Synthesis and Characterization of Al-Cu Nanocomposite and its Water Based Nanofluid

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Abstract: In this paper, synthesis of ultrafine Al-Cu nano composite and its stable dispersion in base fluid is carried out in two step method. Ultrafine powders were prepared by milling elemental Al and Cu powders for nearly 50 hours in a planetary mill. Powder nano composite is dispersed in de-ionized water to produce nanofluid by ultrasonication method. The prepare powder was studied by using XRD, SEM and TEM techniques, water based nanofluids were prepared and their stability has been studied by nano zeta meter at different pH of nanofluids for constant ultrasonication time and magnetic stirring. The results reveal that dispersion stability of Al-Cu ultrafine nano composite in base fluid is better than their fluids of individual Al and Cu particles. This dispersion stability enhances the thermal conductivity and convective heat transfer of nanofluids.

Keywords: Nanocomposite, nanofluid, dispersion stability, thermal conductivity

1. Introduction

Heat transfer is one of the most important processes in many industrial and consumer products. The inherently poor thermal conductivity of conventional fluids puts a fundamental limit on heat transfer. Therefore, for more than a century since Maxwell [1], scientists and engineers have made great efforts to break this fundamental limit by dispersing millimeter- or micrometer-sized particles in liquids. However, the major problem with the use of such large particles is the rapid settling of these particles in fluids, clogging and erosion of pipes and channels. Nanofluids are engineered by suspending nanoparticles with average sizes below 100 nm in traditional heat transfer fluids such as water, oil, and ethylene glycol. Nanofluids (nanoparticle fluid suspensions) is the term coined by Choi [2] to describe this new class of nanotechnology based heat transfer fluids that exhibit thermal properties superior to those of their host fluids or conventional particle fluid suspensions.

The concept of nanofluid has attracted considerable interest because of reports of great enhancement of heat transfer [3, 2], mass transfer [4], and wetting and spreading [5]

For example, the thermal conductivity of copper at room temperature is about 700 times greater than that of water and about 3000 times greater than that of engine oil, Based upon the above discussion of the versatile properties and applications of nanofluids, we have made an approach to synthesize Al, Cu, Al-Cu based nanofluids which can transfer heat more effectively. In the present study, the primary objectives are to synthesize elemental ultrafine particles Al, Cu and Al-Cu alloy & then preparation of stable dispersion of ultrafine particles in base fluid to develop heat transfer fluids.

2. Experimental Method

2.1 Synthesis of ultra fine Al, Cu and Al-Cu nanocomposite

The primary objectives are to synthesize elemental ultrafine particles Al, Cu and Al-Cu nanocomposite & then preparation of stable dispersion of particles in nanofluid to develop heat transfer fluids. There are mainly two techniques for synthesizing nanofluids, which are **2.1. Single-step process** which simultaneously makes and disperses the nanoparticles directly into the base fluids [4-5].

2.2. The Two-Step Method

It represents the formation of nanoparticles and subsequent dispersion of the nanoparticles in the base fluid [6, 7]. Many other routes are used for production of ultrapure nanoparticles like physical and chemical vapour deposition [8],coprecipitation [9], sonochemical technique [10], sol–gel [11], hydrothermal [12,],solution phase method [13], electrochemical synthesis [14], laser ablation [15,].

In the present study nanofluid is produced by using two step method, at first Milling was carried out in Pulverisette-5 planetary ball mill with steel vials and steel balls to prepare ultrafine particles. Starting materials used for milling were elemental Al & Cu powder with 99% purity for the synthesis of ultrafine Al and Cu particles. Powder particles were milled for 50h in two vials- each containing 35g powder and 350g steel balls. In another set of experiment, 50 atomic wt % of Cu and Al powders were mixed and milled for 50 hours to prepare ultrafine Al-Cu powder. The ball to powder weight ratio (BPR) was 10:1. Milling was conducted at 300 rpm in wet medium (about 50 ml of toluene) to prevent undue oxidation and agglomeration of powder. Steel balls of diameter 10 mm were used for milling. Powder samples were picked up from the vials after selected interval of milling time to see the change in shape and size reduction of powder samples. A very small amount of milled powders

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Department of Physics, Rashtrasant Tukdoji Maharaj Nagpur University, Nagpur, Maharashtra, India Licensed Under Creative Commons Attribution CC BY (approximately 0.04g) were dispersed in de-ionized water (150 ml) by ultrasonication and subsequently magnetic stirring for about 30 minute each to prepare the desired nanofluid.

3. Results and Discussions

3.1 X-Ray Diffraction (XRD)

The prepared samples were characterized using laboratory setup of X-ray diffraction with Cu-K α radiation (λ =1.54Å). The XRD patterns of Al-Cu alloy powder particles at different intervals of milling time as shown in fig 1.The XRD pattern of as received powder shows the peaks of Cu and Al, whereas the final milling product is a single phase nanocrystalline Al-Cu alloy which is clear from the graph. It is evident from the figure that after 10 hours of milling, Al-Cu has started to form. It is also clear from the figure that Bragg peaks for milled product (after 50h of milling) are broad, suggesting accumulation of lattice strain and reduction in crystallite size.



Figure 1: The XRD patterns of Al-Cu powder particles and its composite at different intervals of milling time.

3.2 Scanning electron microscopy (SEM) study:

This microscopic study helps to analyze particle size, morphology, types of other materials present in the structure. SEM study was done for powder different milling time at different magnifications. At the initial stage of milling, the powders of Al and Cu are bulky, with random shape and size. As the milling progresses the powders become more homogeneous and compact. From below pictures it can be seen that with increasing milling time the grain size decreases and particles become flat because of straining.



Figure 2: SEM micrographs of Al-Cu alloy at different milling times: 0 hrs, 10 hrs, 25 hrs, 50hrs

Transmission Electron Microscopy (TEM)

The sample for TEM was prepared by adding a pinch of milled elemental metallic powder particles in the beaker containing acetone and kept in an ultrasonic bath for about 15 minute to get uniform dispersion of powder particles in the liquid. After that 2 drops of fluid containing dispersed particles were added in carbon coated Cu-grid and then dried. The desired sample was fixed in the sample holder of TEM for analyzing the internal structure of mechanically alloyed powder. Fig.3 shows the bright field TEM micrograph and corresponding SAD pattern of 50 hours milled powders. It is evident from the figure that the particle size is around 300nm and contains large number of crystallites (size around 15-20 nm) with difference in contrast due to the variation of orientation.

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Figure 3: Bright Field TEM micrograph and corresponding SAD pattern: (Al-Cu)

Dispersion stability of Al, Cu and Al-Cu ultrafine particles in nanofluid

The stability of Nanofluid was determined by measuring zeta potential values of elemental metallic and alloy powder dispersed in deionized water. The values of zeta potential ζ can be calculated by the Helmholtz-Smoluchowski equation.

$\boldsymbol{\zeta} = \mu \boldsymbol{U}/\boldsymbol{\varepsilon}$

Where U is the electrophoretic mobility, and μ , ϵ are the viscosity and the dielectric constant of the liquid in the boundary respectively. In case of Cu powder particles, the zeta potential is zero at pH= 5.1, which is isoelectric point as shown in Fig 5.1. Therefore the force of electrostatic repulsion between particles is not sufficient to overcome the attraction force between particles and hence the dispersion is least stable. At pH = 2.3, the zeta potential becomes higher; the electrostatic repulsion force between particles is stronger, and the coagulated particles can redisperse through mechanical force. Therefore the dispersion stability of copper (Cu) is best at pH = 2.3 and corresponding zeta potential value is 14.6 mV. If pH-value is less than 2.3, then the zeta potential of particle surface and electrostatic repulsion force decreases due to compression of electrical double layer. Therefore, the suspension exhibits a poorer dispersion. Similarly, for Al powder particles, the zeta potential is zero at pH = 8.9 which is isoelectric point as shown in Fig 5.2 and hence the dispersion is least stable. With decreasing pH value by adding reactant reagent, the stability tends to increase and therefore at pH = 2.5 the zeta potential becomes higher; the electrostatic repulsion force between particles is stronger, and the coagulated particles can redisperse through mechanical force. Therefore the dispersion stability of Al is best at pH=2.5 and corresponding zeta potential value is 54.63 mV. If pH value is less than 2.5, then the zeta potential of particle surface and electrostatic repulsion force decreases due to compression of electrical double layer. Therefore, the suspension exhibits a poorer dispersion.In case of Al-Cu alloy powder particles, the zeta potential is zero at pH= 9.7, which is isoelectric point as shown in Fig 4.1. Therefore the dispersion stability of Al-Cu alloy is best at pH=10.3 and 4.96 corresponding to zeta potential value of 49 and -27.7 mV.



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Figure 4: The evolution of zeta potentials of the deionized water-based elemental metallic and alloy powder nanofluids as a function of pH without surfactants. Top left fig 4.1 (Cu); Top right fig 4.2 (Al) and Bottom fig 4.3 (Al-Cu).

4. Conclusions

The following conclusions can be drawn from the present investigation:

- 1) It is possible to prepare ultrafine Al-Cu particles through mechanical alloying process by 50 hours of planetary ball milling.
- 2) The crystallite size decreases and internal strain increases rapidly with milling time up to about 25 hours. With further milling, the crystallite size remains almost constant but the lattice strain appears to increase. It is found from XRD that the crystallite size it is around 31nm after 50 hours of milling. 3. In case of Al-Cu composite, the composite formation starts after 10 hours of milling. The crystallite size decreases and internal strain increases with milling time up to about 50 hours. It is found from XRD that the crystallite size is around 6 nm and lattice strain is 1.434 % for Al- Cu alloy. The dispersion of these stable nanostructures in water will improve the thermal conductivity appreciably and heat transfer capacity.

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