2011

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Ryglowski, Brian K.; Pollak, Randall D.; Kwon, Young W.

ASME

B.K. Ryglowski, R.D. Pollak, Y.W. Kwon, "Characterizing the stability of carbon nanotube-enhanced water as a phase change materials for thermal management systems," Journal of Thermal Science and Engineering Applications, v.2, December 2010, 041007-1/6

http://hdl.handle.net/10945/55672

Characterizing the Stability of Carbon Nanotube-Enhanced Water as a Phase Change Material for Thermal Management Systems

Brian K. Ryglowski
e-mail: brian.ryglowski@navy.mil

Randall D. Pollak
e-mail: randall.pollak@us.af.mil

Young W. Kwon
e-mail: ywkwon@nps.edu

Department of Mechanical and Aerospace Engineering,
Naval Postgraduate School,
700 Dyer Road,
Mail Stop ME/Kw,
Monterey, CA 93950

Carbon nanotube (CNT) suspensions have shown promise as a heat transfer nanofluid due to their relatively high thermal conductivity and ability to remain in stable suspension for long durations. To assess their potential as a phase change material for thermal management systems, the stability of such suspensions under repeated phase change cycles is investigated. Electrical resistance testing was used to monitor stability of the CNT network during freeze-thaw cycling. With distilled water as the base fluid, the effects of CNT size and type, CNT concentration, surfactant type and concentration, and processing parameters were investigated. Nanofluids tested included laboratory-prepared and commercially supplied samples. Experiments showed breakdown of the nanofluid in less than 12 phase change cycles for all samples tested. Ultrasonication after breakdown was shown to restore resistance values to prebreakdown levels. The results suggest the use of CNT-enhanced water as a phase change material presents a significant operational challenge due to instability of the CNT network during phase change cycling. Should the use of such nanofluids be warranted as a phase change material, electrical resistance testing along with repeated ultrasonication may be considered as a means to control and monitor stability of the nanoparticle suspension in service. [DOI: 10.1115/1.4003507]

Keywords: nanofluid, carbon nanotubes, thermal storage, phase change material

1 Introduction

One of the primary challenges in the development of many modern technologies is thermal management. Power-dense electronics and directed energy systems, in particular, must dissipate very large heat loads to avoid operating limitations and failure. One means of heat dissipation in such systems is the transfer of thermal energy to coolant fluids, which may then be piped to melt a phase change material (PCM). The latent heat of melting associated with the phase change stores energy dumped by the coolant fluid. The PCM then must be resolidified before the next operating cycle. Demanding applications require such a thermal storage system to be compact, thus minimizing the volume of PCM and associated tubing. It is thus desirable for high thermal conductivity of both coolant and PCM for efficient heat transfer. Unfortunately, the inherent properties of conventional heat transfer fluids, such as water, engine oil, ethylene glycol mixtures, and toluene render them poor thermal conductors. Hence, the search for heat transfer fluids with improved thermal conductivity has been of keen interest for such applications.

In the 1990s, it was discovered by Choi and Eastman [1] that copper nanoparticle suspensions may significantly enhance thermal conductivity, perhaps more so than predicted by classical mixing rules suggested by Maxwell in the late 19th century. Since these initial studies, colloidal nanoparticle suspensions (dubbed "nanofluids") have emerged as an exciting new class of materials for heat transfer research. A wide variety of solid nanoparticles have been investigated to date, to include copper, silver, gold, aluminum, iron, diamond, silicon, alumina, copper oxide, silicon dioxide, silicon carbide, titania, magnetite, ceria, carbon nanotubes (CNTs), fullerenes, and graphene oxide [2–12]. In particular, CNTs (consisting of a number of concentric graphene sheets rolled into tubes) have received much attention due to their very high thermal conductivity and ability to remain in stable suspension for considerable time. Studies in the early 2000s helped fuel this interest, as increases in thermal conductivity ranging from 20% to 250% (relative to the base fluid) were reported [13–16]. The variability in results is due to differences in CNT size and type, nanoparticle concentration, base fluid, processing variables, test parameters (such as temperature), and testing techniques. To help guide research and design, a recent international effort has sought to establish benchmark thermal conductivity values for such nanofluids to mitigate this variability [17]. Results of this and other recent efforts suggest the thermal conductivity enhancements due to nanoparticle additions may, in fact, be in good agreement with those predicted by classical theory [18].

Motivated by the potential for improved thermal conductivity, there has been a plethora of recent studies of heat transfer nanofluids for various applications. A brief sampling includes investigation of nanofluids flow through microchannels [19–21], in heat pipes and thermosyphons [22–27], under radial flow [28,29], and other applications including use in an automotive radiator [30–33]. In general, these studies address application-centric performance rather than basic properties such as thermal conductivity. Such performance issues may include increased pumping requirements due to higher viscosity through microchannels or enhancement of boiling heat transfer coefficients in a thermosyphon. However, there have been seemingly few investigations of nanofluids as a PCM for thermal storage, for which repeated

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Contributed by the Heat Transfer Division of ASME for publication in the JOURNAL OF THERMAL SCIENCE AND ENGINEERING APPLICATIONS. Manuscript received July 29, 2010; final manuscript received January 10, 2011; published online February 18, 2011. Assoc. Editor: Lili Zheng.

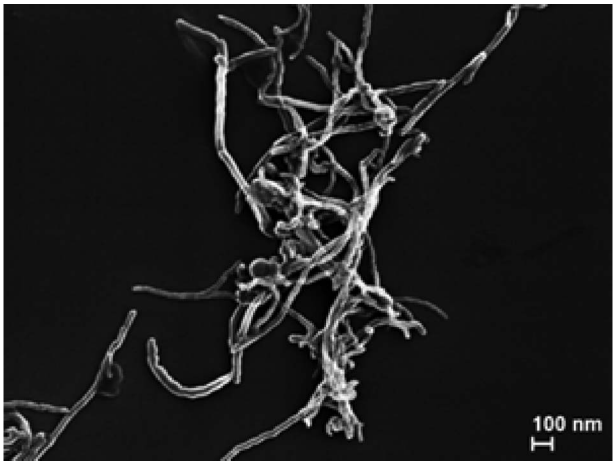

Fig. 1 Scanning electron micrograph of clustered carbon nanotubes

cycles between solid and liquid states are necessary. Stability of CNT-based nanofluids under such cycling is the concern addressed in this paper.

The attractive forces between CNTs lead to their tendency to form clusters (Fig. 1), which decreases performance for many CNT-based applications. For heat transfer nanofluids, this agglomeration decreases thermal conductivity as the CNT paths through the less conductive fluid become fewer and more widely spaced. Dependence of thermal performance on the contiguous path of nanoparticles has been demonstrated through continuum-level and atomic-level analyses by Kwon et al. [34]. Various dispersion methods have been employed to promote a more homogenous distribution. Physical dispersion techniques include high speed shearing of the nanofluid using mechanical mixing techniques, ball milling (grinding) of CNTs prior to their addition into the nanofluid, and ultrasonication of the nanofluid. Chemical dispersion techniques include the addition of side groups to the CNT outer surfaces (i.e., functionalization) and use of surfactants to lower the interfacial tension of the base liquid and CNTs. Stable suspensions typically require a combination of both physical and chemical dispersion techniques [35]. More exotic dispersion methods include the use of a magnetic field to align metal-coated CNTs within the nanofluid [36].

Assessing the stability of a CNT-based nanofluid through repeated cycles using thermal conductivity testing may be impractical due to the need for specialized equipment and variability in results due to different testing techniques. However, since both thermal and electrical conductivities of a CNT-enhanced nanofluid are dependent on the CNT network as the primary conduction path, then electrical conductivity testing has potential for monitoring nanofluid stability in a relative manner, as electrical conductivity tests are quite simple in theory and practice. Furthermore, degradation of the nanofluid (e.g., due to thermal cycling, phase changes, and/or storage) may be detected through comparison of electrical resistance measurements to initial values. It should be noted that translation of electrical conductivity measurements to thermal conductivity measurements is not direct, as these properties may show different thresholds, as shown by Glory [37].

2 Objectives

The aim of this study was to evaluate the potential for CNT-enhanced, water-based nanofluids to be used as a PCM. The focus was on characterizing the stability of nanofluids prepared with varying parameters, rather than determining the actual values of thermal conductivity. "Stability" here refers to the ability of the nanofluid to form a generally homogenous mixture and remain as such after undergoing freeze-thaw cycling of the nanofluid. This stability was to be assessed by measuring the values of electrical resistance after each phase change cycle, a simple yet unexplored approach. If the dispersed nanotubes maintained their network, both the thermal and electrical conductivities of the nanofluid should remain stable, barring any other changes within the sample. Electrical resistance changes after cycling should indicate disruptions to the CNT network. These changes may be confirmed through visible inspection as agglomeration and sedimentation. In addition to investigating the nanofluids, the use of electrical resistance testing itself is thus assessed as a means of monitoring nanofluids.

Parameters were varied to investigate the effects of CNT concentration, type of CNT, type of surfactant, and nanofluid processing temperature on the electrical conductivity and stability of the nanofluid. The impact of combining different types of CNTs in the nanofluid was also investigated.

In short, the project aimed to investigate the use of electrical conductivity testing as a surrogate for thermal conductivity testing, and then use such electrical tests to study the effects of various nanofluid and processing parameters on the stability of static nanofluids made of CNTs dispersed in distilled water. The results help address whether CNT-enhanced nanofluids are practical to use as a PCM in a thermal management system.

Table 1 Nanofluid design parameters

Parameter	Levels	Settings
CNT type	2	Single-walled CNT (SWCNT) Multiwalled CNT (MWCNT)
CNT concentration	2	0.1% volume 0.2% volume
Surfactant type	2	Lithium dodecyl sulfate (LDS) Igepal
Surfactant concentration	1	3.0% volume
Base fluid	1	Distilled water
CNT size	1	SWCNT: $1-10\ \mu\text{m}\times 1\ \text{nm}$ MWCNT: $5-20\ \mu\text{m}\times 15\pm 10\ \text{nm}$

3 Experimental Approach

3.1 Test Design. Based on the results of previous research using CNT-enhanced nanofluids, the variables shown to have a significant impact on the thermal conductivity properties of the nanofluid include base fluid, CNT type, surfactant type, CNT and surfactant concentrations (volumetric loading percentages), and aspect ratio (length divided by diameter) of the CNTs. A factorial approach was used to develop an experimental matrix. Given the time required for nanofluid processing and need to conduct phase change cycling over many days, some parameters were fixed to reduce the total number of samples to be prepared and tested. The base fluid, surfactant loading percentage, and CNT size were fixed. Two levels were used for the other specified parameters. Choices for factor settings were influenced by previous unpublished research [38], which included investigation of a number of parameters to ensure nanofluid stability after preparation and long-term storage. Table 1 lists the design parameters for the nanofluids prepared in-house for this study.

3.2 Materials. Distilled water was selected as the base heat transfer fluid as its thermal conductivity, specific heat, and latent heat of fusion make it a favorable choice for many heat transfer applications. The single-walled and multiwalled CNTs (SWCNTs and MWCNTs, respectively) used in this study were purchased from Nano-Lab, Inc. (Newton, MA). The CNTs were synthesized through chemical vapor deposition. CNTs with a high aspect ratio were selected based on their ability to form better conductive networks. Residual impurities from the CNT synthesis process include less than 1% weight of iron and sulfur according to manufacturer specifications. Chemical dispersion of the CNTs was accomplished using lithium dodecyl sulfate (LDS), an anionic compound in the form of a white powder, as well as nonionic Igepal CO-630, a clear liquid. Both surfactants were manufactured by Sigma-Aldrich, St. Louis, MO, and are soluble in water.

Additionally, prepared and processed CNT-enhanced nanofluids were provided by Luna Innovations, Inc. (Danville, VA). Six of these SWCNT-based nanofluids were evaluated, with two CNT purity levels (117% and 144% according to the Haddon method [39]) and three different CNT concentrations (0.05%, 0.1%, and 0.2%) for each purity level. The CNT synthesis and dispersion techniques used by Luna Innovations were not fully specified for proprietary reasons, but the CNTs were ultrasonically dispersed in distilled water using a 2.0% volumetric loading of gum Arabic surfactant. These samples were designated LnW-402–LnW-407 in accordance with the manufacturer-supplied nomenclature.

3.3 Equipment. For the nanofluids prepared for this study, physical dispersion was initiated using a Stir-Pak mechanical homogenizer with single propeller attachment. The homogenizer did not have an isolation assembly to prevent atmospheric gases from dissolving into the nanofluid during operation. The next step in physical dispersion was ultrasonication (transmission of ultrasonic waves to break up agglomerates), accomplished with a Sonicator 3000 ultrasonic liquid processor manufactured by Misonix, Inc., Farmingdale, NY. The available power settings ranged from 6 W to 42 W in 3 W increments. A temperature probe was used to monitor the temperature of the nanofluid during ultrasonication. An industrial freezer was used to enable the liquid-to-solid phase change of the nanofluid. The freezer was set to −13°C. All electrical resistance measurements were taken using an 8840A digital multimeter manufactured by Fluke Industries, Everett, WA. A two-wire setup was selected since the lead resistance was negligible when compared with the overall electrical resistance of the nanofluid.

3.4 Nanofluid Preparation. The basic procedures used for preparation of nanofluids are outlined as follows. First, 20 ml of distilled water was poured into a glass beaker. Surfactant was measured and placed directly into the distilled water. The distilled water and surfactant mixture were then homogenized at 600 rpm until the surfactant was completely dissolved into the distilled water. This process took about 5 min for samples containing LDS and 30 min for those with Igepal. CNTs were then measured in accordance with the prescribed test matrix and added to the distilled water and surfactant mixture, thereby forming a nanofluid. The nanofluid was again homogenized at 600 rpm; this time for 30 min. Next, the nanofluid was ultrasonicated.

The primary challenge encountered during ultrasonication was controlling the mixture temperature. The energy input from the ultrasonicator causes an increase in mixture temperature, which resulted in excessive foaming and boiling/evaporation. This may significantly alter the volume concentration of surfactant and nanoparticles in the mixture. To avoid excessive boiling, four different ultrasonication power and time settings were preliminary evaluated using distilled water with a 3.0% (by volume) concentration of surfactant. Both LDS and Igepal surfactants were evaluated separately. Nanotubes were not added to the fluid for this preliminary testing. Results showed that less than 1 ml of fluid evaporated at a power setting of 15 W applied for 15 min, which was then used as the protocol for this study. Samples were ultrasonicated at these settings both under ambient conditions and with the beaker set in a water/ice mixture (“ice bath”) to reduce processing temperature. Further optimization of processing parameters was not attempted.

Thus, a total of 16 nanofluid samples were prepared (eight possible combinations from Table 1, each produced with and without an ice bath). Those nanofluids provided by Luna Innovations were not modified and were used as received, with 50 ml of nanofluid drawn for each sample into a glass beaker for resistance testing and phase change cycling.

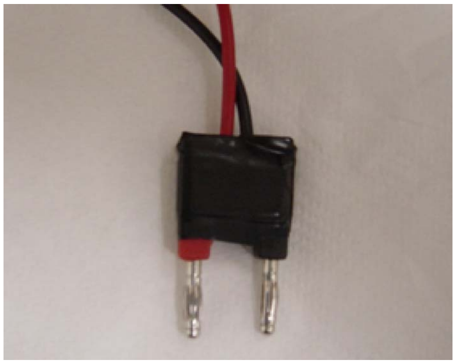

Fig. 2 Electrical leads used for resistance measurements

3.5 Electrical Resistance Testing. Several means of resistance testing of the nanofluid resistance were investigated, starting with a parallel metal plate assembly separated by an insulator, forming a cavity into which nanofluid was poured. Variability and current-induced capacitive effects led to a new design, in which the beaker was capped by a rubber stopper with conductive metal probes inserted. The electrical leads of the multimeter were then attached to the probes, but it was found that too much variability was introduced due to slight changes in the contact points each time the leads were attached. Finally, it was determined that the most reliable means of making resistance measurements was the simplest, namely, inserting the electrical leads from the multimeter directly into a nanofluid-filled container and recording direct-current electrical resistance values. The leads were set at a fixed separation to ensure a consistent path length for each resistance test (Fig. 2).

Initial tests showed that resistance was dependent on the depth to which the leads were inserted, with higher resistances near the surface and lower resistances near the bottom. This result is due to an increasing concentration of the highly conductive CNTs toward the bottom of the container. For consistency, a test protocol was established in which each reported resistance value was actually the average of 15 measurements, with leads inserted approximately halfway deep into the nanofluid. The 15 measurements were taken after cleaning of the leads and reinsertion. Validation tests showed that resistance values varied by less than 3% depending on orientation angle of the leads, so long as the depth of insertion was consistent. Thus, there was no further attempt to control for orientation angle in resistance measurements. In practice, there was generally little variability in each set of 15 resistance measurements for each nanofluid. The test procedures ensured that observed differences in electrical resistance values could be attributed to changes in the morphology of the CNT network rather than external factors. The initial resistance measurements for each sample were taken after 5 min of room-temperature cooling following ultrasonication for those samples prepared in-house.

Resistance measurements were repeated after each phase change cycle (freezing to −13°C overnight and then thawing to room temperature under ambient conditions). Nanofluids prepared in-house were cycled five times or until the sample showed complete CNT network breakdown (as evidenced by layered appearance and large resistance increase). The nanofluids supplied by the vendor were cycled up to 12 times to evaluate the stability through extended cycles. In addition, these samples were re-ultrasonicated at 15 W for 5 min after breakdown to investigate the ability to rehomogenize the sample. Resistance measurements for these samples were taken up to the 6th cycle, and then only after the 12th cycle.

In addition to resistance measurements, samples were held up

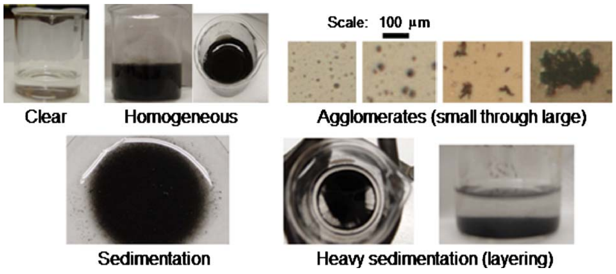

Fig. 3 Reference images for the visual appearance of nanofluids during phase change cycling

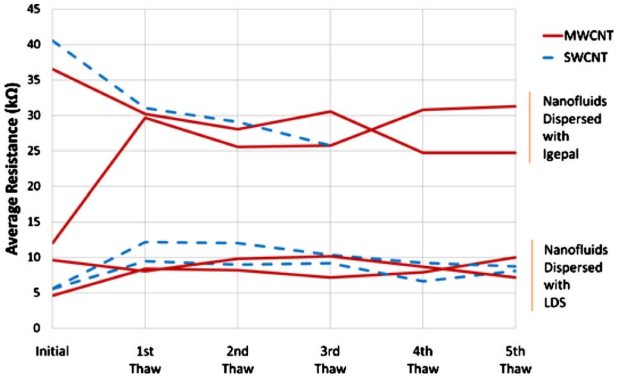

Fig. 5 Resistance values for nanofluids prepared with 0.1% CNT loading by volume

to white light after each phase change to look for observable sedimentation or agglomeration. A rating scale was developed to classify the nanofluid appearance (Fig. 3).

3.6 Additional Testing. Ancillary tests were also conducted in which MWCNTs were added to the SWCNT-based Luna samples to investigate whether a combination of CNT types may yield better stability. This investigation was spurned by the idea that MWCNTs may be stiffer and less prone to agglomeration, while SWCNTs are more conductive. Thus, a combination of the two types may yield better results as MWCNTs provide a network for which more conductive SWCNTs may become entangled. However, the combined-type suspensions segregated after just a single phase cycle, and these tests will not be discussed further. It is possible that refinement of nanofluid parameters and preparation procedures may yield more stable suspensions in the future.

4 Results

4.1 Laboratory-Prepared Samples. Of the 16 samples prepared in accordance with the test matrix, two were unable to form stable suspensions following ultrasonication. Neither of the two samples with SWCNTs and Igepal prepared without ultrasonication temperature control formed a homogeneous mixture. The other 14 samples were successfully subjected to phase change cycling, each for 5 cycles. The resistance values for each are plotted by cycle in Fig. 4. Only two samples experienced complete separation before 5 cycles. These were the two samples with SWCNTs and Igepal using ice bath control. Thus, none of the samples with SWCNTs and Igepal was very stable. It should be noted that all samples experienced some visually identifiable sedimentation and/or agglomeration after the first phase change cycle.

4.1.1 Ultrasonication Temperature Control. Using an ice bath during nanofluid ultrasonication did not appear to have a major effect on the nanofluid resistance throughout thermal cycling, although it did seem to enhance stability for the SWCNTs dispersed with Igepal. Differences in the initial readings between samples with and without an ice bath (Fig. 4) are due to temperature differences, as initial readings were made 5 min after cooling, rather than waiting until complete thermal equilibrium with the environment. Nanofluids prepared with no temperature control reached temperatures in excess of 70°C compared with 21°C using an ice bath. The conductivity of CNTs is notably higher at warmer temperatures. It is recommended that future tests ensure thermal equilibrium for baseline resistance measurements rather than a prescribed waiting interval.

After the first phase change, all nanofluids were tested at the same temperature and resulted in similar resistance measurements depending on the surfactant. Nanofluids dispersed using the anionic LDS had lower resistances than those dispersed with nonionic Igepal, as expected. No apparent relationship between the effects of controlling nanofluid temperature during ultrasonication on the physical stability of the nanofluid could be identified. However, slightly larger agglomerates formed in nanofluids prepared using an ice bath.

4.1.2 CNT Concentration and Type. The results are arranged in Figs. 5 and 6 to show the effects of CNT concentration and CNT type. Nanofluids prepared both with and without an ice bath are plotted together. Based on the data, the type of CNT did not play a major role in the overall electrical resistance of the nanofluid. As expected, the concentration of CNTs had a more noticeable effect on the resistance of the nanofluid. The 0.2% concentration of MWCNTs and SWCNTs showed a decrease in the nanofluid resistance in both types of surfactants. However, larger agglomerates formed as thermal cycling progressed for higher CNT concentrations.

MWCNTs exhibited more noticeable growth of agglomerate

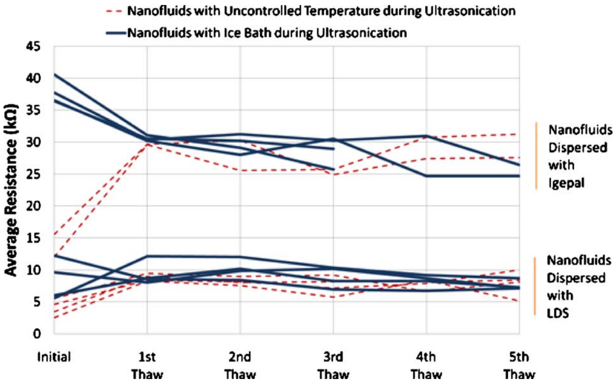

Fig. 4 Electrical resistance values for all in-house prepared nanofluids

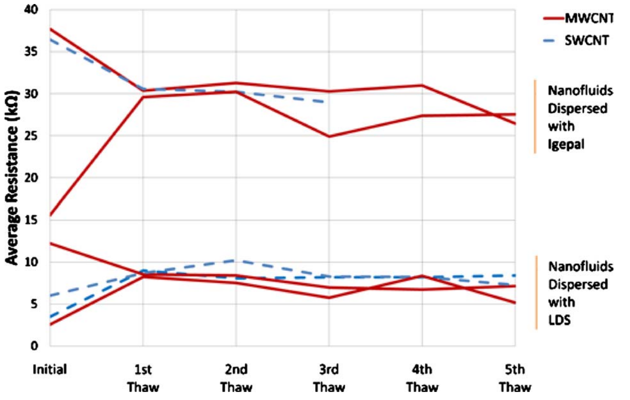

Fig. 6 Resistance values for nanofluids prepared with 0.2% CNT loading by volume

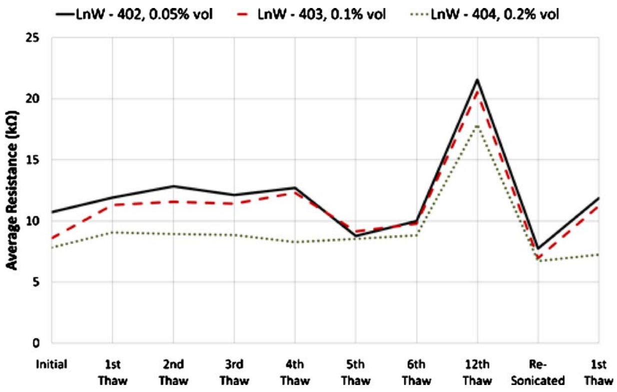

Fig. 7 Resistance measurements for lower-purity commercial nanofluids at three different concentrations of SWCNTs

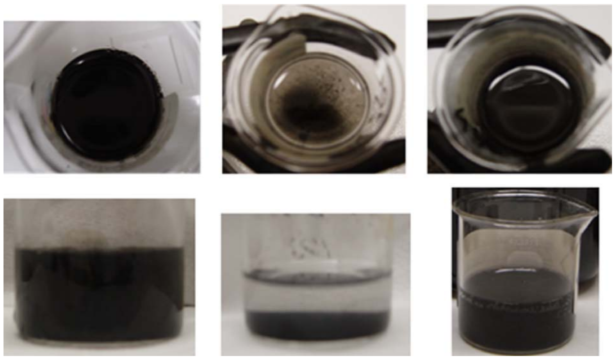

Fig. 9 Comparison of a commercial nanofluid before phase change cycling (left), after 12 cycles (middle), and after re-ultrasonication (right)

size when dispersed in LDS than in Igepal. However, this agglomeration was still mild enough to avoid any noticeable increases in electrical resistance or complete separation of the nanofluid. SWCNTs dispersed in Igepal proved to be more unstable than those dispersed in LDS, as SWCNTs in Igepal separated on the fourth phase change cycle for those samples prepared with an ice bath.

It is important to note that the sizes of the MWCNTs and SWCNTS were not identical, as their structures are quite different. Thus, size effects are slightly confounded with type effects. The aspect ratios were as close as possible given the selection of nanoparticles available, but due to the larger diameter of MWCNTS, their length was also longer, which could account for their propensity to form larger agglomerates.

4.2 Commercially Prepared Nanofluids. The SWCNT-based nanofluids tested as delivered showed exceptional stability throughout the first five thermal cycles, as shown in Figs. 7 and 8 in which samples are grouped into lower-purity and higher-purity groups according to manufacturer-specified data. For all six samples, the nanofluids remained visually homogeneous until the sixth cycle, when light sedimentation and/or small agglomerates were first observed in each of the samples. The resistance measurements were taken through the sixth cycle and remained generally stable compared with initial values. The degree of cycle-to-cycle fluctuation before breakdown was comparable to the variability observed in the laboratory-prepared samples. By the 12th cycle, all six nanofluids progressed to moderate agglomeration and heavier sedimentation, and the resistance of each sample increased noticeably. At this point, each sample was ultrasonicated at 15 W for 5 min, and the resistance levels returned to prebreakdown levels and the samples again appeared homogenous, and remained so after an additional phase change. Figure 9 shows sample LnW-402 upon initial testing, after the 12th thermal cycle, and after re-ultrasonication. The nanofluid was effectively able to be returned to its original status by the 5 min ultrasonication treatment.

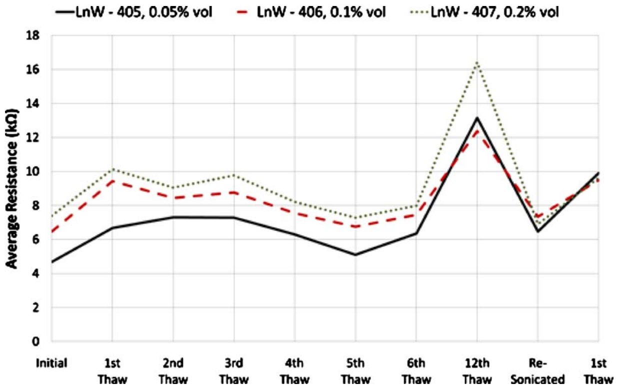

Fig. 8 Resistance measurements for higher-purity commercial nanofluids at three different concentrations of SWCNTs

The nanofluids with a lower purity (Fig. 7) exhibited a decreasing resistance with higher CNT concentrations just as the in-house nanofluids, but the resistance of the higher-purity samples (Fig. 8) increased as CNT concentration increased. No distinct rationale could be drawn to explain this behavior, and it was suspected that the vendor perhaps mislabeled the jars, but discussions with the vendor and analysis of the nanofluids could not confirm this suspicion. Regardless, the commercial nanofluid samples all exhibited comparable behavior, and neither CNT concentration nor purity affected the overall stability.

5 Conclusions

Because both the electrical conductivity and thermal conductivity of a CNT-based nanofluid are dependent on maintaining a dispersed network of CNTs, the electrical conductivity testing proved a useful and simple means of monitoring the state of the CNT network during repeated phase changes. Significant increases in electrical resistance corresponded to cycles at which the nanofluid experienced significant agglomeration and sedimentation. It should be noted, however, that the electrical resistances generally changed little despite observed light to moderate agglomeration and sedimentation. This result suggests that even with a mild amount of breakup in the nanotube network, electrical conductivity paths remained relatively intact. It is not clear whether thermal conductivity would remain as stable under such mild deterioration and this may be an area of interest for further study.

After demonstrating the utility of electrical resistance testing in lieu of thermal conductivity testing for nanofluid monitoring, the primary objective of this research was to evaluate the stability of CNT-enhanced nanofluids as a phase change material. Regardless of CNT type, size, concentration, preparation procedure, or surfactant type, those CNT-enhanced nanofluids undergoing phase change cycling in this study were stable for at least 3 cycles, but each showed significant agglomeration and/or sedimentation by 12 cycles. The effects of CNT type or concentration did not seem to have a major impact on stability over the range of values tested. However, the use of temperature control during processing may reduce agglomeration tendency. The major finding here is that nanofluids, which may be in stable suspension on the shelf under ambient conditions for months, can very quickly show signs of agglomeration and sedimentation under just a few phase change cycles. Their consideration as a phase change material must account for this lack of stability.

The use of re-ultrasonication after CNT network breakdown (indicated by markedly increased electrical resistance and increased agglomeration and sedimentation) was shown to return resistances and appearances to the original state of the nanofluid. Thus, the use of electrical resistance monitoring may provide a simple means to detect breakdown, and ultrasonication may provide the simple means to reverse breakdown. Thus, thermal performance could be ensured, although the system-level benefits must be weighed against the system-level impacts of such monitoring and processing equipment.

In conclusion, this research has shown that CNT-based nanofluids can withstand a limited number of phase changes before significant agglomeration and sedimentation occur. The number of phase changes before such breakdown was dependent on the quality of initial nanofluid processing and parameters of the nanofluid, but the nanofluids used in this study generally deteriorated significantly within 12 cycles. Direct correlation between electrical and thermal conductivities may be possible but was beyond the scope of this study. Such work is recommended for continued research in this area.

Acknowledgment

This project was sponsored through a cooperative research agreement between the Naval Postgraduate School (NPS) and Northrop Grumman Electronic Systems (NGES) in Sunnyvale, CA. The authors appreciate the project leadership of Mr. David Rummler of NGES and the microscopy assistance of Professor Sarath Menon of the Department of Mechanical and Aerospace Engineering at NPS.

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