

## Dependence of thermal conductivity in micro to nano silica

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**Abstract.** This work presents the measurement of thermal conductivity of nano-silica particles using needle probe method. The validation test of thermal probe was conducted on ice and THF hydrates using our experimental set up and the results are satisfactory when compared with the literature data. The nano silica used in this study is with particle sizes in the range 50–1000 nm. The sand powders sieved in different sizes  $<75$  and  $75 \mu\text{m} > d > 250 \mu\text{m}$  were also studied to probe the particle size dependence on thermal conductivity. Thermal conductivity decreased by about 70% in silica nano powders.

**Keywords.** Thermal conductivity; micro and nano particle size; needle probe method.

### 1. Introduction

The thermal conductivity in sand depends upon various factors, i.e. on particle size distribution, structure, porosity, saturation degree and temperature. Amongst all these factors, porosity and saturation degree have remarkable effects on thermal conductivity (Chen 2008). Our study focuses mainly on the effects of particle size on thermal conductivity of silica. Several methods have been used to measure thermal conductivity of soils and details of these methods have been presented (Donazzi 1977). The methods can be divided into two major categories: steady heat-flow method and transient heat-flow method. The steady heat-flow method generally requests a long testing time, consequently, steady heat-flow method is not suitable in some cases. On the contrary, the transient heat-flow method is rapid and only a small temperature increment occurs in the process of test. The transient thermal probe method is a transient heat-flow method. It determines the thermal conductivity of medium through measuring the rate of temperature rise caused by an infinite line heat source within an infinite homogeneous medium. Thermal probe method of determining conductivity is the best method available because of its easy design and usage. The thermal probe can be used to test specimens in the laboratory or soils *in situ*.

### 2. Thermal probe construction and method

The thermal probe used in this work was constructed from seamless thin-walled stainless steel tubes, 100 mm long and 3 mm outer diameter. The main probe comprises of a nicrome

heater wire and a thermocouple as shown in figure 1. The probe assembly design is in accordance with ASTM D 5334 (Cars law and Jaeger 1964). Insulated nicrome heater wire, 0.5 mm in diameter, was looped and inserted into the full length of the tube to form the heating element; Pt100 thermocouple was inserted midway along the length of a tube. Both the ends of heating element and thermocouple wires protruded from the same end of the tube. The tube was filled with thermal binding system (TBS20S, Electrolube, UK) which is thermally active but electrically insulated and a metal tip was placed on an open end of the tube. The epoxy was allowed 24 h for setting and the extended heater wire ends were cut close to the thermal probe and power supply leads were soldered to them. The probe normally consisted of an electrical heater, a temperature sensor and a thermocouple. The probe was inserted into an isothermal material and powered. The time–temperature data from the probe were analysed to obtain the mean apparent thermal conductivity,  $\lambda$  for the material over the temperature range introduced in the experiment. Apparent thermal conductivities are obtained from the data using (3):

$$\lambda = (Q/4\pi)/(dT/d \ln t), \quad (1)$$

where  $Q$  is heat generated,  $dT$  the change in temperature and  $d \ln t$  the natural log of time. Equation (1) indicates an attractive method for thermal conductivity measurements with the advantages of low cost and short test time.

The general solution for the temperature variation with time of a cylinder of a perfect conductor with small radius transferring heat at a constant rate to the surrounding isothermal infinite medium with constant properties indicates that in all the cases, a plot of temperature against  $\ln t$  has a linear asymptote of slope  $(Q/4\pi A)$  (Cars law and Jaeger 1964).

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Therefore, if the value of  $Q$  is known, apparent thermal conductivity  $\lambda$  is determined. A constant current,  $I$ , is supplied to a heater wire with resistance  $R$ , per meter. The heater wire's output per meter,  $Q$ , is determined from  $Q = 2I^2R$ , where factor 2 accounts for the heater wire being a loop running the length of the needle probe. From Blackwell (1954), the heater wire's output causes the temperature change measured within the probe,  $\Delta T$ , to vary as:

$$\Delta T = A \ln(t) + B, \tag{2}$$

where  $t$  is the time in seconds since the probe began heating the sample,  $\lambda$  the thermal conductivity and  $A$  the slope.

$$\lambda = 1/A \cdot Q/4\pi. \tag{3}$$

Equation (3) indicates an attractive method for thermal conductivity measurement with the advantages of low cost and short test time. Hence, this method would be ideal for conducting many tests per day (Manohar et al 2000). In practical situations, the finite radius of the probe has the effect of time delay before the theoretical rate of radial heat flow through the surface of the probe is equal to the heat dissipated by the

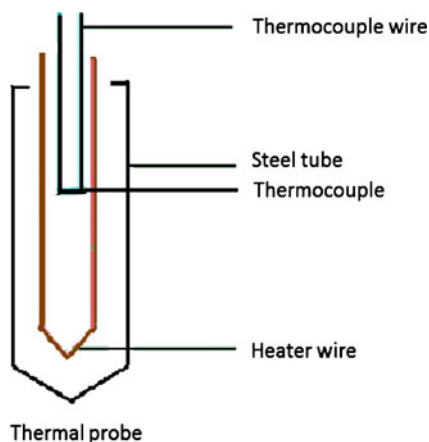


Figure 1. Needle probe arrangement.

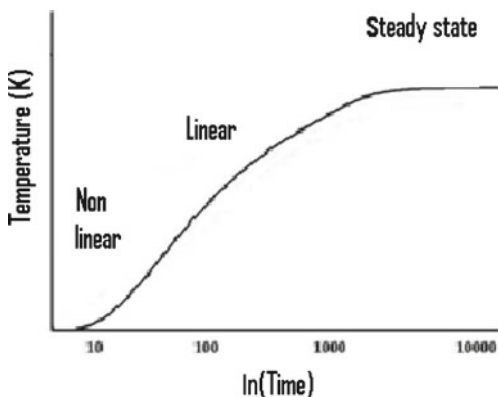


Figure 2. Graph between time and temperature.

heater filament (Winterport 1970). Therefore, for practical situations, the general shape of  $T-\ln t$  plot includes an initial nonlinear region followed by a linear region. With the passage of time, the probe temperature will level off to a steady state value because of an isothermal specimen boundary. The general shape of a typical  $T-\ln t$  plot is shown in figure 2 (Annual Book of ASTM Standards 1995).

### 3. Experimental

We assembled an experimental set up based on line heat source and an automated data logging system to measure thermal conductivity. Thermal probe method of determining conductivity is the best method available because of its easy design and usage. The thermal probe was powered by a regulated d.c. power supply. The electrical circuit used is shown schematically in figure 3. The system employed Aplab 7642 precision power supply voltage from 0 to 20 V. Temperature-time variation of the thermocouple in the thermal probe was monitored and recorded by the computer through I/O board. The computer was programmed to sense one temperature signal per 3 s, computes the average and display it for each 3 s and stores display readings of time and temperature for a total of 200–400 entries. Reliability tests were conducted on ice and THF hydrates to validate the experimental set up and the results are corroborated with the literature. The silica nano powder used was obtained from Intelligent Materials Pvt. Ltd. (NANOSHEL) and used as such. SEM pictures of the silica samples are shown in figure 4. In our experiments, we used 50 g of silica in each particle size. Silica filled into the cell with as minimum as possible air gaps. The probe was inserted exactly into the middle of the cell (sample) and the probe is energized with d.c. voltage. The analysis characterized by (1) required that the specimen be isothermal, therefore, during the experimental study, the total experimental set up was covered with thermally insulated box, thus limiting *in situ* usage.

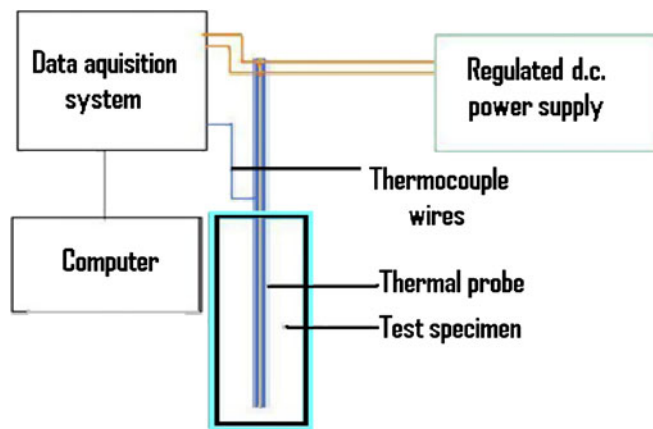
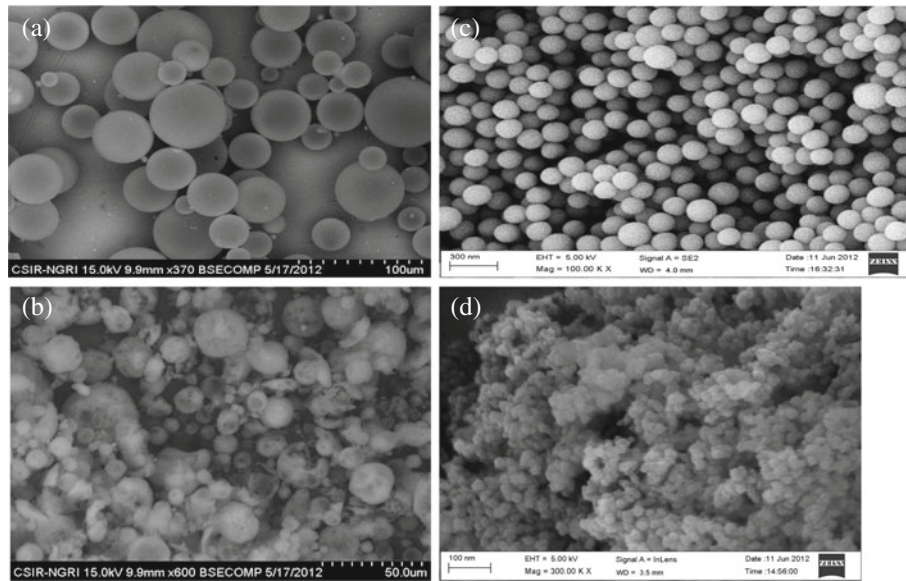
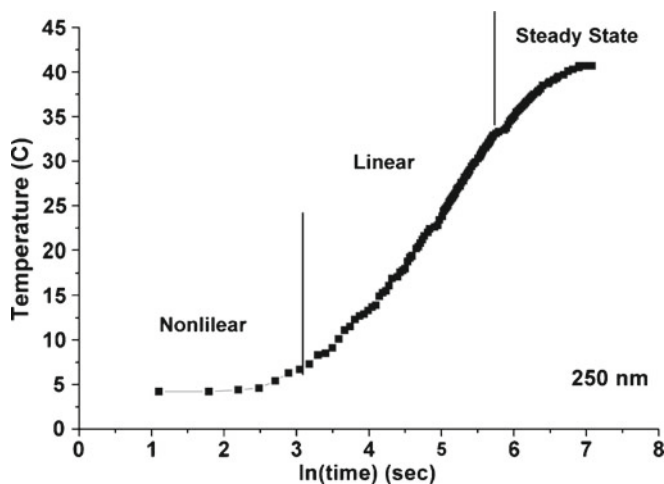


Figure 3. Schematic diagram of electrical circuit and data acquisition system.

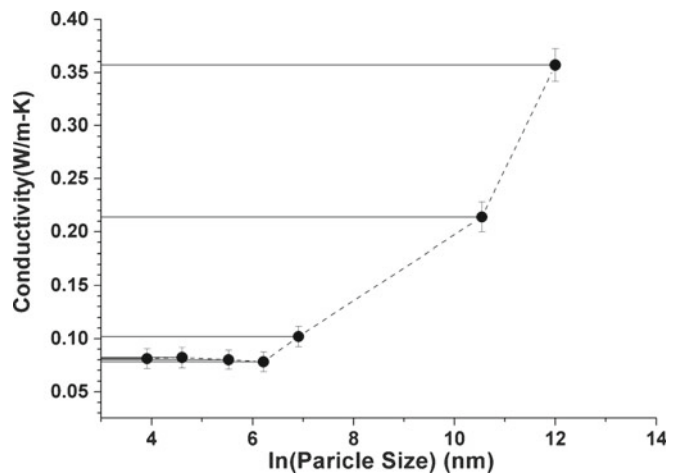


**Figure 4.** (a)  $<75 \mu\text{m}$ , (b) 1000 nm, (c) 250 nm and (d) 50 nm are SEM images of samples used.



**Figure 5.** Typical thermal conductivity curve of 250 nm silica.

The experimental procedure involved, inserting the probe in the material to be tested after both were in thermal equilibrium with the surroundings. Since the friction associated with inserting the probe may cause a measurable temperature increase, the probe temperature was monitored to make certain that isothermal conditions were attained before the heater was powered. This thermal equilibration usually takes 5–10 min. The data acquisition system and heater power were turned on together once the thermal equilibrium had been achieved. The data acquisition system was used to record probe current, temperature and time for ~600–1200 s. Since nichrome wire has a negligible change in electrical resistance with temperature (Weast 1982), the power to the probe heater remains constant. A linear fit to a linear expression for  $T$  in terms of ‘ $\ln t$ ’ was used to describe linear



**Figure 6.** Graph between log of particle size and thermal conductivity.

segment of the heat-up curve which is used while calculating the slope of the curve.

#### 4. Results and discussion

Thermal conductivity and viscosity affect heat transfer behaviour of nanofluids in opposite ways. As a result, combination of thermal conductivity enhancement and increment of the viscosity can give either enhancement or deterioration of the heat transfer coefficient (Ding *et al* 2007). The thermal conductivity of the nano-scale silicon particles increases with their diameter and is two orders of magnitude lower than that of bulk silicon, this may be due to particle phase boundary (Fang *et al* 2006). From the studies of

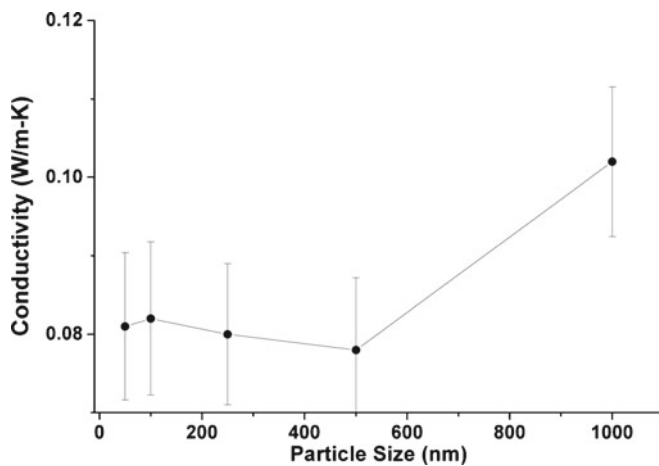


Figure 7. Thermal conductivity at nano level.

Table 1. The dependency of thermal conductivity with different silica particle sizes.

| Particle size (nm) | Conductivity (W/m-K) | std error ( $\pm$ ) |
|--------------------|----------------------|---------------------|
| 162500             | 0.357                | 0.0592              |
| 38000              | 0.214                | 0.0301              |
| 1000               | 0.102                | 0.00855             |
| 500                | 0.078                | 0.00821             |
| 250                | 0.080                | 0.0081              |
| 100                | 0.082                | 0.00877             |
| 50                 | 0.081                | 0.00823             |

Al Nakshabandi and Kohnke (1965), it was observed that the variation in the thermal conductivity was negligible when the particle size exceeded  $1000 \mu\text{m}$ , whereas in case of nano size a significant variation in thermal conductivity was observed. We measured thermal conductivity in dry samples of nano silica to observe how the conductivity varies from micro to nano. It was observed that there is a decrease in thermal conductivity with decrease in particle size of nano-sized particles as compared to micro sized particles and this increase in thermal conductivity of micro sized particles can be attributed to their high surface to volume ratio. Moreover, the decrease in thermal conductivity with decrease in particle size is probably due to the created void spaces in the sample which is completely dependent on the particle size.

The smaller grains will pack closer, forming a more homogenous specimen, so we expect higher conductivity but because of air trapped in void spaces of the system which

is a bad conductor, therefore, the conductivity is reduced. The typical conductivity curve of silica, 250 nm, is shown in figure 5, which is divided into three parts. Nonlinear (time is too short to satisfy (1)), linear data used for linear fit which is useful for calculation of thermal conductivity and steady state (dominated by edge and end effects). Test results on silica and sand size in the range 50–1000 nm and different sizes  $<75 \mu\text{m}$  and  $75 \mu\text{m} > d > 250 \mu\text{m}$  showed a decrease in thermal conductivity. In the graph between log of silica size vs thermal conductivity (figure 6), we observed that when silica size is decreased, thermal conductivity also decreased by about 70% from micro to nano and observed to be saturated at around 500 nm nano level with small variations up to 50 nm (shown in figure 7). Table 1 shows values of particle size and the corresponding thermal conductivity with associated standard error.

## 5. Conclusions

We studied thermal conductivity measurements using thermal probe method. Test results on silica sand size in the range 50–1000 nm and different sizes  $<75$  and  $75 \mu\text{m} > d > 250 \mu\text{m}$  showed a decrease in thermal conductivity. We observed that the thermal conductivity decreases by about 70% upon reducing silica particles size from micro to nano.

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