IMPROVEMENT OF THERMAL CONDUCTIVITY OF WATER BY ADDITION OF IRON POWDER

A THESIS SUBMITTED IN PARTIAL FULLFILLMENT

OF THE REQUIREMENT FOR THE DEGREE OF

Bachelor of Technology

In

Metallurgical and Materials Engineering

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May, 2014

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Certificate

This is to certify that the thesis entitled "Improvement of thermal conductivity of water by addition of iron powder" being submitted by Antariksh Anupam (110MM0094) and Sivasis Dash (110MM0408) for the partial fulfillment of the requirements of Bachelor of Technology degree in Metallurgical and Materials engineering is a bona fide thesis work done by them under my supervision during the academic year 2013-2014, in the Department of Metallurgical and Materials Engineering, National Institute of Technology Rourkela, India.

The results presented in this thesis have not been submitted elsewhere for the award of any other degree or diploma.

Date: 7.5.14

(Prof. Anindya Basu)

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Acknowledgment

We would like to express our sincere gratitude to our guide Prof. Anindya Basu, Metallurgical and Materials Engineering, NIT Rourkela, for giving us the opportunity to work with him and also providing excellent guidance and continuous assistance throughout the project work. His constant criticism, advice, assertions, appreciation were very vital and irrevocable, giving us that boost without which it wouldn't have been possible for us to finish our project. There's not a single day we can remember during our work days which went by without his scrutiny and guidance. We have received endless support and guidance from him, right from the development of ideas, deciding the experiments and methodology of work and this presentation. We are thankful to him for his encouragement throughout the project. We wish to express our deep sense of gratitude to Dr. B.C.Ray, HOD, Metallurgical and Materials Engineering, NIT Rourkela for giving us an opportunity to work on this project. We would be highly obliged to extend our thanks to Mr. Uday Kumar Sahu for his immense support and help rendered while carrying out our experiments, without which the completion of this project would have been at stake. We would also like to thank all the staff members of MME Dept., NITR and everyone who in some way or the other have provided us valuable guidance, suggestion and help for this project

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Abstract

The current work aims to improve the thermal conductivity of distilled water by dispersing electrolytic grade iron powder. Thermal conductivity of fluids is an important parameter in deciding their usability in various commercial applications. Nanoparticles dispersed in fluids generally show interesting properties with respect to thermal conductivity. Iron particles were dispersed in distilled water in different volume fractions (1, 2 and 3 percent respectively) and the resultant fluids were analysed in terms of their thermal conductivity. To study the effect of particle size, the as received iron powder was also ball milled and the same set of studies were repeated with the milled powder. All the conductivity measurements were carried out at room temperature the data were compared with the conductivity value of the pure distilled water. The effect of solid powder additions on distilled water results the increase in thermal conductivity with increase in concentration of iron powder. Effect of milled powder was also compared with that of un-milled powder

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1. Introduction

Nanofluids, are the colloidal suspensions of nanoparticles in fluids. It is known that they exhibit unusually large thermal conductivities. Hence, nanofluids have attracted interest from many researchers due to their potential advantages for numerous uses such as microelectronics, energy supply, transportation, and to increase the heat transfer phenomena of fluids. Based on constant Nusselt number, the convective heat transfer coefficient is directly proportional to the thermal conductivity. Before calculating the heat transfer coefficient of any nanofluid, it is important to estimate their thermal conductivity. It has recently been observed that a two stage approach of synthesizing nano-sized powders by mechanical alloying and subsequently dispersing the same in a given fluid could be a better method of producing nanofluid with a greater scope of scaling up the process of synthesis. The performance of nanofluid invariably depends upon the size and distribution of dispersed phase and their ability to remain suspended and chemically unreacted in the fluid. It is well observed that the uniformity and stability of suspension can be ensured by maintaining appropriate pH, using surface activators or surfactant and employing ultrasonic vibration. Though temperature dependence of the thermal properties of solid particles and liquid can have a remarkable effect on conductivity of nanofluids at increased temperature or higher thermal gradient, as our work concentrates only on the designing of nanofluids at low temperature range and small thermal gradient areas, hence, studies on those aspects are beyond the scope of our project.

2. Literature Survey

2.1 Thermal Conductivity

Thermal conductivity (often denoted as *k*) represents the ability of a material to conduct heat fluids are important media of heat transfer in industrial applications. Increasing the thermal conductivity of fluids can have amazing advantages in terms of increased productivity and efficiency. Heat transfer coefficient of fluids is the parameter which is used in industrial uses, but thermal conductivity is an important laboratory parameter that closely parallels the value of heat transfer coefficient.

2.2 Influencing factors

Temperature

The influence of temperature on thermal conductivity is different for metals and non metals. In metals conductivity happens mainly due to free electrons. The electrical conductivity in metals closely parallels the thermal conductivity. In metals, the thermal conductivity remains almost constant with increase in temperature. In alloys the change in electrical conductivity is considerably lower, and hence thermal conductivity increases with temperature, often proportionally to temperature.

Lattice vibrations, (phonons) are responsible for heat transfer in non-metals. The phonon mean free path doesn't undergo any significant changes at higher temperatures. Hence, the thermal conductivity of non metals is almost constant at low temperatures. This property of non metal has interesting industrial applications.

Chemical phase

Thermal conductivity of a material may change upon phase change. For example, thermal conductivity changes when ice (thermal conductivity of 2.18 W/(m·K) at 0 °C) melts to form liquid water (thermal conductivity of 0.56 W/(m·K) at 0 °C).

Thermal Anisotropy

Different crystal directions in crystalline substances may show different thermal conductivity due to different properties and anisotropy. Sapphire is a notable example of variable thermal conductivity based on orientation and temperature, with 35 W/(m·K) along the c axis and 32 W/(m·K) along the a axis.

Electrical conductivity

In metals, thermal conductivity almost parallels electrical conductivity as freely moving valence electrons transfer electric current as well as heat energy. However, the general dependence of electrical conductance on thermal conductance does not hold for other materials, due to the increased importance of phonon carriers for heat in non-metals. Diamond is an electric insulator but good heat carrier.

Magnetic field

The influence of magnetic fields on thermal conductivity is known as the Righi-Leduc effect.

2.3 Nano fluids

The transfer of heat using fluids is important in several engineering areas like automobiles, nuclear power plants, etc. The efficiency, in terms of design and performance, is increased by the ability to transfer heat across a pre decided thermal gradient. Convection is the main mode of heat transfer through fluids. Solids have far greater thermal conductivity coefficients than fluids, hence the addition of solids to fluids will definitely improve the thermal conductivity of fluids.

Nanofluid is a stable colloidal

suspension of low (<1%) volume fraction of ultra-fine solid particles in nanometric dimension dispersed in conventional heat transfer fluid to offer a dramatic enhancement in conductivity of the fluid , and the problems associated with milli and micro sized particles also do not occur. The exact mechanism for this phenomenon has not been explained yet.

2.4 Particle size reduction by ball milling

It is a process in which a powder mixture placed in a machine called a ball mill is reduced in size due to continuous collisions with small metallic balls.

Earlier, high temperature synthesis was the most widely used method for manufacturing materials.

Phase transformations can also be induced powders with same chemical composition particles: amorphization or polymorphic transformations of compounds, disordering of ordered alloys, etc. However, the principles of these operations are same for all the techniques. Successful alloying is subject to balancing the two processed of cold welding and fracturing.

Planetary ball mill has the advantage of using only a small amount of powder. The ball mill system consists of one turn disc (turn table) and two or four bowls. The turn disc rotates in one direction while the bowls rotate in the opposite direction.the powder experience the effects of the centrifugal force due to the rotation of the mill and the movement of the balls. High energy impact causes cold welding and fracturing.

The figure below shows the motions of the balls and the powder.

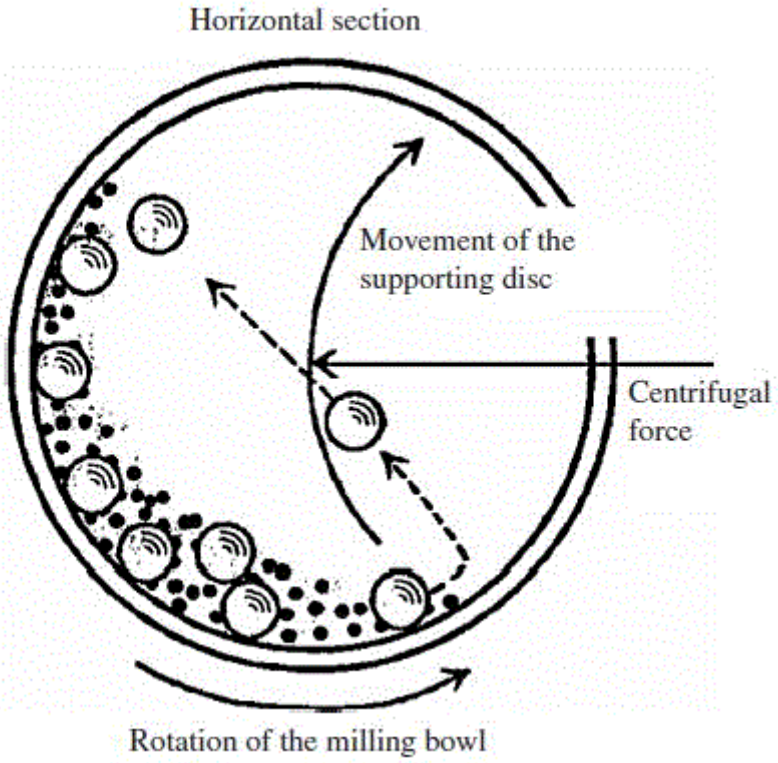

Fig 1 – Movement of milling bowl and balls

The bowl and the turn disc rotate in opposite directions; hence the centrifugal forces are synchronized.

The impact energy of the milling balls in the normal direction attains a value of up to 40 times higher than that due to gravitational acceleration. Hence, the planetary ball mill can be used for high-speed milling.

Fig 2 – Planetary Ball mill

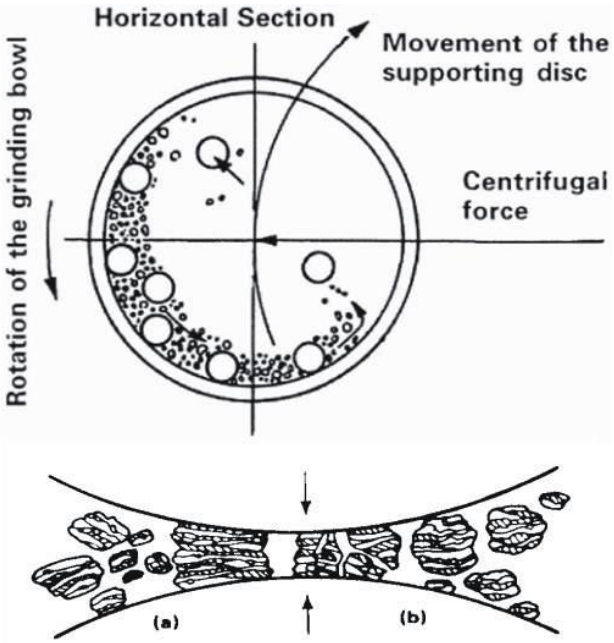

Fig – 3 Milling mechanism

Microstructurally, the mechanical alloying process can be

divided into four stages: (a) initial stage, (b) intermediate stage, (c) final stage, and (d) completion stage.

(a) At the initial stage of ball milling, the powder particles are flattened by the compressive forces due to the collision of the balls. Changes in the shape f the powder particles may occur due to micro forging. There is no change in net mass.

(b) At the intermediate stage of the mechanical alloying process, significant amount of cold welding and fracturing occurs. The intimate mixture of the powder constituents decreases the diffusion distance to the micrometer range. The alloyed powder is still not homogenous in terms of chemical composition.

(c) In the final stage of the process, microstructure of the particle appears to be more homogenous in microscopic scale than those at the initial and intermediate stages.

(d) At the completion stage of the mechanical alloying process, the powder particles possess an extremely deformed metastable structure. At this stage, the lamellae are no longer resolvable by optical microscopy. Further mechanical alloying beyond this stage cannot physically improve the dispersoid distribution. Real alloy with composition similar to the starting constituents is thus formed.

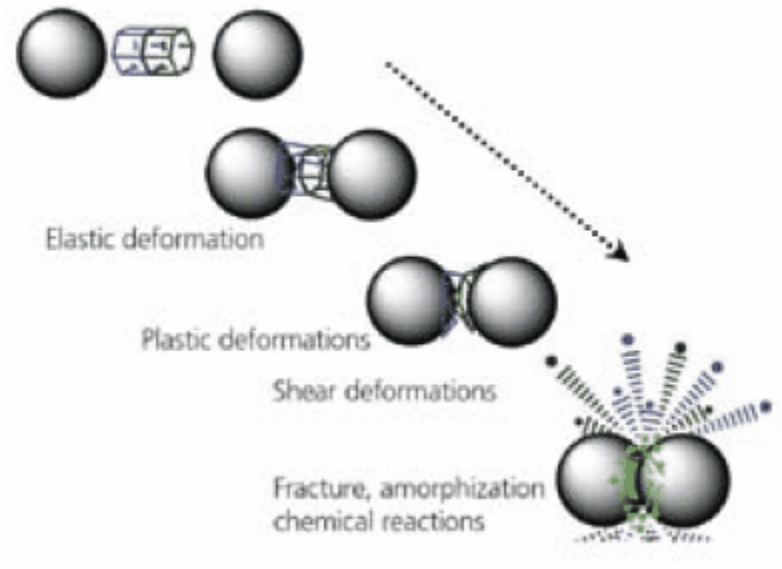

Fig – 4 – Size reduction mechanism

3. Experimental Procedures

3.1 Ball Milling

- Iron (electrolytic, purity = 99.5%) powder of 250 - 300 mesh size, was added to distilled water, was first grinded in a planetary mill for 2.5 hours.
- 25 g of iron powder was taken in each container of the milling machine with a ball to powder ratio of 1:10. The balls taken were of chrome steel.
- Toluene was added to the mixture to provide temperature stability as well as to prevent oxidation of the iron powder.
- The milling was carried on for intermittent periods of 30 minute each, with 30 minutes of gap in between due to the requirements of the machine. On the whole, the milling was carried out for a total time of 2 hours and 30 minutes.
- The powder was taken out and dried to get rid of the remaining toluene. It was then taken for further processing.

3.2 X – Ray Diffraction

- XRD analysis of the powder before milling and after milling for 2.5 hours was done.
- The machine used was a 'Philips X'PERT' X Ray Diffraction machine which used a copper target.
- The scanning range was from 30 degrees to 100 degrees, and the step size was 2 degrees.

Scherrer equation, gives the relation the size of particles, or crystallites, to the broadening of a peak in a diffraction pattern. It is used to determine the size of particles of crystals in the form of powder.

The Scherrer equation can be written as:

$$\tau = \frac{k\lambda}{\beta cos\theta}$$

where:

- τ is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size;
- K is a dimensionless shape factor, with a value close to unity. The shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite;
- λ is the X-ray wavelength;
- β is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians. This quantity is also sometimes denoted as $\Delta(2\theta)$;
- θ is the Bragg angle.

The crystallite size of the powder was calculated using the Scherrer equation.

3.3 Sonication

Sonication is the process of application of sound energy for the agitation of particles in a sample. The process is also aometimes called ultrasonication because ultrasonic frequencies are employed. It is usually applied using an ultrasonic bath or an ultrasonic probe, known as a Sonicator.

Fig 5 – Ultrasonication machine

- The iron powder of both types was dispersed in distilled water in 1, 2 and 3 percent volume fractions.
- Each of these dispersions were then subjected to ultrasonication in sonicating machine for 15 minutes.

3.4 Thermal Conductivity Measurements

Thermal conductivity measurements were made using the device KD2 pro Thermal analyser,

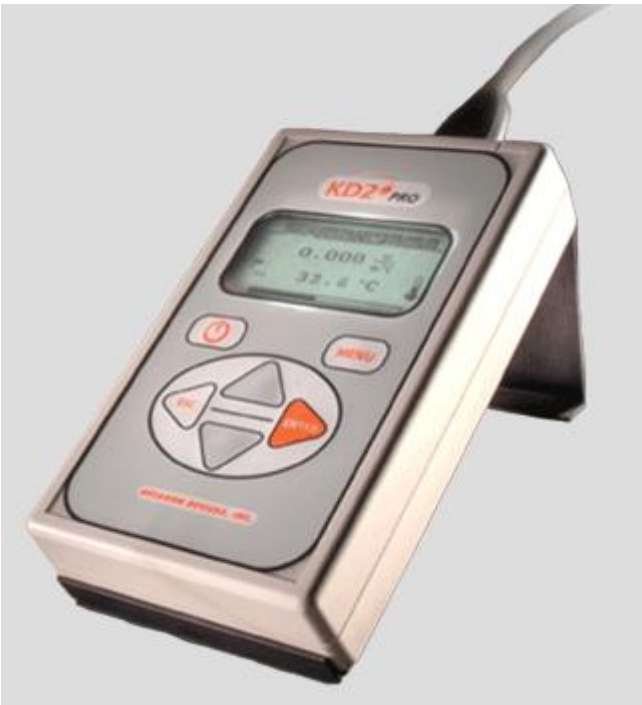

Fig 6 – KD_2 Pro Thermal Conductivity Analyser

This hand held device uses the technique of the transient line heat source to evaluate the thermal properties of the fluid. The unit has 5 % accuracy over the 5 ^{o}C to 40 ^{o}C temperature range and also meets the standards of both ASTM D5334 and IEEE 442-1981. A needle sensor is inserted into the fluid. A single reading takes 2 minutes. The first ninety seconds are sude to endure thermal stability. After that heating of the probe is done for half a minute using a known amount of current. A thermistor is also present to take care of changes in temperature.

Figure below illustrates the experimental setup used for nanofluid thermal conductivity measurements.

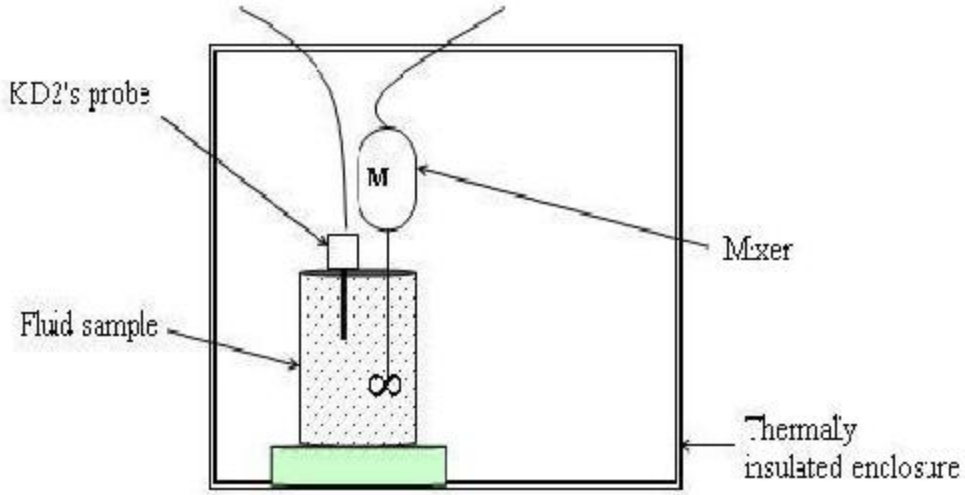

Fig 7 – Experimental Setup

- The thermal conductivity of the dispersions having different volume fractions of iron powder were measured in the KD2 Pro measuring instrument.
- The thermal conductivity data was analysed and compared to each other to observe the general trend.

4. Results and Discussion

4.1 XRD Analysis Data

The plots obtained after the XRD of the two types of powders are presented in the form of plots as shown here.

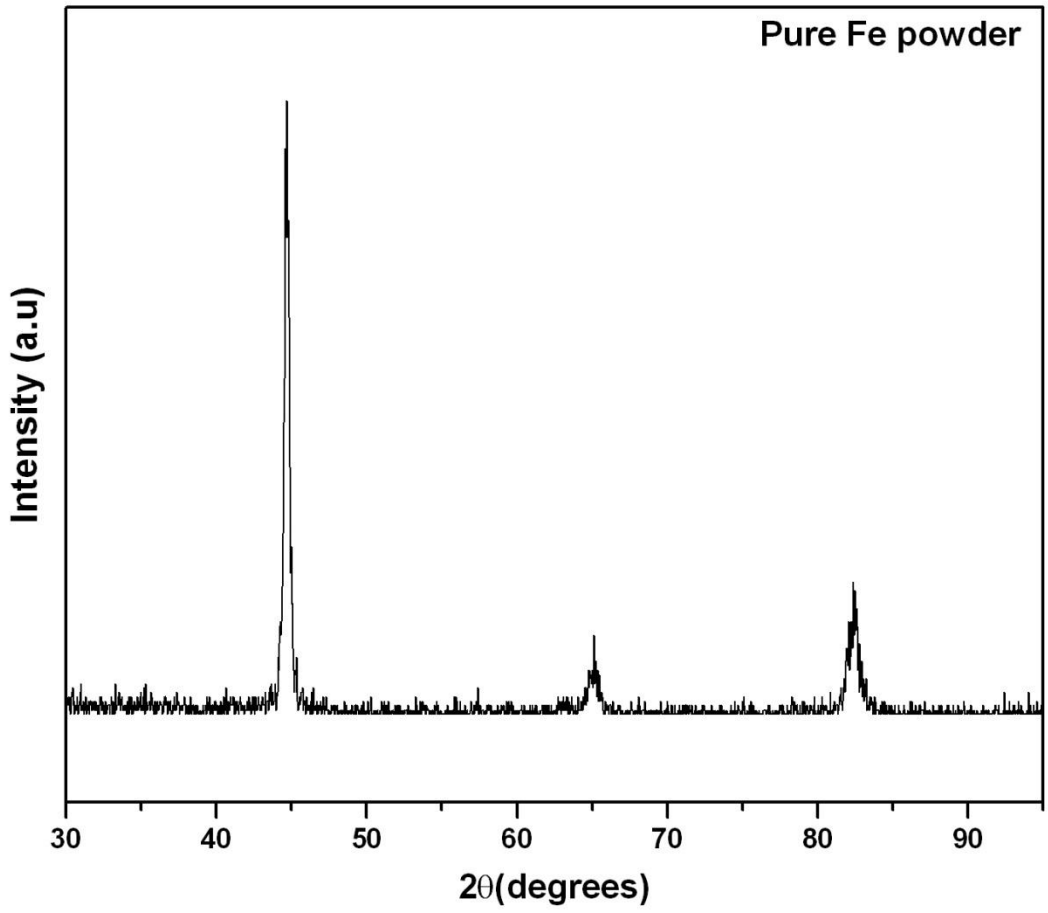

Fig 8 – X-Ray Diffraction plot of pure electrolytic iron powder

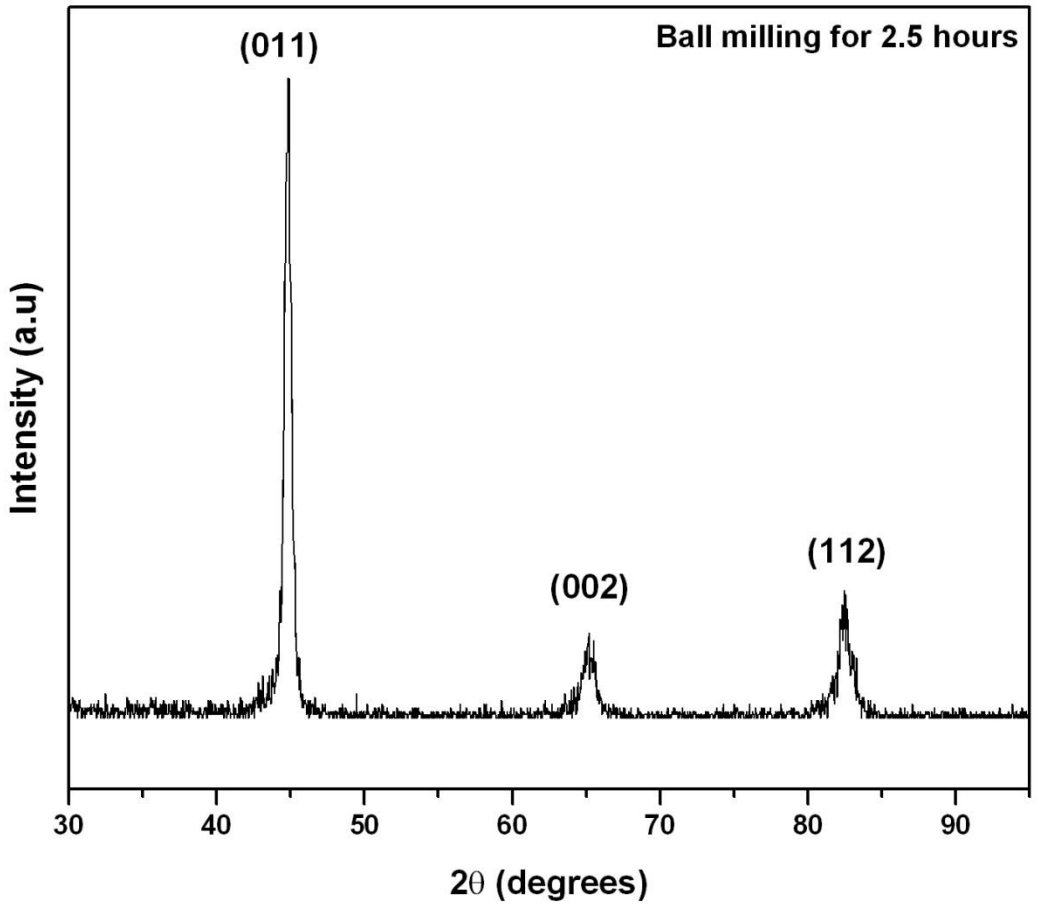

Fig 9 – X-Ray Diffraction plot of 2.5 hr milled iron powder

From the figures it can be observed that only peaks of pure Fe (BCC) is visible. From the plots by Scherrer equation the crstlallite/grain size of the powder was estimated. Instead of particle size estimation, grain size was calculated before and after of milling so that an idea can be obtained about the severity of the milling.

4.2 Crystallite size calculation using XRD data

Grain size obtained by Scherrer equation is tabulated as below:

$K = 0.9$, $\lambda = 1.541 \text{ X } 10^{-10}$ m (Cu target K_α line wavelength)

Sample	β (degrees)	Θ (degrees)	τ (nm)
As taken iron powder	0.3731	22.35	22.68
2.5 hr milled iron powder	0.507	22.432	16.69

From the table it can be observed that after 2.5 hr of milling there is reduction in grain size which is also an indication of reduction in particle size. The actual size calculated here is an indication only as there may be strain components which can increase the peak width.

4.3 Thermal Conductivity Data

The variation of thermal conductivity of the distilled water in as received condition and with addition of different volume % of iron powder is shown in Fig10. It can be observed that with increase in Fe content the conductivity increases, but not linearly. This is due to the higher heat conductivity property of metallic Fe powder.

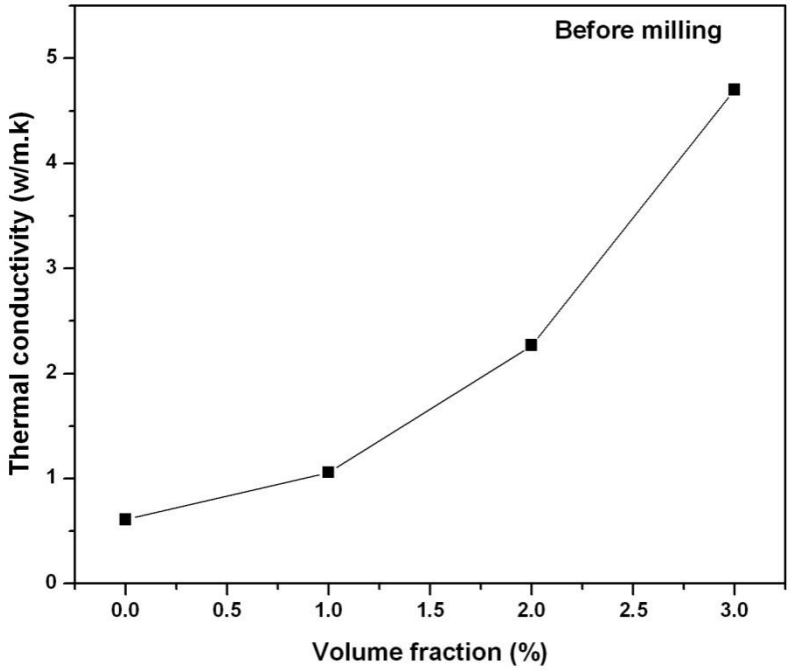

Fig 10 – Thermal Conductivity of as-taken iron powder dispersion

Fig 11 shows the conductivity data for 2.5 hr milled powder and it shows similar trend with the earlier plot.

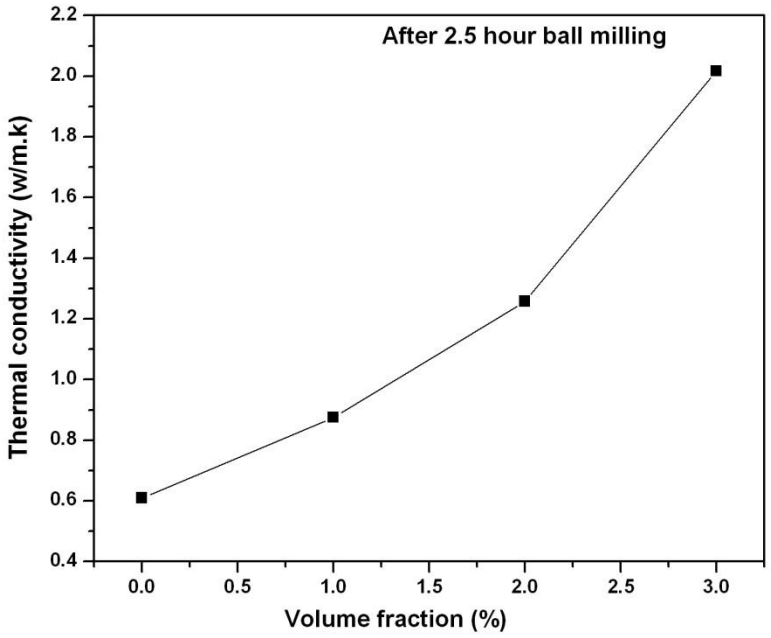

Fig 11 – Thermal Conductivity of 2.5 hr milled iron powder dispersion

Fig 12 shows the combined data of thermal conductivity of all the powder added distilled water. Here it can be observed the with milled powder, the conductivity value of the dispersed fluid decreases at same level of dispersion addition. This trend is somewhat different from earlier report. This can be due to the floatation of fine powder at the air fluid interface.

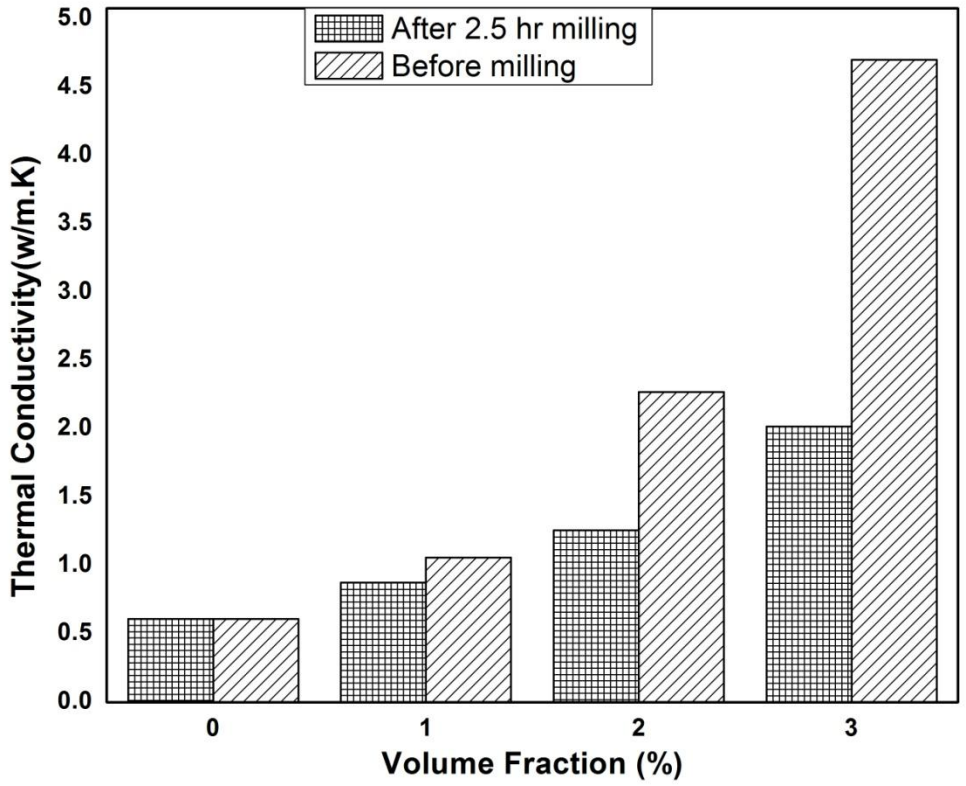

Fig 12 – Comparison of thermal conductivity of two different dispersions

5. Conclusions

i) Pure Fe powder was ball milled and after milling the grain size was decreased due to incorporation of heavy energy.

ii) Fe powder dispersed fluid was prepared and it was observed that with increasing Fe content of the fluid (distilled water), its thermal conductivity was increased due to metallic dispersion.

iii) In case of dispersion with ball milled powder the conductivity was low which may be due to floatation of fine powders.

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