

Thermal Conductivity Enhancement by Al₂O₃@Cu, core@shell Nanoparticle Suspensions in Nanofluid Coolant

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ABSTRACT

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The objective of this research work is to synthesize Alumina@Copper, core@shell nanoparticles suspended nanofluid coolant, possessing high thermal conductivity. The Alumina@Copper nanoparticles were synthesized by two step chemical process. The methodology used to synthesize core@shell nanoparticles includes the synthesis of base alumina nanoparticles by sol-gel method in first step, which was coated with a thin shell of copper using electroless plating technique. The study investigates thermal conductivity enhancement of prepared nanofluid coolant, upon suspension of these nanoparticles for the selected volume of Strub Vulcan Futura coolant oil. Nanoparticles addition to the coolant oil was 0.025 % to 0.3 % by weight. Results indicated that, alumina@copper nanoparticles addition enhanced the thermal conductivity value up to 23.39 %. Results were validated using Hamilton and Crosser (HC) theoretical model assuming nanoparticles to be spherical. An empirical correction factor was applied for HC model which fits the thermal conductivity values to justify the deviation. The findings of this research may serve for faster heat dissipation during any metal cutting operation, since the synthesized nanofluid coolant possesses enhanced thermal conductivity.

1. INTRODUCTION

In order to develop an industry by applying new methods for improving the production processes, calls for exhaustive studies on diverse field. Such fields include availability of machines, human resources, quality of manufactured components, expenditure incurred etc. Machining is one of the largest and universally used techniques in manufacturing sector. A major issue related to machining is the heat generated on the machine, at machine tool interface due to friction during metal cutting operation. Significant developments over the past two decades have motivated researchers all over the world, to perform several studies to identify new methods for enhancing the heat dissipation rate at the work-tool interface. Excess heat during metal cutting operation results in poor surface finish and hence lower quality products. The dissipation of heat at work tool interface for aggressive machining still remains a challenge [1-4].

As per the existing studies to solve the heat dissipation problem, traditional methods of flooded lubrication technique is being practiced by industries, over the last few decades. This technique involves supplying large quantity of coolant [50-60 liters/hour] at the work tool interface to dissipate the heat generated. However since the last decade, due to stricter environmental norms, this method is less recommended and a new technique called Minimum Quantity Lubrication (MQL) [1] is proposed. MQL technique basically refers to supplying a metered quantity of coolant at the work-tool interface during metal machining that carries away the heat generated. Since the quantity of coolant is drastically reduced from 50 to 60

liters per hour to 50-500 mL per hour, the coolant is expected to possess enhanced heat carrying capability.

This enhancement of coolant ability to carry away more heat at machining zone, is obtained using the concept of nanotechnology involving suspension of solid nanometer sized metallic particles into a liquid coolant. With the latest advancement in nanotechnology, application of nanofluid coolant assisted MQL, has further enhanced the heat carrying capacity at machining zone [2].

A conventional fluid containing nanometer sized particles suspended in it, is defined as 'nanofluid' [3]. These fluids used for machining applications are referred to as nanofluid coolants.

In the present decade, scope for nanofluid assisted MQL technique for machining is gaining more importance [4]. Various researchers have experimented nanoparticle suspended coolants for enhancing heat carrying capacity at the machine tool interface. Yoo et al., [5] with his experiments showed that by maintaining pH value of alumina nanofluid heat conduction capacity is improved at the machining zone. Al₂O₃ nanofluids have been applied for high temperature field applications as per studies of Jwo et al., [6]. Kandekar et al., [7] performed machining experiments, by adding 1 % by volume alumina nanoparticles to coolant and observed enhanced wettability during turning of AISI 4340 as compared to, by using conventional fluid supply method. Mao et al., [8] performed investigations on adding alumina nanoparticles to deionized water and canola oil used as coolants for machining AISI 52100 steel and found reduction in forces and temperature. Several parameters were investigated by Mao et

al., on AISI 52100 steel using water as cutting fluid/coolant embedded with alumina nanoparticles, which benefitted in terms of optimizing nozzle angle to gain minimized grinding force and roughness [9]. Other researchers like Khalil et al., [10] and Setti et al., [11] conducted experimentation using alumina nanoparticles to reduce tool wear during turning and reduction of coefficient of friction for grinding of Ti-6Al-4V respectively. Usage of copper nanoparticles to prepare nanofluid has also proven to be beneficial, to enhance thermal conductivity of the fluid as per Chol. S et al., [12]. Copper nanoparticles use has been investigated by Liu et al., [13] for mending effect. The enhancement of thermal conductivity upto 23.8 % with 0.1 % addition to water and upto 20 % of 35 nm copper oxide nanoparticles was noted by Liu M. S. et al., [14]. Newtonian behavior was exhibited, with 12.7 % enhancement in heat transfer coefficient for 2 % by weight nanofluid addition was observed by Saeedinia et al., [15].

From the above literature, it is observed that researchers have experimented with single nanoparticle suspension to enhance the thermal conductivity of the coolant. No innovation or studies were found, that discussed the effect combination of more than one type of nanoparticles, that have been used for machining applications.

As the research progressed, a new type of nanoparticles namely, core@shell nanometer sized particles were used for purposes like chemical and colloidal stability, preparation of bio-conjugates, optimization of magnetic properties etc. [16]. Tom et al., in the year 2003 came up with a one-step method synthesis of Au@ZrO₂, Au@TiO₂ particles of nanometer size that were coated with a shell of controllable thickness. A typical size of core ranges from 30-60 nm with a shell of thickness 3 nm [17]. Ahn et al. was the first to synthesize by low temperature plasma enhanced chemical vapor deposition method, copper nanowire graphene (CuNW-G) core@shell nanostructure at around 400 °C [18]. The concept of preparation of core@shell nanoparticles gained more importance due to its "physical synergistic approach". However all the above core@shell nanoparticles take significant time and involve sufficient cost for their synthesis.

To solve this problem the present paper, focuses on synthesis of low cost Alumina (Al₂O₃) core, coated with Copper (Cu) shell nanoparticles that are suspended in the selected coolant, to prepare a new nanofluid coolant.

The remainder of the paper is organized as follows: Section 2 explains the method for synthesis of alumina@copper nanoparticles and describes experimental measurements of nanofluid coolant thermal conductivity, Section 3 deals with the characterization using XRD, SEM, TEM and EDX analysis and covers the experimental results with modelling, Section 4 deals with applicability of this nanofluid coolant for metal cutting applications, finally Section 5 describes the conclusion and paves a way for future researchers.

2. METHODS (SYNTHESIS AND CHARACTERIZATION OF AL₂O₃@CU CORE-SHELL NANOPARTICLE)

Initially 16.66 gm of Aluminum Sulfate Pentahydrate Al₂(SO₄)₃. 5H₂O is dissolved in a 100 ml beaker containing distilled water. This solution is then transferred to the 250 ml volumetric flask. To this mixture, distilled water is added up to 250 ml mark on flask. The mixture in the volumetric flask is stirred properly to get a homogenous mixture. The

concentrated ammonia is taken in the beaker and diluted to 25 %. The 25 % ammonia is transferred to the burette and fixed to the stand. The prepared 250 ml Al₂(SO₄)₃. 5H₂O solution is transferred to the 500 ml beaker. Around 2-3 gm of Polyethylene glycol is added to the 250 ml solution in the beaker and stirred properly for 5 minutes. Polyethylene glycol (PEG) is added to solution because it avoids agglomeration by acting as a surfactant. The beaker containing mixture is placed on magnetic stirrer with rpm set to around 700 rpm. The titration is carried out until white color milky precipitate (maintained at pH=9) is formed. The reaction mixture is placed on the fume hood for about 3 to 4 hours until sol-gel is obtained. The sol-gel is washed with distilled water for about 3 to 4 times. Followed by this, sol-gel is washed with ethanol at once to remove polyethylene glycol and other impurities. Further, the sol-gel is heated for about 6-7 hours on a fume hood to obtain a paste of it. The obtained paste is transferred to the crucible and placed in furnace. The temperature of furnace is set to around 800 °C and heated for one hour. The white powder obtained is aluminum oxide nanoparticles.

The coating process of copper as a shell, on this alumina powder starts with activation of the surface of the substrate. The particles are subjected to coursing in HNO₃ solution for 15 minutes. The solution is stirred well for 15 minutes at room temperature. Later the particles are subjected to sensitizing in an aqueous solution of SnCl₂ for 20 minutes. Activation of particles is done in an aqueous solution of PdCl₂ for 20 minutes and stirred well at room temperature. Copper plating solution composed of 35-50 gm/l of copper sulfate solution; 18-36 gm/l of Formaldehyde, 66 gm/l of EDTA are added. NaOH is used to adjust the pH value of plating solution. The plating solution is to be stirred continuously and maintained at 30 °C. The process of washing with distilled water is repeated 5-6 times. The powder is then dried in an incubator maintaining a temperature of 60 °C. Typical Al₂O₃@Cu core@shell nanoparticles are obtained.

Al₂O₃@Cu core@shell nanoparticle suspended nanofluid coolant, were prepared with Strub Vulcan Futura oil as base coolant oil [Make: Swiss Tribology], used as a coolant for Minimum Quantity Lubrication machining applications. A total of 12 samples were prepared for testing at the nanotechnology lab, NITK Surathkal. The Al₂O₃@Cu nanoparticles were added in proportion of 0.025 %, 0.05 %, 0.75 %, 0.1 %, 0.125 %, 0.15 %, 0.175 %, 0.2 %, 0.225 %, 0.25 %, 0.275 % and 0.3 % by weight concentration to proportionate fixed amount of selected coolant. Poly Ethylene Glycol (PEG) is added as a dispersant. Stability of a nanofluid plays a vital role in attributing the required thermal characteristics, to carry away heat during machining of metals [19]. The prepared nanofluid was stable, only upon proper ultrasonification time of 1 hour with the stability retained for 24 hours.

3. RESULTS

3.1 XRD analysis

X-ray diffraction patterns of prepared core-shell nanoparticles were collected using X-ray diffractometer (Model: Bruker AXS D8 Advance). XRD analysis from figure 1 reveals that there were no peaks observed for Al₂O₃@Cu nanoparticles. This is expected as the prepared Al₂O₃ nanoparticles were in the amorphous phase. The peaks due to

copper were also absent as thickness of shell is very thin, as very low concentration of copper precursor was used. Hence

amorphous nature of particles was exhibited which is suited for tribological/coolant applications.

CU A10

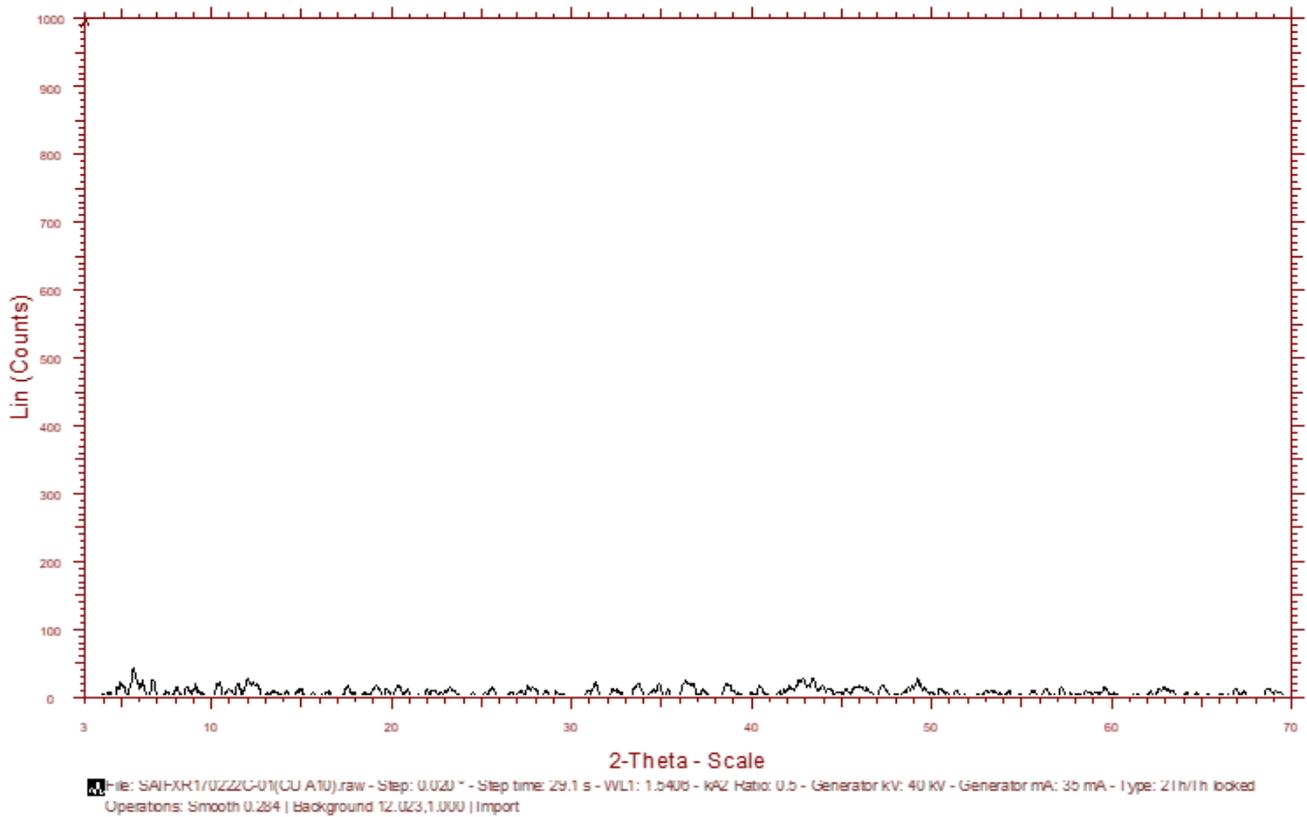


Figure 1. X-ray diffraction pattern of prepared Al₂O₃@Cu nanoparticles

3.2 SEM analysis

The surface morphology of the prepared core-shell nanoparticles was investigated using the scanning electron microscope (SEM) (Model: JEOL 6390LV). The SEM image in Figure 2 shows that the agglomerates have irregular (non-spherical) shape and size. SEM analysis reveals that the agglomerates of prepared nanoparticles are porous with different sizes and shapes, which leads to larger surface area, thus contributing to enhanced heat transfer.

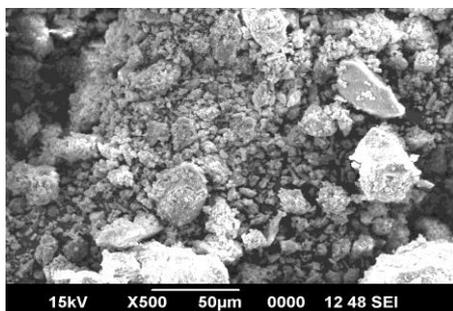


Figure 2. Scanning electron microscope image

3.3 TEM analysis

The Transmission Electron Microscopy testing was conducted at IIT Bombay, on TEM Model PHILIPS CM 200.

The image in Figure 3 shows that the average size of the particles obtained was around 20 nm. The dark portion in the image indicates the deposition of copper layer on amorphous Al₂O₃ nanoparticles. Hence core@shell nanostructures are successfully synthesized.

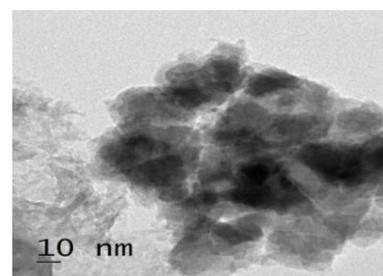


Figure 3. Transmission electron microscopic image

3.4 EDX analysis

The Energy Dispersive Spectroscopy analysis (Model: Oxford INCA X-Act) of prepared nanoparticles was done as shown in figure 4 to study the elemental composition. Peaks were observed at 0.5 keV, 0.9 keV and 1.5 keV corresponding to oxygen, copper and aluminum respectively. This shows that no other peaks were found, thereby confirming the presence of oxygen 52 % by weight, aluminum 34.13 % by weight and copper 13.87 % by weight.

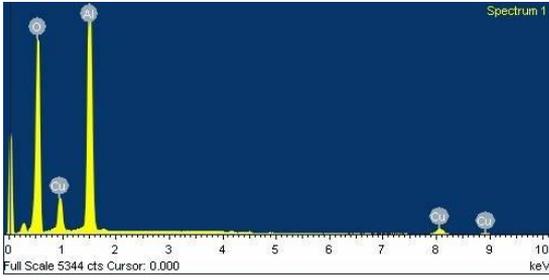


Figure 4. EDX image of Al₂O₃@Cu nanoparticles

3.5 Thermal conductivity measurements

The prepared stable nanofluids by suspending these core@shell nanoparticles were tested to determine the non-dimensional thermal conductivity (NDTC) value experimentally, using standard measuring instrument KD-2-Pro thermal property analyzer (Make: Decagon Devices, USA), using needle attached with stainless steel KS-1 sensor. The instrument provided an accuracy of 5 % that meet the standards of ASTM D5334 and IEEE 442-1981 [21], providing the measuring range of 0.2 W.m⁻¹. K⁻¹ to 2 W.m⁻¹. K⁻¹. The equipment was calibrated before taking the measurement using glycerol solution. The thermal conductivity measurements were carried out, for varying proportion of Al₂O₃@Cu nanoparticles from 0.025 % to 0.3 % to the nanofluid Strub Vulcan Futura Oil coolant. Proportionate amount of Poly Ethylene Glycol (PEG) was added as a surfactant. Widely accepted, theoretical thermal conductivity Model Hamilton and Crosser [HC] model [22] was used for calculating the thermal conductivity of prepared nanofluid at different concentrations by weight % of nanoparticle addition. The model provides non-dimensional thermal conductivity value, assuming nanoparticles to be spherical as in Eq. (1),

$$NDTC_{HCM} = \frac{k_{eff}}{k_f} = \frac{k_p + (n-1)k_f + (n-1)\phi(k_p - k_f)}{k_p + (n-1)k_f - \phi(k_p - k_f)} \quad (1)$$

where k_{eff} / k_f represents the non-dimensional thermal conductivity value, k_p represents the thermal conductivity of nanoparticle with a value of 381 W.m⁻¹. K⁻¹ (of copper), k_f is the thermal conductivity of nanofluid coolant (Strub Vulcan Futura Oil coolant) with a value of 0.11 W.m⁻¹. K⁻¹, Φ is the weight percentage of nanoparticle addition, n is the empirical shape factor equals to $3/\psi$, where ψ = sphericity with a value of 1 for spherical particles. Figure 5 shows the plot of NDTC value Vs the percentage weight of nanoparticle addition for the selected nanofluid coolant.

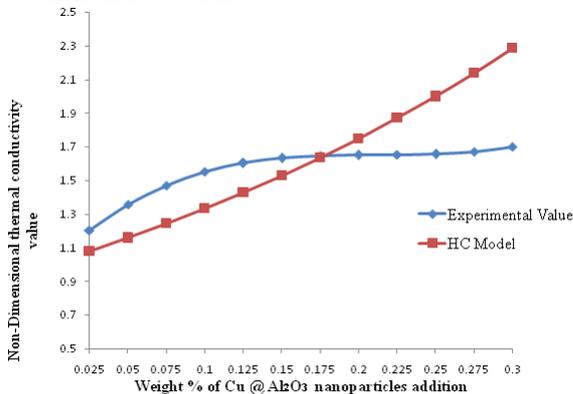


Figure 5. Plot of nanoparticle addition (in weight %) Vs NDTC value

From Figure 5 we observe that with the addition of nanoparticles in weight percentage from 0.025 % to 0.175 %, the theoretical HC model underestimates the experimental values and for further addition above 0.175 % by weight upto 0.3 %, the HC model overestimates the experimental values. This may be accounted to the fact, HC model assumed that nanoparticles are spherical in shape for regular other nanoparticles. HC model also take into account value of ‘ n ’ with $\psi=1$ for spherical nanoparticles. Since the Al₂O₃@Cu, newly synthesized core@shell nanoparticles are non spherical and of irregular shape HC model does not fit here. To account for this deviation and behavior, due to non-availability of a standard suitable model for thermal conductivity [23], a modification to Hamilton and Crosser model is needed. Hence an empirical correction factor has been proposed to HC model considering random shapes of the nanoparticles. The modified HC model (with proposed correction factor) for NDTC, fits the experimental values within an error margin of 5 %. This is shown in figure 6. Incorporation of empirically derived proposed correction factor, provides better results. The new equation with correction factors is in equation (2),

$$NDTC_{HCM} = \frac{k_{eff}}{k_f} = \frac{k_p + (n-1)k_f + (n-1)\phi(k_p - k_f)}{k_p + (n-1)k_f - \phi(k_p - k_f)} * CF \quad (2)$$

where CF= correction factor = [1.32 *exp (1.86)] in the above equation (2).

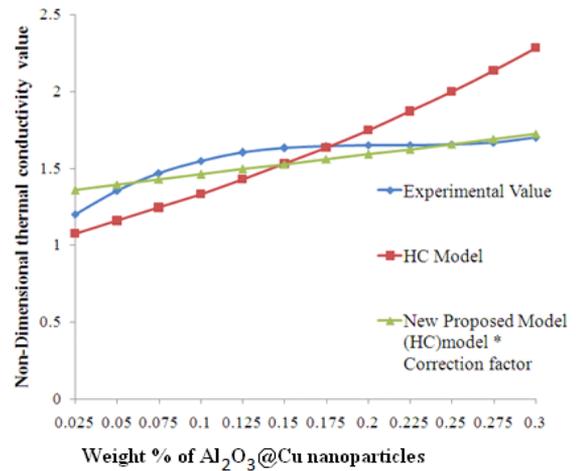


Figure 6. Plot of nanoparticle addition (in weight %) Vs NDTC value [With correction factor taken into account]

By incorporating empirically derived and proposed correction factor (CF) in equation (1), for the Hamilton and Crosser model equation (2) is obtained. The new plot is as shown in figure 6 that almost fits the obtained experimental values of thermal conductivity.

4. APPLICABILITY OF CORE@SHELL NANOPARTICLES SUSPENDED COOLANT FOR METAL MACHINING

Most of the earlier experimental studies deal with the addition of individual/single nanoparticles to different coolants, used for MQL machining applications. However for machining of difficult to cut metals, MQL technique faced a challenge. Hence the present Al₂O₃@Cu, core@shell nanoparticles possessing different morphologies and structures

with nanospheres, can be tested for wide range of cooling applications [24]. With enhanced thermal conductivity of the coolant, this nanofluid coolant used at the machine tool interface during machining, mainly contributes in carrying away the heat generated due to friction. Suspension of solid nanometer sized core@shell nanoparticles leads to brownian motion contributing to faster heat dissipation. Using ecofriendly coolants like Strub Vulcan Futura Oil coolant, with $\text{Al}_2\text{O}_3@\text{Cu}$, core@shell nanoparticle suspensions, with outer shell copper possessing high thermal conductivity value and the inner core being made of low cost material alumina, will definitely contribute to enhance the heat transfer ability at the machining zone. As seen from the graphical plot in figure 6, using $\text{Al}_2\text{O}_3@\text{Cu}$, core@shell nanofluid further enhancement of heat transfer can be achieved. Using such new $\text{Al}_2\text{O}_3@\text{Cu}$ nanoparticle suspensions in coolants, will promote in addressing the issue of less heat carrying capacity of coolants, during MQL assisted metal cutting techniques.

5. CONCLUSIONS

The present work focused upon the novel idea of coating a low thermal conductivity core aluminum oxide nanoparticle with high thermal conductivity thin shell of copper, has proven successful. The coating enhances the heat carrying capacity of the metal nanoparticle thus enhancing thermal conductivity of coolant upon its suspension. The synthesized nanoparticles are of average size 20 nm. The maximum value of thermal conductivity rise of 23.39 % is achieved at 0.3 % by weight addition of these $\text{Al}_2\text{O}_3@\text{Cu}$ nanoparticles. This indicates that addition of even small amounts of upto 0.3 % by weight of $\text{Al}_2\text{O}_3@\text{Cu}$ nanoparticles to coolant, increases its heat carrying capacity, suitable for application of this nanofluid coolant at the machine tool interface during metal machining. The research paves a way for imminent researchers in performing future studies, to synthesize different metallic core@shell nanoparticles of various combinations, that can be applied for cooling applications. Further studies on heat transfer capability could be done.

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NOMENCLATURE

SEM	Scanning Electron microscope
TEM	Transmission Electron microscope
EDX	Energy Dispersive X-ray
XRD	X-Ray Diffraction
NDTC	Non-Dimensional Thermal Conductivity constant
k_{eff} / k_f	ratio of effective thermal conductivity to thermal conductivity of nanofluid (constant)
k_p	thermal conductivity of nanoparticle in solid form in $W.m^{-1}.K^{-1}$
k_f	thermal conductivity of nanofluid coolant, in $W.m^{-1}.K^{-1}$
n	empirical shape factor
MQL	Minimum Quantity Lubrication
PEG	Poly Ethylene Glycol
CF	Correction Factor

Greek symbols

ϕ	weight % of nanoparticle
ψ	Sphericity of nanoparticle
%	percentage

Subscripts

eff	effective
f	nanofluid
p	nanoparticle