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Enhanced thermal conductivity of nano-SiC dispersed water based nanofluid

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Abstract. **Silicon carbide (SiC) nanoparticle dispersed water based nanofluids were prepared using up to 0·1 vol% of nanoparticles. Use of suitable stirring routine ensured uniformity and stability of dispersion. Thermal conductivity ratio of nanofluid measured using transient hot wire device shows a significant increase of up to 12% with only 0·1 vol% nanoparticles and inverse dependence of conductivity on particle size. Use of ceramic nanoparticles appears more appropriate to ensure stability of dispersion in nanofluid in closed loop single-phase heat transfer applications.**

Keywords. **Nanoparticle; SiC; nanofluid; thermal conductivity.**

1. Introduction

While the exploits of nanoscience and nanotechnology mostly concern solid state, components or devices, attempts to use ultrafine particles to enhance thermal, rheological or magnetic property of fluid have recently been initiated with immediate positive outcome. For instance, nanofluid, a tailored quasi-single phase medium comprising stable colloidal dispersion of ultra fine or nanometric (< 100 nm) particles in very low concentration (< 1 vol %) have shown promises to enhance thermal conductivity and eventually heat transfer coefficient of the fluid to an extent that defies the rule of average or conventional physics of heat conduction (Lee *et al* 1999).

Efficient transfer of sensible heat, from a hot body or location to another is often required in thermal power plants, refrigeration, heat exchanger systems, electronic devices, automobiles and chemical industries. Usually, a fluid is chosen as an agent for transferring heat mostly through convection in all these applications using the Newtons's law:

$$q = hA\Delta T,$$

where q is the rate of heat transfer, h the coefficient of convective heat transfer, A the surface area (perpendicular to heat flow) and ΔT the temperature gradient across the distance of heat flow. For a given ΔT and A, q is directly related to h and is a complex function of velocity and thermophysical properties of the fluid including thermal conductivity (Xuan and Li 2000). Since thermal conductivity of solids is invariably orders of magnitude greater than that of liquids, suspension of solid particles is bound to enhance thermal conductivity of the fluid. However, coarse particles tend to sediment or clog and erode the pipes or ducts. In contrast, appropriate combination of nanometric solid particles and a fluid can form a stable colloidal suspension (by using a suitable surfactant) and offer significant enhancement in thermal conductivity (Chen 1996; Eastman *et al* 2001; Das *et al* 2003; Patel *et al* 2003; Wang *et al* 2003).

While nanoparticles can be produced by several routes like inert gas condensation, mechanical attrition/alloying (Suryanarayana 2001) and chemical precipitation techniques (Fecht 2002), nanofluids can be prepared either by a single-step process of directly collecting metallic/ceramic nanoparticles synthesized by chemical precipitation in a heat transfer fluid (Wang *et al* 1999; Chopkar *et al* 2006; Paul *et al* 2010a, b), or through a two-step process of first preparing nanoparticles separately and then dispersing them in an appropriate fluid in a given quantity (Xie *et al* 2004; Paul *et al* 2011). Ultrasonic vibration and/or use of surfactant may be effective to ensure homogeneous dispersion of particles in the base liquid (Xuan and Li 2000; Das *et al* 2003). Thermal conductivity of nanofluids can be measured by a number of methods (Paul *et al* 2010a, b) viz. the transient hot-wire (Lee *et al* 1999), temperature oscillation (Wang *et al* 2003), steady-state parallel plate method (Challoner and Powell 1956) and improvised thermal comparator method (Manna *et al* 2005).

Studies involving the use of several base fluid mediums, viz. water, ethylene glycol, pumps oil, ethanol, refrigerant and toluene for the preparation of nanofluids have been reported in literature. But among these fluids, water and ethylene glycol are the most extensively used base fluid mediums. The influence of base fluid medium on the thermal conductivity ratio of nanofluids can be predicted from the

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enhancements reported by Kim *et al* (2007), Lee *et al* (1999) and He *et al* (2007). It may be pointed out that, in general, the ethylene glycol based nanofluids show higher thermal conductivity ratio compared to water based nanofluids, all other parameters being kept constant. Though water has the highest thermal conductivity among fluids, but nanofluids with other base fluid mediums show higher thermal conductivity ratio. This result is encouraging because heat transfer enhancement is often most needed when poorer heat transfer fluids are involved. Ethylene glycol alone is a relatively poor heat transfer fluid compared to water, and mixtures of ethylene glycol and water fall between the two in effectiveness of heat transfer. Though this is the general trend, it is not the overall trend since some articles report thermal conductivity ratio of water based nanofluids as higher compared to ethylene glycol based nanofluids (Chopkar *et al* 2006, 2007).

In the present study, a systematic effort has been made to characterize as received and mechanically milled SiC nanoparticles, disperse the latter in water in very low volume fraction following a special routine to prepare nanofluid and carry out thermophysical studies including thermal conductivity measurement of this nanofluid. The thermal conductivity of the nanofluid has been studied as a function of particle concentration and particle size in the nanofluids.

2. Experimental

2.1 *Preparation of nanofluid and particle characterization*

Nanofluid is not simply a solid–liquid mixture. It must ensure a uniform and stable suspension (like a colloid), and preclude agglomeration or chemical change of the particles or fluid. Nanofluids with dispersion of nanometric copper, aluminum and their oxides have been widely investigated by many researchers (Chen 1996; Lee *et al* 1999; Xuan and Li 2000; Eastman *et al* 2001; Das *et al* 2003; Patel *et al* 2003; Wang *et al* 2003; Chopkar *et al* 2006; Paul *et al* 2010a, b). However, silicon carbide, which has an excellent chemical and physical stability, low density and fairly high thermal conductivity (among ceramic compounds) has not yet been exploited although it is cheap and commercially available in ultrafine particle/crystallite size. Silicon carbide powder having particle size in the micrometer range was used as the dispersiod for the present study. The as-received powder had particle size of the order of micrometer. In order to reduce the size, the as-received sample was subjected to mechanical milling/attrition for 30 h at room temperature in WC media (vials and balls) at 300 rpm and 10:1 ball to powder weight ratio. Milling was carried out in wet medium using toluene to prevent undue oxidation, agglomeration/welding of powders and coating of the balls and vials with the powder. Nanofluids were prepared by dispersing 0·01 to 0·1 vol. % of nanocrystalline SiC powder in deionized water. Ultrasonic vibration and magnetic stirring were used for nearly 4 h to ensure proper and stable dispersion of different volume fractions of SiC nanoparticles in the base fluids.

Prior to dispersing the nanoparticles in the base fluid, the powder was characterized by X-ray diffraction (XRD) using a BRUKER AXS instrument at a scan speed of 0·05°/s with Co-K_α radiation (0·179 nm) for phase identification and crystallite size determination. A field emission gun assisted scanning electron microscope (SEM) (SUPRA 40, CARL ZEISS) was used in the secondary and back scattered modes to investigate shape and size of the nanoparticles. A JEOL JEM-2100 transmission electron microscope (TEM) was used to study size, shape and phase purity of isolated nano-SiC particles at higher resolution albeit with poorer statistics than that possible in SEM. The purity of the particles was examined using an OXFORD INCA energy dispersive spectroscopy (EDS) instrument attached to both SEM and TEM.

2.2 *Measurement of thermal conductivity of nanofluids*

Transient hot-wire technique was adopted to measure the thermal conductivity of nano-SiC dispersed water based nanofluids using a KD2-Pro thermal analyzer (Decagon Services, USA) which was based on the principle of calculating the transient temperature field around a hot-wire which can be treated as a line source. In this widely used technique, the wire served as both the heat source and thermometer. The hot-wire was completely immersed in a vessel containing nanofluid and a constant heat was supplied to measure the necessary temperature rise. For a given applied heat input (q), the thermal conductivity, k, was calculated from the Fourier's law as:

$$k = \left[\frac{q}{4\pi\,(T_2 - T_1)}\right] \ln\left(\frac{t_2}{t_1}\right), \tag{1}$$

where T_1 and T_2 are the temperatures at times, t_1 and t_2, respectively. Experiments to study the effect of particle concentration and size were conducted at room temperatures. The time required for each measurement was 1·5 min and the recorded data (repeated at least 5 times with adequate interval) showed a maximum uncertainty of 5%. About 30 ml of the prepared nanofluid was taken in a test tube and placed within an isothermal bath, which was suitably insulated to prevent errors due to heat dissipation. Figure 1 shows schematic details of the experimental set up used for measuring thermal conductivity of nanofluids. The KD2 Pro thermal analyser consisted of a probe which was inserted into the nanofluid for k measurement and a unit for reading out the k value. A single reading generally took 1·5 min. The first 30 s were used to ensure temperature stability, after which the probe was heated for 30 s using a known amount of current. The remaining 30 s were used to cool the probe back to the ambient temperature. The probe also contained a thermistor which measured the changing temperature while the microprocessor stored the data. At the end of reading, thermal conductivity of the fluid was computed using the

temperature difference vs time data. The nanofluid was taken in an appropriate test tube for k measurement at room temperature. The probe of the KD2 Pro device was placed inside the test tube and k measurement was done.

3. Results and discussion

3.1 *Characterization of nanoparticles*

The microstructure of the as received SiC powder samples has been studied to analyze the initial size, shape and morphology. Figure 2 a shows SEM image of the as received SiC sample dispersed in fluid medium. Lumps of particles forming large agglomerates are visible from the secondary electron image observed under SEM. It may be pointed out that size of the particles varied in the range of several nanometers to micrometers and the particles exhibit irregular shape and morphology as observed from figure 2a. In order to reduce size of particles and make them homogenous and ultrafine in nature, the SiC powder samples have been subjected to mechanical milling for a total duration of 30 h and samples have been collected at regular intervals during mechanical milling to study the variation in size and the effect of size on thermal property of nanofluids.

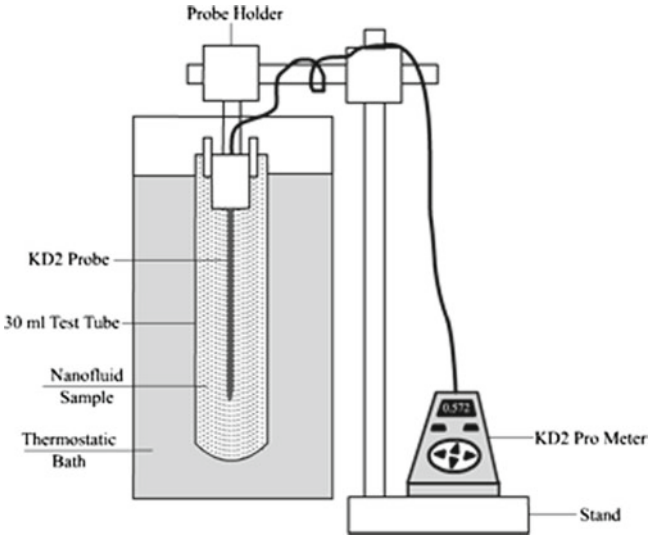

Figure 1. Experimental set up for thermal conductivity measurement.

Figure 2b shows XRD profiles at 1, 10 and 30 h of mechanical milling. As observed from the XRD profile of 1 h mechanically milled powder sample, peaks of SiC and SiO_2 are visible. The customary Bragg reflections match with the most intense peaks of hexagonal structured silicon carbide sample (JCPDS file no: 74-1302) and with monoclinic silica (JCPDS file no: 82-1575). XRD profiles of the subsequent hours of mechanical milling shows no shift in peak position and sufficient peak broadening is observed which indicates that the sample turns nanocrystalline. The crystallite/grain size has been calculated from the full width at half maximum of the most intense peaks observed from XRD profiles using the Scherrer formula after eliminating the instrumental and strain broadening errors. It may be observed that the crystallite/grain size decreases from 165 nm at 1 h with the progress in mechanical milling reaching a minimum of 28 nm after 30 h of milling. It may be mentioned that lattice strain induced during mechanical milling calculated from the Williamson–Hall plot, increases nonlinearly with increase in milling duration. Hence elimination of strain effect from peak broadening is essential to calculate the effective crystallite size.

The change in size, shape and morphology of the samples during the course of mechanical milling has been monitored and analyzed under SEM. Figure 3a shows mechanically milled SiC particles as near-spherical ultrafine powders without significant agglomeration. Compared to the initial or as-received large sized agglomerates in lumpy form (figure 2a), the level of particle size reduction in the 30 h milled sample (figure 3a) is quite significant. Since cold welding and fragmentation are concurrent during mechanical milling/attrition, the degree of particle size reduction evidenced in figure 3a obviously suggests that comminution in wet condition has been effective to ensure greater degree of fragmentation/fracturing of coarse SiC particles into fine powder than cold-welding them during continued

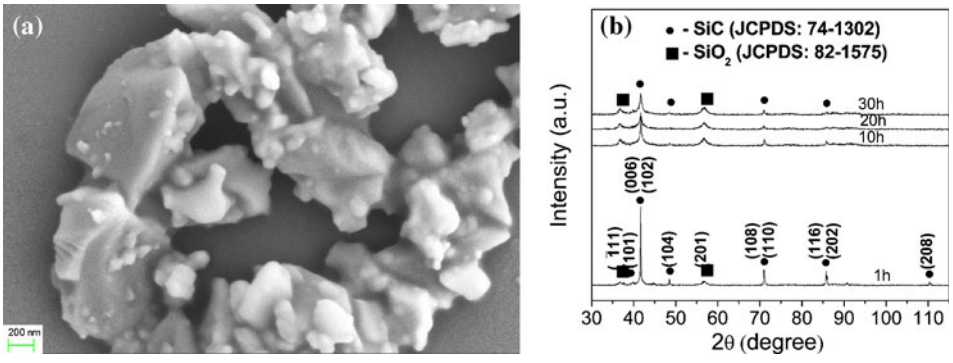

Figure 2. **(a)** SEM image of as received SiC powder sample dispersed in fluid medium and **(b)** XRD profiles of mechanically alloyed SiC powder samples at cumulative hours of milling.

milling. The size distribution histogram shown as the inset of figure 3a constructed from the image analysis of a series of SEM images of 30 h mechanically milled samples shows that the particle size varies in the range 37–110 nm, with a mean value of 60 nm. The standard deviation of data has been calculated as 6·774. Thus it may be summarized that the size of the particles gradually reduces from agglomerates having size in the range of several micrometers to isolated particles with an average of 60 nm during the course of 30 h of mechanical alloying.

The 30 h mechanically milled SiC sample has been observed under TEM after dispersing in fluid medium to analyse morphology and size of the particles. Figure 3b shows bright field TEM image of SiC sample which exhibits some amount of agglomeration but isolated particles are also visible towards edges of the clusters. The inset of figure 3b shows size distribution histogram of 30 h mechanically milled SiC sample which reveals that the particle size varies in the range of 47–92 nm with a mean size of 55 nm. Comparison between statistical results observed for SEM and TEM shows that the size distribution range recorded for TEM is much tighter compared to that of SEM though the mean size observed are comparable. The size distribution determined from SEM and TEM suggests that the particles are polycrystalline as the crystallite size calculated from XRD is much smaller compared to the particle size.

3.2 *Thermal conductivity of water based nanofluids*

3.2a *Effect of concentration*: The as-received SiC particles have been dispersed in water to prepare nanofluids having 0·1–0·8 vol% concentration. Figure 4a shows variation of thermal conductivity ratio (with respect to water) as a function of particle concentration. The thermal conductivity ratio has been observed to increase linearly with increase in concentration and the maximum level of enhancement observed is 26% at 0·8 vol% concentration. Despite significant enhancement in conductivity, this fluid with micrometer sized particle dispersion exhibited very poor results in terms of stability. Since size of the as received particles is in the micrometer range, the fluids prepared by dispersing

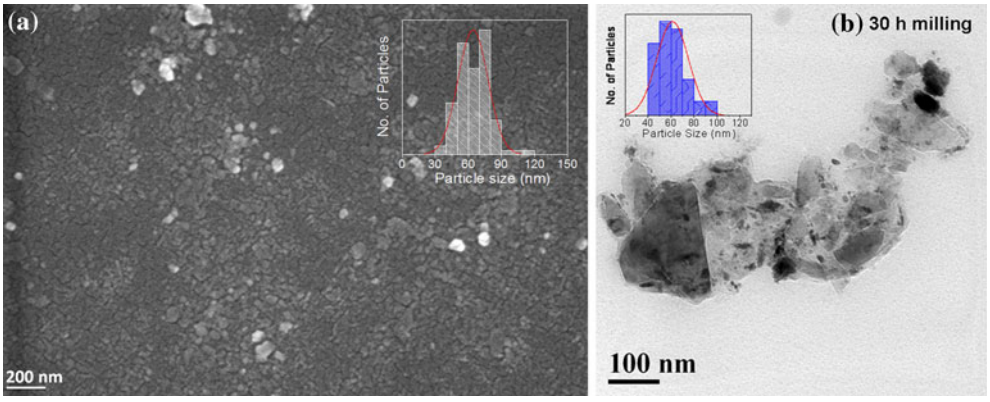

Figure 3. **(a)** SEM image of SiC samples at 30 h of mechanical alloying (with size distribution shown as the inset) and **(b)** TEM of 30 h mechanically alloyed sample with size distribution shown as the inset.

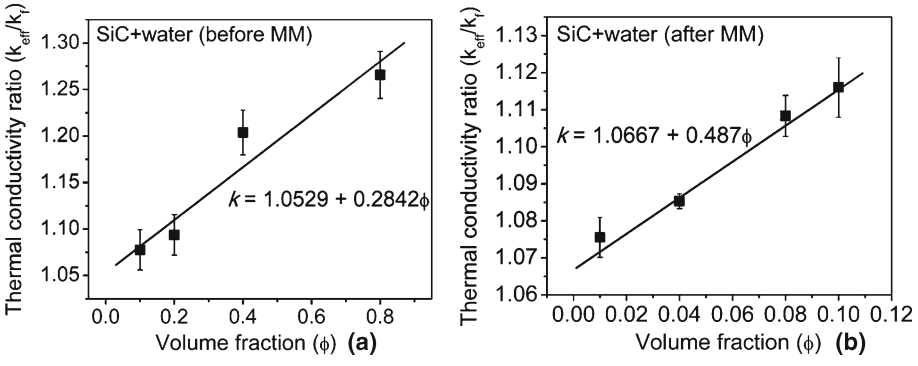

Figure 4. **(a)** Effect of concentration of particles on thermal conductivity of water based nanofluids dispersed with as received SiC particles and **(b)** variation of thermal conductivity ratio of mechanically alloyed SiC dispersed water based nanofluids with respect to concentration of dispersed nanoparticles.

these particles do not satisfy the size dependence criterion for nanofluids. In order to address this stability issue, the particles have been mechanically milled to reduce the size to nanometer range and nanofluids have been prepared in water in the concentration range of 0·01–0·10 vol%. Figure 4b shows effect of concentration of dispersed nanoparticles on the thermal conductivity ratio of mechanically milled SiC dispersed nanofluids. The thermal conductivity ratio has been observed to increase linearly with increase in concentration of dispersoid reaching a maximum of ∼12% at 0·1 vol%. Regression analysis of the recorded experimental data shows a linear fit with a R^2 value of 0·98507 expressed as

$$k_{\text{eff}}/k_{\text{f}} = 1{\cdot}0667 + 0{\cdot}487\phi. \quad (2)$$

It may be noted that the nanofluids prepared with 0·1 vol% mechanically alloyed SiC nanoparticles show a much higher thermal conductivity enhancement as compared to that observed for fluids prepared with as received SiC particles at 0·1 vol% (∼7%) (shown in figure 4). Thus it may be pointed out that mechanical milling improves the level of enhancement observed in thermal conductivity at comparable level to particle concentration. It may be pointed out that the thermal conductivity enhancement reported in the present study is higher than that reported by Xie *et al* (2002).

3.2b *Effect of particle size*: Figure 5 shows variation of thermal conductivity ratio of SiC dispersed water based nanofluids with respect to the particle size calculated from XRD profiles of powder samples collected at different hours of mechanical milling. It has been observed experimentally that the thermal conductivity ratio has increased as the particle size becomes smaller reaching a maximum of ∼12% at 27 nm size. All the experiments have been conducted at the same concentration (0·1 vol%) of dispersoid to neglect the effect of varying concentration. The crystalline/grain size calculated from peak broadening analysis of relevant XRD data appears smaller than the average particle size measured from SEM and TEM images suggesting that the SiC particles used in the present study are poly-nanocrystalline. Yet the particle size is considered more relevant for analysing the size dependence issue (figure 5) because surface atoms are primarily responsible for phonon or heat transport from particle to particle within the fluid than the atoms at the core of the nanoparticles. The thermal conductivity enhancement ratio of nano-SiC dispersed water based nanofluids have been compared with the reported values of other ceramic dispersed nanofluids in water and ethylene glycol (EG), as shown in figure 5. It is worth mentioning that the enhancement reported in the present study of 0·1 vol% nanofluids is comparable to that reported by Kim *et al* (2007) at 3 vol% of nano-TiO_2 dispersed in EG. The enhancements at other concentrations reported by Kim *et al* (2007) is considerably lower than that of the present study, although the concentrations of nanoparticle loading are considerably higher than that reported in the present study. Similarly for water based nano-TiO_2 dispersed nanofluids, the enhancement reported by He *et al* (2007) are lower than that reported in the present study. From comparison, it may be pointed out that nano-SiC particles prepared by mechanical alloying show considerably higher enhancement than that of nano-TiO_2 dispersed nanofluids.

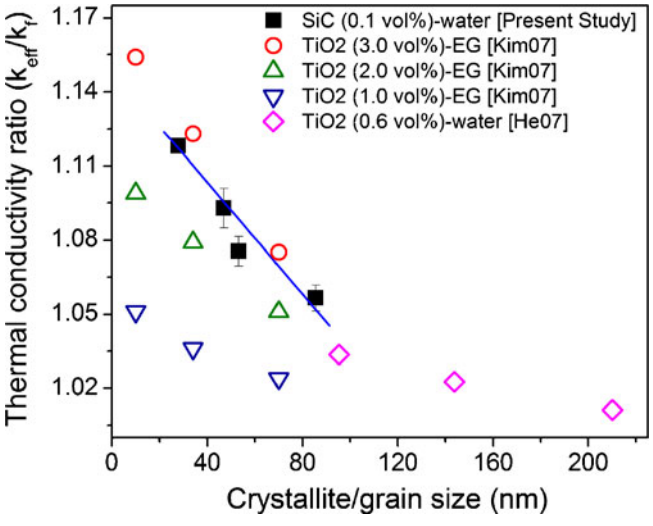

Figure 5. Effect of crystallite/grain size on thermal conductivity ratio of SiC dispersed water based nanofluids.

4. Conclusions

The present study shows that the thermal conductivity of water can be enhanced by 12% by dispersing only 0·1 vol% nanometric (∼27 nm) SiC particles. Even greater enhancement (∼26%) is possible with higher (0·8 vol%) particle concentration and bigger particle size albeit poor stability of dispersion. This improvement in thermal conductivity records an inverse dependence on particle size. In general, it appears that nano-SiC dispersed water based nanofluid may offer attractive scopes of application for heat transfer in single phase regime.

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