

Enhancement of thermal conductivity with Cu for nanofluids using chemical reduction method

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Received 1 June 2005; received in revised form 3 February 2006

Available online 4 April 2006

Abstract

The enhancement of the thermal conductivity of water in the presence of copper (Cu) using the chemical reduction method is presented in this study. It is the first time that the chemical reduction method for synthesis of nanofluids containing Cu nanoparticles in water is reported. No surfactant is employed as the dispersant. The volume concentration of Cu–water nanofluids is below 0.2 vol.%. Without the addition of dispersant and surfactant, the thermal conductivity of the produced nanofluids reveals a time-dependent characteristic. The thermal conductivity is the largest at the starting point of measurement and decreases considerably with elapsed time. The results show that Cu–water nanofluids with low concentration of nanoparticles have noticeably higher thermal conductivities than the water base fluid without Cu. For Cu nanoparticles at a volume fraction of 0.001 (0.1 vol.%), thermal conductivity was enhanced by up to 23.8%. © 2006 Elsevier Ltd. All rights reserved.

Keywords: Nanofluid; Thermal conductivity; Cu nanoparticle

1. Introduction

The thermal conductivity of thermofluid plays an important role in the development of energy-efficient heat transfer equipments. Passive enhancement methods are commonly utilized in the electronics, HVAC&R, and transportation devices. However, the thermal conductivities of the working fluids such as ethylene glycol, water, and engine oil are relatively lower than those of solid particles. In that regard, the development of advanced heat transfer fluids with higher thermal conductivity is thus in a strong demand.

Nanoparticle technology is of considerable interest for a large number of practical applications. A new approach to nanoparticles in nanofluid was proposed by Dr. Choi at the USAs Argonne National Laboratory in 1995 [1]. Consider-

able research and development focusing on nanofluids has been recently conducted [2–6].

In 1857, Michael Faraday first reported the study of the synthesis and colors of colloidal gold [7]. After the first report on the chemical reduction of transition metal salts in the presence of stabilizing agents by Faraday [8], Turkevich et al. [9] and Turkevich [10] established the first reproducible standard protocol for the preparation of metal colloids [11].

More recently the chemical approach using wet chemistry is emerging as a powerful method for growing nanostructures of metals, inorganic semiconductors, organic materials, and organic–inorganic hybrid systems. The advantage offered by nanochemistry is that surface functionalized nanoparticles and nanorods of metals or inorganic semiconductors, dispersible in a wide variety of media such as water can be readily prepared. Furthermore, nanochemistry also lends itself to a precise control of conditions to produce monodispersed nanostructures [12].

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Copper nanoparticles are of great interest because of their use as coolant and its application in heat exchanger. The synthesis methods for copper nanomaterials include physical method and chemical method. The physical method used for copper nanofluids has been reported [2,3]. Nanofluids consisting of Cu nanoparticles directly dispersed in ethylene glycol have been observed to exhibit significantly improved thermal conductivity enhancements compared to nonparticle-containing fluids or nanofluids containing oxide particles [3]. On the other hand, gold and silver nanofluids synthesized using chemical method has been investigated [4]. However, copper nanofluids formed by chemical method are not reported yet.

Many studies on the thermal conductivities of nanofluids had focused on the nanofluids synthesized from physical method. Recently, our group has also reported the results of CuO–ethylene glycol nanofluids based on this method [6]. However, the reported enhancement of thermal conductivity for nanofluids via two-step method is usually not exceptionally high. As a consequence, the present study reports for the first time the chemical reduction method for synthesis of nanofluids containing Cu nanoparticles in water. The thermal conductivities of the Cu suspensions are measured by a transient hot wire method.

2. Experiments

In the present work, chemical reduction method is used to synthesize copper (Cu) nanoparticles in the presence of water as solvent under nitrogen atmosphere. This process is different from the previous method. The physical method is adopted in previous study [5,6].

Copper acetate ($\text{Cu}(\text{CH}_3\text{COO})_2$) was used as the precursor. The copper acetate was dissolved in deionized water. The solution was stirred uniformly at a temperature of 55 °C under nitrogen. Hydrazine (N_2H_4) acts as a reducing agent. A predetermined quantity of hydrazine solution was then added slowly to copper acetate solution with stirrer being operated simultaneously till the solution became uniform. The mixture was cooled down to 0 °C then kept deposited. The mixture was slowly washed with deionized water to remove impurity. The product was then obtained. The volume fraction of Cu nanoparticle suspensions in water liquid is below 0.2 vol.%.

All chemicals were used as received. Water has a high permittivity which makes it a good solvent for polar or ionic compounds. Therefore, many chemical reactions take place in aqueous media [13]. Highly pure water was obtained from a Millipore Milli-Q plus system.

Taguchi's method was also applied to study the synthesis factors. Effects of variables including concentrations of precursor, amount of reducing agent, and amount of water solvent were investigated. The precursor concentrations are in the ratio of 1, 2, and 4 while the concentrations of reducing agent are in the ratio of 1, 2.5, and 5, respectively. The mass ratios of water solvent are 1–3 at increment of one.

The copper nanoparticle was measured with scanning electron microscopy (SEM) to determine its microstructure. The thermal conductivity of Cu–water nanofluid was measured with a computer-controlled hot wire system. The details have been described elsewhere [5,14]. Thermal conductivity was measured at room temperature.

For the transient hot wire system, a thin platinum wire was immersed in the fluid using a vertical cylindrical glass container. The hot wire served as an electrical resistance thermometer. A Wheatstone bridge heated the platinum wire and simultaneously measured its resistance. The electrical resistance of the platinum wire varies in proportion to changes in temperature. The thermal conductivity was then estimated from Fourier's law. The transient hot wire system was calibrated with deionized water at room temperature. The Cu–water nanofluids were filled into the glass container to measure the thermal conductivity. The inner diameter and length of long glass container are 19 mm and 240 mm, respectively.

A time sequence of thermal conductivity measurement of the copper nanofluids was conducted at intervals of 1–5 min. The measurement ended up at 30 min when there was no apparent change in the thermal conductivity. The time profile of the thermal conductivity distribution of the copper nanofluids then can be examined.

3. Results and discussion

Copper nanoparticles are produced in water by reduction of copper acetate. The Cu^{2+} ions are converted into copper atoms. The copper atoms then precipitate to form copper nanoparticles. The color of the solution will gradually change from light brown to brown and even to dark brown. The volume fractions of Cu nanoparticle suspensions in water liquid are from 0.05% to 0.2 vol.% with an interval of 0.05%.

Typical SEM micrographs of Cu nanoparticles are shown in Fig. 1. Fig. 1(a) shows a micrograph of a Cu–water nanofluid at 0.1 vol.% (specimen No. 4). Fig. 1(b) exhibits a micrograph of a Cu–water nanofluid at 0.1 vol.% (specimen No. 5). Copper nanoparticles synthesized by chemical reduction shows the monodispersed distribution of particle sizes. The grain size and shape of Cu–water nanofluids as a function of nanoparticle volume fraction is shown in Table 1. The agglomerated particle sizes of the Cu nanoparticles range from 50–100 nm with spherical and square shapes. On the other hand, Fig. 1(c) illustrates a micrograph of a Cu–water nanofluid at 0.2 vol.% (specimen No. 9). The typical particle sizes of the copper nanoparticles are >250 nm with spherical, square, and needle assorted shapes.

Fig. 2 displays the energy dispersive spectroscopy (EDS) patterns of Cu nanoparticles. In tandem with the scanning electron microscope, it is used to determine the chemical composition of a microscopic area of a solid sample. The two strong peaks point out the element of Cu [15]. This indicates that Cu particle solid nanoparticles are synthesized from their precursors using a chemical reaction. On

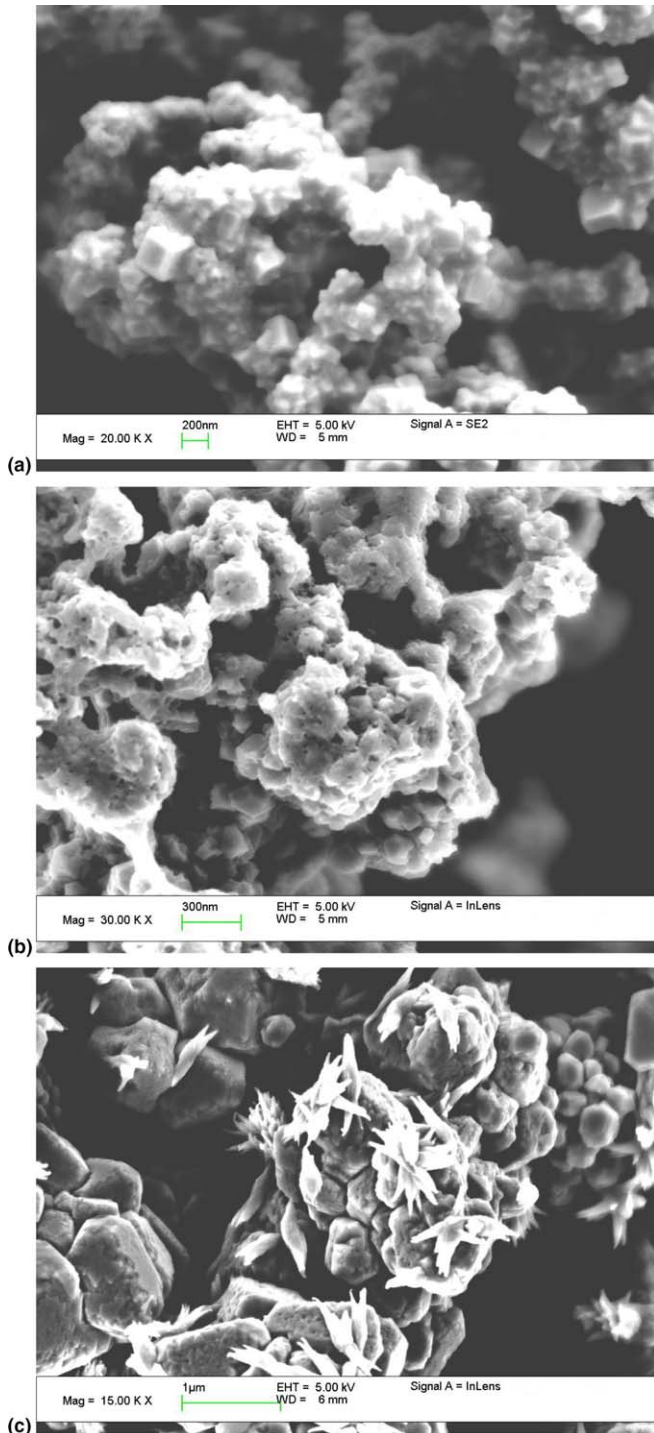


Fig. 1. Typical SEM micrographs of Cu nanoparticles for (a) specimen No. 4, (b) specimen No. 5, and (c) specimen No. 9.

the preparation of Cu nanoparticles for SEM, the Cu nanoparticles are coated with Au and Pd to increase the electrical conductivity before sent to vacuum chamber of SEM for EDS measurement. The additional peaks of Au and Pd are thus observed.

The thermal conductivity increased ratio of Cu–water nanofluids as a function of nanoparticle volume fraction is shown in Table 1. The thermal conductivity ratio of

Table 1
Enhanced thermal conductivities of Cu–water nanofluid

Specimen No.	Volume fraction (vol.%)	Thermal conductivity increased ratio (%)	Grain size and shape from SEM
1	0.05	11.6	100–200 nm, spherical and square
2	0.05	3.6	Not available
3	0.05	8.5	130–200 nm, spherical and square
4	0.1	23.8	75–100 nm, spherical and square
5	0.1	23.8	50–100 nm, spherical and square
6	0.1	11.0	100–300 nm, spherical, square, and needle
7	0.2	9.7	130–300 nm, spherical
8	0.2	13.2	200 nm × 500 nm needle
9	0.2	3.6	250 nm, spherical, square, and needle

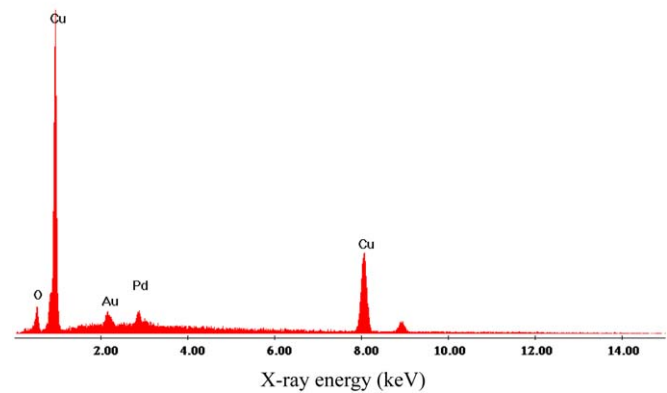


Fig. 2. Typical EDS patterns of Cu nanoparticles.

Cu–water nanofluids increases with the increase of volume fraction of Cu until it reaches a maximum value at 0.1 vol.%. It is interesting to note that above this volume fraction thermal conductivity decreases. This behavior indicates the interesting aspect of Cu–water nanofluids synthesized from chemical reduction method. It is likely that the decrease of thermal conductivity ratio above this threshold fraction is caused by the larger size of Cu nanoparticles (>100 nm).

The normalized thermal conductivity data for the Cu–water nanofluids as a function of the measured time is shown in Fig. 3. The k denotes the thermal conductivity of Cu–water suspensions and the k_{base} is the thermal conductivity of the water base fluid. The as-synthesized Cu nanofluid is added into the vertical cylindrical glass container of the transient hot wire (THW) system after cooling down. By prescribing the input temperature to the Cu nanofluid, the associated thermal conductivity was measured directly by THW system and adjusting the variable resistor of the Wheatstone bridge circuit accordingly, the measurement of thermal conductivity could begin without

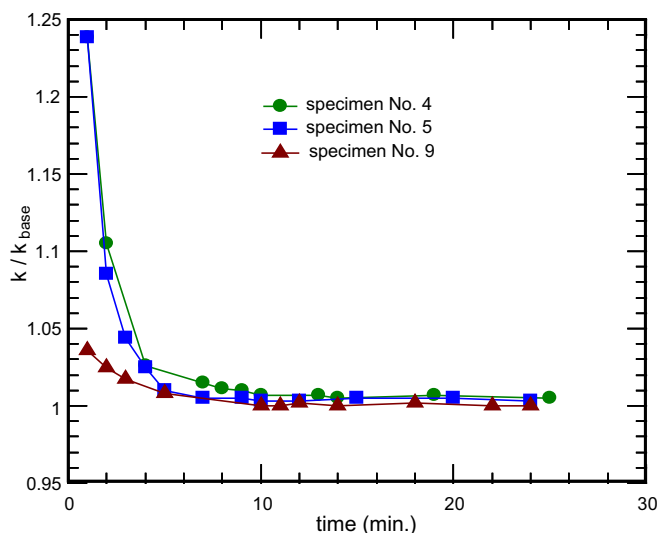


Fig. 3. The normalized thermal conductivity data for the Cu–water nanofluids as a function of the measured time.

further agitating in the glass container. Therefore, it takes about 1 min to get the first point of thermal conductivity after the Cu nanofluid is added. This point is regarded as the first measured point at time of 1 min. During thermal conductivity measurement, the Cu nanofluid is kept as it is with no further action.

On the other hand, it is also possible that the Cu nanoparticles synthesized from chemical reduction method without addition of dispersant and surfactant could be agglomerated to some certain extent, not separated individually. Therefore, the thermal conductivity of the Cu nanofluid shows the time-dependence behavior. However, the addition of dispersant and surfactant would make the Cu surface coated, thereby resulting in the screening effect on the heat transfer performance of Cu nanoparticle.

From this figure one can see that Cu–water nanofluids with a low concentration of nanoparticles have considerably higher thermal conductivities than the identical water base liquids without solid nanoparticles. A strong dependence of thermal conductivity on the measured time is observed. In addition, one also can see that at a constant volume fraction, k/k_{base} is the largest at the starting point of measurement and drops considerably with elapsed time. For Cu nanoparticles at a volume fraction of 0.001 (0.1 vol.%), thermal conductivity is enhanced by 23.8%. The ratio of k/k_{base} is almost unchanged when the elapsed time is above 10 min. The value of k/k_{base} is slightly above unity, indicating no appreciable enhancements due to particles agglomeration.

Fluids with solid particles on a nanoscale show better thermal conductivities than fluids with coarse solid particles on a microscale. This is due to the larger total surface areas of nanoparticles. Some possible explanations of the enhancement of nanoscale particles can be found from Lee et al. [14]. The particle sizes of the Cu nanoparticles for specimens Nos. 4 and 5 range from 50–100 nm with

spherical and square shapes and those of specimen No. 9 are larger than 250 nm with spherical, square, and needle assorted shapes. It can therefore be expected that their respective nanofluids' thermal conductivity behave differently. Note that Cu nanoparticles are kept in their original form as synthesized by chemical method. This is attributed to the standard SEM micrographs of Cu nanoparticles in the following:

- (1) Obtain Cu nanofluid in solution form.
- (2) Dropped one drop of the solution on sticky tape of top surface of SEM specimen holder.
- (3) Heated in vacuum oven to dry the liquid drop or dried in air naturally.
- (4) Obtain solid Cu particle.
- (5) Sent to vacuum chamber of SEM for picture after coated with Au and Pd.

The value of thermal conductivity increased ratio (%) has strong relationship with the grain size of Cu nanoparticle. Generally, smaller size gives rise to in higher thermal conductivity increased ratio. For Cu nanoparticles, the primary particle sizes range from 50–100 nm with spherical and square shapes. On the microstructure characterization of Cu nanoparticles with SEM, the water of Cu nanofluid is removed from the top surface of SEM specimen holder by heating in vacuum oven. The solution is being dried and Cu nanoparticles agglomerated together. The agglomerated secondary particle sizes could be thus over one micron. In addition to agglomeration without any surfactant, the large Cu agglomerates would result in bad dispersion and would further accelerate the drop of thermal conductivity with time.

The reaction conditions including concentrations of the precursor and reducing agent that can be used to control nanoparticle sizes and nanoparticle shapes. Taguchi's method is applied to study the process factors to reach small size and narrow distribution of copper nanoparticles. The copper size depends on the precursor concentration and reducing agent concentration. Different combinations of concentrations produce different size nanoparticles. The Cu nanoparticles with the highest thermal conductivity enhancement are formed with higher concentration of copper acetate precursor and hydrazine reducing agent. The reducing agent will not be enough to reduce all the copper precursors if less reducing agent is added.

Suspensions containing a small amount of Cu nanoparticles have also been reported to have considerably higher thermal conductivities. Xuan and Li [2] showed that the ratio of the thermal conductivity of the copper–water nanofluid to that of the base liquid varies from 1.24 to 1.78 when the volume fraction of the nanoparticles increases from 2.5 vol.% to 7.5 vol.%. The corresponding Cu nanoparticles were about 100 nm diameter and were directly mixed with deionized water. The laurate salt at several weight percents was used to enhance stability of the suspension.

Chemical reduction method is used for the preparation of copper–water nanofluids. Comparison with the present experimental results and the results by Xuan and Li [2] shows that copper–water nanofluids synthesized from chemical reduction method display much better properties than the nanofluids synthesized from physical method. The thermal conductivity enhancement is found to be of same order for Cu nanoparticles in water at different volume fractions. However, the volume fraction of Xuan and Li [2] is one order of magnitude higher than the volume fraction obtained in this study. This observation may be attributed to the difference in the size of Cu nanoparticles.

Eastman et al. [3] showed that copper–ethylene glycol nanofluid has a much higher thermal conductivity than either pure ethylene glycol or ethylene glycol containing the same volume fraction of dispersed copper oxide nanoparticles. The thermal conductivity is shown to be increased by up to 40% for a nanofluid containing approximately 0.3 vol.% Cu nanoparticles at a mean diameter less than 10 nm. They developed a one-step physical method to produce the associated nanofluids. This technique involves the direct evaporation of Cu sample and the condensation of vapor into nanoparticles by contact with a flowing low vapor pressure liquid. Cu nanoparticles with little agglomeration having an average diameter of less than 10 nm were produced. For some samples, a small amount of thioglycolic acid (<1 vol.%) as a stabilizing agent was added to the nanofluid to improve the particle dispersion behavior.

In this study, chemical reduction method is used for the preparation of copper–water nanofluids. Comparison with the present experimental results and the results of Eastman et al. [3] shows that both results for Cu–water nanofluids and Cu–ethylene glycol nanofluids, having Cu particle concentration well below one volume percent, exhibit the same trend. The thermal conductivities of Cu–ethylene glycol nanofluids and Cu–water nanofluids are shown to be increased by up to 40% at 0.3 vol.% and 23.8% at 0.1 vol.%, respectively.

Patel et al. [4] showed that thermal conductivity enhancement of 5–21% in the temperature range of 30–60 °C at a concentration loading of 0.026 vol.% for gold–water nanofluid. The gold nanoparticles of 10–20 nm in mean diameter were made with citrate stabilization. On the other hand, comparatively lower thermal conductivity enhancement is observed for larger silver particles for their larger particle size.

Fig. 4 shows a plot of thermal conductivity as a function of volume fraction for gold–water nanofluid [4], Cu–water nanofluid (present study), and Cu–ethylene glycol nanofluid [3]. The thermal conductivity enhancement is found to be of different order at different volume fraction. From this figure, however, one can see that there is a linear relationship between thermal conductivity and volume fraction approximately.

The typical value of thermal conductivity is $0.25 \text{ W m}^{-1} \text{ K}^{-1}$ for ethylene glycol, $0.6 \text{ W m}^{-1} \text{ K}^{-1}$ for water, $300 \text{ W m}^{-1} \text{ K}^{-1}$ for gold, and $400 \text{ W m}^{-1} \text{ K}^{-1}$ for copper.

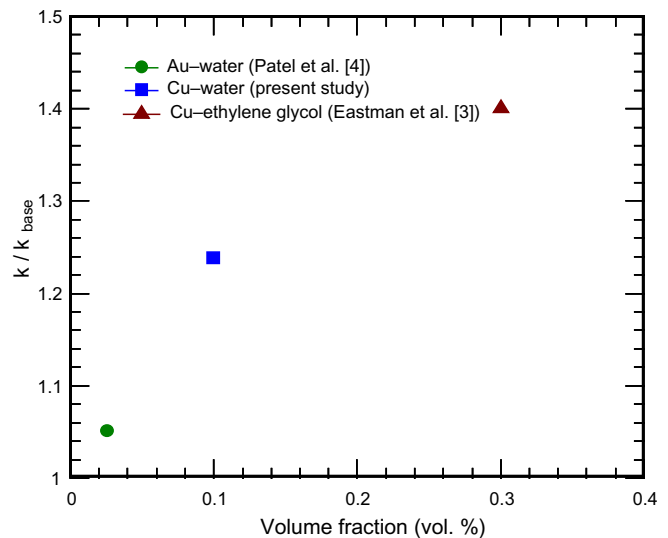


Fig. 4. The thermal conductivity as a function of volume fraction for the Au and Cu water base nanofluids.

There are three orders of magnitude difference between liquids and metals for thermal conductivity. Therefore, fluids containing solid particles can be anticipated to show appreciably enhanced thermal conductivities compared with pure fluids. The typical values of thermal conductivity for Au and Cu are in the same range and thus those nanoparticles have similar effect on the thermal conductivity enhancement of nanofluids at comparable volume fraction.

Liu et al. [6] studied the enhancement of the thermal conductivity of ethylene glycol in the presence of copper oxide. The CuO nanofluids were prepared in a two-step method. No surfactant is employed as the dispersant in the water-based nanofluid. For CuO nanoparticles at 5 vol.%, thermal conductivity is enhanced by up to 22.4%. On the other hand, for the oil-based nanofluid, Li and Chang [16] used the dispersants of oleic acid and sodium oleate to disperse CuO nanoparticles in *n*-alkanes. The dispersion process with sodium oleate has more adsorptions of oleate on CuO nanoparticles than oleic acid does. The prepared organic suspensions from sodium oleate-modified CuO nanoparticles are much more stable. Thermal conductivity measurement is not reported.

Stabilizing agents are usually added into the solution to prevent the nanoparticles from aggregating in chemical method. The protective enveloping of nanoparticles may be achieved by use of organic polymers such as PVP [17]. However, the stabilizing agents tend to associate with the surface of the nanoparticle. In this study, without the addition of surfactant, the surface of Cu nanoparticle contacts the water solvent directly to enhance the thermal conductivity effectively. The heat transfer at the free surface of the nanoparticles is more active than coated surface with stabilizing agents.

Recently, Jang and Choi [18] proposed a theoretical model for the role of dynamic nanoparticles in nanofluid. The Brownian motion on the nanoparticles at the molecular and nanoscale level is a major mechanism controlling

the thermal conductivity of nanofluid. The fundamental difference between solid/solid composites and solid/liquid suspensions is identified. Thus solid/liquid interface characteristics need to be exploited. On the other hand, Vadasz et al. [19] investigated the heat transfer enhancement revealed experimentally in nanofluids suspensions by applying the hyperbolic heat conduction constitutive relationship. The experimental results of Cu–ethylene glycol nanofluid [2] and Cu-deionized water nanofluid [14] prepared from physical mixing method were evaluated. It is observed that the hyperbolic heat conduction could be the source of the excessively high effective thermal conductivity of the suspensions.

At present study, Cu nanofluids are prepared using chemical method and the volume fraction of Cu has been increased to 0.1 vol.% (~1 wt.%). A significant increase in thermal conductivity up to 23.8% is observed at single measurement point. The thermal conductivity decreases with elapsed time and the thermal conductivity remains almost constant after a period of time (~30 min). For durability and reliability concerns, the synthesis of nanoparticle utilizing stabilizing agent is an important subject for future developments. Furthermore, a mixing tool could be used to agitate the Cu nanofluid continuously to achieve good dispersion dynamically for application of nanofluid in a thermofluid device.

4. Conclusions

This study conducts a chemical reduction method to produce Cu–water nanofluids. The chemically synthesized copper nanoparticles in water without surfactant significantly improve thermal conductivities, compared to pure fluids. Copper nanoparticles show the characteristics of small particle sizes and uniform size distribution. The typical copper nanoparticles are around 50–100 nm in diameter with spherical and square shapes. The strong peaks of EDS indicate the Cu solid nanoparticles are synthesized from their precursors using a chemical reaction. The volume fractions of Cu nanoparticle suspensions in water liquid are in the range from 0.05% to 0.2 vol.%. Without the addition of dispersant and surfactant, the thermal conductivity of the produced nanofluids reveals a time-dependent characteristic. The thermal conductivity is the largest at the starting point of measurement and decreases considerably with elapsed time. The ratio of k/k_{base} is almost unchanged when the elapsed time is above 10 min. The value of k/k_{base} is slightly above unity, indicating no appreciable enhancements due to particles agglomeration. Initially, the thermal conductivity of Cu/water suspension can be enhanced by 23.8 percent at a volume fraction of 0.001 (0.1 vol.%). The higher thermal conductivities of Cu solid materials together with the larger surface area of Cu nanoparticles are keys to the enhancement of thermal conductivity. The present study presents Cu–water nanofluids synthesized from chemical method, for the first time, to improve the thermal conductivities of conventional heat transfer water-based fluids.

Acknowledgements

Support of this research through a grant from Bureau of Energy, Ministry of Economic Affairs, Taiwan, is greatly acknowledged. The authors also wish to express their sincere appreciation to Dr. Choi of Argonne National Laboratory for his help.

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