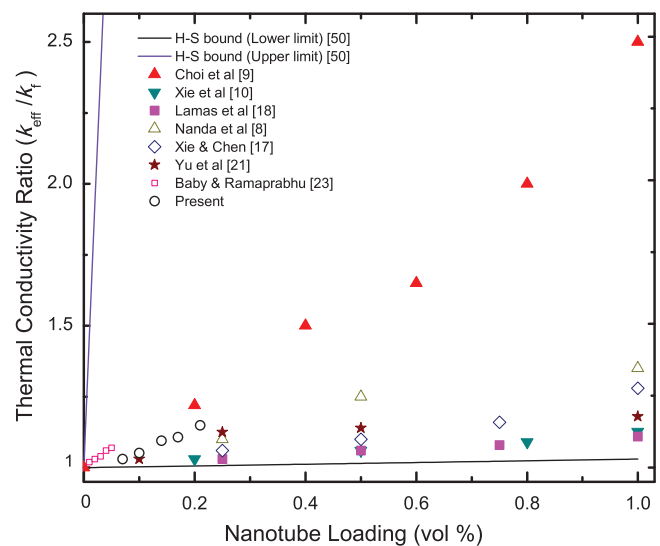


Fig. 7. Effective thermal conductivity of SWNT/EG dispersions-comparison with the H–S model [49] and other reported values.

to be noted that Cherkasova and Shan [54] reported interfacial resistance of similar order of magnitude for SDBS encapsulated MWCNTs in water by fitting their experimental data with the effective medium theory. It may be possible that the interface resistance varies depending on the surfactant used and the dispersion medium. Lack of clear knowledge about the interfacial thermal resistance remains a hindrance to completely rely on this model. However, it helps to understand the critical role of interfacial thermal resistance in the degradation of thermal conductivity enhancement.

Fig. 7 shows the theoretical bounds of the H–S model [50] along with the present experimental and literature results. In two recent studies, Fan and Wang [55,56] argued that when the ratio of the thermal conductivity of the nanomaterial to the base fluid conductivity is greater than unity, the separation between upper and lower limits of this model becomes more pronounced. They also found that particles with higher conductivities tend to interconnect and disperse in the base fluid. As a result, the energy transport across the interface is greatly enhanced, making it possible for the nanofluids to achieve remarkable enhancements as predicted by the



upper bound of the H–S model by simply tuning the morphology of the nanomaterial [55,56]. Xie and Chen [57] optimized the thermal conductivity enhancement by cleverly tailoring the morphology of the MWNTs. They adjusted the thermal conductivity enhancement by cutting the acid functionalized MWNTs to different length by ball milling technique. Their results suggest that the straight MWNTs tend to form percolating network more easily and transport heat more efficiently than bent MWNTs. The authors concluded that the straightness factor of CNTs is an important factor which determines the thermal conductivity enhancement [57].

The majority of the experimental data in the prevailing literature fit very well within the upper and lower limits set by the H–S model [50]. However, for the case of CNTs, the upper bound set by this model is exceptionally high. This model has a major drawback that it fails to take interfacial thermal resistance into account. Further work need to be carried out in the future to improve this model or to develop a new model to accurately estimate the thermal conductivity of carbon nanotube-based nanofluids.

The mechanism behind the thermal conductivity enhancement also remains unclear. Many mechanisms, such as Brownian motion, micro convection, formation of ordered liquid layer, ballistic motion and particle clustering have been proposed to explain the conductivity enhancement [3,5]. Tsai et al. [58] reported that in high viscous fluids Brownian motion is greatly inhibited. Hence, it is unlikely that Brownian motion and Brownian motion assisted micro convection are responsible for the conduction enhancement noticed in this work. In the presence of surfactant-encapsulated nanotubes, the formation of an ordered liquid layer may be difficult [14]. Enhancement observed in the present work can be attributed to the percolation of CNTs to form a network [14,59]. However, experimental evidence for this mechanism is still lacking. Further experiments need to be carried out to determine the underlying mechanism behind the thermal conductivity enhancement noticed in nanofluids.

#### 4. Conclusions

Single walled carbon nanotubes were synthesized by alcohol catalytic chemical vapour deposition method, and nanofluid dispersions were created using a bile salt as the surfactant. The effective thermal conductivity of the nanofluids was measured using the transient hot wire technique. The effective thermal conductivity increased in nonlinear fashion with respect to the nanotube loading. Classical models failed to predict the experimental results. Hashin–Shtrikman model sets very unrealistic upper bound for the thermal conductivity enhancement. Effective medium theory seems to perform better than rest of the models. Formation of networking structure was considered to be the potential reason behind the conductivity enhancement observed. However, further work need to be carried out to provide experimental evidence to explain the mechanism behind the heat conduction in nanofluids.

#### Acknowledgments

Part of this work was financially supported by Grant-in-Aid for Scientific Research (22226006 and 19054003). Part of this work was supported financially by the Japanese Government Monbukagakusho (MEXT) Scholarship and the “Global Center of Excellence for Mechanical Systems Innovation (GMSI)”. The authors acknowledge C. Kramberger at the University of Tokyo for helpful discussions about Raman characterization.

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