

Characterization and Performance of Eco-friendly Coolants in Sustainable Machining of Inconel 718

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CERTIFICATE

This is to certify the thesis entitled "**Characterization And Performance Of Eco-friendly Coolants in sustainable Machining of Inconel 718**" submitted by **Mr. Mechiri Sandeep Kumar Venugopal** for the award of the degree of **Doctor of Philosophy in Mechanical Engineering**, in the Faculty of Engineering of **National Institute of Technology, Warangal** is a bonafide research work carried out by him under my guidance in the **Department of Mechanical Engineering, National Institute of Technology, Warangal, Telangana**.

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DECLARATION

I, hereby declare that the matter embodied in this thesis titled **“Characterization And Performance Of Eco-friendly Coolants in sustainable Machining of Inconel 718”** is the result of research carried out by me under the guidance of **Dr. V. Vasu** and **Prof. A. Venugopal**, Department of Mechanical Engineering, National Institute of Technology (Deemed University), Warangal, Telangana. This work or any part of this work has not been submitted to any other University or Institute for the award of any other degree or diploma.

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Dedicated To My Parents,
And
Friends

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ABSTRACT

Manufacturing by mechanical machining has historically benefited from the use of cutting fluid. Cutting fluids help to reduce temperature, friction, flush away chips, and hence prolong tool life and improve machining performance. However, uncontrolled use of cutting fluid raises concern in respect of cost and environmental burden. For these reasons, dry machining is used in conjunction with high speed machining to reduce cycle times and simultaneously deliver a greener process. However, for some workpiece materials full implementation of dry machining is not economically viable due to the absence of the essential cooling and lubricating functions delivered by cutting fluids. The most feasible bridging technology is minimum quantity lubrication (MQL) where a very small flow rate of coolant/lubricant is delivered to the cutting zones. In terms of machinability, the application of MQL is promising.

New trend concerning the cutting fluids in machining is to replace hazardous components with environmentally friendly compounds. These new compounds not only must show the same properties to cutting fluids but also must improve the machining performance such as productivity. Literature studies about machining using vegetable based cutting fluids are limited. When lubricant is supplied to the cutting zone, it reduces the friction between the chip and the workpiece. The heat produced during the machining can be reduced by using lubricants which possess high thermal conductivity. Due to emerging of nanotechnology, fluids possessing high thermal conductivity called nanofluids have evolved.

The motivation for this PhD work was to prepare a novel hybrid nanoparticle, selection of basefluid and testing them in machining. In this work synthesis of various combinations of hybrid Cu-Zn nanoparticles and characterization is explored. A two-step approach is considered for synthesis of Zn, Cu, and Cu-Zn (50:50) nanoparticles by mechanical alloying, followed by dispersing them in a base fluid (vegetable oil) to prepare nanofluids. The thermophysical properties like thermal conductivity and viscosity were examined, where hybrid nanofluids showed better enhancement than individual nanoparticles.

For selection of basefluids, studies were carried out by incorporating hybrid nanoparticles in to bio degradable oils (vegetable oils) and conventional metal cutting oils. Vegetable oil as basefluid showed better increment in flash point, thermal conductivity and showed marginal less stability. On the basis of integrated study of thermal conductivity and

viscosity, in the point of cost and higher relative thermal conductivity to relative viscosity for effective heat transfer vegetable oil based nanofluid showed better results.

Further investigation was carried out for various combinations of Cu-Zn (75:25, 50:50 and 25:75). Outcome of these combinations showed hybrid Cu-Zn (50:50) alloy combination showed better result. For further enhancement Cu-Zn-Ag (40:50:10) was prepared with addition of Ag to Cu-Zn (50:50) combination. With this combination only 9% enhancement was obtained, compared to Cu-Zn (50:50). This enhancement in thermal conductivity comes at higher cost; therefore Cu-Zn (50:50) yields the purpose of enhancing the thermal conductivity at lower cost. Machining was carried out on Inconel 718 with hybrid nanofluids (MQL/nanofluid) as cutting fluids with hybrid Cu-Zn (50:50) nanoparticles dispersed. Tests for cutting forces and surface roughness were carried out and compared with dry and MQL/vegetable oil. This work also deals with residual stress measurement for all three lubricating conditions. MQL/nanofluid showed better results when compared to dry and MQL/vegetable oil.

A new hybrid nanoparticle and choice of basefluid which is bio degradable is developed in this present work. The work is important to industry in that it contributes to develop tailored material that are inexpensive and possess high thermal conductivity.

Keywords: Hybrid Nanoparticles, Bio degradable oils, thermophysical properties, Inconel-718, Cutting forces, Surface roughness, Residual stresses.

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ABBREVIATIONS

FE-SEM	Field emission Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
XRD	X-ray Diffraction
ANN	Artificial Neural Network
ANOVA	Analysis of variance
SDS	Sodium Dodecyl Sulfate
DLS	Dynamic Light Scattering
SAE	Society of Automotive Engineers
MQL	Minimum Quantity Lubrication
PCR	Percentage Contribution
CF	Cutting Force
CS	Cutting Speed
SR	Surface Roughness
EDAX	Energy Dispersive Spectroscopy

NOMENCLATURE

W_p	Weight of Particles
ρ_p	Density of Particles
W_{bf}	Weight of Base fluid
ρ_b	Density of Base fluid
ϕ	Volume fraction
T	Temperature
D_p	Diameter of particle
K_p	Thermal conductivity of Particle
K_{bf}	Thermal conductivity of Base fluid
K_{NF}	Thermal conductivity of Nanofluid
μ_{bf}	Viscosity of Base fluid
μ_{NF}	Viscosity of Nanofluid
DOC	Depth of Cut

CHAPTER 1

INTRODUCTION

1.1 Research Background

United Nations world commission on environment and advancement has defined sustainability as the capacity to experience the need of the present without compromising the ability of future eras to address their own issues. Nowadays metal cutting area is under immense pressure to enhance environmental performance because of the implementation of strict environmental regulations. With the addition of sustainable practices in metal cutting sector, environmental performance can be enhanced under economic conditions. The possibility of sustainable manufacturing manages viable utilization of energy, vitality, health safety, material flow, well being security, and environmental concerns. Sustainability in manufacturing sectors can be achieved by employed by reducing the use of toxic and non-biodegradable chemicals, minimizing resources, efficiently designing life cycles and improving working conditions.

The concept of sustainable manufacturing, aims toward the reduction of greenhouse gas emissions and the ecological footprint. It is also important to consider the surface integrity of manufactured parts to promote sustainability practices. Most of the metal working fluids are hazardous in nature, thus limiting their usage is necessary. With this, alternate methods like near dry and minimum quantity lubrication techniques are being developed to reduce the use of flood lubrication.

1.2 Machinability of difficult-to-cut materials

The machinability of materials depends on their mechanical, chemical and thermal properties. Some are not always easily machinable; these materials are called difficult-to-cut materials. Inconel 718 is one such material that possesses high strength at elevated temperature, high chemical affinity and low thermal diffusivity. It is the most commonly used nickel base alloy used in manufacturing aerospace components such as engine cases, combustors, blades and seals are common. In addition to these it is uses include submersible well pump motor shafts, turbocharger rotors and seals, heat exchanger tubing, chemical

processing and pressure vessels and steam generators in nuclear pressurized water reactors. The Pratt and Whitney aircraft division and research Centre of United Techno Corporation [6], assigned a machinability rating of less than 25% for Ni based superalloys and for Inconel 718 was rated as 14%. Due to rapid work hardening ability and also due to presence of hard carbides in its microstructure, the machinability of Inconel 718 is poor. It also possesses poor thermal diffusivity and high dynamic shear strength which increases the temperature at the machining zone. The rise in temperature, in the cutting zone cannot be totally avoided, but can be minimized by the use of proper environment. There are several methods to achieve proper cooling and lubrication of the cutting zone. Today's industry performs machining in flood cooling environment. The machining industry has been using conventional cutting fluids like oil based and water based fluids, to lubricate and cool the cutting zone. These conventional cutting fluids create environmental and health problems.

Machining at high speeds can lead to shorter cycle time, rapid material removal rates, better surface quality and low cutting forces. However, machining at high cutting velocities offers challenges such as high interface temperatures and short tool life. Cutting fluids that can be utilised to reduce heat generation and temperature can be ineffective when running in at high speeds, as it is difficult for the cutting fluids to gain access to the secondary contact zone due to highly localized stress and elevated temperatures. Elevated temperatures, as experienced in high-speed machining, can make the presence of cutting fluids detrimental to the cutting tool due to thermal cracking. Therefore, there are fundamental process drivers for HSM without the use of cutting fluids (i.e. dry cutting). Moreover, it is assumed that, machining in the absence of cutting fluids can be environmentally friendly.

1.3 Role of Cutting fluids in Machining

The process of machining involves a shearing mechanism to transform a workpiece to an end shape. This fundamental mechanism creates a high friction load between the cutting tool and the workpiece, which significantly increases the cutting temperature. In addition, friction dissipates energy thus generating heat, which if it is not properly controlled might have a detrimental effect on the cutting tool and machined component. The main sources of heat are normally generated in two areas known as the primary or shear zone (1) and tool-workpiece interface (2). Moreover, a third zone (3) of heat is generated where friction between the tool and the chip occurs (Figure 1.1).

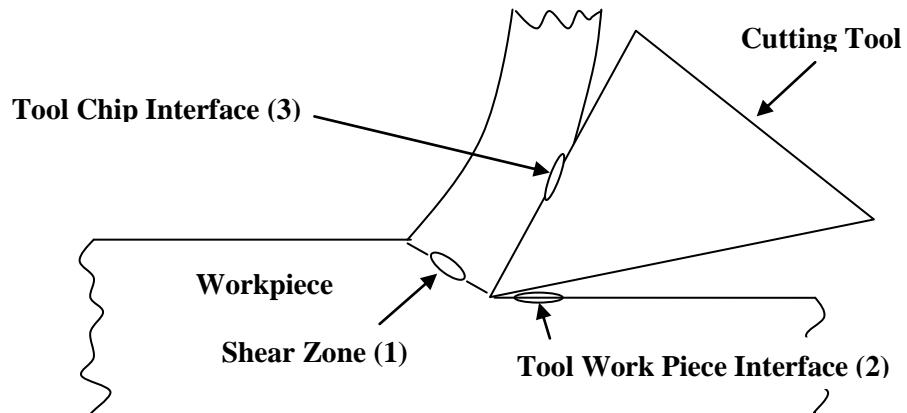


Figure 1.1: The Sources of heat in machining

Control of the heat generated can be achieved by reducing cutting temperature and friction. Cutting temperature has a positive and negative impact. A higher cutting temperature would be necessary, to some extent, to make the deformation process occur easily; as in hot machining processes. On the other hand, it generates excessive heat, which will affect tool life and in some cases the surface integrity of components. Heat induced by temperature rise is cutting speed dependent i.e. increasing the cutting speed increases the cutting temperature.

In addition to its cooling action, the lubricating effect of cutting fluid makes a significant contribution in friction reduction. In machining, where the heat generated is foremost at the interfacial face of the tool and chip, the presence of cutting fluid particles will enhance the lubricating action over the face. In addition, the use of cutting fluid in cutting operations is also beneficial in order to;

1. Flush the chips off the machined surface to prevent the chips from scratching the machined surface.
2. Reduce friction so that the cutting force is lower and chatter can be minimised.
3. Create a thick film to provide a layer of protection for the machined surface to prevent chemical reaction that could promote corrosion. The lubricant film also prevents the chips from being welded to the machined surface.
4. Avoid a welding effect on the rake face, which could promote tool failure.

However, most cutting fluid applications are performed by flooding. There are several mechanisms identified in which cutting fluid works [1]. These mechanisms are briefly described below;

1. *Capillary action*: the asperity of two vicinity solid bodies moving relatively would tend to create tiny channels that are recognised as minute capillaries. This capillary action helps the spread of fluids between two vicinity surfaces.
2. *Diffusion mechanism*: this refers to penetration through the metal lattice caused by different fluid concentrations. With this mechanism, the fluid particles infiltrate to form bonds with the workpiece's metallurgical structure.
3. *Volatilisation*: vaporisation of fluid that changes the viscosity as well as the state, increasing the penetration capability.
4. *Rehbinder effect*: interaction of surface-active species (usually chlorine) with the workpiece material tends to reduce the shear strength in the primary deformation zone.

Additionally, all these mechanisms can work well if cutting fluid possesses good cooling and lubricating capabilities. Cooling capability is a predominant necessity at high cutting speeds; yet cutting fluid fails to provide proper lubricating actions at high cutting speeds. However, because of the elevated temperatures during the high speed cutting operation, the right combination between the type of cutting fluid used and the cutting tool material has to be taken into consideration to prevent tool failure.

However, effective cutting fluid application cannot be achieved if it fails to meet some prerequisites such as easy access to the source of heat, high thermal capability for heat dissipation, high flash point, stable against oxidation, corrosion protection, free from unpleasant odour and must not cause skin irritation. So, by fulfilling these conditions the main advantages of cutting fluid application can be achieved.

1.4 Types of cutting fluid and their performance

A perfect cooling action can be gained if cutting fluids have high thermal conductivity and high specific heat. Water has these special characteristics besides being economically inexpensive. However, water is a poor lubricant and in addition promotes corrosion on ferrous material more easily. Therefore, the use of pure water as a coolant tends to be avoided but is still used to some extent by adding an emulsion that can overcome the poor lubricating action and corrosive tendency of pure water. This type of coolant is known as water soluble oil. Alternatively, to gain good lubrication on interfacial contacts in machining operations mineral lubricating oil can be used. However, in comparison to water, mineral oil has poor thermal conductivity and specific heat therefore; additives are normally incorporated to enhance the

performance of mineral oil. Accordingly, extreme pressure agents, known as Extreme Pressure, are sometimes employed in severe cutting operations. Based on the brief description above, cutting fluids can be classified into three main groups as shown in Figure 1.2.

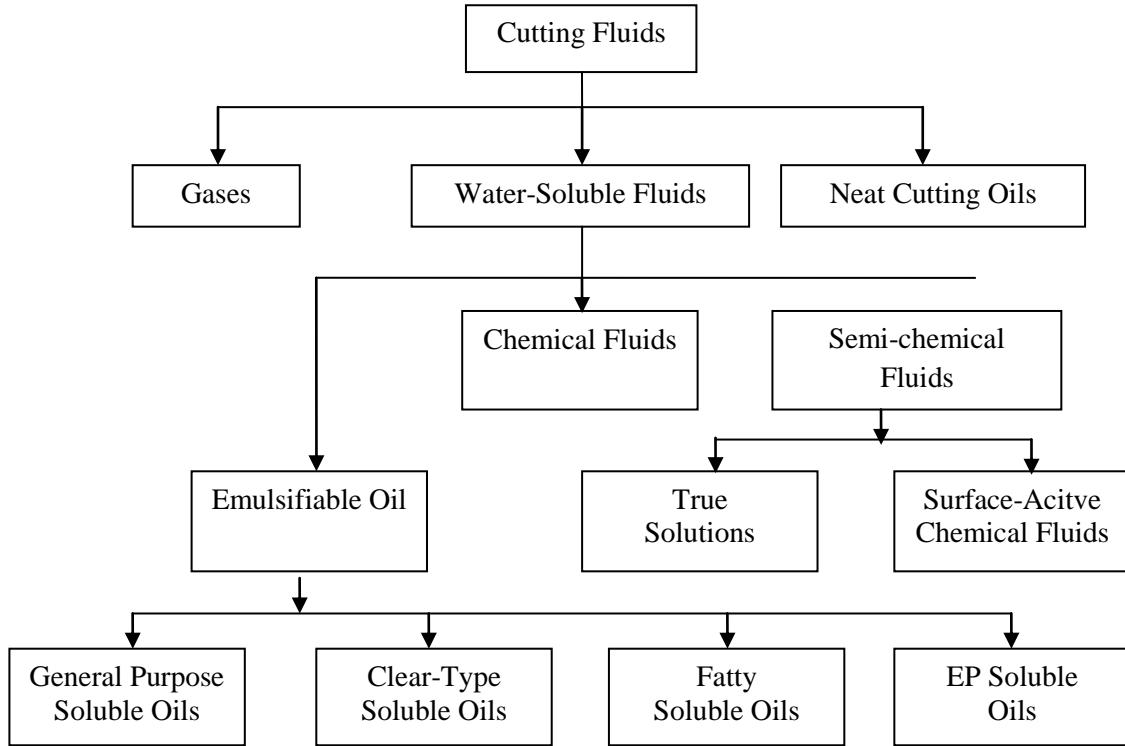


Figure 1.2: Classification of cutting fluids (source El Bardie [2])

1.4.1 Water-soluble fluids

Water soluble fluids are a type of cutting fluid comprised of an emulsifier to blend oil and water. However, due to adverse characteristics they do not dissolve. None the less, created from two substances that possess good cooling and lubricating capabilities makes this type of cooling media preferable for metal removal processes running at high speed and low pressure.

1.4.2 Soluble oils

Soluble oils, as the emulsions are popularly known, are bi-phase composites of mineral oils added to water in proportions that vary from 1:10 to 1:100. It contains additives (emulsifiers) to allow the fusion of oil particles and water. These additives decrease the surface tension and form a stable monomolecular layer in the oil/water interface thus generating the formation of small particles of oil, which results in transparent emulsions.

1.4.3 Semi synthetic fluids

Semi synthetic fluids have 5% to 50% mineral oil plus additives and chemical composites, which dissolve in water forming individual molecules of micro-emulsions. The presence of a large amount of emulsifiers, compared to soluble oil, provides a more transparent appearance to the fluid. The low volume of mineral oil and the presence of biocides increase the life of the fluid and reduce health risks, compared to emulsions.

1.4.4 Solutions

Solutions are mono-phase composites of oils that are completely dissolved in water, so they do not require emulsifiers, because the composites react chemically forming a mono-phase.

1.4.5 Synthetic fluids

This type of cutting fluid does not have mineral oil in its composition. It is based on chemical substances, which form a solution with water. They are made of organic and inorganic salts, lubricant additives, biocides, etc. They have a longer life than other fluids due to bacteria resistance, thus reducing the number of replacements required. They enable the formation of transparent solutions which offer good views of the machining process and have additives which can improve wet ability and, therefore, high cooling ability.

1.4.6 Neat cutting fluids

Vegetable and animal oils were the first lubricants used as pure oil in metal cutting. However, their use became impractical due to high costs, unstable oxidation and quick deterioration; but they are still used as additives in mineral fluids, aimed at increasing the lubrication properties. Neat oils are basically mixed either with pure mineral oils or with additives, generally of the extreme pressure type. The use of this type of cutting fluid has been reduced due to inherent problems of high purchase costs, disposal processing costs, fire risk, inefficiency at high cutting speeds, poor cooling ability, smoke formation and high risk to the human health when compared to water based cutting fluids.

1.5 Environmental Friendly machining Processes

For decades, machining processes have relied on the use of cutting fluids to extract the heat generated during the process. The heat generated at the primary and secondary cutting zones has a negative effect on the cutting tools, promoting wear. Increasing wear degrades the

cutting tools performance, consequently affecting the quality of machined parts/components, which is essential in machining processes. Therefore, the use of cutting fluids is an important part of a machining process system. Without cutting fluid, tools have only a short life, which makes the machining process costly. Cutting fluid usage becomes crucial in cutting operations and this sometimes leads to uncontrolled use of cutting fluid which in turn increases direct fluid usage costs along with other costs such as, recycling, maintenance and fluid disposal.

For example, in Germany, in 1998, there was an increasing trend for cutting fluid utilisation from 7 - 17%. A worldwide producer of cutting fluid, conducted a survey of typical end-use of coolant. They discovered that utilisation of coolant in machining processes could reach 16% of total manufacturing cost see Figure 1.3. This magnitude was higher than the cost of cutting tools.

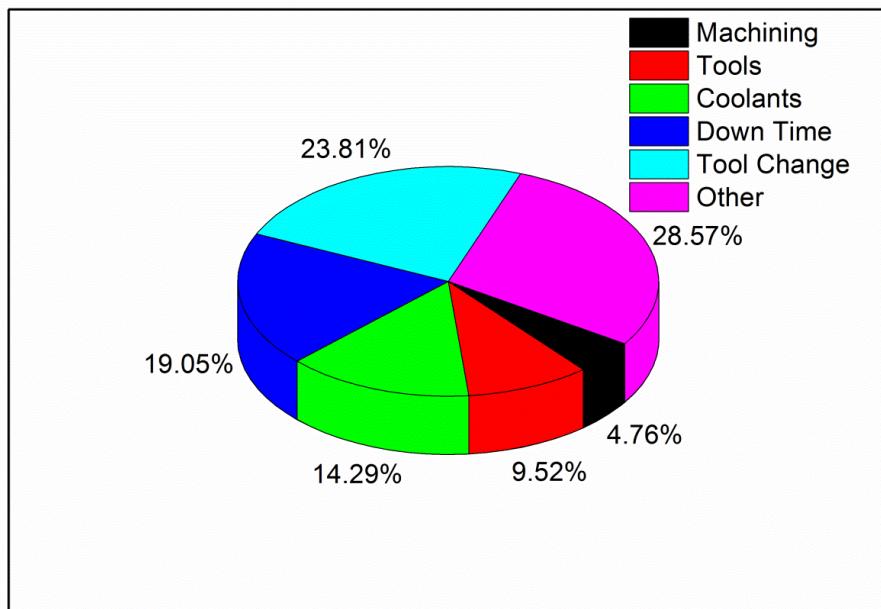


Figure 1.3: Typical end-user machining cost [3]

Increasing cutting fluid consumption increases associated costs and it creates problems in the environment. Improper use of cutting fluid will subject individuals, working on the shop floor, to health problems and daily exposure to air borne contaminants gradually increases the health risk for workers, causing severe health problems such as respiratory disorder, skin disease and increasing the risk of cancer. In addition, besides causing detrimental health problems, improper treatment of cutting fluid residue could also increase the environmental impact in categories such as acidification, ecotoxicity etc.

Increasing cutting fluid consumption and related costs and the detrimental impact on the environment are the drivers for machining scientists and practitioners around the globe to address sustainable development strategies. In order to do this a proposal was initiated to eliminate the use of cutting fluid use on the shop floor. This method is recognised as dry machining. Studies have shown that dry machining can be employed for certain processes; while investigations are still underway in other areas to break down the obstacles to full implementation of dry machining, a bridging method known as near dry machining has been introduced.

1.6 Cooling Methods

The use of cutting fluids to reduce elevated temperatures during machining operations is believed beneficial for improving machining performance. However, the increasing consumption of cutting fluids nowadays has become of great concern to practitioners and scientists in machining industries. This trend triggers economic and environmental issues. The high volume of cutting fluid used on the shop floor will increase the cost of its acquisition, in addition to the treatment costs for its disposal. Nevertheless, quite apart from the economic issues the utilisation of cutting fluid damages the environment and causes detrimental health problems.

Furthermore, the negative effects of using cutting fluid, as previously described, have forced the use of alternative, new technology that is more sustainable and economically feasible; particularly in industrialised countries with strict environmental regulations. This technology is known as the dry machining method. In dry machining no cutting fluids are used. Due to constraints in applying this technology across all the processes, full implementation is restricted to certain processes; however, development of dry machining technology (MQL) is currently in progress. Near dry machining is another method which has been extensively used and studied. Though not as radical as dry machining, near dry machining uses cutting fluid, but in amounts much lower than that of conventional applications. This technology is the most feasible technology that could be implemented on the shop floor. Despite its feasibility for improving machining performance while using a small volume of cutting fluid, it also benefits from the simplicity of its delivery systems. The MQL strategy can be further enhanced using nanofluids as cutting fluids.

1.7 The concept of MQL

In minimum quantity lubrication, a small volume of cutting fluid is transported to the cutting zone assisted by air and converted via orifices into small particles (atomisation). These small particles are delivered to the cutting zone in the form of air borne particles, a gaseous suspension of liquid particles. The working principle of MQL can be explained by using simple model depicted in Figure 1.4.

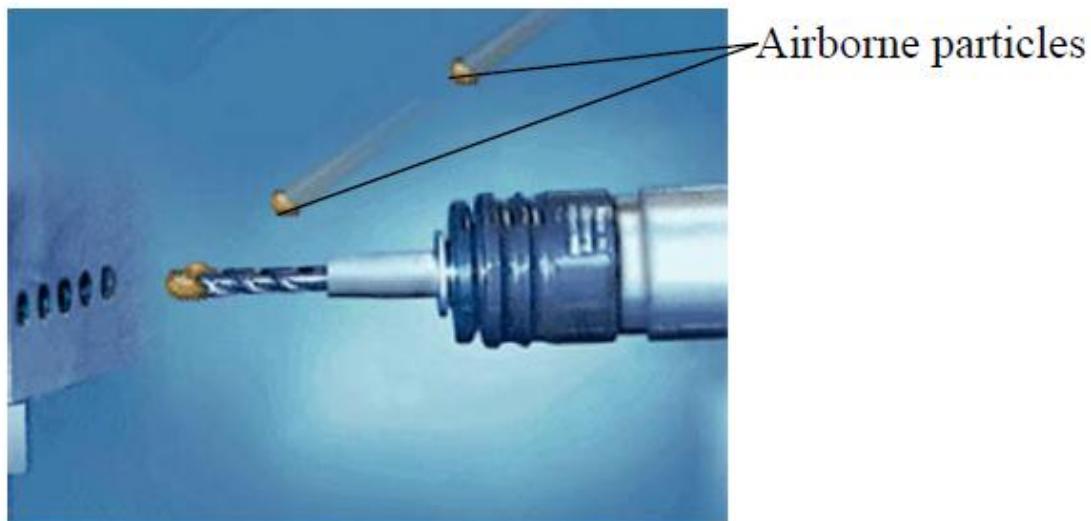


Figure 1.4: Idealized concept of MQL (source Astakhov [4])

Oil in the reservoir is kept under constant pressure higher than in the mixing chamber. This pressure difference will make the oil flow up to the end of the nozzle where an orifice is located. The orifice has the main function of accelerating the particles' movement. As the particles' speed increases, the particles' size will be reduced. Smaller oil particles discharged from nozzle tip will subsequently be accelerated by the compressed air hence creating air borne particles. The lubricant in the form of air borne particles will have increased penetrability, especially to intrinsic (i.e. difficult to reach) areas. In this way, minute capillary created by two different surface asperities will help the small size particles of lubricant to gain access to the margins of the sliding and sticking areas. Additionally, the lubricant in the form of air borne particles will increase cooling capacity due to vaporisation of lubricant particles.

1.8 Development and Concept of Nanofluids

Since Nobel prize winner Richard P. Feynman presented the concept of micromachines in his seminal talk, “There’s Plenty of Room at the Bottom—An Invitation to Enter a New Field of Physics,” in December 1959 at the annual meeting of the American Physical Society at the California Institute of Technology, miniaturization has been a major trend in modern science and technology. In 1962, the Japanese thermodynamicist Kubo showed that the electronic level of a fine metallic grain could change according to the difference in the particle size. Gerd Binnig and Heinrich Rohrer of the IBM laboratory in Switzerland developed a new scanning tunneling microscopy (STM) technology in early 1980, developing images of the atomic structure on the surface of objects, which enabled further development for the foundation of nanomaterials. Nobel Prize winner, H. Rohrer, presented the chances and challenges of the “nano-age”. Nano is a prefix meaning one-billionth, so a nanometer is one-billionth of a meter. Nanotechnology is the creation of functional materials, devices, and systems by controlling matter at the nanoscale level, and the exploitation of their novel properties and phenomena that emerge at that scale. The steady miniaturization trend has dropped from the millimeter scale of the early 1950s to the present-day atomic scale. Nanofluid technology will thus be an emerging and exciting technology of the twenty-first century. With the continued miniaturization of technologies in many fields, nanofluids with a capability of cooling high heat fluxes exceeding 1000 W/cm^2 would be paramount in the advancement of all high technology.

Nanofluids have attracted attention as a new generation of heat transfer fluids with superior potential for enhancing the heat transfer performance of conventional fluids. These fluids are obtained by a stable colloidal suspension of low volume fraction of ultrafine solid particles in nanometric dimension dispersed in conventional heat transfer fluid such as water, ethylene glycol or propylene glycol in order to enhance or improve its rheological, mechanical, optical, and thermal properties. The concepts of nanofluid was first coined by S.U.S Choi at Argonne National Laboratory and have attracted considerable interest because of reports of great enhancement of heat transfer, mass transfer, and wetting and spreading. It is well known that at room temperature, metals in solid form have orders-of magnitude higher thermal conductivities than those of fluids. For example, the thermal conductivity of copper at room temperature is about 700 times greater than that of water and about 3000times greater than that of engine oil. The thermal conductivity of metallic liquids is much greater than that of nonmetallic liquids. Therefore, the thermal conductivities of fluids that contain suspended

solid metallic particles could be expected to be significantly higher than those of conventional heat transfer fluids.

1.9 Importance of Project (Nanofluids) to Industry

In the last two decades, Minimum Quantity Lubrication (MQL), also known as Near Dry Lubrication or Micro Lubrication has been introduced to improve machining problems arising from the usage of cutting fluids. It offers reasonable steps to reduce the consumption of cutting fluid in metal cutting processes. Positive effects were obtained due to the present of lubricating oil in the cutting zone, which is able to penetrate deep into the tool-chip and tool-workpiece interfaces, thus reducing friction between them during turning. Despite its feasibility for improving machining performance while using a small volume of cutting fluid, it also benefits from the simplicity of its delivery systems. The growing industrial and manufacturing activities have urged the researchers and the manufacturers to look for new and state-of-the-art heat transfer fluids with superior cooling capacity.

Due to the emergence of nanotechnology, fluids possessing high thermal conductivity called nanofluids have evolved. The applicability of the fluids as coolants is mainly due to their enhanced thermo-physical properties resulting from nanoparticle inclusion. On the whole, the research studies have focused on suspending either metal or metal oxide based nanostructured materials into the base fluid using surfactants for effective dispersion of nanoparticles. For further enhancing the thermal properties of the nanofluids, there is a need for tailored material incorporation. Tailored material is a blend of two or more dissimilar solid nanoparticles, which are effectively dispersed in the fluid medium. In MQL systems, the nanoparticle incorporated in fluids cannot be retrieved or reused, which in turn increase the overall cost of lubrication. Hence, there is a need to develop hybrid nanoparticle that are inexpensive and possess high thermal conductivity.

1.10 The main objectives of the present work are as follows:

1. Selection of Nanoparticles
2. Synthesis of Hybrid Nanoparticles by Mechanical alloying
3. Characterization of Hybrid Nanoparticles
4. Preparation of Nanofluids
5. Analyzing thermo physical properties of Nanofluids
6. To study the effect of various parameters affecting the Thermal conductivity.

7. The effect of Hybrid Nanofluids as cutting fluids for Machining difficult to cut metals like Inconel-718

1.11 Work Plan:

The work plan for the thesis is as follows.

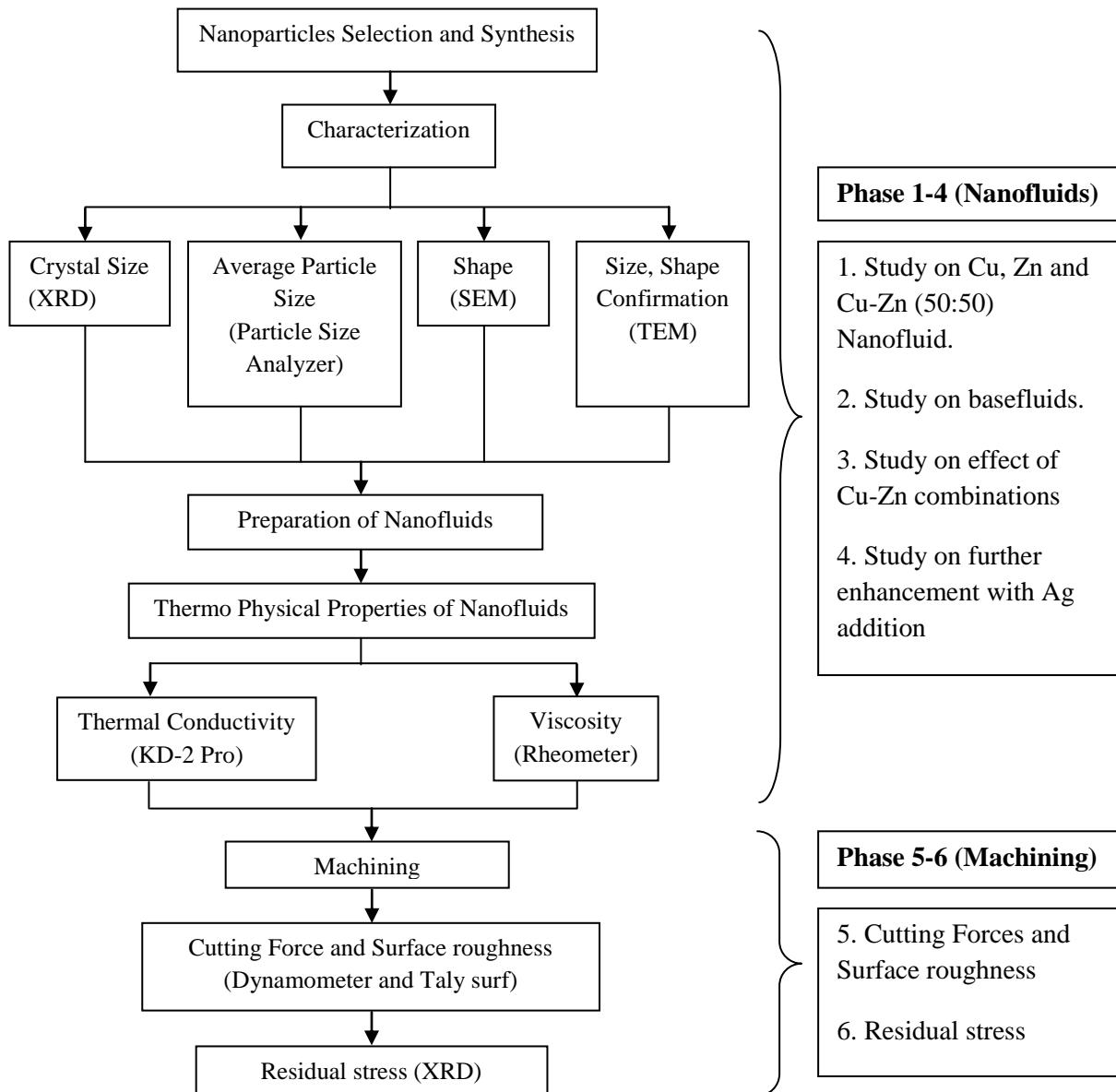


Figure 1.5: Flow chart of the work plan

1.12 Thesis Outline

This thesis contains six chapters.

Chapter 1: Introduction

A brief description of the project background, importance of hybrid nanoparticles to industries is documented here. Also summarises the fundamental knowledge gap that needs to be filled to improve cutting fluids with MQL performance. Based on this, the aim and objectives of this work were developed.

Chapter 2: Literature Review

A literature review particularly focused on enhancement of thermal conductivity of nanofluids with inexpensive materials and machining of difficult to cut materials using MQL are presented in this chapter. Furthermore, the environmental burden in respect of the use of MQL and vegetable oil as base fluid is reviewed in the context of manufacturing in sustainability.

Chapter 3: Nanofluids (Synthesis, characterization, preparation and thermophysical properties)

A description of the equipment used for all research activities, material and the type of equipments used for Synthesis, characterization, preparation and finding the thermophysical properties of the in-situ prepared hybrid nanofluids.

This section is divided in to four phases,

1. Selection and Study on Cu, Zn and Cu-Zn (50:50) Nanofluid.
2. Study and selection on basefluids.
3. Analyze on effect of Cu-Zn combinations.
4. Analyze on further enhancement with Ag addition.

Chapter 4: Machining

This chapter presents the equipment used for all research activities, workpiece material selection and the type of cutting tool used. Machining is carried out with the hybrid nanofluids developed, mentioned in chapter 3. In addition, Design of Experiments is also included in this chapter. Additionally, the methodology used to measure cutting forces, surface roughness and residual stresses under dry, MQL/Vegetable oil and MQL/Nanofluid lubricating conditions is documented.

Chapter 5: Conclusions and future scope

Chapter 6: References

CHAPTER 2

LITERATURE REVIEW

2.1 Machining and Sustainability

According to the United Nation, sustainable development can be defined as an activity to meet the needs of the present without compromising the ability of future generations to meet their own needs [5]. More specifically, the United States Department of Commerce defines sustainability for the manufacturing sector as the creation of manufactured products that use processes that are non-polluting, conserve energy and natural resources and are economically sound and safe for employees, communities and consumers[6].

Machining processes, as an important element in manufacturing, has shown rapid growth, especially in emerging countries like China. This was indicated by the growth in the machine tool market sales in China as observed by the Freedonia group, a market research institution. They predicted that China's machine tool industries had to be well prepared to manage a 14.2 % increase in demand annually by 2014 [7]. Additionally, European Union producers of machine tools who are members of the CECIMO group also experienced an increase in demand for machine tools in 2011 following the economic downturn of 2009 [8]. They supplied approximately 33% of the global machine tool market share, in particular highly innovative, diversified and high-precision machine tools. Increasing demand for machine tools around the globe indicates that the demand for components produced by using machine tools is undergoing rapid increases.

This positive trend would indeed lead to economic benefits. However, it would also trigger an increase in consumption, for instance, the consumption of cutting fluids increased as well as demand for energy. Cutting fluids and energy supplies are mainly met from natural resources. Although cutting fluid, made from chemical substances has been widely used, dependence on mineral-based cutting fluids is still prominent. Meanwhile, the availability of natural resource has declined persistently. Besides limited availability of natural resources, the impact caused by the use of cutting fluids and increasing consumption of energy would create negative effects on the environment. During machining processes, the cutting zones are traditionally enveloped with huge volumes of cutting fluid to avoid temperature elevation that

can distress cutting tools and shorten service life. After fulfilling its function, cutting fluid is normally circulated back to a storage tank. However, the percentage that flows back to the fluid storage tank has reduced due to vaporisation, contaminating chips, the deposits covering machine tool components and the machined surface. Vaporised cutting fluid creates a chemical suspension in the air; if it is inhaled by people working in enclosed machine areas it will cause health problems. In addition, the chips contaminated by the cutting fluid require purification and re-processing before they can be recycled as new raw material. Meanwhile, after use, cutting fluid has to be disposed of; which requires the correct treatment to be undertaken before it can be discarded to prevent harm being caused to the environment and living organisms. Provision of extractors, so that the machine tool ambient environment is safe, and purification processes and treatment of cutting fluid disposal need extra budgets, which affect production costs. The energy to power up machine tools is supplied by power stations that are primarily generated by natural resources such as fossil fuel, coal, etc. The increase in demand for products manufactured by the machining process not only increases energy consumption and in turn the cost of energy; but also has implications on global warming due to the carbon released by power generators. Thus, a stringent policy to avoid any detrimental effects on the environment is needed. In addition, the International Standard Organization (ISO) has originated an environmental management standard by releasing ISO 14000 series. Therefore, to produce highly competitive machined products, the machining industries had to address all the problems of machining processes and to seek appropriate strategy to satisfy environmental standards [9].

By implementing the sustainable practices in the metal cutting sector, environmental performance and economics can be improved considerably. Sustainability concepts can be incorporated in manufacturing sector by implementing the following practices [10 - 12];

1. Reducing amount of input resources like energy, material and water.
2. Improving environmental quality of resources, reducing the use of toxic and non-biodegradable chemicals.
3. Efficient designing of life cycles.
4. Adopting eco-friendly manufacturing technologies.
5. Improving ergonomic, health and safety requirements, equity and fairness, and employee personal development.

To make it environmentally friendly, machining operates without the use of cutting fluid. However, there are many problems associated with full implementation of this technology, and therefore, efforts for technological improvement is ongoing. Thus, a back-up technology, such as near dry machining, becomes a suitable alternative.

2.1.1 Dry machining and semi-dry machining

The use of cutting fluids to reduce elevated temperatures during machining operations is believed beneficial for improving machining performance. However, the increasing consumption of cutting fluids nowadays has become of great concern to practitioners and scientists in machining industries. This trend triggers economic and environmental issues. The high volume of cutting fluid used on the shop floor will increase the cost of its acquisition, in addition to the treatment costs for its disposal. Nevertheless, quite apart from the economic issues the utilisation of cutting fluid damages the environment and causes detrimental health problems [13 - 16]. Furthermore, the negative effects of using cutting fluid, as previously described, have forced the use of alternative, new technology that is more sustainable and economically feasible; particularly in industrialised countries with strict environmental regulations. This technology is known as the dry machining method. In dry machining no cutting fluids are used. Due to constraints in applying this technology across all the processes, full implementation is restricted to certain processes; however, development of dry machining technology is currently in progress. Near dry machining is another method which has been extensively used and studied. Though not as radical as dry machining, near dry machining uses cutting fluid, but in amounts much lower than that of conventional applications.

2.1.1.1 Dry machining

The term dry machining is associated with machining processes without the presence of cutting fluids. In this technology, the elevated temperature is reduced by lessening the friction at interfacial faces (i.e. tool-workpiece interface and tool-chip interface). The friction reduction in this technology can be achieved by the use of a coating layer onto tool substrate material. The coating layer must have a low friction coefficient. This relies on the choice of coating materials as well as coating technology. In their study involving dry machining, Cassin and Boothroyd [17] used this method to compare the lubricating action of several types of cutting fluid that were mixed with carbon tetrachloride as an lubricant improving agent. They found that CCL4 (i.e. carbontetrachloride) treatment on carefully ground off

workpiece material can result in the same cutting force magnitude as in dry machining. This finding indicates that to some extent the application of dry machining is feasible, especially in terms of lowering cutting forces at a low cutting speed of 0.178 m/min. It is apparent that at low cutting speeds, the superior coating layer provides a lower friction coefficient, however, dependent upon the combination of tool type and workpiece material, as the cutting speed increases the heat generated will render the effect of the coating layer inferior. Accordingly, a study was undertaken to find an appropriate coating material and new technology, which will improve the coating material's characteristics. The study will look at coating materials and on how to coat the substrate material properly to improve performance. As such, coating methods such as Physical Vapour Deposition (PVD) and Chemical Vapour Deposition (CVD) have made the prospect of dry machining on the shop floor becoming more feasible. Recently the search for suitable material for tool coating has shown a markable uphill trend and many studies are towards the discovery of an appropriate coating material that can be used to improve the performance of the dry machining process [18 - 22]. Thus, the range of cutting speeds that can be applied to the dry machining application can be increased.

Mativenga and Hon [23] identified that a better surface finish on end milling of H13 tool steel can be obtained by appropriate selection of tool coatings as well as coating technology. They suggested that effective tool coatings would enable wear zone protection thus increasing the feasibility of dry machining implementation at higher cutting speeds. Furthermore, Dudzenski [24] highlighted the importance of suitable combinations of tool material and its coating material asserting that the right combination can result in better dry machining performance at different cutting speeds when cutting Inconel 718 alloy.

Diniz and Micaroni [25] found that the careful selection of cutting parameters could improve machining performance in terms of surface finish, tool life and even exhibit superior performance compared to the use of cutting fluid. This principle was confirmed following the turning of two AISI 1045 steel cylinders that were hardened to 55 HRd and 59 HRd respectively. However, as indicated by Galanis et al [26] in a comparison study between dry machining and wet machining of AISI 422 stainless steel workpiece material, there is an optimum condition where the machining performance using dry cutting was acceptable.

On the other hand, heat generation caused by shearing of cutting tools and workpiece material cannot be neglected in analysis of machining performance using a dry cutting operation. It is well known that cutting temperature is cutting speed dependent. As the cutting

speed increases, the cutting temperature will increase comparatively hence increasing the heat generated at the contact zone. The heat generated will dissipate through the solid body involved in removal of material, including workpiece material, which will encounter microstructure changes. Consequently, it can lead to weak workpiece material structural integrity, thus it would be possible to increase burr size[27]. Furthermore, it would also affect the strength of machined part/ components due to residual stress as caused by elevated temperatures [28].

From the above-mentioned studies, it is conceivable that successful application of dry machining for a broad range of machining conditions, including workpiece material, can be achieved if the superior friction characteristics of cutting tools can be perfected so that the function of cutting fluid can be completely altered. In addition, it has to be able to surpass part quality and machining time of that which can be achieved when employing cutting fluid [29,30].

Recently, the implementation of a near dry machining that has proved popular is minimum quantity lubrication (MQL). This is supported by commercially available MQL delivering system equipment. The term minimum quantity lubrication (MQL) has become widely used in academia and research. Some researchers use micro litre lubrication [31] and others use near dry machining [32-34]. MQL is a misnomer because dry machining is feasible and the lower limit for minimum quantity lubrication is dry machining. Ideally, it should be called low quantity lubrication. There is need for international consensus on which terminology to use. Hence, in line with popular terminology, MQL is used in this thesis.

2.1.1.2 Semi-dry machining

Dry machining is a technology believed to be most sustainable. However, constraints in its application due to technological limitations of tooling have prevented full implementation, especially when high cutting speeds are required. While improvements to related aspects of dry machining are being developed, a bridging method is necessary to fill the gap between wet machining and dry machining. One view about sustainable machining processes has urged further development of a technology widely known as the near dry machining. The terminology of the near dry machining method is associated with the use of the minimal quantity of liquid as a cutting fluid and/ or any type of liquid or gas used to replace existing cutting fluids. Some technologies have been explored as the prospective

candidates for near dry machining methods. Among these are cryogenic machining, high-pressure jet machining (HPJ) and minimum quantity lubrication (MQL). These need study to evaluate the requirement of full mitigation of the use of cutting fluid. In cryogenic machining, an attempt is made to reduce the temperature as low as possible in the cutting zones by employing liquid gas such as nitrogen, helium, hydrogen, neon, air and oxygen as a coolant. Recently four methodologies were applied in relation to cryogenic machining. These are pre-cooling the workpiece [35], indirect cryogenic cooling, cryogenic spraying with jets and direct cryogenic treatment of cutting tools[36].

Studies to find the best cryogenic machining application have been conducted using different materials ranging from low hardness [37 - 39] to hard-to-difficult materials. Cryogenic machining is fast becoming one of the more feasible methods for cutting difficult-to-cut aerospace alloys and other hard-to-cut materials due to its capability for maintaining temperatures at the cutting interface below the critical temperature of cutting tool materials [40, 41]. In addition, besides having the best cooling capability, using liquid nitrogen in cryogenic machining processes provides a significant reduction in tool-chip contact length thus reducing heat generation on the rake face [42]. Thus, it improves machining of hard-to-cut material at higher cutting speeds. Although the cooling action in cryogenic machining can offer a significant reduction in temperature at the cutting zone and shorten the contact length there are still some difficulties anticipated in the lubricating action. In order to prove that cryogenic machining using liquid nitrogen can provide lubrication at the cutting interface, Hongand Ding [43] did a sliding test. They found that a combination of various temperatures dependent on a micro scale of hydrostatic effect might have a role to play in achieving a lubricating action. However, it fails to provide a lubricating action as with traditional cutting fluids because liquid nitrogen can easily vaporise due to low viscosity and chemically stable [44].

Furthermore, the lack of lubrication in cryogenic machining cannot stop it being viewed as a sustainable method due to the potential benefits that can be gained by its application. Among those benefits are being environmentally friendly, having a higher material removal rate, longer tool life, higher throughput and improvement to machine part quality owing to a cleaner environment. However, the high investment and operating costs of cryogenic machining makes it impractical for low production volumes [45].

Other sustainable methods that have been attracting interest among machining scientists is high-pressure jet machining (HPJ). This method is similar to the traditional overhead method but uses a different delivery method. In the traditional way, the cutting fluids are transported to the contact zone helped by a little low air pressure, whereas in high pressure jet machining, the cutting fluids are delivered at higher air pressure. Higher air pressure increases the particles speed hence reducing the particles size. Higher particles speeds and minute particle size will enhance the penetration ability of cutting fluids, thus it is easier for the particles to enter the operational area and establish the lubricating action. In addition, it also increases the cooling capacity of the cutting fluids due to moving heat predominantly by evaporative mode, which is more efficient than the convective mode [46]. The main achievement of high-pressure jet coolant in machining is its ability to improve cutting tool performance. A 250% improvement in tool life was achieved by using cutting fluids such as water-soluble oil [47]. This tremendous achievement was further investigated by Wertheim et al [48] when studying the effect of high-pressure flushing through the rake face of tools, though at the time they only increased the air pressure by up to 25 atm.

Moreover, Rahman et al [49] observed that by identifying effective zone high-pressure coolant, a better surface finish could also be obtained. It should be borne in mind that HPJ machining cannot fully be recognised as a sustainable method due to the use of large amounts of cutting fluids as a cooling/lubricating media. Kovacevic et al [50] suggested applying a narrow jet in order to reduce the amount of cutting fluids usage in high-pressure jet machining. Unfortunately, implementation of this method is still constrained by high investment costs.

Another technology that could be categorised as a bridging method for sustainable development is popularly known as minimum quantity lubrication (MQL). This technology is the most feasible technology that could be implemented on the shop floor. For example, the Ford Motor Company has, since May 2005, fully implemented the MQL system on the shop floor [51]. Despite its feasibility for improving machining performance while using a small volume of cutting fluid, it also benefits from the simplicity of its delivery systems. The table 2.1 below lists the advantages and disadvantages of different strategies employed during machining of Nickel based alloys.

Table 2.1: Advantages and disadvantages of different environmental friendly cooling strategies employed in the machining of Nickel alloys under continuous cutting processes.

Dry	Minimum Quantity Lubrication (MQL)	High Pressurized Cooling (HPC)	Cryogenic Cooling
No lubricant/ coolant required, no need for coolant disposal	Very small amount (ml/min) of lubricant/ coolant is involved	Very high quantity of lubricant/ coolant is required	Generally lubricants/ coolants are compressed air or liquid nitrogen
Poor temperature control in the cutting zone	For machining difficult to cut materials not effective due to low cooling capacity [54]	Superior temperature control in cutting zone	Superior temperature control in cutting zone [53]
Poor chip removal and dust formation	Better chip removal as compared to dry	Superior chip removal	Better chip removal as compared to dry
Poor tool life	Extended tool life at higher cutting speeds as compared to dry but highly dependent on arrangement of nozzle	Improved tool life [52]	Improved tool life [53]
Poor surface finish	Improved surface finish but highly dependent on arrangement of nozzle	Improved surface finish but highly dependent on arrangement of coolant delivery system and pressure involved	Inconsistency in surface integrity as it is highly dependent on the pairing of tool and workpiece materials [54]

2.2 Application of MQL in machining processes

The application of the MQL method can be found in a variety of machining processes, especially in primary processes such as turning, drilling, milling, and grinding. In addition, cutting of wide varieties of workpiece material, such as aluminium, steel, hardened material as well as hard-to-cut material can also be done using the MQL method.

Table 2.2: Report of various articles about application of MQL in machining processes

Author	Application	Parameter investigated	Observation
Sadeghi et al [55]	Grinding	Forces and surface quality	Tangential cutting forces were lower using the MQL application compared to flood cooling and better surface finish can also be obtained with this method
Tawakoli et al [56]	Grinding (100 Cr6 hardened steel)	Forces and surface quality	Identified that a combination of the MQL method and resin bond corundum gave the best grinding performance in comparison to wet and dry machining
[58]	Drilling	Cutting temperature	Reduce cutting temperature by approximately 50% of compared to external coolant supply
Heinemann et al [59]	Drilling	Tool life and lubricants	Increase the cooling capacity thus helping tool wear reduction Low viscosity cutting fluid can further improve penetration ability hence enabling cooling and lubricating action simultaneously

[60]	Drilling high thermal conductivity material (aluminium AA1050)	Different lubricating conditions	Appropriate combination of machining variables, the performance can be equal to that of flood cooling for drilling aluminium
Dhar et al [61]	Turning (AISI 1040 steel)	Cutting temperature, chips and dimensional accuracy	Identified favourable tool chip interaction using the MQL application
Varadarajan et al [46]	Turning	Different lubricating conditions	MQL performance better than wet and dry machining on hardened AISI 4340 steel

2.2.1 MQL and machining parameters

MQL equipment is simple, that is designed to be easily attached to any kind of machine tool. On the other hand, there is MQL equipment that has to be properly installed on the machine tool. Therefore, the selection of the equipments is dependent on the needs and investment cost constraints.

However, the performance of some of the MQL equipment has been inadequate because the equipment has not been fully adapted for the specific requirement of the shop floor, although the manufacturer has provided all the merits that can be gained by its acquisition. As stated by Astakhov [62], these were intended for promotional effect. Therefore, tests are still necessary to seek the best performance for all specific needs and with this in mind, tests to find the optimum set of MQL parameters (i.e. flow rate and nozzle positions) and the optimum cutting condition for given workpiece material and selected cutting tool yet needs to be undertaken.

2.3 Machining with Vegetable Based Cutting Fluids

Although attempts in manufacturing research are focused to diminish the use of cutting fluids, the present state-of-the-art technologies do not seem to assure that cutting

fluids will be entirely eliminated in the next future [63]. Some machining operations and workpiece materials still required the use of cutting fluid. New trend concerning the cutting fluids in machining is to replace hazardous components with environmentally friendly compounds. These new compounds not only must show the same properties to cutting fluids but also must improve the machining performance such as productivity. Literature studies about machining using vegetable based cutting fluids are limited. Higher cost of vegetable based oils relative to mineral based oils is the principal limitation of them but this drawback will be diminished in the future as the petroleum prices increase [64].

2.4 Turning with vegetable based cutting fluids

Khan and Dhar [65] investigated the effect of MQL by vegetable oil on cutting temperature, tool wear, surface roughness and dimensional deviation in turning of AISI 1060 steel. MQL reduced the cutting temperature, tool wear and surface roughness as compared to dry machining. The reduction of about 5–12 % at average cutting temperature using MQL by vegetable oil as compared to using dry machining was observed depending upon the levels of the process parameters (cutting speed, feed rate). Cutting forces decreased by about 5–15 % using MQL by vegetable oil. Ozcelik et al. [66] reported the experimental studies of sunflower and canola oils based cutting fluids including different percentage (8 and 12 %) of extreme pressure additive and two commercial cutting fluids (semisynthetic and mineral based) in turning of AISI 304L stainless steel with respect to surface roughness, cutting force, feed force and tool wear. Experiments were also conducted at dry cutting conditions which caused rapid tool wear and fracture. Tool life below 200, 1,000 and 2,000 s was recorded under dry cutting, semi-synthetic and mineral based cutting fluids, respectively. The higher tool life in vegetable based cutting fluids was due to the fatty acid content. Canola based cutting fluids showed better performance than sunflower based cutting fluids because of different length of carbon chains. Canola oil has three carbons more in formulae and longer carbon chain outstand high cutting temperature, thus improving the surface protection. Moreover, the high viscosity of canola oil had a tendency to resist flow. This high viscosity provides more effective lubricating at the tool-chip interface, thus reduces the friction between the tool and workpiece and removes heat developed at the interface easily. High percentage of extreme pressure additive in vegetable based cutting fluids showed the higher surface roughness values. 8 % of extreme pressure additive included canola based cutting fluid performed better than the rest [60]. Higher rate of EP in sunflower and canola based

cutting fluids reduced cutting and feed forces during turning of AISI304L austenitic stainless steel, however the increment of EP rate affected surface roughness values negatively. As a result, mineral and semi-synthetic cutting fluids can be replaced by vegetable based cutting fluids in turning [67]. Ojolo et al. [68] used vegetable based cutting fluids (groundnut oil, coconut oil, palm kernel oil and shear butter oil) during turning of mild steel, aluminium and copper and measured cutting force. Although, it was found that the effects of vegetable based cutting fluids were material dependent, groundnut oil showed the best performance among the four vegetable based cutting fluids investigated. Xavior and Adithan[69] used coconut oil during turning of AISI 304 stainless steel and measured tool wear and surface roughness. The performance of coconut oil was compared with an emulsion and neat cutting oil. They found that coconut oil reduced the tool wear and improved the surface finish. In another study they measured temperature and cutting force [70]. Coconut oil outperformed the other two cutting fluids (soluble oil and straight cutting oil) in terms of reducing the cutting force and temperature. Paul and Pal [71] investigated the performance of different types of cutting fluids (karanja oil, neem oil, conventional fluid) as compared to dry cutting condition during turning of mild steel. The use of vegetable based cutting fluid improved surface quality as compared to dry turning and conventional cutting fluid. They explained the lower temperature of neem vegetable oil than that of karanja vegetable oil with the lower viscosity of neem oil with respect to karanja oil.

2.5 Turning of Inconel 718:

Nickel based alloys are extensively used in aerospace industries, marine equipments, nuclear reactors etc. as they exhibit higher strength, resistance to corrosion, fatigue, creep and erosion [72]. In nickel based alloys Inconel 718 is the most frequently used metal due to its mechanical properties and thermal properties. The complexity of machining of difficult to cut material resolves itself into two primary problems: short tool life and poor surface finish of machined workpiece [73,74].

Due to low thermal conductivity of nickel based alloys more heat is generated and plastic deformation takes place resulting in poor surface finish. Also the microstructure of the alloy is altered due to heat generated. Alteration in microstructure and due to plastic deformation, induce residual stresses [75]. Residual stresses affect the mechanical behaviour of the material, specially the fatigue life of the workpiece [76,77]. The dimensional instability also occurs due to induced residual stresses, which in turn can lead to difficulties during

assembly [78, 79]. The major parameters to achieve adequate tool lives and surface integrity of the machined surface which include machining method, cutting feed, feed rate, depth of cut, coating material, lubrication.[75,77]

2.6 Mechanism of minimum quantity lubrication

There are four mechanisms for flood cooling application, which include capillary action, diffusion, volatilisation and the Rehbinder effect. Among these, with regard to the nature of MQL application, the capillary action and volatilisation are more representative of MQL application. Astakhov's [80] view was that the Rehbinder effect is the most plausible method. However, Astakhov's postulation was not supported by any evidence. As suggested by Williams [81], "*molecular transport within a network of interfacial capillaries could be a significant mechanism that could improve the effectiveness of cutting fluid and as long as they could react with metal surface*". This statement refers to how to create an effective overflow cutting fluid application to enhance machining performance. Therefore, this brings up the question of whether this similar condition could also be applied to the MQL application. MQL, as generally known, uses a very small quantity cutting fluid in the form of air borne particles delivered directly to the contact zones. Thus, it guarantees cutting fluid molecules are smaller than conventional cutting fluid application. However, the reality of this statement would need to be clarified by the presence of actual evidence to prove its applicability to MQL. Childs [82] reviewed friction contact between tool and chip when modelling friction in metal machining; especially continuous chip formation. Childs reviewed what had been postulated by William [81] and presented a metal machining model for several conditions of cooling methods using different cutting speeds. This model was proposed to help in selecting appropriate conditions when cutting fluid can be applied effectively. Unfortunately, this model was based on the conditions mostly used in metal machining which use low cutting speeds and flood cooling applications. The model has limitation for the use of advanced technology or advanced methods in the new era of metal machining, such as high-speed cutting and minimum quantity lubrication. Therefore, this model needs to be justified by employing high cutting speeds and minimum quantity lubrication; because the nature of continuous cutting, as used in Child's study, was different to the nature of interrupted cutting. It is, therefore, still questionable in milling processes using the MQL application. Likewise, the study of Childs used a very low cutting speed threshold of 10 m/min, which is too low for industrial application. Figure 2.15 Idealised channels by which lubricant penetrates the lightly

loaded part of a chip-tool contact and the consequent limitation of fluid lubrication to low cutting speed (after Childs [82]) Using aluminium as the workpiece material, Itoigawa et al [83] investigated the effect and mechanism of MQL in intermittent turning. They used two types of cutting fluid as MQL media, mineral oil and a combination of synthetic oil and water. The results illustrated that synthetic oil is preferable as an MQL media particularly when it is mixed with water. This combination could eliminate tool damage and material pick-up on the tool surface. This is because water affirmatively possesses good cooling effects that can maintain the film strength rather than producing a film chemical absorption reaction. In addition, by adding water to synthetic ester the cooling-lubricant mechanism could be improved significantly and therefore provide the best performance during application of MQL. This might be due to the cooling and lubrication action working simultaneously. Furthermore, the presence of water could increase the oxygen in the cutting environment and thereby the cutting performance would be enhanced as concluded by Min et al [84] when investigating the absorption behaviour of lubricants using near dry machining.

However, the mechanism proposed in this study correlated with the ability of workpiece material to form a lubricant film that would help MQL to improve its performance. The workpiece material used in this study was aluminium, which is known to have high thermal conductivity. This type of material when heat conduction was stimulated would have good absorption capacity. Therefore, it can be argued that this mechanism cannot be applied to low thermal conductivity workpiece material such as titanium alloy, tool steel etc.

Liao and Lin [85], using minimum quantity lubrication (delivered at a flow rate of 10ml/h) found that the material diffusion across the tool-chip interface was responsible for weakening the cutting tool. A hypothesis was proposed that if the protective layer could be formed in tool-chip interface then this would retard tool wear. Using MQL provided extra oxygen to the tool-chip interface and promoted the formation of a protective layer. However, it was identified that the stability of the oxide layer was cutting speed dependent and, hence, presented a relatively poor performance at a higher cutting speed of 500 m/min, compared to a lower cutting speed of 300 m/min.

Unfortunately, Liao and Lin did not try to examine the effect of different MQL flow rates and air pressure on the formation of the oxide layer. From Liao's and Lin's work, air pressure combined with oxygen might guarantee the availability of oxygen; but that effect was not explored in the study and therefore, it was not clear whether the formation of a

protective layer was caused either by the influence of cutting fluid or air pressure. In addition, they used less viscous cutting fluid, which has less lubricant capacity; so that it would be difficult to maintain the protective layer under severely elevated temperatures, as cutting fluid tends to vaporized easily. Moreover, it might be possible that the protective layer coalesced with a corrosive reaction occurring at the hot surface (i.e. the tool faces) following exposure to the fluid. In general, the cutting tool in milling processes is still exposed to the fluid at the end of the cutting length; therefore, it can form a corrosive layer at the tool face.

Obikawa and Kamata [86] conducted an experiment to investigate the performance of the super lattice coating of TiN/AlN in cutting Inconel 718 using MQL and at high cutting speed regime. They also identified that the presence of oxygen could be a major controlling factor in turning. Synthetic ester was pulverized at a rate of 16.8 ml/h through an internal delivery system. However, they did not mention in their paper how oxygen could exist at the cutting zones, particularly when the cutting speed increases .On the other hand, they reported that increasing the air pressure from 0.40 to 0.60 MPa could increase the oxygen in the cutting environment. The rise in air pressure used in this study was too small, so it cannot be assumed that it could significantly increase the oxygen.

Therefore, it is still unclear why the MQL works, providing an even better cutting performance when compared to fluid cooling. This remains a challenge in research because understanding the MQL mechanism will lead to significant improvement and help in selecting appropriate types of cutting fluid for given workpiece materials.

2.7 Improving MQL Lubrication

MQL showed better results than dry lubrication, when used for machining purposes. These results were due to the micro liter lubrication provided with the aid of MQL system to the cutting zone. When lubricant is supplied to the cutting zone, it reduces the friction between the chip and the workpiece. The heat produced during the machining can be reduced by using lubricants which posses' high thermal conductivity.

2.7.1 Thermophysical properties of Nanofluids

Due to emerging of nanotechnology, fluids possessing high thermal conductivity called nanofluids have evolved. Nanofluids are a stable colloidal suspension of ultrafine solid particles (10-100 nm) in base fluids. With addition of ultrafine particles (higher thermal conductivity of particles compared to basefluid) in the base fluid the properties of the fluid

can be enhanced [87]. The applicability of the fluids as coolants is mainly due to their enhanced thermo-physical properties resulting from nanoparticle inclusion [88]. On the whole, the research studies have decisive on suspending either metal or metal oxide based nanostructured materials into the base fluid [89, 90] using surfactants for effective dispersion of nanoparticles.

Ceramic nanomaterials exhibit dreadfully good stability and chemical inertness, but possess low thermal conductivity as compared to metallic nanoparticle, when incorporated in basefluids to increase the thermal conductivity. Metallic nanoparticles like copper possess high thermal conductivity with less stability and reactivity. Therefore, to overcome this problem a hybrid nanofluids is prepared by incorporating a small amount of copper particles were added to alumina matrix, which considerably improved the thermal properties without affecting the stability of the nanofluid [91]. Table 2.2 reports the various articles on nanofluids used as metal working fluids.

Table 2.3: Report of various articles about nanofluids as metalworking fluid Thermophysical properties of Nanofluids

Type of Nanoparticles	Base fluid	workpiece	Investigated parameters
Al ₂ O ₃ [92]	Vegetable oil	Inconel 600 alloy	- Cutting temperature - Surface roughness, - Tool wear
Graphite [93]	Water soluble oil	AISI 1040	- Average chip–tool interface Temperature, - Tool wear
CuO [94]		AISI 1040	- Workpiece temperature
CNT [95]		AISI 1040	- Cutting temperature - Surface roughness, - Tool wear
Nano graphite [96]	Water soluble oil	AISI 1040	- Cutting temperature - Surface roughness, - Tool wear
MWCNT [97]	Coconut oil	Martensitic Stainless Steel	- Cutting temperature - Surface roughness
MWCNT [98]		AISI 1040	- Cutting temperature - Finite, element analyses (FEAs)
Al ₂ O ₃	Water	Nimonic 90	- Cutting forces - Surface roughness - Tool wear

Silver [99]	Water	Nimonic 90	- Cutting forces - Surface roughness - Tool wear
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As we have discussed earlier, the performance of MQL systems can be enhanced by using nanofluids. The important properties of nanofluids to serve as cutting fluids are mainly thermal conductivity of nanofluids and the viscosity. The conductivity enhancement depends on the particle volume fraction, particle size, temperature and interfacial properties on the particle-fluid interface. Below listed are the observed thermal conductivity enhancement of particles in percentages, depending on the type of nanoparticle addition and the respective basefluids.

2.7.1.1 Non- metallic Nanoparticle dispersion:

Earlier work on nanofluids was done with non-metallic nanoparticle dispersion. The commonly used non-metallic nanoparticles are Al_2O_3 , CuO , TiO_2 , MgO , ZnO , SiC etc. Although these nanoparticles possess less thermal conductivity than metallic nanoparticle it has the advantage over it in case of density. They are easy to suspend and the stability of the nanofluids is also better. Below mentioned (table 2.3), is the summary of nanofluid thermalconductivity enhancements overbase fluids with Ceramics Nanoparticles dispersion.

Table 2.4: Summary of nanofluid thermalconductivity enhancements over base fluids with Ceramics Nanoparticles incorporation

Investigator	Nanofluid	Size (nm)	Concentration (vol%)	Observation
Masuda H <i>et al.</i> [100]		13	4.3%	30
Lee S <i>et al.</i> [101]		33	4.3	15
Xie.Het <i>et al.</i> [102]		68	5	21
Lee <i>et al.</i> ,[101]		38.4	4.3	9
Lee <i>et al.</i> , [101]		30±5	0.3	1.44
Masuda H <i>et al.</i> ,[100]		13	4.3	30
N.Putra <i>et al.</i> ,[103]		38.4	4	9.4% -21°C/24.3% - 51°C
		131	4	24%-51°C

C.H.Chonet <i>et al.</i> ,[104]	Al ₂ O ₃ + water	11	1	24.8%-70 ⁰ C
		47	1	10.2%-70 ⁰ C
		150	1	4.8%-60 ⁰ C
		47	4	28.8%-70 ⁰ C
X. Wang <i>et al.</i> ,[105]		28	5.5	16
C.H.Lieet <i>et al.</i> ,[106]		36	6	28.2
		47	6	26.1
X.Zhang <i>et al.</i> [107]		20	5	15
E.V.Timofeeva <i>et</i> <i>al.</i> ,[108]		11	5	8
		20	5	7
		40	5	10
Lee <i>et al.</i> [101]	Al ₂ O ₃ + EG	38.4	4.3	18
Wei Yu <i>et al.</i> [109]			5	28.2
Zhou L P <i>et al.</i> [110]		50	0.4	16
Das <i>et al.</i> ,[111]		28.6	4	13
Masuda H <i>et al.</i> [100]		24	3.4	11
Wang <i>et al.</i> ,[105]		23	10	30
Masuda H <i>et al.</i> [100]	CuO + EG	24	4	20
Wang <i>et al.</i> ,[112]		23	15	50
Murshed SMS <i>et</i> <i>al.</i> [113]	TiO ₂ + water	15	5	30
Duanfthongsuket <i>et al.</i> [114]		21	2	7
Murshed <i>et al.</i> [113]		10	5	32.8
Wei Yu <i>et al.</i> [115]	TiO ₂ + EG		5	27.2
Wei Yu <i>et al.</i> [115]	MgO+ EG		5	40.6
	ZnO+ EG		5	26.8
	SiO ₂ +EG		5	25.3
Xie.Het <i>et al.</i> [116]	SiC + water	26	4.2	16
Xie.Het <i>et al.</i> [116]	SiC + EG	600	4	23

2.7.1.2 Metallic Nanoparticle dispersion

Metallic nanoparticles have found their place in enhancing the thermal conductivity of fluids, as it possess a higher order of thermal conductivity than basefluids. The commonly used metallic nanoparticles are Cu, Al, Fe, Ag etc. Below mentioned table 2.4, is the summary of nanofluid thermal conductivity enhancements over basefluids with metallic Nanoparticles dispersion.

Table 2.5: Summary of nanofluid thermal conductivity enhancements over base fluids with Metal Nanoparticle dispersions

Investigator	Nanofluid	Size (nm)	Concentration (vol%)	Observation Thermal conductivity enhancement in %
Choi SUS <i>et al.</i> [117]	Cu+EG	10	0.3	40
Assae <i>et al.</i> ,[118]		120	0.48	3
Eastman <i>et al.</i> [119]		<10	0.3	40
Xuan Y <i>et al.</i> ,[120]	Cu+water	100	7.5	78
Hong T K <i>et al.</i> [121]	Fe + EG	10	0.55	18
Patel H E <i>et al.</i> [122]	Au + water	10-20	0.026	21
Putnam <i>et al.</i> [123]	Au + Ethanol	4	0.6	1.3±0.8
	Au + Tolonene	1.65	-----	-----
Patel H E <i>et al.</i> [122]	Ag + water	60-80	0.001	17
Patel H E <i>et al.</i> [122]	Ag + Citrate	10-20	0.001	3
Murshed <i>et al.</i> [124]	Al + water	80	5	>40

2.7.1.3 Carbon Nanotubes Dispersion.

Carbon nanotubes are unique tubular structures of nanometer size diameters and length. It consists of hundreds of concentric shells of carbons and the network of the shells is closely related to honeycomb arrangement. These particles possess highest thermal conductivity and have not as much of density. Below mentioned (table 2.5), is the summary of nanofluid thermal conductivity enhancements over basefluids with carbon nanotubes dispersion.

Table 2.6: Summary of nanofluid thermal conductivity enhancements over basefluids with Carbon Nanotubes incorporation

Investigator	Nanofluid	Size (nm)	Concentration (vol%)	Observation Thermal conductivity enhancement in %
Choi SUS <i>et al.</i> , [117]	MWCNT+ water	D=100 nm,L= 70 μ m	0.6	38
Xie.Het <i>et al.</i> ,2003		D=15 nm,L= 30 μ m	1	7
Assael MJ <i>et al.</i> [125]		D=15-130 nm,L= 50 μ m	0.6	38
Ding <i>et al.</i> ,2006		D=20-30 nm,L= -----	1	27
Xie.Het <i>et al.</i> [116]	MWCNT+E G	D=15 nm,L= 30 μ m	1	13
Liu <i>et al.</i> [126]		D=20-30 nm,L= ---	1	12.4
Xie.Het <i>et al.</i> [116]	MWCNT+ decene	D=15 nm,L= 30 μ m	1	20
Assael MJ <i>et al.</i> [125]	MWCNT+ oil	D=25 nm,L= 50 μ m	1	150
Liu <i>et al.</i> [126]		D=20-30 nm,L= -----	2	30
Biercuket <i>et al.</i> [127]	CWCNT+ Epoxy	D=3-30	1	175
Wei Yu <i>et al.</i> [115]	GNS		5	61

2.7.1.4 Literature on hybrid nanofluids:

Hybrid nanofluids are a rank of fluids containing a blend of two or more dissimilar solid nanoparticles, which are effectively dispersed in the fluid medium. Many researchers

have studied Heat transfer and rheological characteristics of Hybrid nanofluid containing hybrid nanoparticles like Al_2O_3 –Cu [6], Silica-Multi-wall carbon Nanotubes (MWCNT) [12], Cu – TiO_2 [13], MWCNT– Fe_3O_4 [14], silica nanosphere/MWCNT [15], Ag /mSiO₂ [16] and compared them with single particle based nanofluids such as Al_2O_3 , TiO_2 and Fe_3O_4 . Below mentioned table 2.6, is the summary of nanofluid thermal conductivity enhancements over basefluids with hybrid Nanoparticles dispersion depending on the size.

They reported that heat transfer coefficient increased with increase in volume concentration of hybrid nanoparticles compared to single particle based nanofluids. At high concentrations, hybrid nanostructures have better influence on the viscosity of the base fluid. The density of hybrid nanoparticle is less when compared to single particle; this lets the nano particle to be suspended in nanofluid for longer time. In addition, the nanofluid behaved as Newtonian fluid in the tested temperature range for various concentrations of nanoparticles [16].

Table 2.7: Summary of nanofluid thermal conductivity enhancements over basefluids with alloy Nanoparticle incorporation

Investigator	Nanofluid	Size (nm)	Concentration (vol%)	Observation Thermal conductivity enhancement in %
Chopkaret al.[128]	Al_2Cu	30-40	2	100
	Ag_2Al	30	2	100
S. Suresh[129]	Al_2O_3 –Cu	17	0.1-2	12
D. Madhesh [130]	Cu – TiO_2	55	0.1-2	52
L. SyamSundar, [131]	MWCNT– Fe_3O_4 /water		0.1-0.3	31
MohammadaliBaghbanzadeh[132]	MWCNT–silica	10	1	16.7

2.7.2 Models for Thermal Conductivity of Nanofluids:

A wide experimental investigation on increasing thermal conductivity of nanofluids had been carried out in the last ten years, including oxides, metallic, SCNT, MCNT and different basefluids. The measurements show inconsistency due to difference in production of nanofluids, nanofluids characterisation and thermal conductivity measurement techniques. However a firm relationship is established from the experimental results between nanofluid thermal conductivity and following parameters: Nanoparticle volume fraction, size, shape, surfactants and additives. Below listed in table 2.7 are some of the models for effective thermal conductivity of solid liquid suspension.

Table 2.8: Models for effective thermal conductivity of solid-liquid suspensions.

Models	Expression
Maxwell (1873)[133]	$\frac{k_{nf}}{k_f} = 1 + \frac{3(\alpha - 1)\phi}{(\alpha + 2) - (\alpha - 1)\phi}$
Hamilton & Crosser [134]	$\frac{k_{nf}}{k_f} = \frac{[k_p + (n-1)k_f - (n-1)\phi(k_f - k_p)]}{[k_p + (n-1)k_f + \phi(k_f - k_p)]}$
Bruggerman [135]	$k_{nf} = (3\phi - 1)k_p + [3(1 - \phi) - 1]k_f + \sqrt{\Delta}$ $\Delta = (3\phi - 1)^2 k_p^2 + [3(1 - \phi) - 1]^2 k_f^2 + 2[2 + 9\phi(1 - \phi)]k_p k_f$
Jeffrey [136]	$\frac{k_{nf}}{k_f} = 1 + 3\beta\phi + (3\beta^2 + \frac{3\beta^2}{4} + \frac{9\beta^2(\alpha + 2)}{16(2\alpha + 3)} + \dots)\phi^2$ Where $\beta = \frac{\alpha - 1}{\alpha + 2}$; $\alpha = K_p/K_f$
Davis [137]	$\frac{k_{nf}}{k_f} = 1 + \frac{3(\alpha - 1)}{(\alpha + 2) - (\alpha - 1)\phi} [\phi + f(\alpha)\phi^2]$ Where $f(\alpha) = 2.5$ for $\alpha = 10$, and $f(\alpha) = 0.5$ for $\alpha = \infty$
Lu & Lin	$\frac{k_{nf}}{k_f} = 1 + \alpha\phi + \beta\phi^2$; Where $\beta = \frac{\alpha - 1}{\alpha + 2}$; $\alpha = K_p/K_f$

[138]	
Landau – Lifshitz/Looyenga [139]	$\frac{k_{nf}}{k_f} = \left[(k_p^{1/3} - k_f^{1/3})\phi + k_f^{1/3} \right]^3$

For solving complex problems in different areas in recent years, A Artificial Neural Network technique has been applied with substantial reduction in computational time. ANN technique has been used for analysis of refrigeration systems, fracture prediction in steel, thermal conductivity estimations of various materials and fluids, for finding convective heat transfer coefficients, dairy products and foodstuffs [Jambunathan et al. (140), Haque e al. (141), Arcaklioglu (142), Ertunc (143), Parlak (144)].

2.8 Literature of nanoparticle based on viscosity

The viscosity of the nanofluids has major effect on the thermal conductivity of the nanofluids. It affects the pumping power, pressure drop and convective heat transfer. Therefore extensive work on the viscosity is quite essential for determining the behaviour of nanofluids [145-146]. The viscosity of fluid depends on many factors such as size of the particle, temperature, viscosity of basefluid, volume concentration of nanoparticle loading. He et al. [147] reported that with increase in size of particle the viscosity of the fluid increases and with the increase in temperature the intermolecular attraction in nanofluids decreases and resulting in decrease in viscosity [148]. Prasher et al. [149] reported on the viscosity change due to change in particle volume fraction. Chevalier et al. [150] noticed that the viscosity of SiO_2 –ethanol nanofluids increases with an increase in volume concentration.

2.8.1 Models for Viscosity of nanofluids

There are many existing formulas for predicting the viscosity of nanofluids. The first formula was developed in 1906 by Einstein [151] with an assumption that the fluid is viscous and with spherical particle. Brinkman [152] in 1952 extended the Einstein's equation for nanofluids with moderate particle concentration. This correlation had more acceptances considering the effect of addition of nanoparticles with less than 4%. In 1959 a model was proposed by Krieger and Dougherty [153] for hard spherical particles. A temperature dependent model was developed by Nguyen et al. [154]. The sets of equations (table 2.8)

available in literature are in efficient to account all the parameters affecting the viscosity of nanofluids.

Table 2.9: Models for viscosity of solid-liquid suspensions.

Models	Expression
Einstein [151]	$\frac{\mu_{nf}}{\mu_{bf}} = 1 + 2.5\phi$
Brinkmann [152]	$\frac{\mu_{nf}}{\mu_{bf}} = (1 - \phi)^{2.5}$
Nguyen [153]	$\frac{\mu_{nf}}{\mu_{bf}} = (2.1275 - 0.0215T + 0.00027T^2)$
Chen et.al. [155]	$\mu_{nf} = \mu_f (0.995 + 3.645\phi + 468.72\phi^2)$
Batchelor [156]	$\frac{\mu_{nf}}{\mu_f} = 1 + 2.5\phi + 6.2\phi^2$

2.9 METHOD OF PREPARATION OF NANOFLOIDS:

There are basically two methods for producing nanofluids;

- (i) The one-step direct evaporation method, which represents the direct development of the nanoparticles within the base fluids.
- (ii) The two-step method represents the formation of nanoparticles and subsequent dispersion of the nanoparticles in the base fluids.

In both the cases, the uniform dispersion of nano particles is essential for obtaining stable physical properties of nanofluids. A two step approach would be adopted here to prepare nanofluids. In the recent past, many techniques are developed to synthesize nano particles such as mechanical attrition, inert gas condensation, mechanical alloying, laser ablation etc [157]. High energy ball mills induce high impact energy on the charged powder by collision between balls and powder; this causes severe plastic deformation, repeated fracturing and cold welding of charged powder leading to formation of nanoparticles.

2.10 Mechanical Alloying

Mechanical alloying (MA) is a powder processing technique, which involves repeated welding, fracturing, and re-welding of powder particles in a high-energy ball mill [158-160]. MA is a different approach than usual techniques which uses chemical reactions and heat treatments to alloy components, as this process mainly depends on deformation processes to blend materials. The powder to be milled or alloyed is mixed together in ball milling machine. These powder particles get trapped between the balls and the vial, which causes their deformation and mixing.

2.11 Motivation

1. Owing to increase in the usage of nickel based alloys day by day due to their superior properties, it is essential to know the machining behaviour of these materials.
2. Carbon nanotubes possess the highest thermal conductivity enhancement Whereas when considering fluid containing Al_2Cu and Ag_2Al alloy nanoparticle possess thermal conductivity nearer to fluids containing carbon nanotubes
3. Considering cost-saving and the thermal conductivity enhancement hybrid nanoparticles gives the best outcome
4. From the literature it is evident that much less amount of work is done on alloys in nanoscale to enhance the thermal conductivity of fluids. Thus there is a scope for study on various alloys, with various combinations.
5. From the available literature, it was observed that, there had been a gap in the application of vegetable-oils based hybrid nanofluid on super alloy materials.

2.12 Research Gaps

Based on the literature survey the following gaps were identified.

1. Conventional cutting fluids cause environmental, occupational health hazard and economical problems: Need for biodegradable cutting fluids have evolved as a replacement for conventional cutting fluids
2. Advances in lubricating techniques, which bridge between flood lubrication and dry lubrication: MQL system is emerged out as a plausible solution to aforementioned problems as it reduces the drawbacks associated with flood cooling and dry machining and even in some cases has outperformed flood cooling

3. Due to emerging of nanotechnology, fluids possessing high thermal conductivity called nanofluids have evolved. Researcher have already studied the properties of nanofluids, with commercially available nanoparticles: There is a need to further enhance the prerequisite properties of nanofluids used as cutting fluids, by tailor made nanoparticles.
4. Owing to increase in the usage of nickel based alloys day by day due to their superior properties, it is essential to know the machining behaviour of these materials: It was observed that, there had been a gap in the application of vegetable-oils based hybrid nanofluid on super alloy materials

CHAPTER -3

HYBRID NANOFLOIDS:

SYNTHESIS, CHARACTERIZATION AND THERMOPHYSICAL PROPERTIES

Chapter synopsis

The increasing attention to the environmental and health impacts of industrial activities by governmental regulations and by the growing awareness level in the society is forcing industrialists to reduce the use of mineral oil-based metalworking fluids as cutting fluid. The trend towards renewable lubricant technology is now irreversible, driven by global warming, dwindling oil reserves, ethical corporate policies and government strategies to encourage biomaterials. Vegetable oils are biodegradable from renewable sources, free from mineral oil, chlorinated additives and have many performance advantages over mineral oil based analogues. With the emerging trends towards nanotechnology, the properties of the basefluid can be enhanced by addition of ultrafine particles. With the enhanced thermophysical properties of fluids, it has found wide applicability as coolants. In this chapter work is carried on development of new hybrid nano- particles with the combinations of Cu, Zn and Ag. Various combinations of Cu and Zn were studied and a thorough investigation of thermophysical properties have been carried out to state which combination of Cu and Zn yield the purpose. Combinations of basefluids, such as SAE oil and paraffin oil were also compared to vegetable oil to check the applicability.

This chapter mainly consists of the following sections.

3.1 Material and methods

In this section materials and methods used for synthesis, characterization, preparation and measurement of thermophysical properties of nanofluids is discussed.

3.2 Phase 1: Synthesis and Characterization of Single and Hybrid Cu and Zn Nano- fluid

This phase deals with the study of Cu, Zn and hybrid combination Cu-Zn (50:50) weight percentage. Comparison study will be carried out between these three types of particles, by checking the thermophysical properties like thermal conductivity and viscosity and the outcomes are reported.

3.3 Phase 2: Selection of Basefluids

This phase focuses on the behaviour of different basefluids. In this case three kinds of basefluids are taken up, such as vegetable oil, paraffin oil and SAE oil. The thermal conductivity and viscosity studies are carried out on these fluids by dispersing preset amount of nanoparticle concentrations. The outcomes of the study will be reported in terms of the exceptional performances of the basefluids.

3.4 Phase 3: Effect of Various Combinations of Hybrid Cu and Zn (50:50, 75:25, 25:75)

This phase highlights the study of thermophysical of various combinations of Cu and Zn. In this work three combinations are taken up, such as Cu-Zn (72:25), Cu-Zn (50:50) and Cu-Zn (25:75). These three combinations of Cu-Zn are synthesized, characterized and nanofluids are prepared. Nanofluids are prepared by dispersing preset amount of nanoparticle concentrations. The thermal conductivity and viscosity test are carried out for these combinations and the outcomes are reported.

3.5 Phase 4: Effect of Ag Particle Addition

In this phase further enhancement of thermal conductivity of nanofluids is carried out by adding Ag to the best outcomes of phase 3. The best combination of hybrid particles from phase 3 is added with Ag and milled to obtain nanoparticles and the percentage enhancement is observed, when compared to the outcomes of phase 3. The results are reported keeping the cost factor to the percentage enhancement in thermal conductivity with addition of Ag.

3.6 Phase 5: Modelling of Nanofluids

Modelling of thermal conductivity and viscosity of nanofluids is carried out using ANN. This developed model is compared to standard models and reported.

3.1 MATERIAL AND METHODS

In this section, material and equipments used for synthesis, characterization, preparation and testing of nanofluids are discussed. The following experimental aspects have been described in this section:

3.1.1 Synthesis of Nanoparticles:

Ball milling was carried out in Retsch planetary ball (PM-100) mill with tungsten vials and tungsten balls. The figure 3.1 shows the image of the ball mill available in NITW laboratory. It has a single vial with capacity of 250 ml. A counter weight is used on the other side to balance the weight of the vial. The micrometre size materials were purchased from Sisco Research Laboratories Pvt. Ltd. The specifications of ball mill is shown in table 3.1. Starting material used for preparing alloys were elemental Cu and Zn. Hybrid alloy powders of various percentage combinations by weight were prepared by mixing required combinations to obtain hybrid nanoparticle. Mechanical alloying is carried out in wet medium, using toluene as process control agent. Milled powder samples were collected for various intervals to test the change in shape and size.



Figure 3.1: Planetary Ball Mill PM-100

Table 3.1: Specification of Planetary Ball mill PM-100

Applications	pulverizing, mixing, homogenizing, colloidal milling, mechanical alloying
Field of application	agriculture, biology, Chemistry, construction materials, engineering / electronics, environment / recycling, geology / metallurgy, glass / ceramics, medicine / pharmaceuticals
Feed material	soft, hard, brittle, fibrous - dry or wet
Size reduction principle	impact, friction
Material feed size*	< 10 mm
No. of grinding stations	1
Speed ratio	1 : -2
Sun wheel speed	100 - 650 min ⁻¹
Effective sun wheel diameter	141 mm
G-force	33.3 g
Material of grinding tools	tungsten carbide
Grinding jar sizes	250 ml
Interval time	00:00:01 to 99:59:59
Pause time	00:00:01 to 99:59:59
Measurement of input energy possible	Yes
Interface	RS 232 / RS 485
Drive	3-phase asynchronous motor with frequency converter
Drive power	750 W
Electrical supply data	different voltages
W x H x D closed	630 x 468 x 415 mm
Net weight	~ 86 kg
Patent / Utility patent	Counter weight (DE 20307741), FFCS (DE 20310654), Safety Slider (DE 202008008473)

3.1.2 Particle characterization techniques

3.1.2.1 X-Ray Diffraction (XRD):

X-ray diffraction of the milled powders was performed using the X’Pert powder XRD (PANalytical, Netherlands). Milled powders were collected at various intervals during milling. These powders are dried and were tested for crystal size with increase in milling time and also the fundamental and superlattice peaks were observed with increase in milling time. The radiations used were CuK_α ($\lambda=1.542$ Å). The results were analyzed using X’Pert highscore plus software with data collection accuracy of 0.01 degree.

3.1.2.2 Particle Size Analyzer:

The average particle size of the particles produced by mechanical alloying is measured using Nano zeta sizer. Figure 3.2 shows the Malvern nano zeta sizer. It can measure particle size ranging from 0.3nm to 10μm. It works on the principle of dynamic light scattering and measures particle size in wet medium. The samples were prepared by adding a pinch of milled powder in to the beaker containing base fluid. This sample was ultrasonicated for 1 hour. The prepared sample was poured into sampling beakers and placed into the equipment to measure the average particle size of the milled powder. Table 3.2 shows the specification of Malvern Zeta Sizer.



Figure 3.2: Nano zeta sizer

Table 3.2: Specification of Malvern Zeta Sizer

Measurement Range	0.3nm - 10.0 microns (diameter)
Measurement Principle	Dynamic Light Scattering
Minimum sample volume	12 μ L
Accuracy	Better than +/-2% on NIST traceable latex standards
Precision/Repeatability	Better than +/-2% on NIST traceable latex standards
Sensitivity	0.1mg/mL (Lysozyme)

3.1.2.3 Scanning Electron Microscopy (SEM):

The SEM micrographs of the prepared powder by milling were obtained using the scanning electron microscope (JEOL). The images were obtained in secondary electron (SE) mode. The SEM setup used is available at NITW.

3.1.2.4 Transmission Electron Microscopy (TEM)

The TEM facility used, was availed from IIT Karagpur. The TEM sample was prepared by dispersing a pinch of prepared powder in acetone. The mixture is sonicated for about 15 minutes for uniform dispersion. Two drops of fluid containing dispersed particles was added in carbon coated Cu-grid and dried.

3.1.3 Equipments used for Preparation of Nanofluid

3.1.3.1 Weighing machine

The precision weighing machine is as shown in figure 3.3, is used to measure the quantity of powder loading in the nanofluids.



Figure 3.3: Precision weighing machine

3.1.3.2 Dispersion of Nanoparticles

A two-step approach is followed for dispersion of nanoparticles into the basefluids. Where the nanoparticles are first produced and then are dispersed in the basefluid with the aid of various physical techniques, such as stirrer, ultrasonic disruptor, ultrasonic bath and high-pressure homogenizer. This approach is used to disperse nanoparticles in vegetable oil to produce nanofluids with different concentrations of solid particle dispersion. The ultrasonicator available at NITW, is used for Sonication process is as shown in figure 3.4. It has a maximum sonicating power of 350 W and frequency of 20 kHz.



Figure 3.4: Ultra sonicator

3.1.4 Measurement of thermophysical properties of Nanofluids:

3.1.4.1 Measurement of thermal conductivity of Nanofluids

The thermal conductivity of the fluids was measured with the aid of KD2 Pro (figure 3.5). Decagon's KD2 Pro (Decagon Devices, USA) design attempts to optimize thermal properties measurements relative to these issues. Its sensors are relatively large and robust making them easy to use. The specifications of KD2 pro are as shown in table 3.3. To lower the time required for measurement, minimize the movement and free convection in liquid, heating times are kept as short as possible. The KD2 pro uses special algorithms to analyze measurements and high resolution temperature measurements techniques to resolve temperatures in the range of ± 0.001 $^{\circ}\text{C}$. The algorithms are capable of analyze measurements made during the heating and cooling intervals, can also separate the effect of the heat pulse

form ambient temperature changes. A KS-1 sensor was used to measure the thermal conductivity of the nanofluids. The accuracy of the sensor needle is $\pm 5\%$ and with measuring range of 0.2–2 W/m K. KD2 pro (KS-1) meets both ASTM D5334 [161] and IEEE 442–1981 [162] standards.



Figure 3.5: KD2 Pro

Table 3.3: Specifications of KD2 Pro

Sensors	6 cm (small) single needle (KS-1) Size: 1.3 mm diameter x 6 cm long Range: 0.02 to 2.00 W/ (m*K) (thermal conductivity)
Power	4 AA batteries
Battery Life	At least 500 readings in constant use or three years with no use (battery drain in sleep mode < 50 μ A)
Case Size	15.5 cm x 9.5 cm x 3.5 cm
Display	3 cm x 6 cm, 128 x 64 pixel graphics LCD
Keypad	6 key, sealed membrane

The special arrangement was designed for measuring thermal conductivity with KD2 pro for varying temperature is shown in figure 3.6 and the schematic diagram is shown in figure 3.7. The nanofluid is poured into the steel container and placed in the insulated box.

This box is closed from all sides except at the bottom from where the heat is supplied to nanofluid from the hot plate.



Figure 3.6: Experimental setup for measuring thermal conductivity at high temperatures

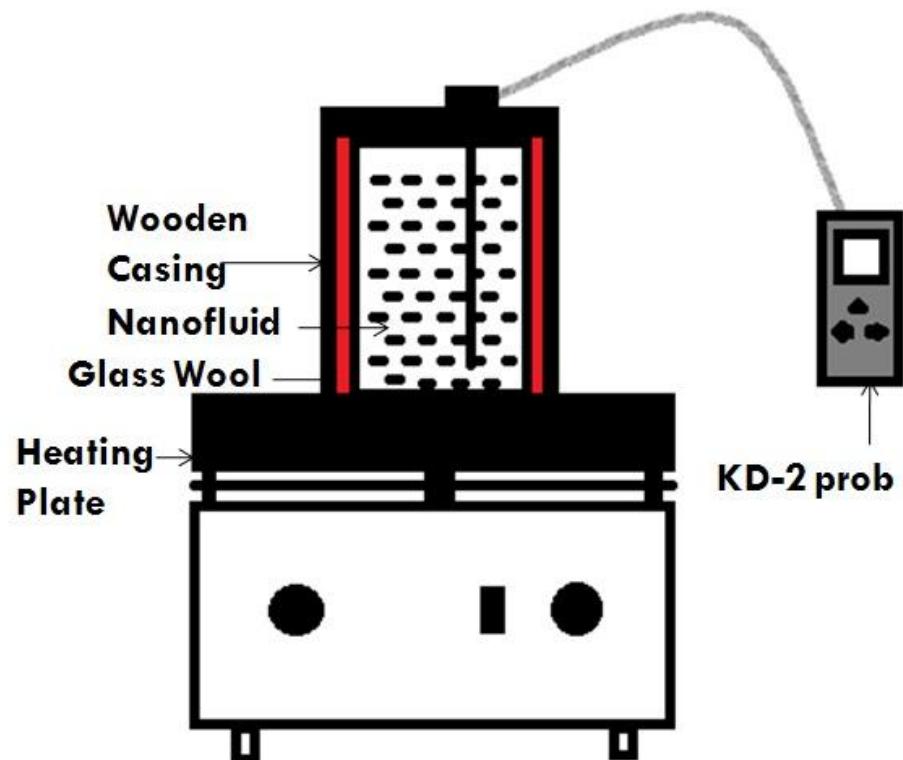


Figure 3.7: Schematic diagram of experimental setup for measuring thermal conductivity at high temperatures

3.1.4.2 Measurement of viscosity

Viscosity of the fluids was measured using a Rheometer (RheolabQC, Anton-Paar, Austria) with a sample holder. The Rheometer used for measuring viscosity is shown in figure 3.8 and the specifications of it are shown in table 3.4. The viscous drag of the fluid against the spindle rotation is used to calculate the viscosity of the fluid. A wide range of viscosity measurements can be done with various combinations of spindle types and speeds. For maintaining constant temperatures for all measurements, a thermostat bath was used. The fluids of which viscosity is to be measured were loaded into the filling cup. Viscosity was measured for a range of temperatures.



Figure 3.8: Rheometer

Table 3.4. Specifications of Rheometer

Speed	0.01 to 1200 1/min
Torque	0.20 to 75 mNm
Shear stress	0.5 to 3×10^4 Pa
Shear rate	10^{-2} to 4000 1/s
Viscosity range (depending on the measuring system)	1 to 10^9 mPas

Temperature range	-20 °C to 180 °C
Angular resolution	2 µrad
Dimensions (W x H x D)	300 x 720 x 350 mm
Weight	14 kg

3.2 PHASE – I: SYNTHESIS AND CHARACTERIZATION OF SINGLE AND HYBRID CU AND ZN NANOFUID

In this phase, Cu, Zn, Cu–Zn nanopowders have been synthesized by mechanical alloying method. Cu/vegetable oil, Zn/vegetable oil, and Cu–Zn/vegetable oil hybrid nanofuids were prepared by dispersing the prepared nanopowders in vegetable oil by the aid of ultrasonication. The present work aim out to explore the properties of atypical hybrid nanofuids. The thermal conductivity and viscosity of Cu, Zn and Cu–Zn vegetable oil hybrid nanofuids were measured at a range of volume concentrations.

3.2.1 Synthesis of Nanoparticles

Mechanical alloying of the micro sized metal powders was carried out in Retsch Planetary Ball Mill PM-100 with Tungsten carbide vials and balls to prepare ultrafine powders. Elemental copper and zinc powder particles were used for preparing ultrafine particles of copper, zinc and Cu-Zn alloy. Cu and Zn ultrafine powders, which in turn milled in a vial containing 80 gram of powder and 390 gram balls for 60 hours. To prepare Cu-Zn ultrafine powder, Cu and Zn powders were mixed in a ratio 1:1 by weight and milled for 60 hours. The ball to powder weight ratio was 5:1. Milling was conducted at a speed of 250 rpm in wet medium (about 50 ml of toluene) to prevent undue oxidation and agglomeration of powder. Tungsten carbide balls of 10 mm diameter were used for milling. The specification of the milling systems are given in Table 3.5. Powder samples were collected from the vials at selected intervals of time to check the reduction in size of the particles. Powders were characterized by X-ray diffraction (XRD) D8-Advance, Brooker, Germany with Cu-K α radiation and Particle size Analyzer, Nano ZS, Malvern.

Table 3.5: The specifications for the milling systems.

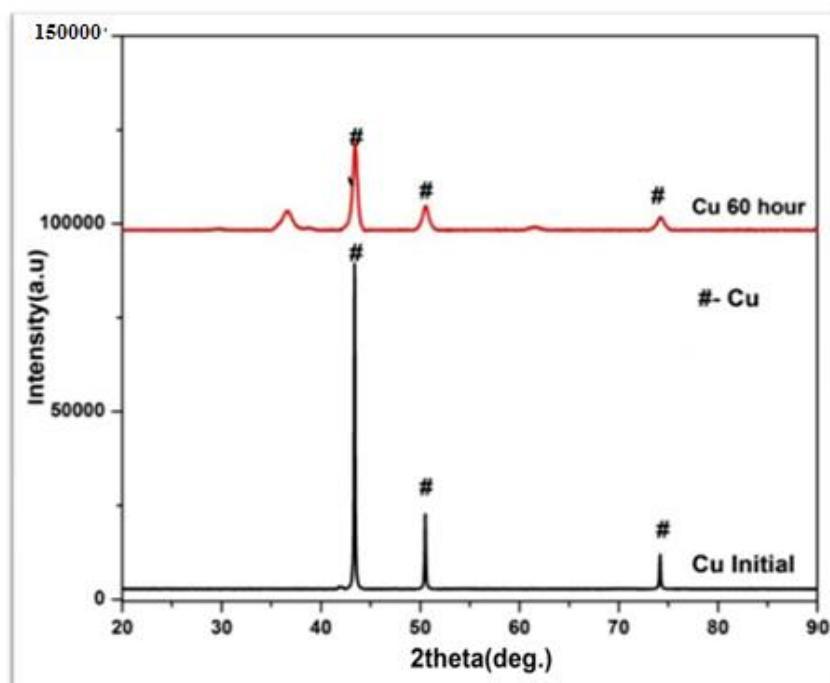
Mill type	Retsch planetary ball mill PM 100
Wet milling medium	Toluene
Milling speed	250 rpm
Grinding media:	
Ball and jar material	Tungsten carbide
Ball size	9.3 mm dia
Ball weight/jar	390g

Ball to powder ratio	5:1
Jar dimensions:	
Length	95 mm
Diameter	75 mm

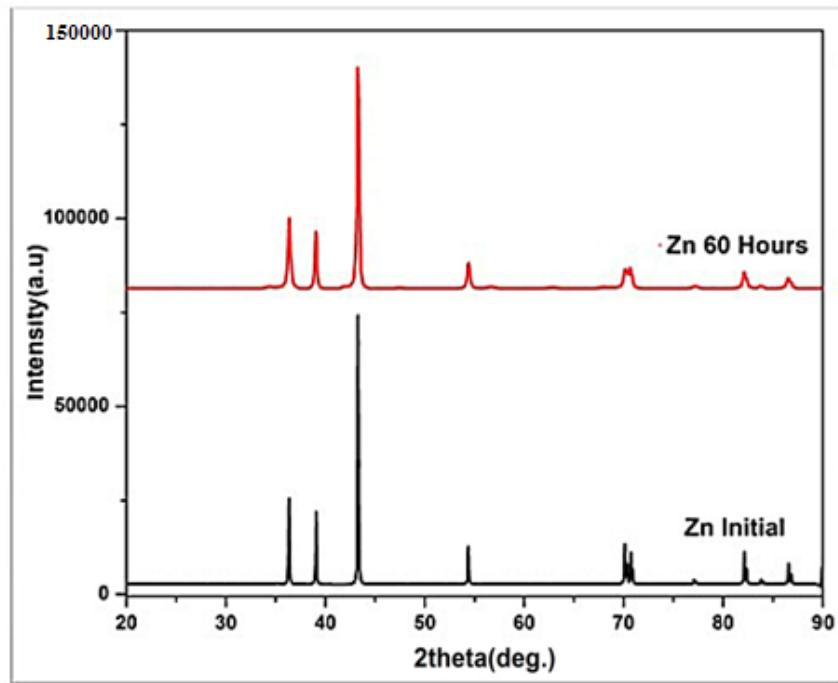
3.2.2 Characterization of Nanoparticles:

3.2.2.1 Characterization of Nanoparticle using XRD:

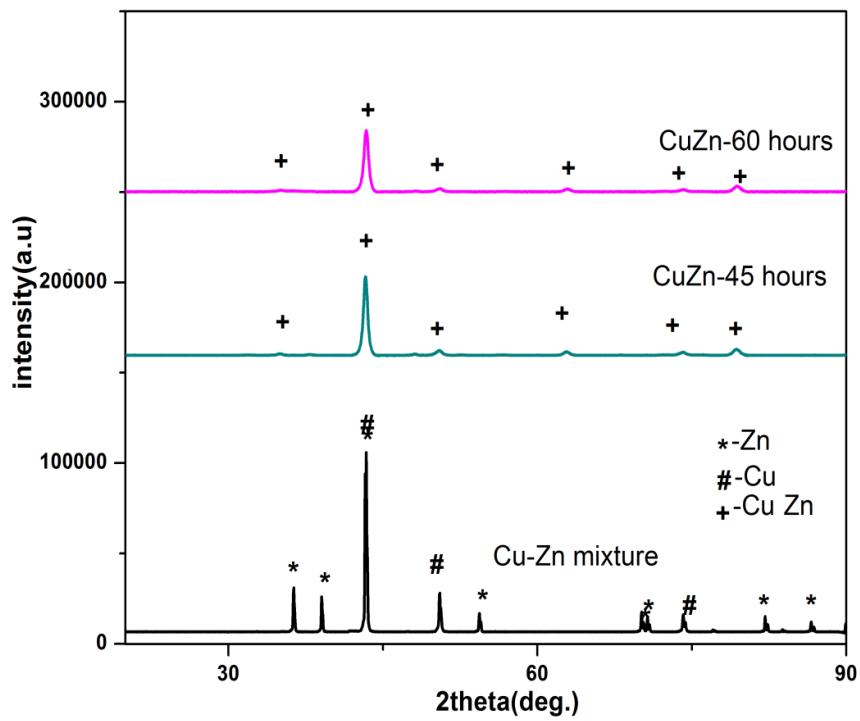
XRD of powder milled for different time period was conducted to study the different phase evolutions during milling. The XRD patterns of the Zn, Cu and Cu-Zn powder particles at selected intervals are shown in Figures 3.9 (a-c). It is evident that the Bragg peaks for milled product (after 60h of milling) are broad, suggesting reduction in crystallite size and accumulation of lattice strain. It is also observed that intensity of the peaks decreases due to the decrease of crystallinity of powder during milling. In case of Cu-Zn alloy powder, the XRD pattern of as received powder shows the peaks of Cu and Zn. The final milling product is a single phase nanocrystalline material which is clear from the graph. It is evident from the Figure. 3.9 (c) that after 45 hours of milling, Cu-Zn alloy has formed. The crystalline size was obtained by using Scherrer equation. The Table 3.6 shows that the Crystalline size of Cu, Zn and Cu-Zn Nanoparticles.



(a)



(b)



(c)

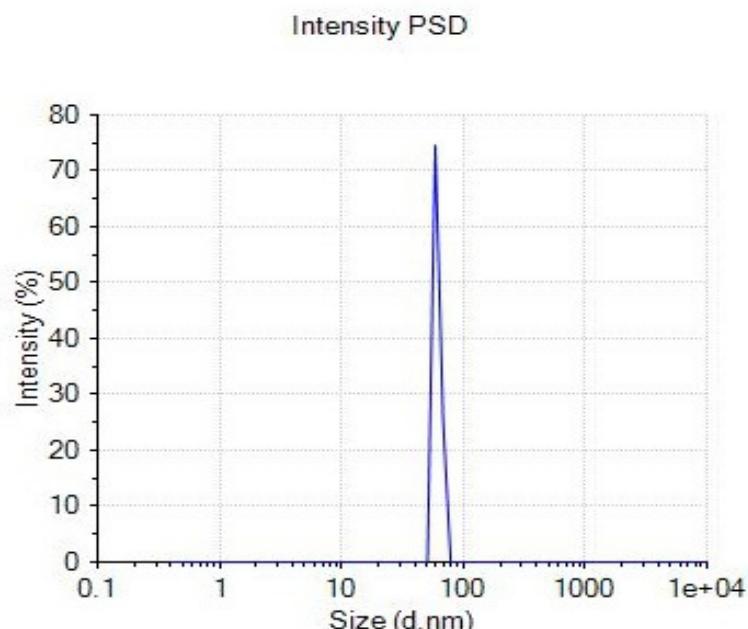
Figure 3.9: XRD pattern of (a) Cu Nanoparticle, (b) Zn Nanoparticle and (c) Cu-Zn Hybrid Nanoparticle at different milling times

Table 3.6: Crystallite size of nanoparticles.

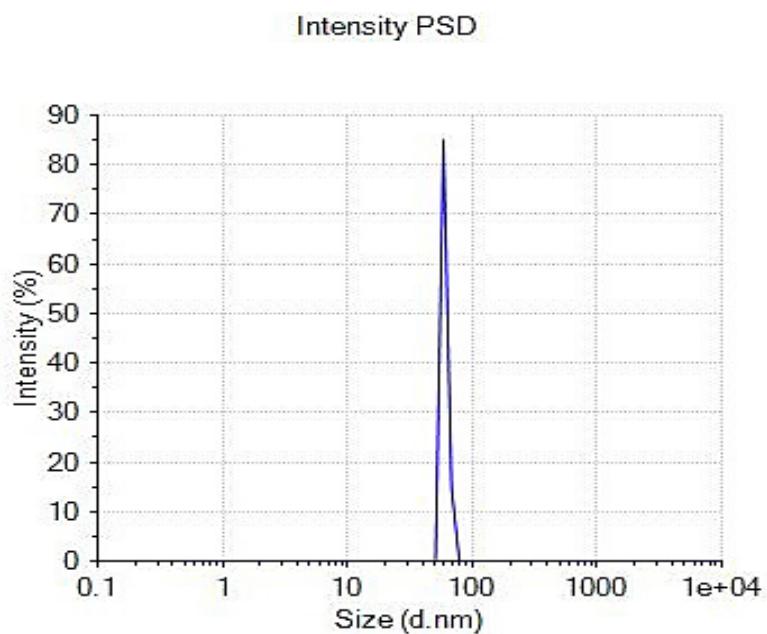
Sample	Crystallite Size (nm)
Zn initial powder	238
Zn after 60 hours	59
Cu initial powder	204
Cu after 60 hours	26
Cu-Zn after 45 hours	28
Cu-Zn after 60 hours	25

3.2.2.2 Particle Size Analyser for Nanoparticle size characterization:

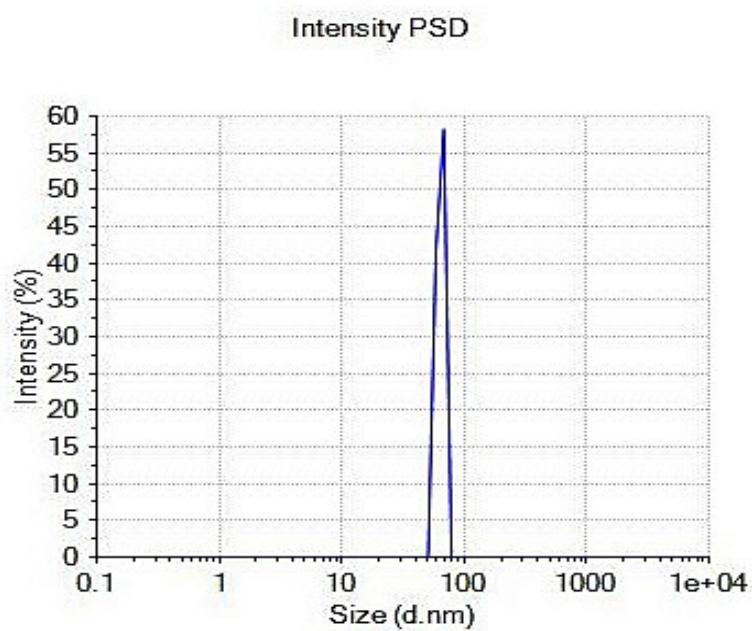
The particle size of the milled powders was determined using Malvern Zetasizer (Model: Nano ZS, Malvern). The particle size analysis was done after 60 hours milling of each powder. The output showing the particle size of each of the Zn, Cu and Cu-Zn hybrid powders are shown in Figures 3.10 (a-c). It is evident from the graph that the particle size of the Zn ultrafine powder is 60nm, Cu powder is 60nm and Cu-Zn powder is 70 nm respectively.



(a)



(b)



(c)

Figure 3.10: Particle size of (a) Cu particles after 60 hrs of milling, (b) Zn particles 60 hrs of milling and (c) Cu-Zn particles after 60 hrs of milling

3.2.3 Preparation of Nanofluids

Copper, zinc and Cu-Zn nanofluids were prepared by dispersing the estimated amount of milled nanopowders in vegetable oil by ultrasonication using an ultrasonic probe sonicator for 2 hours each. The amount of the nanoparticles essential for preparation of nanofluids is calculated using the law of mixture.

$$\% \text{ vol concentration} = \frac{\left[\frac{W_p}{\rho_p} \right]}{\left[\frac{W_p}{\rho_p} + \frac{W_{bf}}{\rho_{bf}} \right]}$$

W_p denotes the weight of the nano particles in grams, ρ_p denotes the density of the nano particles in kg/m^3 , W_{bf} denotes the weight of the base fluid in grams and ρ_{bf} denotes the density of the base fluid in kg/m^3 . The density of Zn, Cu and Cu-Zn hybrid powders are 7140, 8900 and 8050 kg/m^3 respectively. The test conditions and the specification of materials used for producing desired nanofluids are shown in Table 3.7. Figure 3.11 to 3.13 shows the prepared Zn, Cu and Cu-Zn nanofluids containing 0.1, 0.2, 0.3, 0.4 and 0.5% volume fraction in 100 ml of the base fluid (vegetable oil) from right to left.

Table 3.7: Specification of the nanofluids prepared.

Nanofluid	Zn-vegetable oil	Cu-vegetable oil	Cu-Zn-vegetable oil
Particles	Zinc	Copper	Cu-Zn
Base fluid	vegetable oil	vegetable oil	vegetable oil
Particle size	60 nm	60 nm	70 nm
Vol. fraction	0.1 – 0.5 %	0.1 – 0.5 %	0.1 – 0.5 %
Surfactant	SDS	SDS	SDS
Thermal conductivity of vegetable oil	0.162 W/mK	0.162 W/mK	0.162 W/mK
Viscosity of vegetable oil	$3.62 \times 10^{-7} \text{ (m}^2/\text{s)}$	$3.62 \times 10^{-7} \text{ (m}^2/\text{s)}$	$3.62 \times 10^{-7} \text{ (m}^2/\text{s)}$

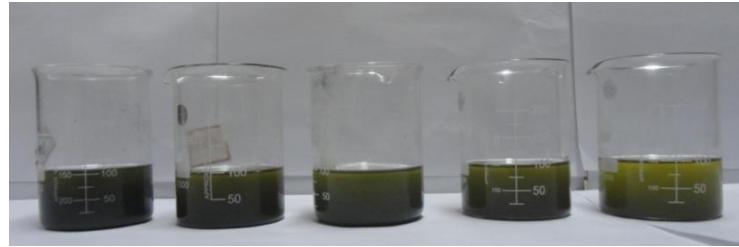


Figure 3.11: Zn –Vegetable oil Nanofluids



Figure 3.12: Cu – Vegetable oil Nanofluids

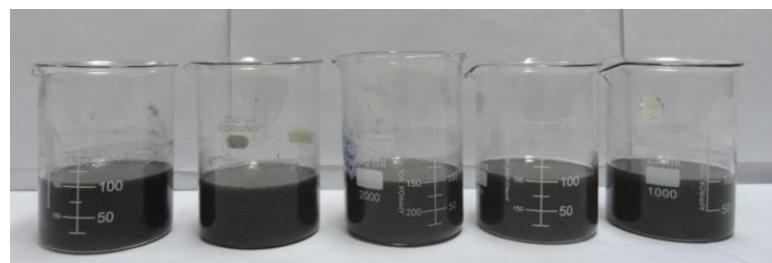


Figure 3.13: Cu-Zn – Vegetable Oil Nanofluids

3.2.4 Thermal Conductivity of Nanofluids

Thermal conductivity of the basefluid and the nanofluids was measured using KD2-Pro thermal property analyze (Decagon Devices, USA) [163-165]. The nanofluids prepared after ultrasonication was brought to room temperature (30°C) and then it was poured into a sample bottle and the KS-1 sensor of the KD2 Pro which is used for measuring the thermal conductivity of fluids was inserted into the bottle and kept stationary for measurement, as even small vibrations can cause error in the measurement. A maximum of 10 measurements were recorded for each volume concentrations. The thermal conductivity of the base fluid was found to be 0.162 W/mK.

From figure 3.14 it is clear that for all the three powders the thermal conductivity increases with increasing volume fraction. The thermal conductivity enhancement for Zn and Cu vegetable oil nanofluid were 36% and 42%, compared to basefluid at 0.5% of volume

fraction. Further increase in particle loading to 0.6%, a minor enhancement in thermal conductivity was found. Therefore, for further investigation of thermophysical properties, particle loading till 0.5% will be considered. The increase in thermal conductivity of the nanofluid is due to the increased area to transfer heat which increases as the particle size decreases, the high thermal conductivity of the nanopowders dispersed in it and due to the Brownian motion of the suspended particles which is inversely proportional to the density of the particles.

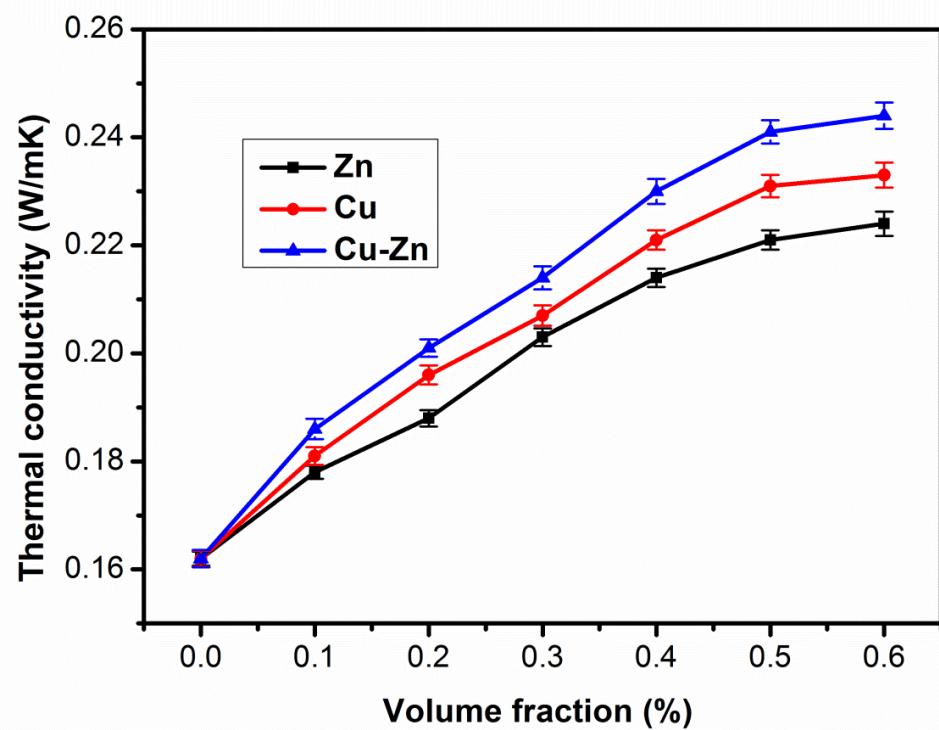


Figure 3.14: Thermal conductivity results.

Elemental Zn has low thermal conductivity and low density; therefore, Zn contributes less to the thermal conductivity of the Zn vegetable oil nanofluid by its low thermal conductivity. The Brownian velocity of the Zn particles is more as its density is low and it contributes more to the stability of the nanofluid. Similarly, Cu has very high value of thermal conductivity; therefore, Cu contributes more to the combined thermal conductivity of the Cu vegetable oil nanofluid. The Brownian velocity of the Cu particles is less as its density is high and it contributes less to the stability of the nanofluid. Cu-Zn hybrid particles have the advantage of high thermal conductivity and high Brownian motion. Therefore, the thermal conductivity of the Cu-Zn vegetable oil nanofluid is high with good stability. The hybrid

nanofluid gave a maximum enhancement of 48 % in thermal conductivity at a volume fraction of 0.5 %.

3.2.5 Viscosity Measurement of Nanofluids:

The internal resistance of a fluid to flow is described as viscosity and it is an important property that is involved in thermal application; affecting the pumping power and convective heat transfer coefficient. The viscosity of the fluids was determined at room temperature with the help of a Rheometer [166-167]. Nanofluids were prepared with 0.1% to 0.5% volume loading. From the figure 3.15 it is observed that as the particle loading increases the viscosity of the basefluid increases. The viscosity is affected by nanoparticle size, volume concentration, nanoparticle material and due to the agglomeration of the nanoparticles dispersed in the base fluid. The results show that an alloy of Cu-Zn showed more increment in viscosity when compared to Cu and Zn nanoparticles. This is due to the size dependency of viscosity. The size of Cu-Zn alloy (70 nm) is larger than Cu and Zn (60 nm). With further increase in particle loading to 0.6%, the increase of about 6% viscosity is observed from 0.5% to 0.6%. This increase in viscosity will cause increase pumping power, particle clogging etc. The increase in thermal conductivity is very less and increase in viscosity is on the higher side, therefore for further studies; particle loading of 0.5% volume fraction will be considered.

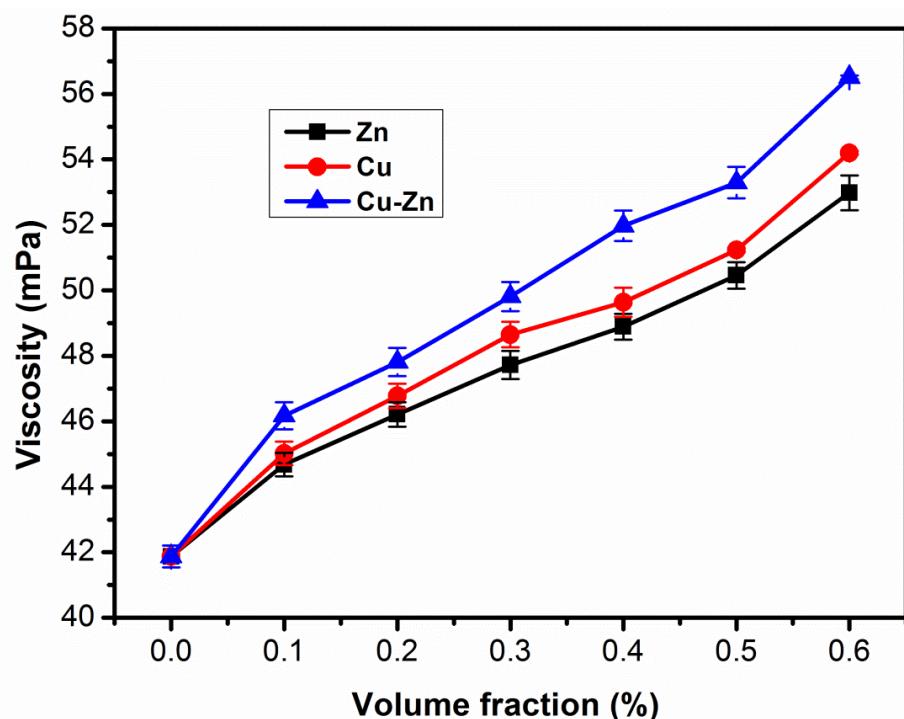


Figure 3.15: Viscosity results

3.2.6 Summary

An alloy of Cu and Zn could be formed through mechanical alloying which has a composition of 1:1. The increase in thermal conductivity and viscosity in the case of Cu-Zn hybrid nanofluid is more compared to that of the Zn and Cu nanofluids. Cu-Zn hybrid nanofluids showed better enhancement in thermal conductivity of 42% at 0.5 % volume. The nanofluid containing Cu-Zn hybrid nanoparticle possessed high viscosity of 36% at 0.5 % volume.

3.6 PHASE – 5: MODELLING OF NANOFLOUIDS

A wide experimental investigation on increasing thermal conductivity of nanofluids had been carried out in the last ten years, including oxides, metallic, SCNT, MCNT and different basefluids. The measurements show inconsistency due to difference in production of nanofluids, nanofluids characterisation and thermal conductivity measurement techniques. However a firm relationship is established from the experimental results between nanofluid thermal conductivity and following parameters: Nanoparticle volume fraction, size, shape, surfactants and additives. All the equations available in literature are in efficient to account all the parameters affecting the thermal conductivity of nanofluids. For solving complex problems in different areas in recent years, An Artificial Neural Network technique has been applied with substantial reduction in computational time.

3.6.1 Modelling of Thermal Conductivity of Hybrid Nanofluid

ANN of model was used to represent the experimental data for the thermal conductivity of the nanofluids.

Table 3.9: Inputs ranges for training the model

Parameters	Ranges
Volume Fraction (%)	0.1 - 0.5
Temperature (K)	303 - 333
Diameter of Nanoparticles (nm)	19 - 60
Ratio of Thermal Conductivity (Particle/Basefluid)	716.04 - 2469.13

The proposed ANN model was designed by software developed using the MATLAB Neural Network Toolbox. The input ranges for training the neural network are as given table 3.9. The structure of the network is as shown in figure 3.43. The experimental data were grouped into training data and testing data. The interconnected group of each artificial neuron receives one or more data and sums them to produce an output. These sums are weights corresponding to the parameters of the model; this is passed through non-linear function known as activation function and in turn processed using connectionist approach to computation. Neurons are organised into inputs, output and hidden layers which provide the

predictions of the model for the variables of interest. Single-layer architecture was chosen for ANN: input neurons are connected to a layer of N hidden neurons, which are connected to the output neurons. The number of input neurons for training the network were tested from 4 to 12 and found that with 10 neurons resulted better outcomes. The activation function is sigmoidal for hidden neurons, and linear for output neurons. Data are first scaled so that each component of the input and output vectors has zero mean and unitary variance across the dataset, as to give to the inputs the same importance in the fitting procedure, independently from the magnitude of their values.

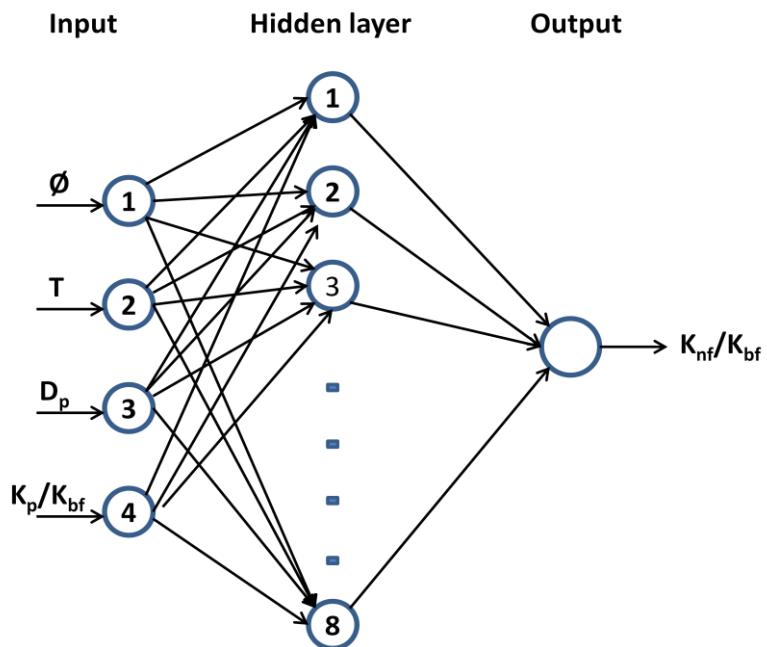


Figure 3.43: ANN architecture selected as the predicting model for Effective Thermal conductivity

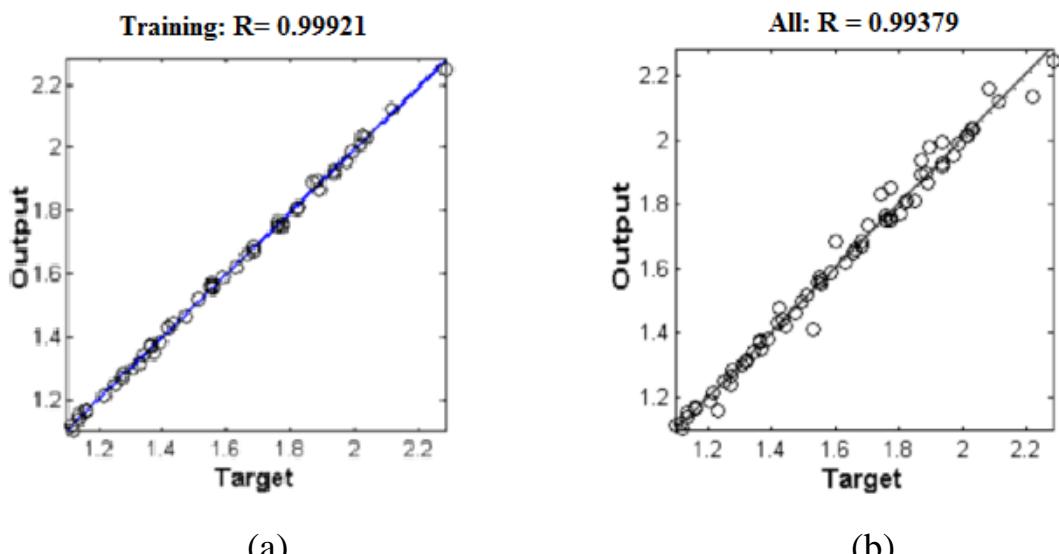


Figure 3.44: ANN Regression results (a) Training regression (b) All Regression

The relationship between measured (experimental) results, training and all regression results by using the ANN prediction model is shown in Figure 3.44. The proposed ANN is compared with the standard theoretical model of

Yu-Choi (168)

$$\frac{k_{nf}}{k_{bf}} = \frac{k_{pe} + 2k_{bf} + 2(k_{pe} - k_{bf})(1 + \beta)^3 \phi}{k_{pe} + 2k_{bf} - (k_{pe} - k_{bf})(1 + \beta)^3 \phi} \quad (1)$$

Patel et al. (169)

$$\frac{k_{eff}}{k_f} = \left(1 + 0.135 \left(\frac{k_p}{k_f} \right)^{0.273} \phi^{0.467} \left(\frac{T}{20} \right)^{0.547} \left(\frac{100}{d_p} \right)^{0.234} \right) \quad (2)$$

Godson et al. (170)

$$\frac{k_{eff}}{k_f} = 0.9692 \phi + 0.9508 \quad (3)$$

Fig 3.45 shows the predicted output of the theoretical models for 50:50 combination (at 303° K), when compared with proposed ANN. The theoretical models either over predicted or under predicted the experimental data. As a result of the study, it can be said that the well-trained ANN model can be used to optimize the effective thermal conductivity of hybrid Cu-Zn for all combinations, at various temperatures, volume fractions, diameter of particles, thermal conductivity of nanoparticles and thermal conductivity of basefluids.

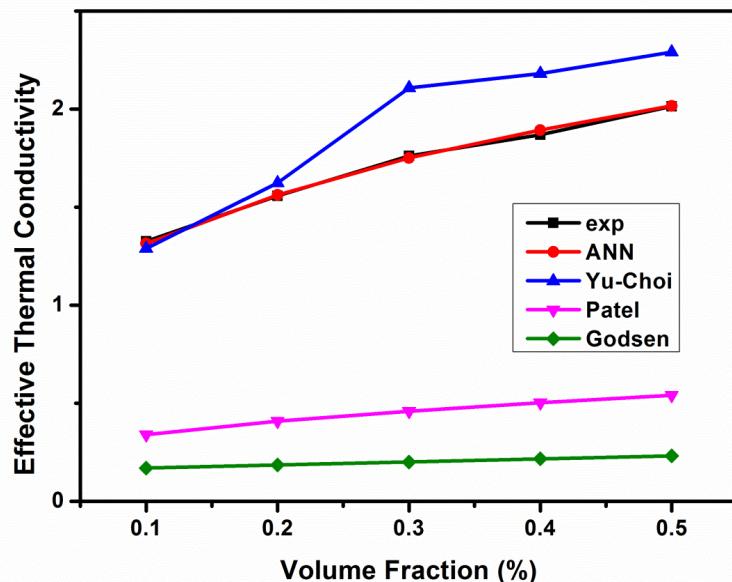


Figure 3.45: Experimental data vs ANN predicted results, Yu-Choi, Patel and Godsen

models

3.6.2 Modelling Technique for Viscosity of Hybrid Nanofluid

ANN of model was used to represent the experimental data for the viscosity of the nanofluids. The structure of the network is as shown in figure 3.46. The input data used to train the network is given is table 3.10

Table 3.10: Inputs ranges for training the Artificial Neural Network model

Parameters	Ranges
Volume Fraction (%)	0.1 - 0.5
Temperature (K)	303 - 333
Diameter of Nanoparticles (nm)	19 - 60

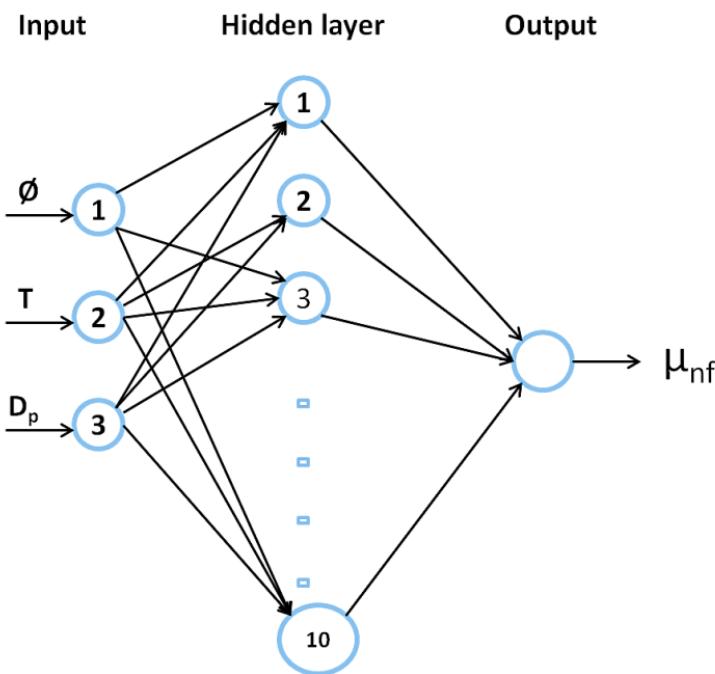


Figure 3.46: ANN architecture selected as the predicting model for viscosity

The relationship between measured (experimental) results, training and all regression results by using the ANN prediction model is shown in Figure 3.47. The value of R^2 in the training indicates that the network obtained explains at least 0.99% of the observed data. The proposed ANN is compared with the standard theoretical model of Einstein, Brinkmann and Nguyen. Figure 3.48 and 3.49 represent the comparison of theoretical models with proposed ANN model. Einstein model equation (4) and Brinkman model equation (5) were developed considering only the volume concentration of the nanoparticles. Both these equations over

predict the viscosity. Here other parameters of viscosity such as temperature, diameter of nanoparticles were not considered. Nguyen (6) considered the effect of temperature, but volume concentration effect was not considered. The theoretical models either over predicted or under predicted the experimental data As a result of the study, it can be said that the well-trained ANN model can be used to optimize the viscosity of hybrid Cu-Zn for all combinations (0:100, 75:25, 50:50, 25:75, and 100:0), at various temperatures, volume fractions, diameter of particles, viscosity of nanofluids and viscosity of basefluids.

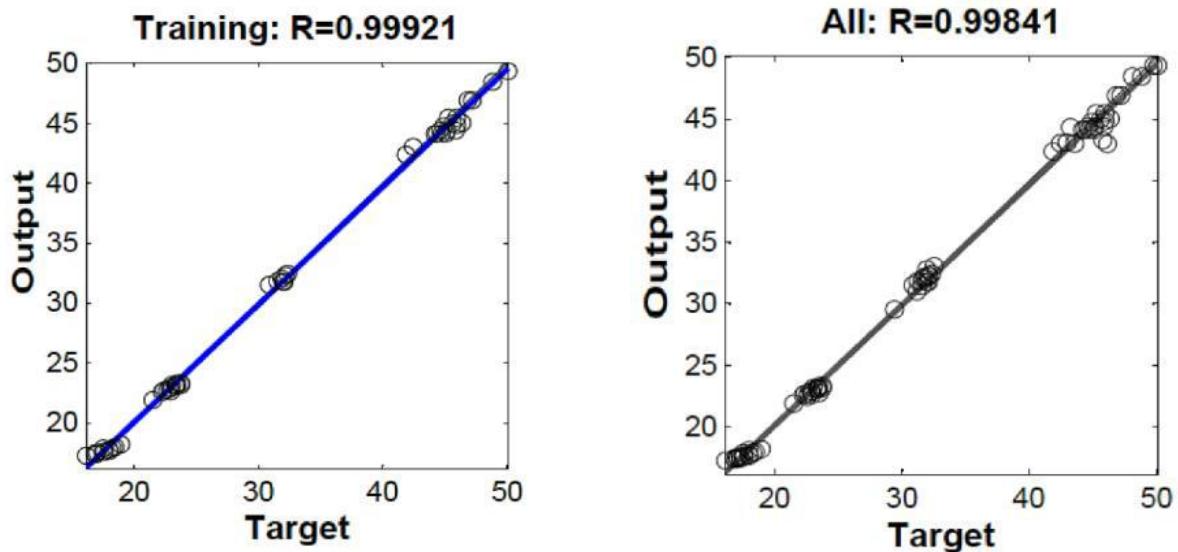


Figure 3.47: ANN regression results (a) Training regression, (b) All regression

Einstein Model (151)

$$\frac{\eta_{nf}}{\eta_{bf}} = 1 + 2.5\phi \quad (4)$$

Brinkman Model (152)

$$\frac{\eta_{nf}}{\eta_{bf}} = (1 - \phi)^{2.5} \quad (5)$$

Nguyen Model (154)

$$\frac{\eta_{nf}}{\eta_{bf}} = (2.1275 - 0.0215T + 0.00027T^2) \quad (6)$$

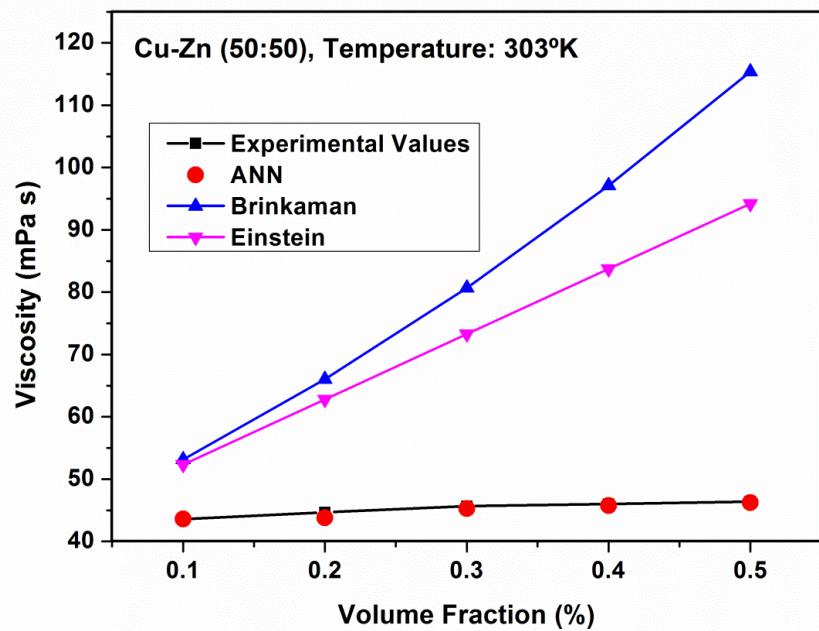


Figure 3.48: Experimental data vs ANN predicted results, Einstein and Brinkman model

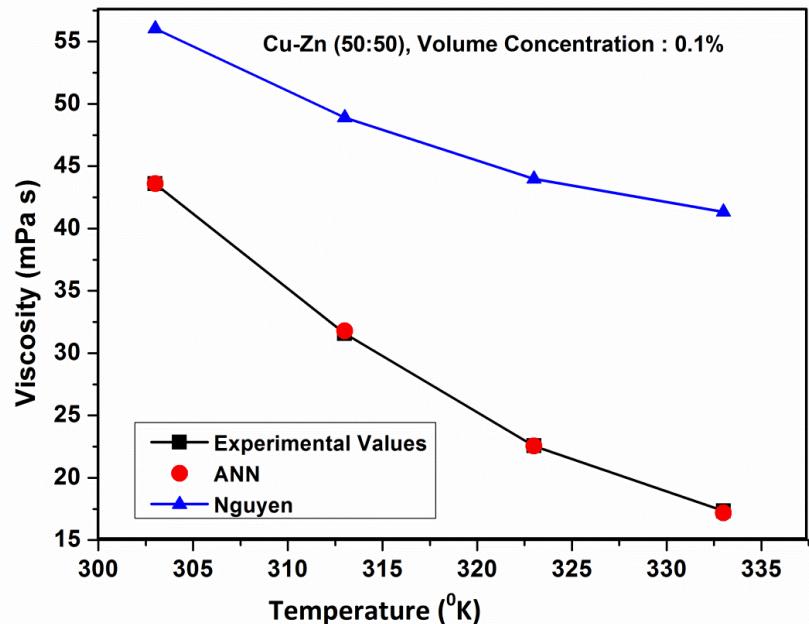


Figure 3.49: Experimental data vs ANN predicted results and Nguyen model

3.6.3 Summary:

Artificial Neural network models were proposed to represent the thermal conductivity as a function of the temperature, nanoparticle concentration, diameter of nanoparticle and the thermal conductivity of the nanoparticles and basefluids. In similar way ANN was proposed to represent the viscosity as a function of the temperature, nanoparticle concentration,

diameter of nanoparticle and the viscosity of the basefluids and compared with existing theoretical models.

3.3 PHASE – 2: SELECTION OF BASEFLUIDS

Lubricants are being utilized in all sectors of industry to reduce the wear by reducing friction by providing a protective layer between two contacting surfaces that have relative motion. In 2015 as reported, that a total of 640 million gallons of MWFs is annually consumed throughout the world, out of which 66 percent is disposed after usage. Out of which 85% of lubricants used around the world are petroleum based oils, causing adverse effect on environment. Conventional oils exhibit unwanted and undesirable elements such as phosphorous, iron, magnesium and calcium due to combustion.

The depleting trend of conventional cutting fluids due to the adverse effect on environment has triggered need to shift towards alternative renewable bio lubricants. Bio lubricant has a greater potential to serve as renewable energy in the present as well as the future. There is a considerable need to focus on improving environmentally friendly bio lubricant which is reliable, durable and efficient.

The conventional metal working fluids are mineral oil based fluids and are not bio degradable. Disposal of these metal working fluids causes diminishing of the environment and has adverse effect on the environment. As cutting fluids are complex in their composition, they may cause irritation or allergy, even microbial toxins are generated by bacteria and fungi present in water soluble cutting fluids, which are unsafe to the operators. Hence there is a need to develop bio lubricant which can triumph over these problems. Vegetable based oils are decidedly attractive alternate to mineral based cutting fluids because they are renewable, highly biodegradable, environment friendly and less toxic.

Compared with conventional metal working fluids, vegetable oil-based bio lubricants exhibit better lubricity, high viscosity index, high flash point, and low evaporative losses. Bio lubricants such as vegetable oils exhibit long fatty acid chains and the presence of polar groups in the structure leads to both boundary and hydrodynamic lubrication. With the emerging trends in nanotechnology, fluids having higher thermal conductivity called nanofluids have evolved. Nanofluids are a stable colloidal suspension of ultrafine solid particles (10-100 nm) in basefluid. The thermophysical properties of the basefluid can be enhanced by inclusion of nanoparticle. The enhanced thermophysical properties of the basefluid lead to the applicability of the fluids as coolants. On the whole, the research studies

have decisive on suspending either metal or metal oxide based nanostructured materials into the base fluid using surfactants for effective dispersion of nanoparticles.

As in phase 1, Cu-Zn hybrid nanoparticles showed better thermophysical properties. In this phase, the thermophysical properties of Cu-Zn (50:50) hybrid nanofluids are studied when incorporated in bio-degradable oils and conventional metal cutting oils for selection of basefluid. In situ preparation of Cu-Zn hybrid nanoparticle is done and dispersed in vegetable oil, paraffin oil and Castrol oil. The stability, behaviour, enhancement in thermal conductivity and the relative viscosity of the hybrid nanofluid prepared by incorporating Cu-Zn hybrid nanoparticle in vegetable oil, paraffin oil and SAE oil were studied to identify best suited basefluid.

3.3.1 Synthesis and Characterization of Hybrid Cu-Zn (50:50) particles:

In phase 1, Cu-Zn hybrid nanoparticle showed better results than individual nanoparticle of Cu and Zn. The size of the hybrid nanoparticle was too high, so this combination (50:50) was again milled to have reduced size. Mechanical milling of the micro sized metal powders was carried out in Retsch Planetary Ball Mill PM-100 with Tungsten carbide vials and balls to prepare ultrafine powders. Elemental copper and zinc powder particles were used for preparing ultrafine particles of Cu-Zn hybrid nanoparticle. The parameters were altered compared to the parameters used in phase 1 for milling alloy of hybrid nanoparticles. Cu and Zn ultrafine powders, which in turn milled in a vial containing 70 grams of powder and 560 gram balls for 45 hours. The ball to powder ratio was 8:1 and Tungsten carbide balls of 5mm and 3mm were used for milling. Milling was conducted at a speed of 250 rpm in wet medium (about 50 ml of toluene) to prevent undue oxidation and agglomeration of powder. Powder samples were collected from the vials at selected intervals of time to check the reduction in size of the particles. Powders were characterized by X-ray diffraction (XRD) D8-Advance, Brooker, Germany with Cu-K α radiation and Particle size Analyzer, Nano ZS, Malvern.

XRD of powder milled for different time period was conducted to study the different phase evolutions during milling. The XRD patterns of Cu-Zn powder particles at selected intervals are shown in Figures 3.16. It is evident that the Bragg peaks for milled product (after 45h of milling) are broad, suggesting reduction in crystallite size and accumulation of lattice strain. It is also observed that intensity of the peaks decreases due to the decrease of

crystallinity of powder during milling. In case of Cu-Zn alloy powder, the XRD pattern of as received powder shows the peaks of Cu and Zn. The final milling product is a single phase nanocrystalline material.

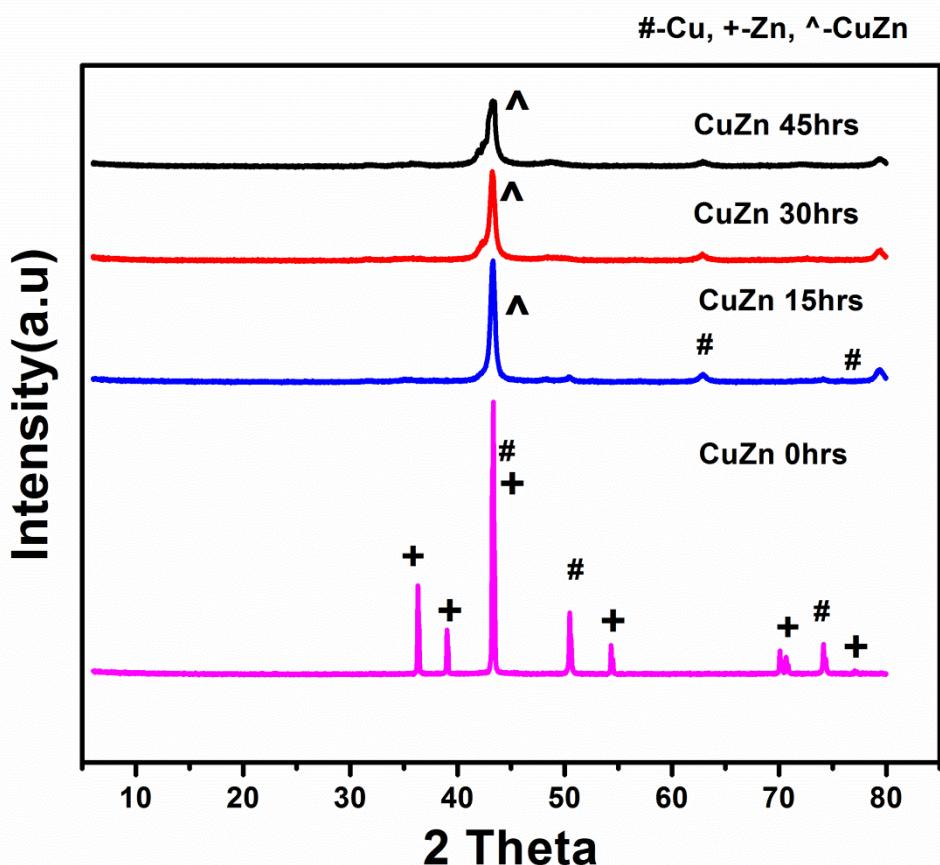


Figure 3.16: XRD graph of Cu-Zn (50:50) hybrid nanopowder

Further the particle size analysis was done after 45 hours milling of powder using particle size analyzer. The output showing the particle size of each of the Cu-Zn hybrid powders is shown in Figure 3.17. It is evident from the graph that the average particle size of the Cu-Zn powder is 25 nm. The FE-SEM image taken (figure 3.18) for powder milled for 45 hours, shows that the shape of the particle is spherical. TEM image (figure 3.19) was also taken after 45 hours, shows that the particle is spherical in shape and nano size is obtained.

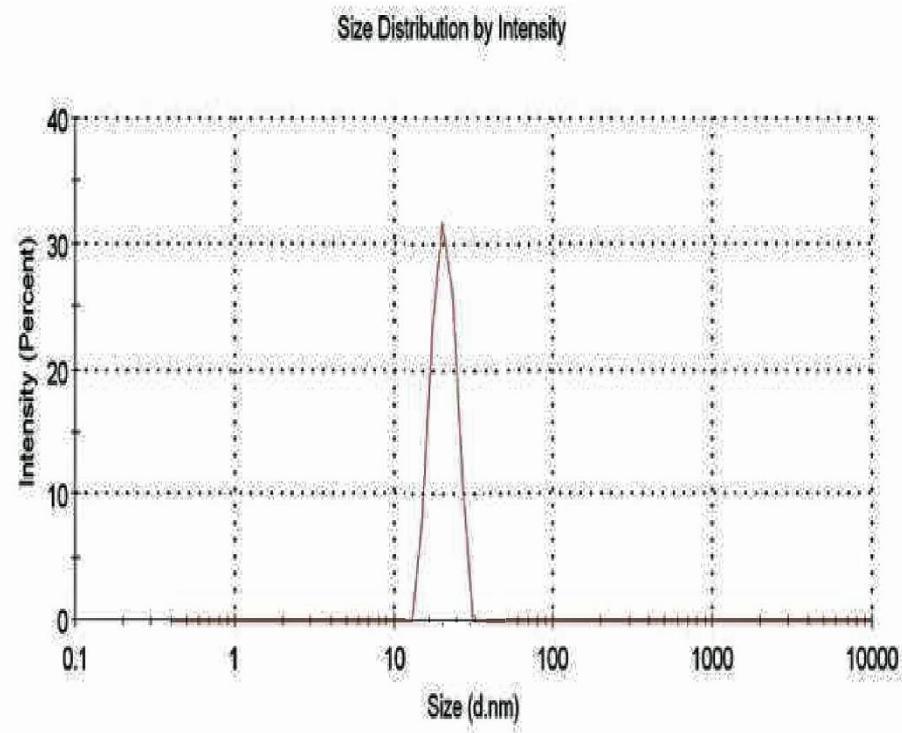


Figure 3.17: Results for particle size of Cu-Zn (50:50) hybrid nanopowder milled for 45 hrs using Particle size analyzer

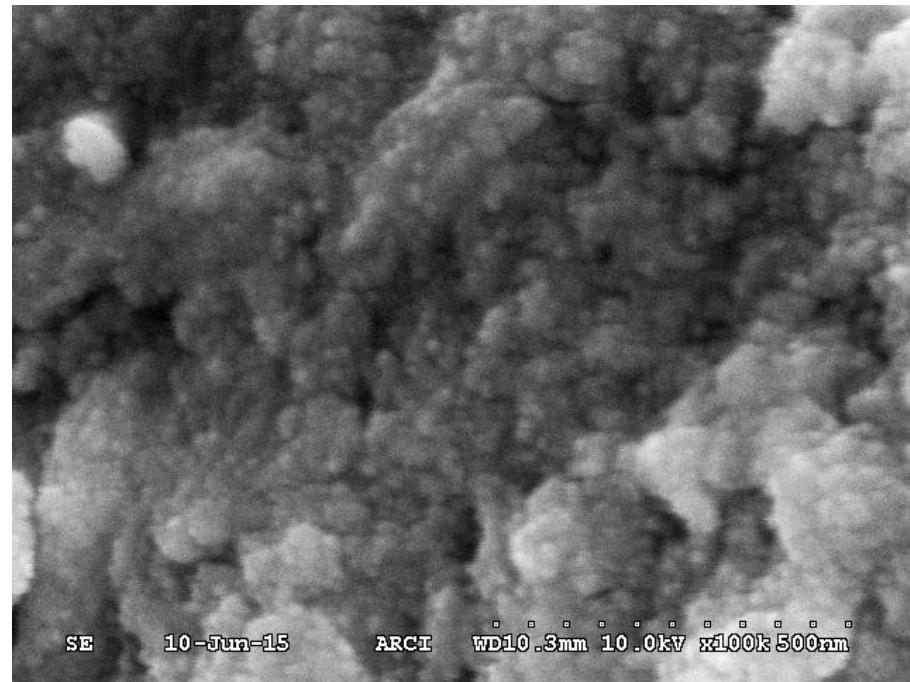


Figure 3.18: FE-SEM microstructure of Cu-Zn (50:50) hybrid nanopowders milled for 45 hrs

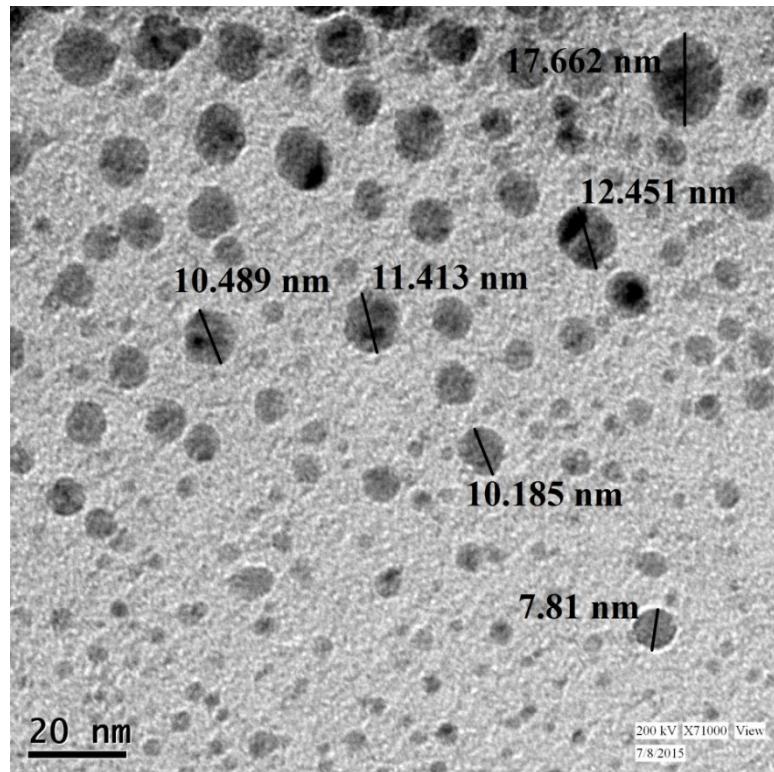


Figure 3.19: TEM microstructure of Cu-Zn (50:50) hybrid nanopowders milled for 45 hrs

3.3.2 Preparation of Nanofluids

Hybrid nanofluids were prepared by dispersing the estimated amount of milled nanopowders in vegetable oil. The procedure mentioned in 3.2.3 was used to prepare the hybrid nanofluids in this phase. The test conditions and the specification of materials used for producing desired nanofluids are shown in Table 3.8.

Table 3.8: Specification of the nanofluids.

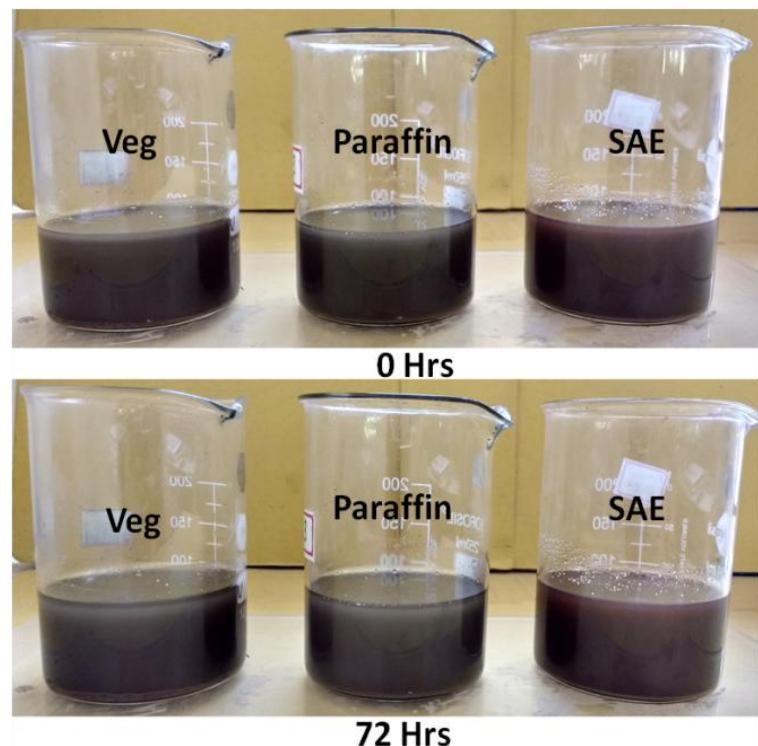
Base fluid	Vegetable oil	Paraffin oil	SAE oil
Particles	Cu-Zn (50:50)	Cu-Zn (50:50)	Cu-Zn (50:50)
Particle size	25 nm	25 nm	25 nm
Vol. fraction	0.1 – 0.3 – 0.5 %	0.1 – 0.3 – 0.5 %	0.1 – 0.3 – 0.5 %
Surfactant	SDS	SDS	SDS
Thermal conductivity of vegetable oil	0.162 W/mK	0.136 W/mK	0.133 W/mK
Viscosity of oils	41.72 (m Pa)	99.76 (m Pa)	161.07 (m Pa)

3.3.3 Nanofluid Stability:

The stability of nanofluids is compared for different basefluids by two techniques. For the study, the volume fraction of Cu-Zn hybrid nanoparticles was selected as 0.5%.

3.3.3.1 Stability analysis of nanofluids using Photo Capturing:

The nanofluids were prepared with three basefluids i.e. vegetable oil, paraffin oil and SAE oil, with this concentration of 0.5%. Photographs of the nanofluids were captured at different time intervals. From figure 3.20 it can be seen that till 72 hrs the all three nanofluids are stable and after that the sedimentation of nanoparticles takes place. As the time increases the nanoparticle tends to agglomerate to each other and the sedimentation of the particle increases and settle down by 168 hrs. Compared to all oils, SAE oil showed better stability.



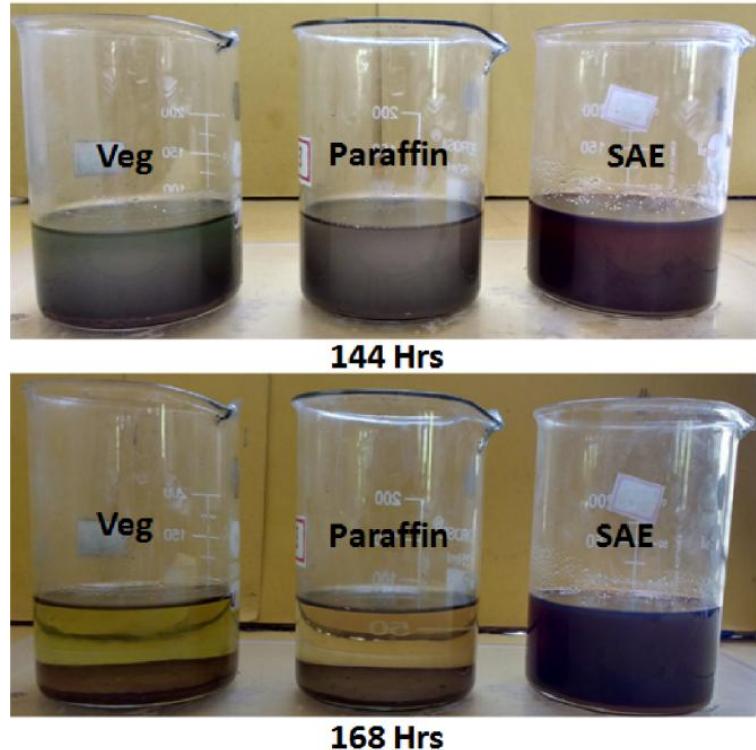
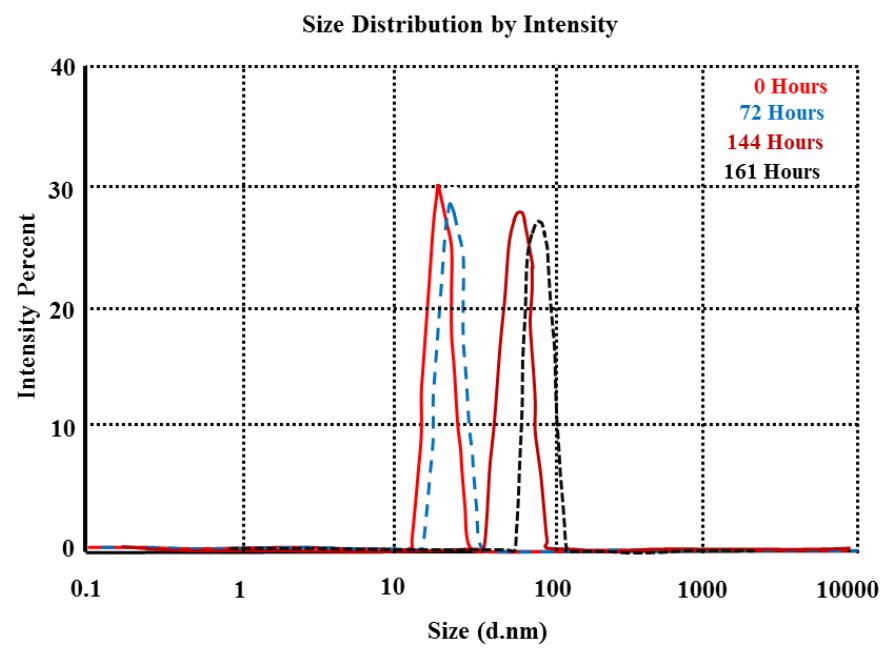


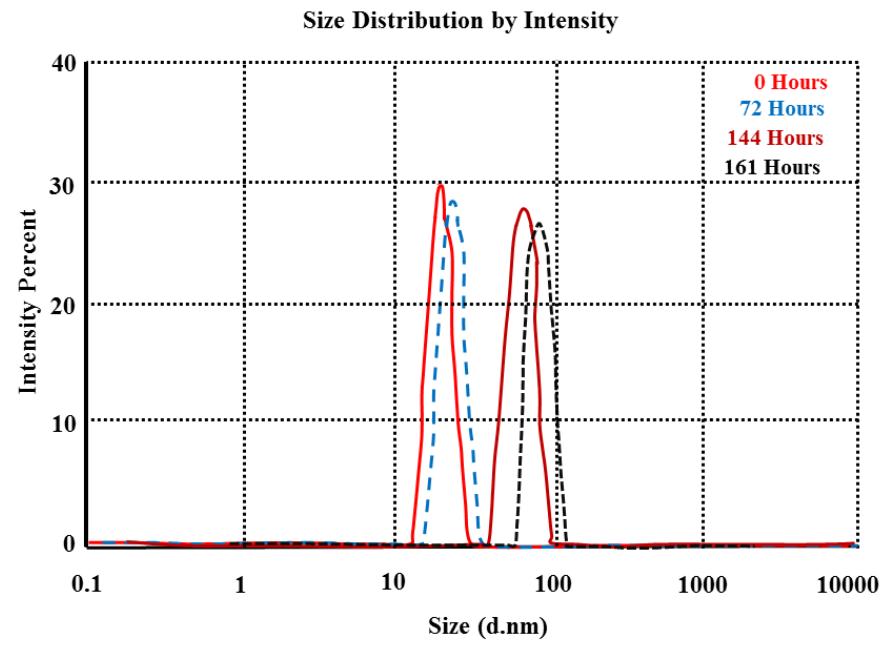
Figure 3.20: Sedimentation Photographs of Nanofluids with three type of basefluids at 0.5% volume concentration

3.3.3.2 Stability analysis of nanofluid using DLS method:

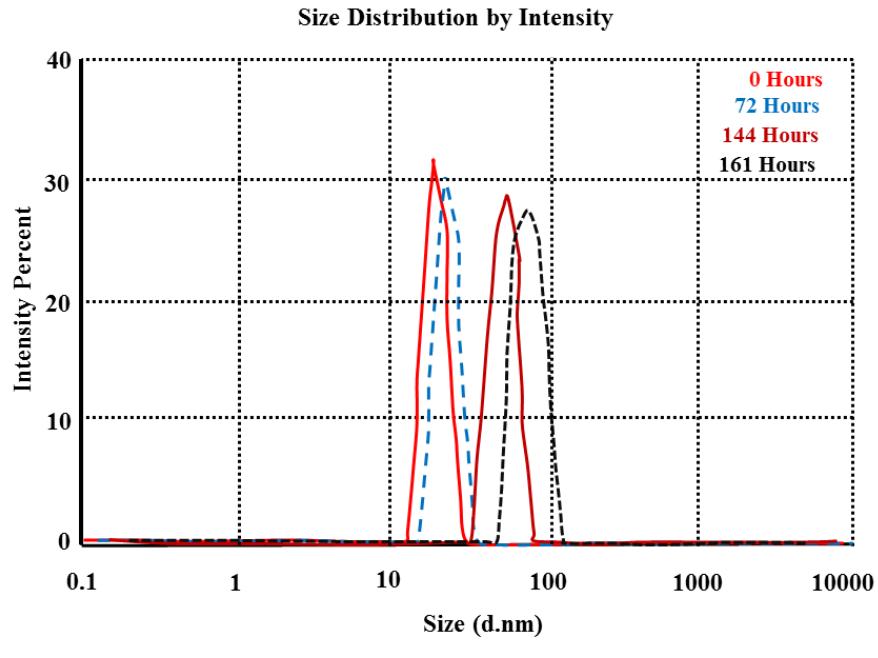
The stability of the nanofluid is evaluated by considering the tendency of the nanoparticle to aggregate over time period as taken as control parameter. A Zetasizer Nano (Malvern) was used to measure the average particle size of the nanoparticle suspended in the fluid solution. The particles in the fluid move randomly, and their speeds of movement are used for determining the size of the particle. Nanofluids with 0.5% volume concentration were prepared using three different basefluids (vegetable oil, paraffin oil and SAE oil) for stability investigation. Three samples of fluid listed in Table 3.7 were placed in three different measurement cuvettes. The samples were measured every day for 7 days without shaking the fluid, to evaluate the size distribution changes due to natural sedimentation. Figure 3.21 (a-c) show the size distribution of three of nanofluids prepared with three kinds of basefluids. Results indicated that all the prepared fluids were stable till 72 hrs and then the agglomeration of particles starts and by 168 hrs almost all the particles tend to settle down. The similar trend can be seen in stability measurement by photographic method. Among the three oils, SAE oil indicates less agglomeration.



(a)



(b)



(c)

Figure 3.21: Nanoparticles size distribution containing 0.5 wt.% Cu-Zn (50:50) hybrid nanoparticle dispersed in (a) Vegetable oil, (b) Paraffin oil and (c) SAE oil

3.3.4 Flash Point of Nanofluids:

Flash point refers to both flammable as well as combustible liquids. Flash point of a volatile liquid is the lowest temperature at which it can vaporize to form an ignitable mixture in air. Flash point of the hybrid nanofluids were measured using Pensky-Martens open cup apparatus as per ASTM D6450. Flash point values of three basefluids and hybrid nanofluids prepared from based fluids are as shown in figure 3.22. The flash point of all three types of nanofluids increased, with the increase in volume concentration of particle loading. The increase in flash point of vegetable oil based nanofluids is more, compared to the nanofluids with other base oils.

3.3.5 Thermal Conductivity of Nanofluids:

KD2 Pro equipment was used to measure the thermal conductivity for different nanofluids. The thermal conductivity of base fluids with hybrid particles with 0.1%, 0.3% and 0.5% volume concentrations was measured. Figure 3.23 shows the plot of effective thermal conductivity of the three base fluids and nanofluids with different concentrations. There is an enhancement in thermal conductivity of the hybrid nanofluids with increase in volume

concentrations in all type of oils. The effective thermal conductivity of vegetable oil is more when compared to paraffin oil and SAE oil.

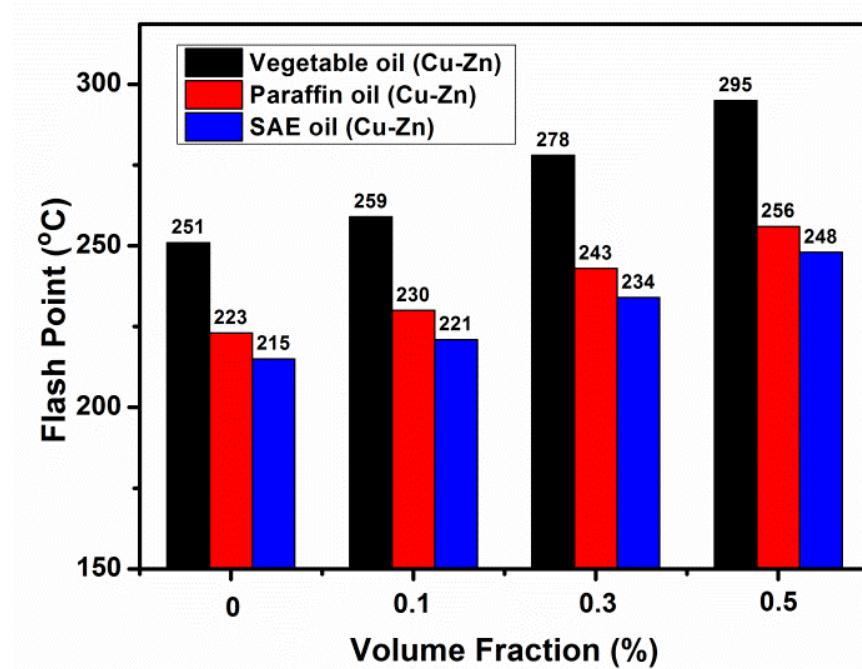


Figure 3.22: Flash point of Hybrid nanofluids

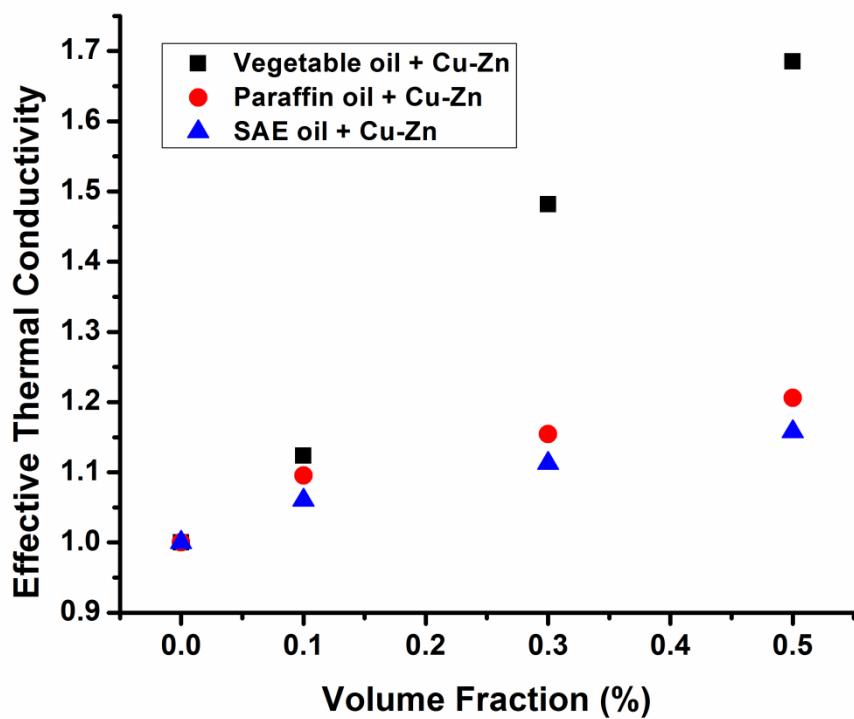


Figure 3.23: Effective Thermal Conductivity Vs Volume Fraction

3.3.6 Viscosity of Nanofluids:

The Viscosity of different base fluids with Cu-Zn hybrid nanoparticles incorporated is measured using a Rheometer (Anton-Paar, Austria). The viscosities and shear rate of different basefluids nanofluid are obtained for various volume fractions and shear rate.

3.3.6.1 Effect of Concentration

Figure 3.24 shows the relative viscosity of vegetable oil, paraffin oil and SAE oil with concentrations of hybrid Cu-Zn nanoparticle. The relative viscosity is the ratio of the viscosity of the basefluid to that of the prepared nanofluid. Vegetable oil base nanofluid exhibit higher relative viscosity with increase in concentration of hybrid nanoparticle incorporation. A relatively less increase in relative viscosity of paraffin oil and SAE oil based nanofluids are found.

Figure 3.25 shows the shear stress to shear rate of vegetable oil, paraffin oil SAE oil with different concentrations of hybrid Cu-Zn nanoparticle. The slopes of the curve indicate the increase or decrease of viscosity and the behaviour of nanofluid. Vegetable oil based nanofluid exhibited linear relation between shear stress and shear rate. Vegetable oil based hybrid nanofluid behaved as Newtonian fluid and with the other oils the slope of the curve is increasing as they exhibit higher viscosity and tend to behave like non-newtonian. Due to this the properties of the vegetable oil based nanofluid properties can be predicted and considered as ideal.

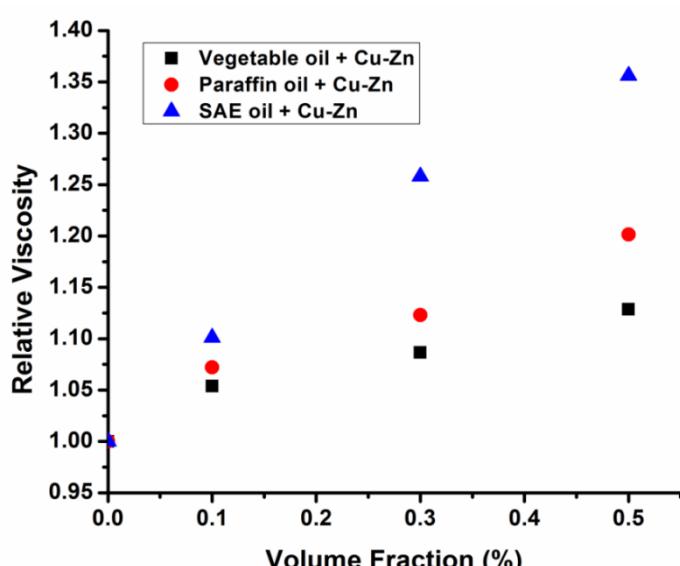


Figure 3.24: Relative viscosity of different basefluid with Cu-Zn hybrid nanoparticle dispersion

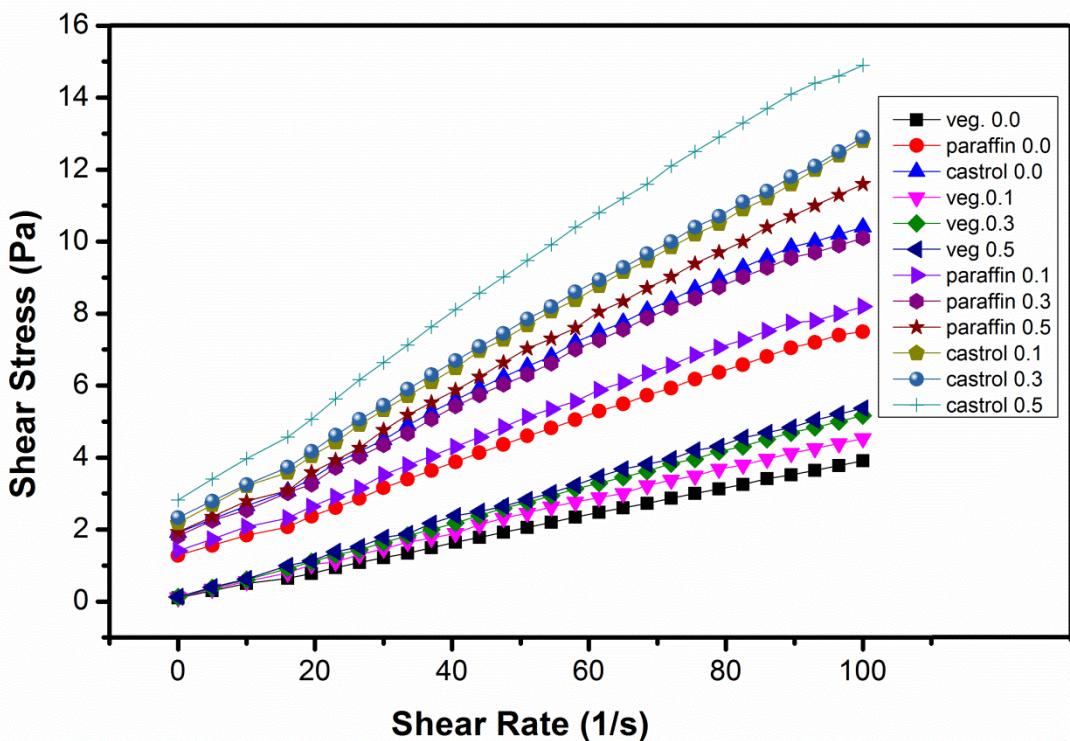


Figure 3.25: Shear stress Vs Shear rate of different basefluids with Cu-Zn hybrid nanoparticle dispersion

3.3.6.2 Effect of shear rate

The variation in viscosity of Cu-Zn (50:50) Vegetable oil/ Paraffin oil/ SAE oil hybrid nanofluid as a function of shear rate is shown in figure 3.26. With application of shear to the oils, deformation of the oils takes place resulting in decrease of viscosity. Vegetable oil based hybrid nanofluid exhibited less amount of deformation or shear thinning with application of shear to it when compared to paraffin and SAE oil. The reason for shear thinning for the paraffin and SAE oils is, as the spindle rotates in the fluid, the structure of the fluid molecule changes temporarily, gradually align themselves in the direction of increasing shear and produce less resistance and hence a reduction in viscosity at low shear rate. When the shear rate is high enough the maximum amount of shear ordering possible is attained, the aggregates are broken down to smaller size decreasing the friction and hence the viscosity increase. This implies that vegetable oil is much stable with application of shear with respect to other two base oils.

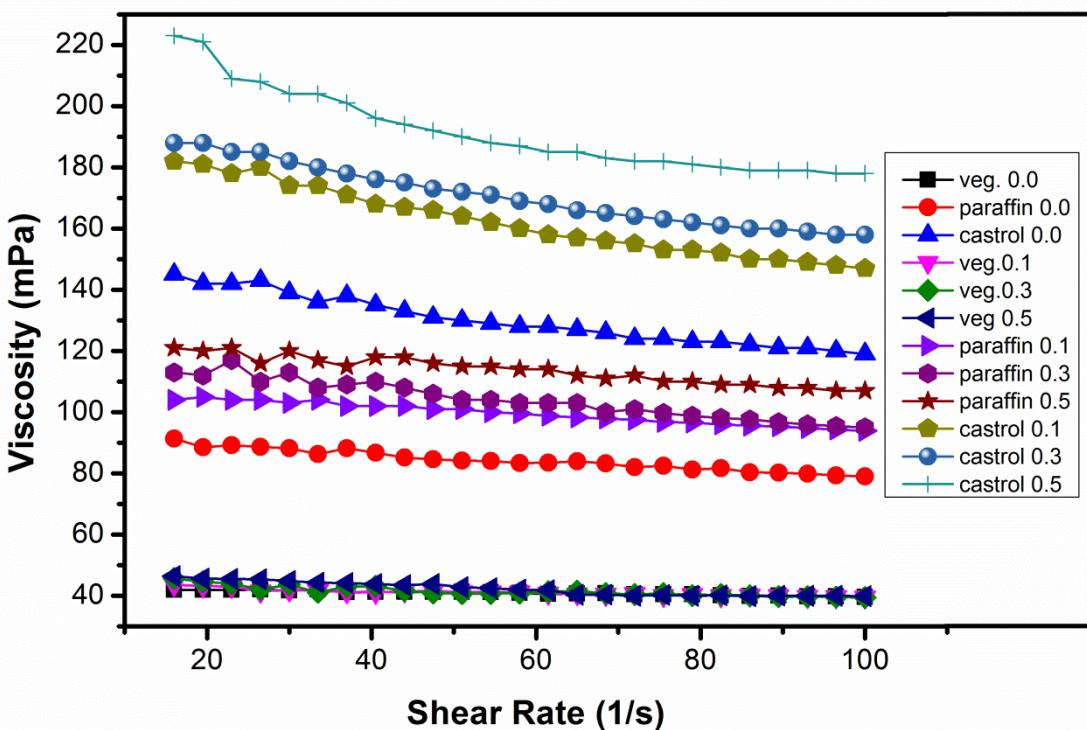


Figure 3.26: Viscosity Vs Shear rate of different basefluids with Cu-Zn hybrid nanoparticle dispersion

3.3.6.3 Relative Thermal Conductivity to Relative Viscosity:

Relative thermal conductivity to relative viscosity defines the rate of increase in thermal conductivity with increase in viscosity. Figure 3.27 shows the ratio of relative thermal conductivity to relative viscosity of all three basefluids with hybrid Cu-Zn nanoparticle dispersion. The ratio of relative thermal conductivity to relative viscosity of vegetable oil increases with addition of nanoparticle and increase in viscosity, while the ratio decreases in case of paraffin oil and SAE oil. This illustrates that addition of nanoparticle in paraffin and SAE oil doesn't affect much on thermal conductivity when compared to vegetable oil. This is due to the internal resistant of the fluid to flow and reduction in convective heat transfer coefficient. With less resistant to internal flow there is reduction in requirement of pumping power and enhanced heat transfer coefficients. The vegetable oils exhibit better enhancement in thermal conductivity, viscosity, stability and ideal behaviour, compared to paraffin and SAE oils. Hence vegetable oil is considered as basefluids for cutting fluids.

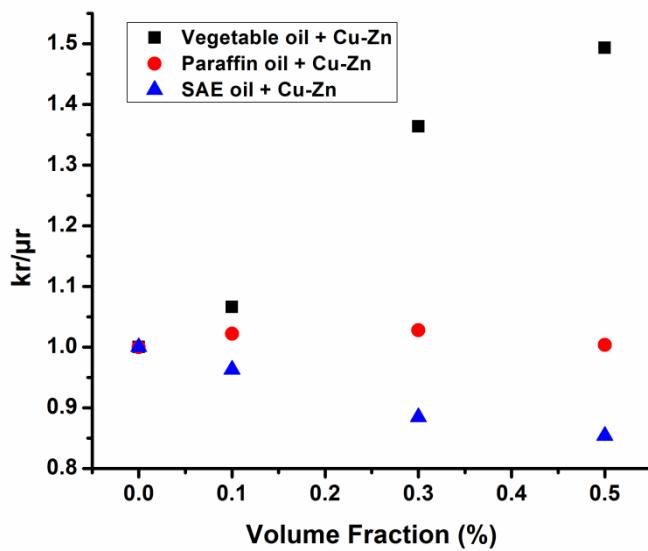


Figure 3.27: Relative Thermal Conductivity / Relative Viscosity vs Volume fraction of different basefluids with Cu-Zn hybrid nanoparticle dispersion

3.3.7 Summary

The thermophysical properties of hybrid nanofluids with different basefluids (vegetable oil, paraffin oil and SAE oil) were studied, with incorporation of hybrid Cu-Zn (50:50) nanoparticles. Flash point test and stability analysis were carried out. Finally integrated study of thermal conductivity and viscosity, in the point of cost and higher relative thermal conductivity to relative viscosity for effective heat transfer was carried out to identify the suitable base oil for cutting fluids.

3.4 PHASE – 3: EFFECT OF VARIOUS COMBINATIONS OF HYBRID Cu AND Zn (50:50, 75:25, 25:75)

From the previous phases, it's seen that a hybrid alloy of Cu and Zn (50:50) showed better results when compared to elemental Cu and Zn. In this phase further investigation is carried out to find out the effect of various combinations of Cu and Zn (75:25; 25:75). These combinations are synthesized using the procedures mention in section 3.3. Various literatures have stated that thermal conductivity of the nanofluid is dependent on the thermal conductivity of the particle and Brownian motion. Brownian motion of the particle is dependent on the density of the particle and viscosity of the fluid. The viscosity of the fluid is dependent on the size of the particle loaded. When the size of the particle loading is similar the viscosity of the fluid should also be identical. On these bases the powders are synthesized that the final product has similar size to that of the powder with Cu and Zn (50:50) combination. The synthesized powder are characterized and used to prepare hybrid nanofluid by adding various volume concentrations of prepared hybrid nanoparticle in vegetable oil (ground nut) and ultrasonication. This section compares the three combinations of Cu and Zn i.e. 50:50; 75:25; 25:75 and their effect on thermophysical properties at a range of volume concentrations and different temperatures.

3.4.1 Synthesis and Characterization of hybrid Cu-Zn nanoparticles (50:50, 75:25, 25:75) combinations

A procedure similar to 3.3.1 is used to for synthesis of powder in this phase. To prepare Cu-Zn ultrafine powder, Cu and Zn powders were mixed in a ratio 25:75 and 75:25 by weight and milled for 45 hours. The ball to powder weight ratio was 8:1. Milling was conducted at a speed of 250 rpm in wet medium (about 50 ml of acetone) to prevent undue oxidation and agglomeration of powder.

XRD of powder milled for different time period was conducted to study the different phase evolutions during milling. The XRD patterns of the Zn, Cu and Cu-Zn powder particles at selected intervals are shown in Figures 3.28 (a-b). It is evident that the Bragg peaks for milled product (after 45 h of milling) are broad, suggesting reduction in crystallite size and accumulation of lattice strain. It is also observed that intensity of the peaks decreases due to the decrease of crystallinity of powder during milling.

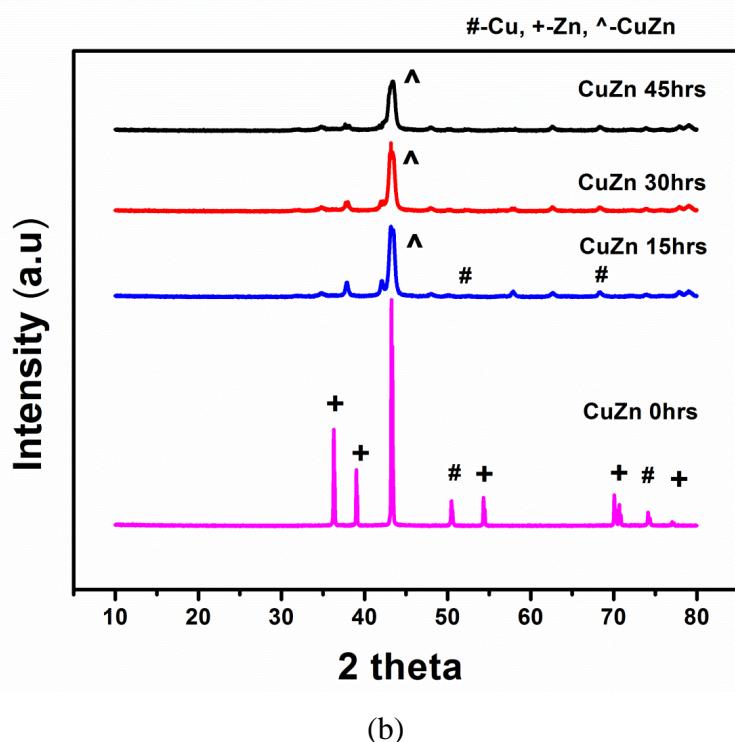
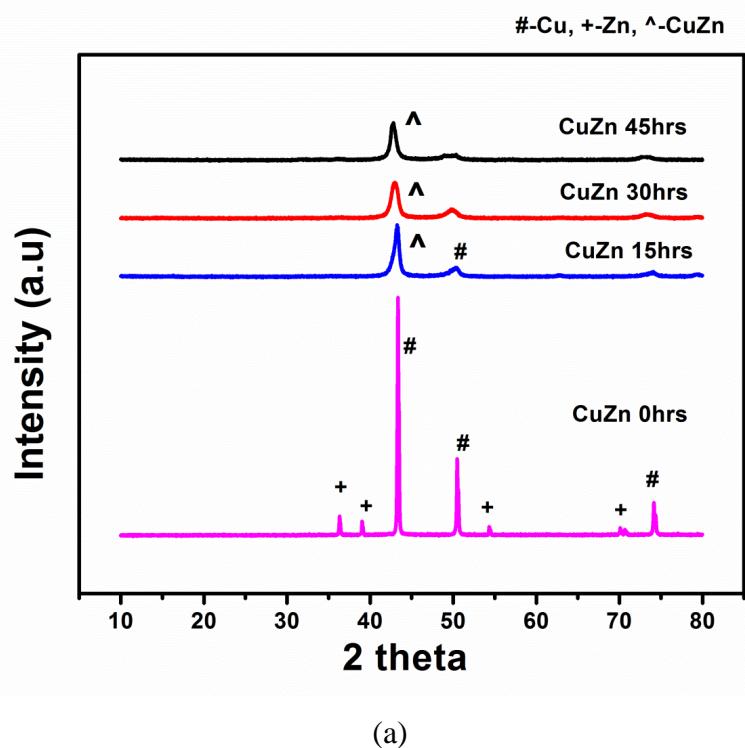


Figure 3.28: XRD pattern of Cu-Zn hybrid particle at time intervals (a) Cu-Zn (75:25),

(b) Cu-Zn (25:75)

The particle size analysis was done after 45 hours milling of powder. The output showing the particle size of each of the Cu-Zn hybrid powders is shown in Figure 3.29 (a-b). It is evident from the graph that the average particle size of the Cu-Zn (75:25) and Cu-Zn

(25:75) powder is 19 nm and 23 nm respectively. The FE-SEM images (figure 3.30 (a,b)) taken for powder milled for 45 hours shows that the shape of the particle is spherical. TEM image (figure 3.31 (a,b)) was also taken after 45 hours, shows that the particle is spherical in shape and nano size is obtained.

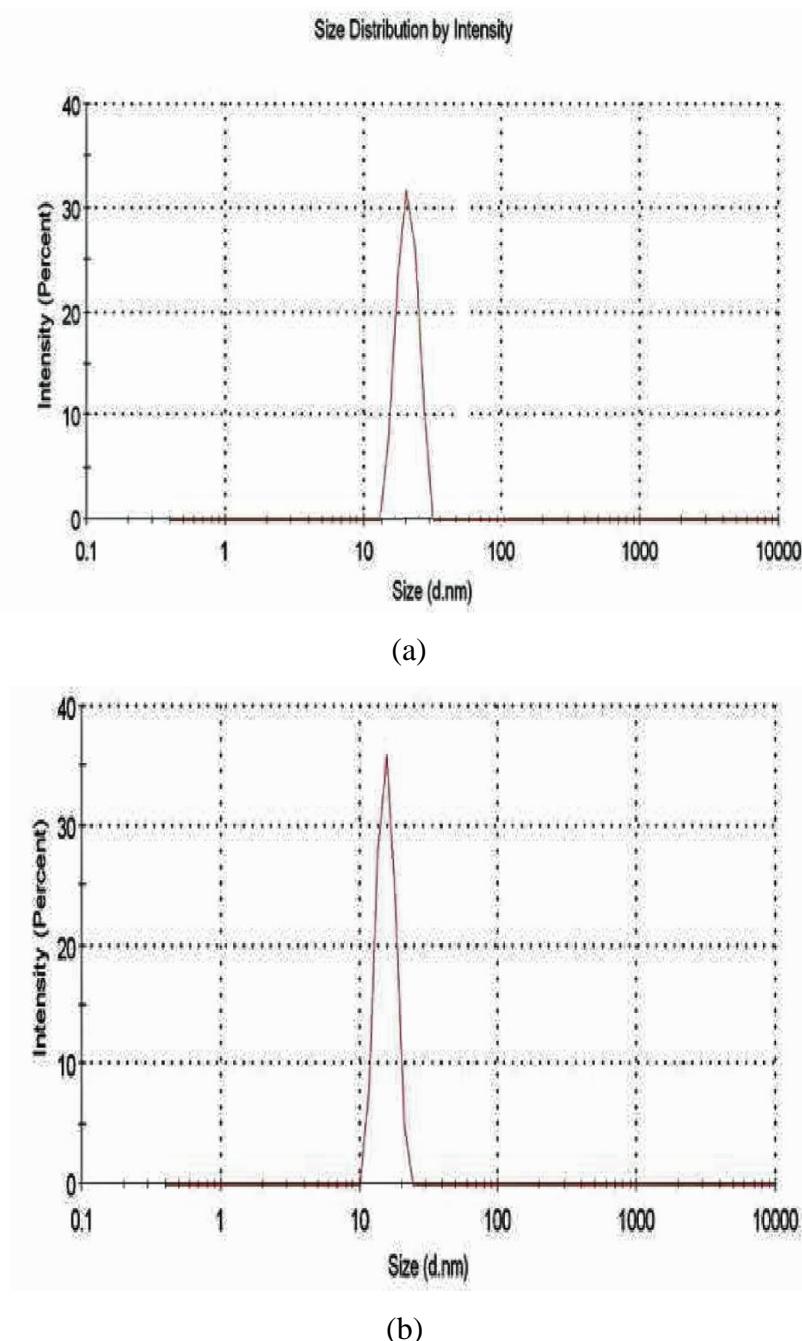
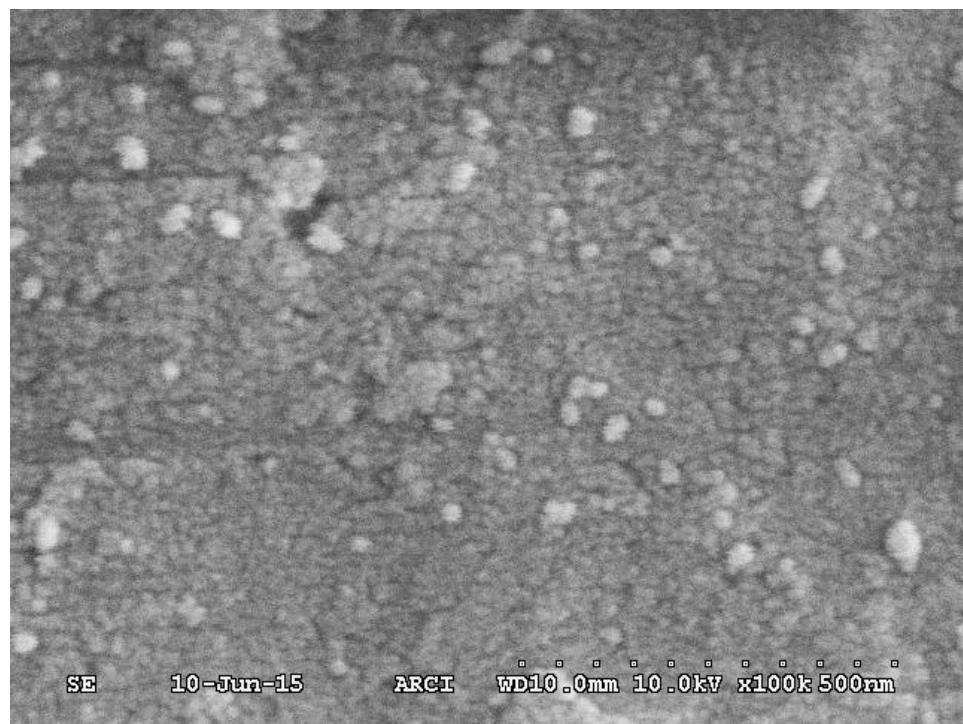
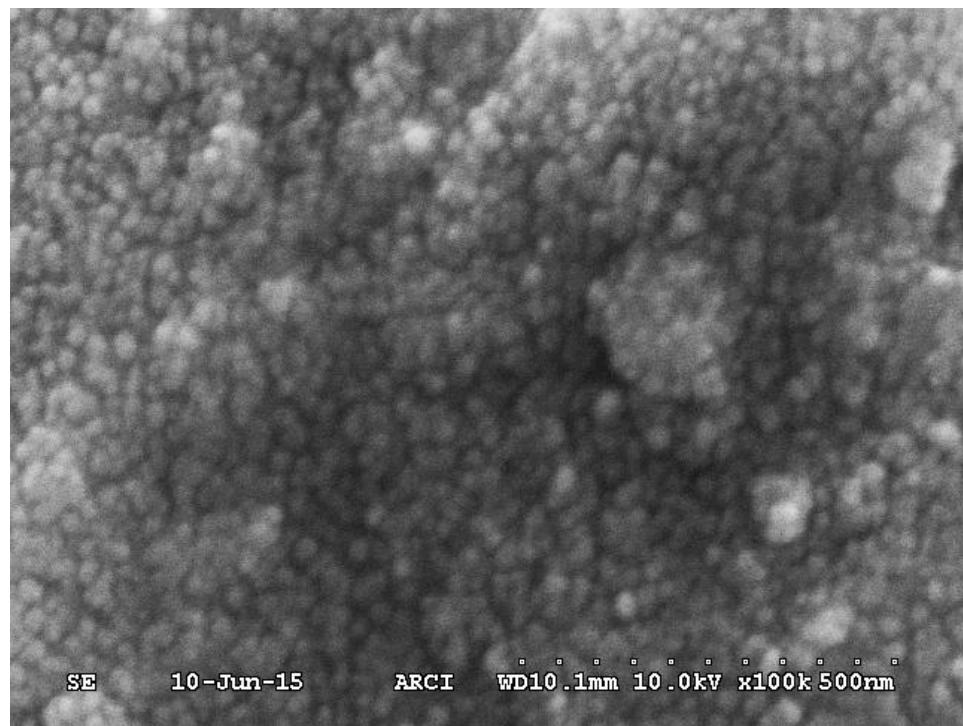


Figure 3.29: Particle size of (a) Cu-Zn (75:25) and (b) Cu-Zn (25:75)

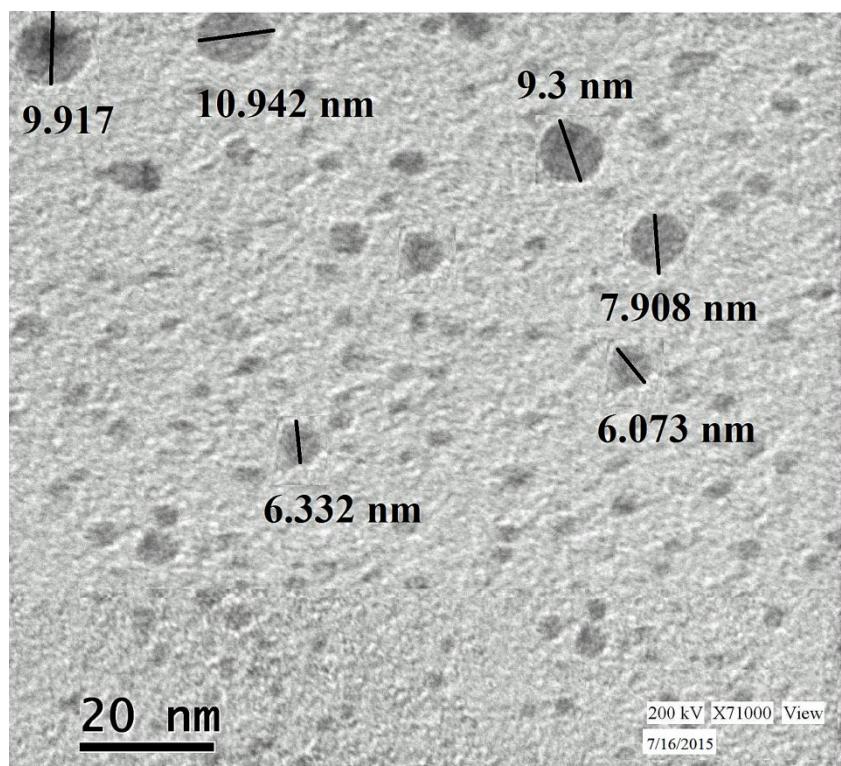


(a)



(b)

Figure 3.30: FE-SEM images of as milled Nanoparticles (a) Cu-Zn (75:25)
(b) Cu-Zn (25:75)



(a)

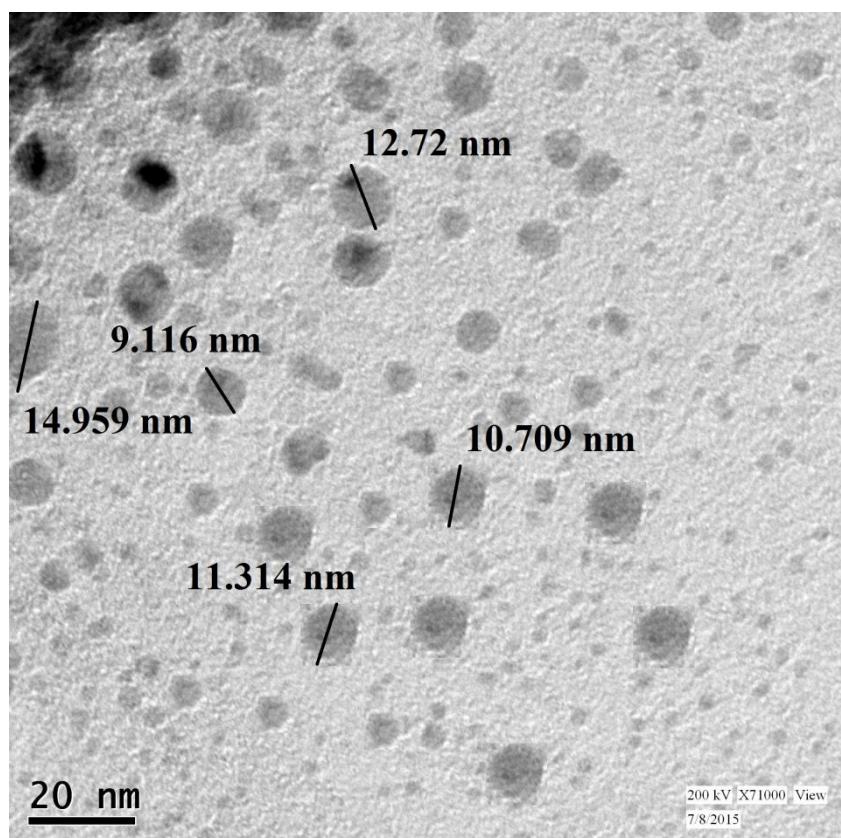
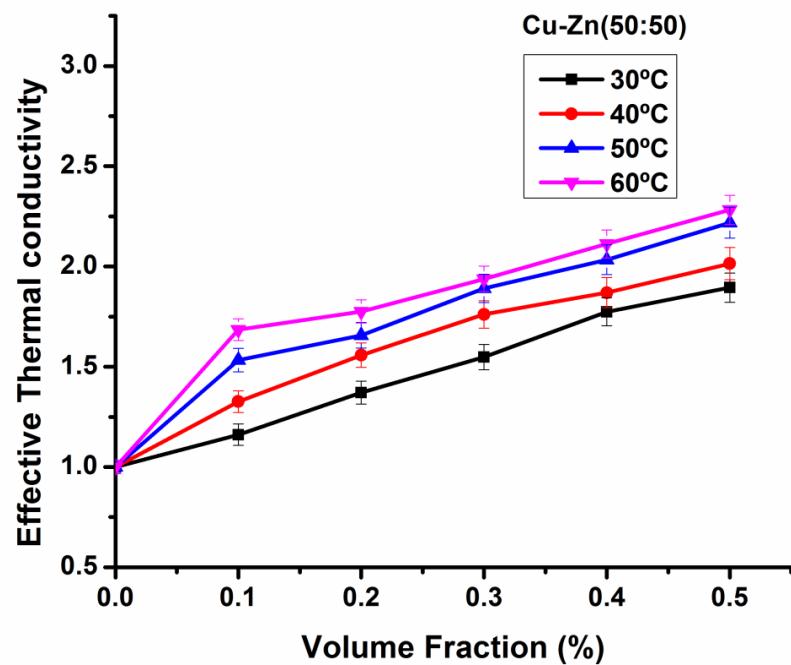


Figure 3.31: TEM images of hybrid nanoparticles (a) Cu-Zn (75:25) and (b) Cu-Zn (25:75)

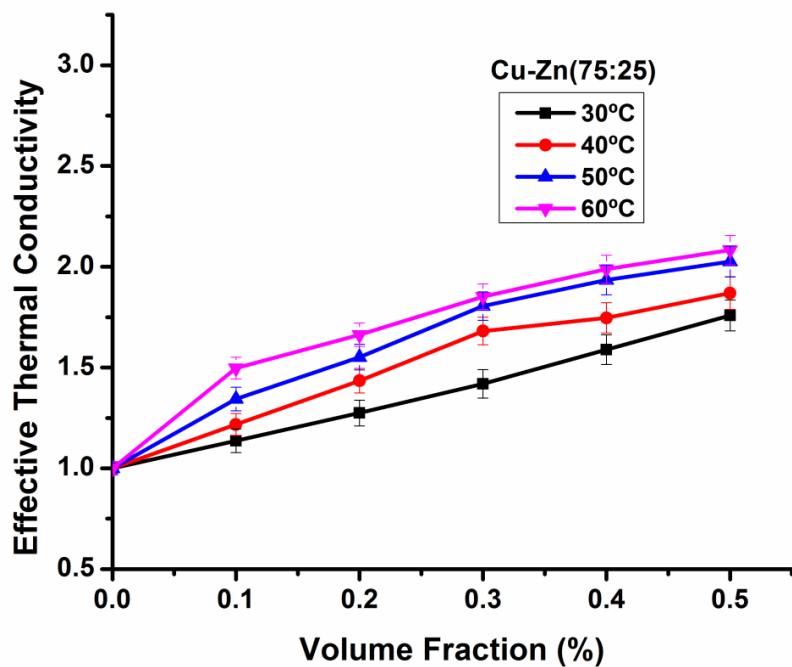
3.4.2 Measurement of Thermophysical properties of nanofluids

3.4.2.1 Thermal conductivity

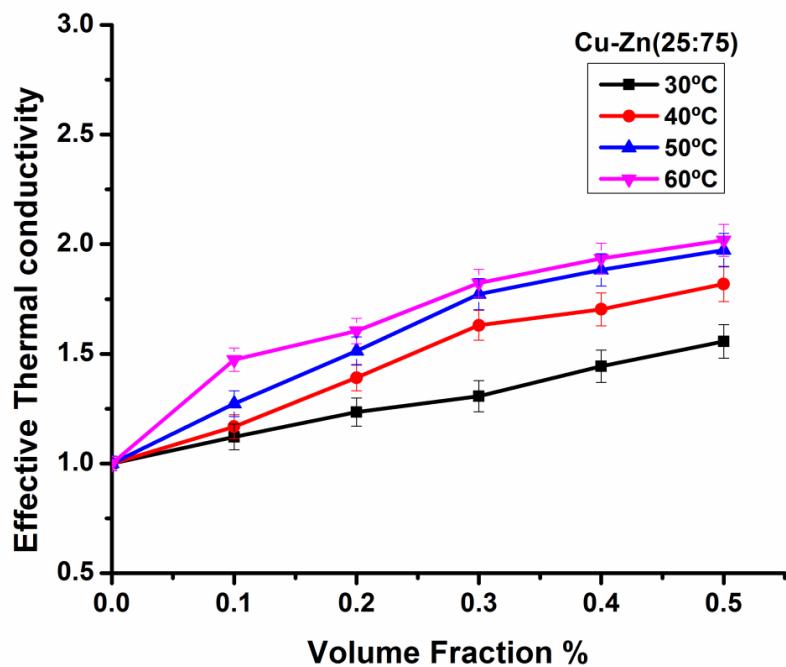
The thermal conductivity of nanofluids with 0%, 0.1%, 0.3% and 0.5% volume concentrations were prepared with 25:75, 75:25 combination and compared it with 50:50 combination. Thermal conductivity was measured in the temperature ranges of 30 °C to 60°C. The temperature of the nanofluid is raised through the heating plate, once the required temperature is reached and maintain equilibrium, a maximum of 10 measurements were recorded for each volume concentrations and temperatures. The thermal conductivity of the base fluid was found to be 0.162 W / m K at 30 °C. From figure 3.32 (a-c), it is observed that thermal conductivity increases with increase in volume fraction and temperature. Enhancement in thermal conductivity of the nanofluid is due to the increased area to heat transfer which increases as the particle size decreases, the high thermal conductivity of the nanopowders dispersed in it and the Brownian motion of the suspended particles which is inversely proportional to the density of the particles. Cu-Zn (50:50) hybrid nanoparticles having equal amount of Cu and Zn by weight possess the advantage of both having high enhancement in thermal conductivity and has lower density as compared to Cu-Zn (25:75) and Cu-Zn (75:25). Being lighter than Cu-Zn (75:25) the motion of the particles when suspended in basefluid was high and at the same time possess better enhancement in thermal conductivity than Cu-Zn (25:75).



(a)



(b)



(c)

Figure 3.32: Thermal conductivity results of (a) Cu-Zn (50:50), (b) Cu-Zn (75:25) and (c) Cu-Zn (25:75)

3.4.2.2 Viscosity Measurement

Initially viscosity is measured for the basefluid i.e., vegetable oil, the results show that shear stress is proportional to shear rate for different temperatures, indicating that the fluid exhibit Newtonian behaviour and the slope represent viscosity which is 41.52 mPa.s. A similar behaviour was observed with nanofluids at 0.1%, 0.3% and 0.5% volume fraction concentration. The viscosity of Hybrid Cu-Zn (50:50; 75:25; 25:75) combinations / vegetable oil nanofluids with different volume fractions were presented in Figure 3.33 and observed that the viscosity of hybrid nanofluid is directly proportional to the volume concentration. When the volume concentration increases the viscosity of the hybrid nanofluid increases. The viscosity is affected by nanoparticle size, volume concentration, nanoparticle material and due to the agglomeration of the nanoparticles dispersed in the base fluid. In this study the hybrid nanoparticles of same size, utmost identical shape (spherical), but having various densities were prepared. It is observed that when the shape and size of the hybrid nanoparticles are utmost identical the viscosity of the hybrid nanofluid is more or less the same at same volume concentrations. From the FE-SEM microstructure of the as milled powder, it is observed that the particles of Cu-Zn (50:50) tend to agglomerate more as compared to other two hybrid nanoparticle, resulting in higher viscosity. The increase in viscosity of Cu-Zn (50:50) is high when compared to the Cu-Zn (25:75) and Cu-Zn (75:25). These nanoparticles when dispersed in liquid tend to arrange into structure that makes the liquid adjacent to the particle surfaces which are less moveable, resulting in increase in viscosity. From the Figure 3.34(a-c) it is also observed that, as the temperature increases from 30 °C to 60°C the slope of the curve reduces, the decrease in viscosity of the hybrid nanofluids with respect to increase in temperature is also identical. With less change in viscosity the motion of the particle is dependent on the densities and heat transfer properties of the hybrid nanofluid are dependent on the thermal conductivity of the hybrid nanoparticles. Hence the hybrid Cu-Zn (50:50) has the advantage of having less density and high thermal conductivity as compared to Cu-Zn (25:75) and Cu-Zn (75:25).

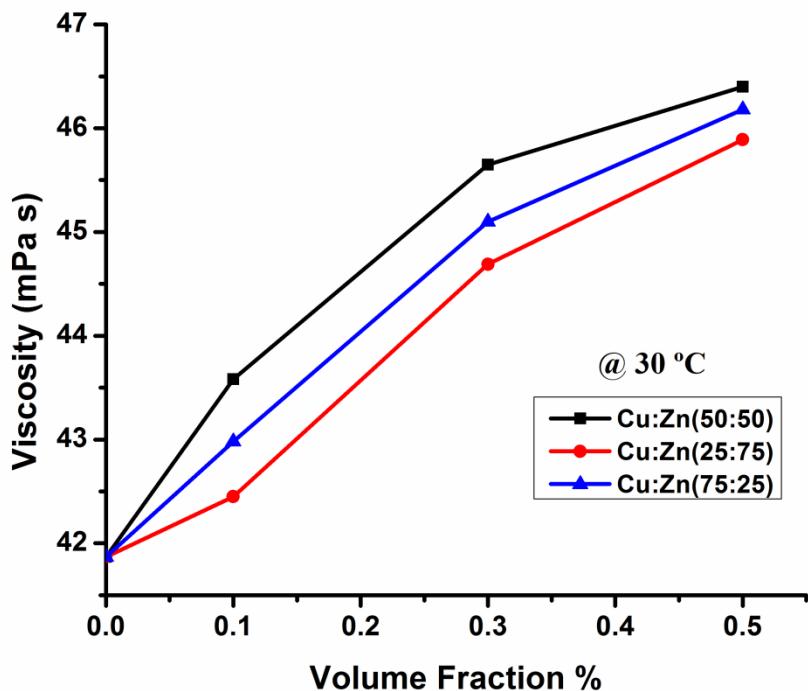
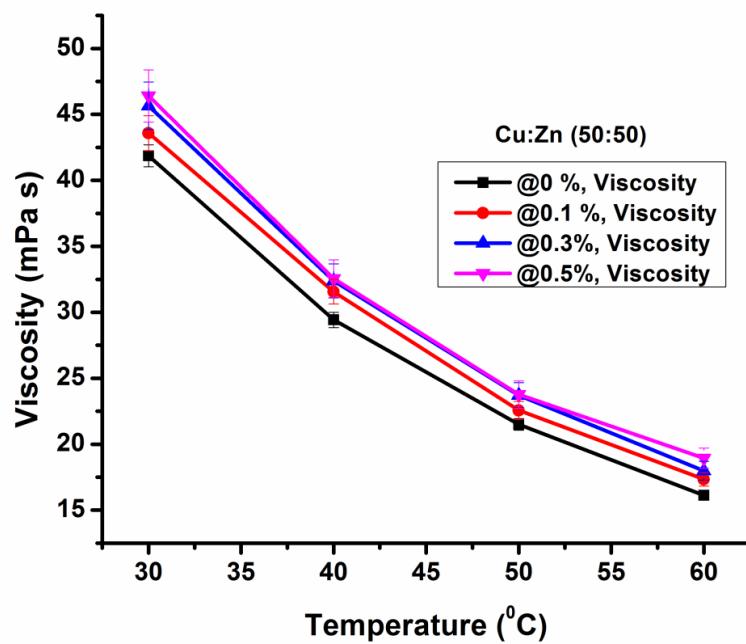
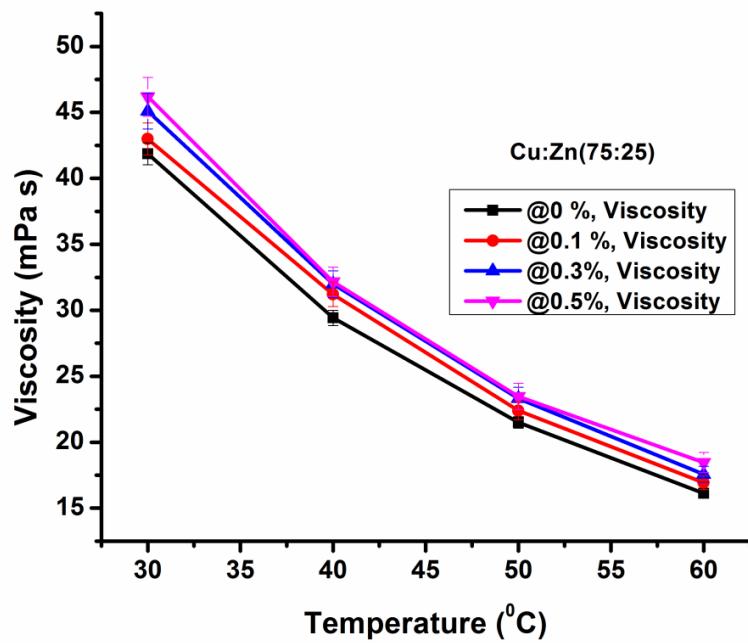


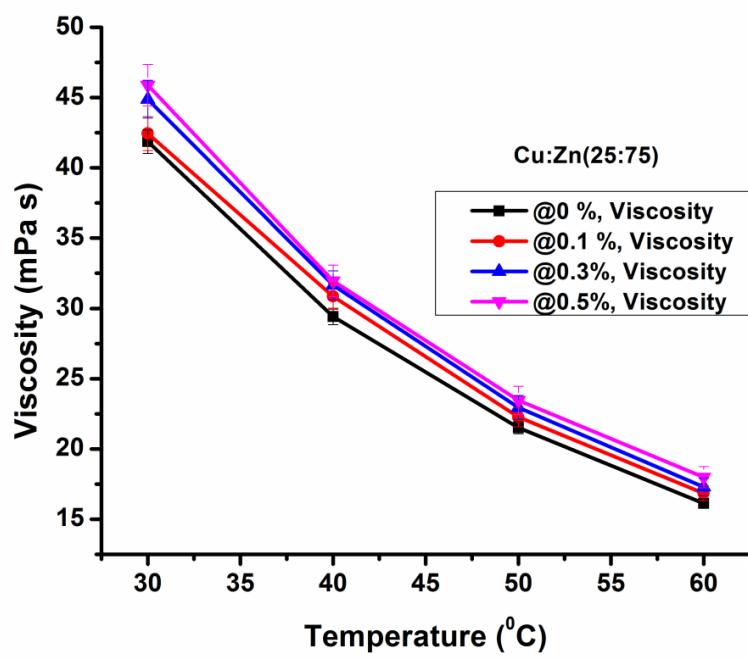
Figure 3.33: Viscosity Results for all three combinations at 30°C



(a)



(b)



(c)

Figure 3.34: Viscosity Results for all three combinations at various temperatures (a) Cu-Zn(50:50), (b) Cu-Zn (75:25) and (c) Cu-Zn (25:75)

3.4.3 Summary

Mechanical milling is used to synthesize Cu-Zn hybrid nanoparticles alloy with 50:50; 75:25; 25:75 combinations using a planetary ball milling. XRD patterns, particle size analyzer and FE-SEM were used for characterization. Detail experimental studies were carried out to examine the thermophysical properties. Cu-Zn with 50:50 combination results in better enhancement in thermal conductivity due to the Brownian motion of the particles.

3.5 PHASE – 4: EFFECT OF Ag PARTICLE ADDITION

In this section, the effect of Ag addition to Cu-Zn hybrid nanoparticle is studied. The intent of this section is to experimentally explore the thermophysical properties of Cu-Zn-Ag/vegetable oil hybrid nanofluids at different volume concentrations and different temperatures.

3.5.1 Synthesis and Characterization of Cu-Zn-Ag hybrid nanoparticles

Procedure similar to 3.3.1 is used to synthesize Cu-Zn-Ag hybrid nanoparticles in this phase.

The XRD patterns of the Cu-Zn-Ag hybrid powder particles at selected intervals are shown in Figure 3.35. It is evident that the Bragg peaks for milled product (after 50 hrs of milling) are broad, suggesting reduction in crystallite size and accumulation of lattice strain. It is also observed that intensity of the peaks decreases due to the decrease of crystallinity of powder during milling. In case of Cu-Zn-Ag alloy powder, the XRD pattern of as received powder shows the peaks of Cu, Zn and Ag. The final milling product is a single phase nanocrystalline material and an alloy is formed after 50 hrs of milling.

The particle size of the milled powders was determined using Malvern Zetasizer (Model: Nano ZS, Malvern). The particle size analysis was carried out for the powder after 50 hours milling by dispersing the powder in acetone. The output showing the particle size of Cu-Zn-Ag hybrid powder is shown in Figure 3.36. It is apparent from the graph that the particle size of Cu-Zn-Ag powder is 28 nm. FESEM (figure 3.37) microstructure was taken of the as milled powder, the morphology of the alloyed nanoparticles showed spherical shape and tiny agglomerates. TEM microstructure (figure 3.38) shows the particle size is less than 30nm and spherical in shape.

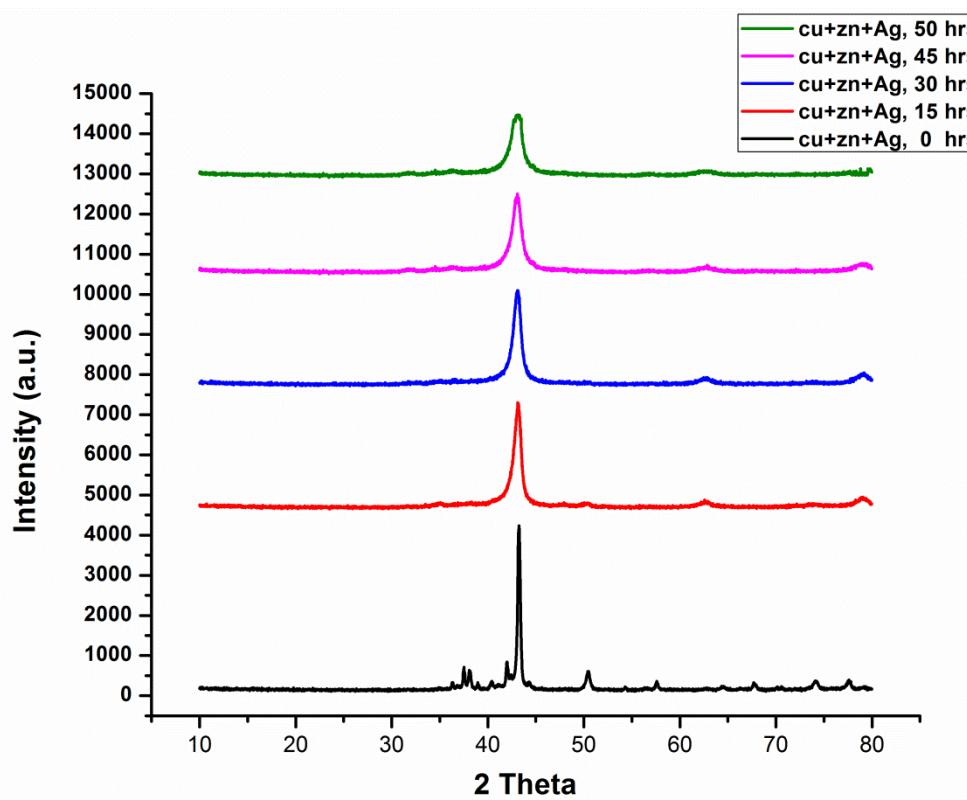


Figure 3.35: XRD pattern of Cu-Zn-Ag hybrid nanoparticle at different time intervals

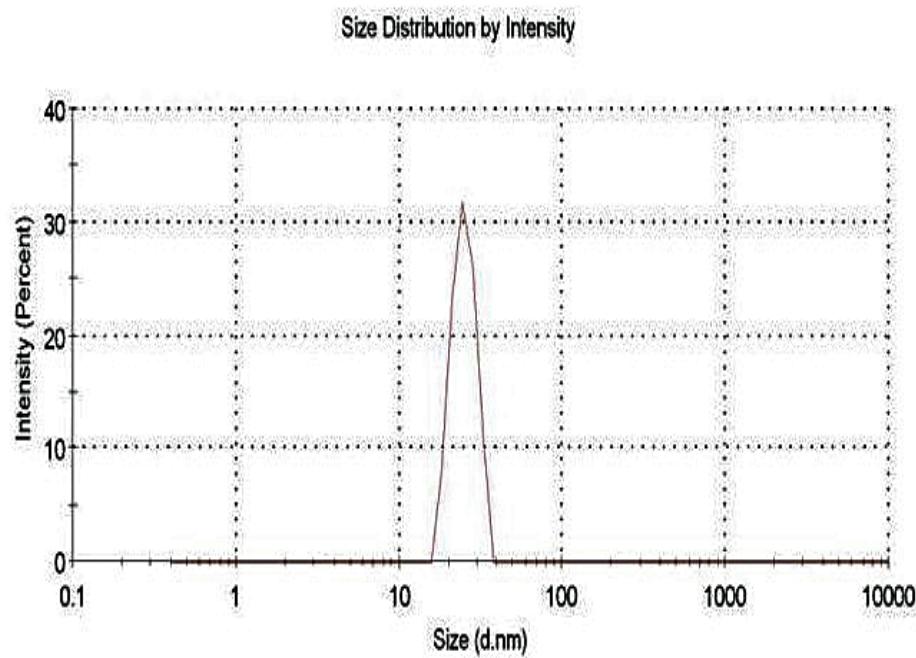


Figure 3.36: Average Particle size of Cu-Zn-Ag hybrid nano powder after 50 hrs of milling

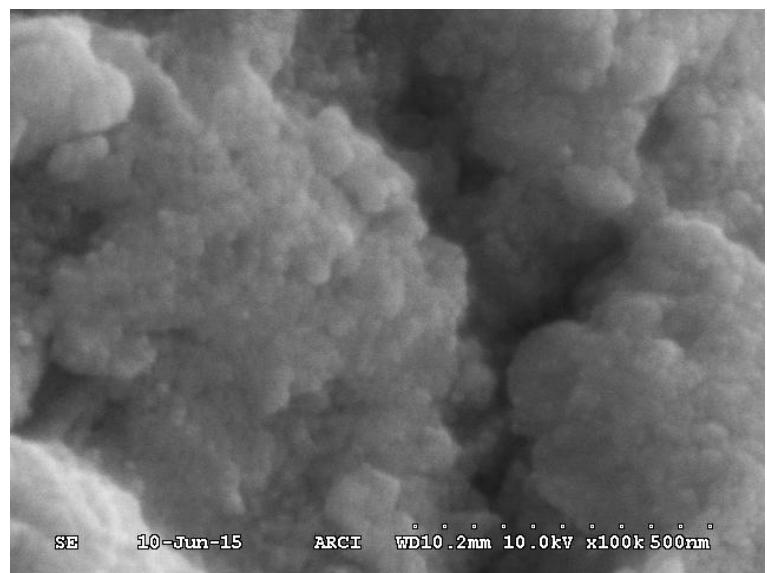


Figure 3.37: FESEM microstructure of Cu-Zn-Ag hybrid nanoparticle after 50 hrs of milling

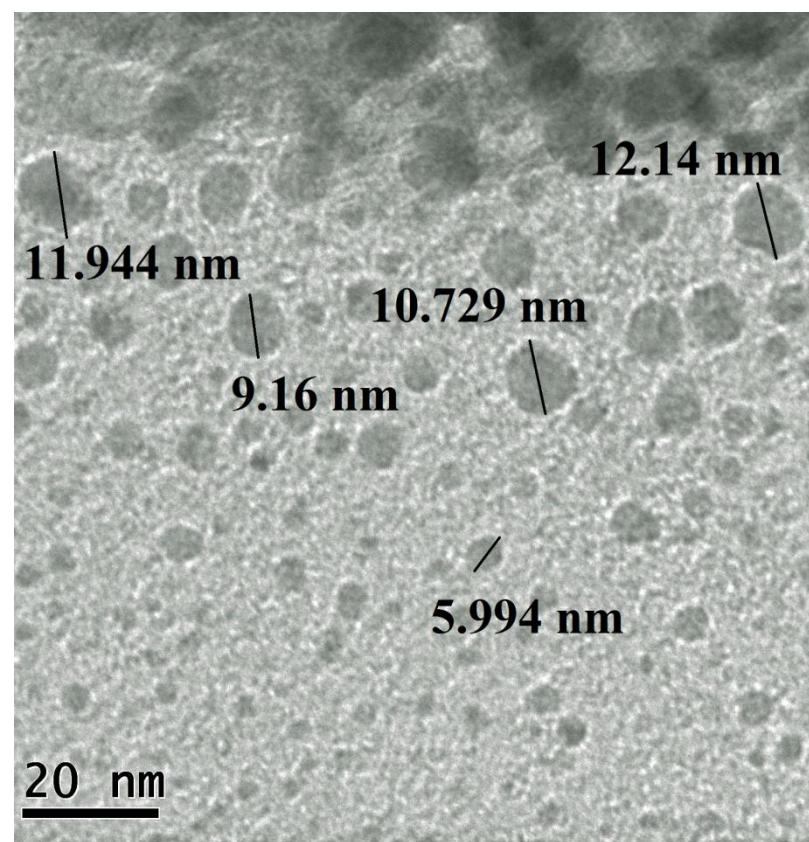
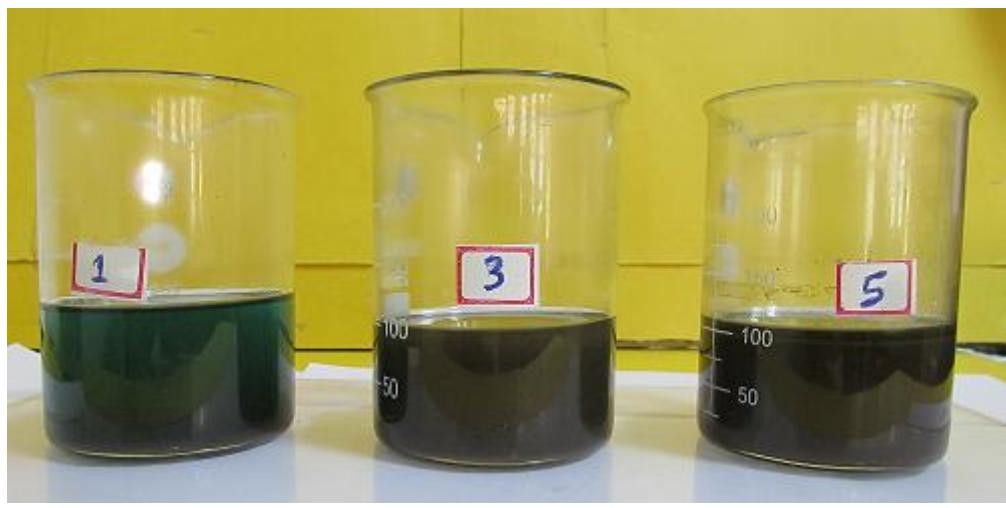


Figure 3.38: TEM microstructure of Cu-Zn-Ag hybrid nanoparticle after 50 hrs of milling

3.5.2 Preparation of nanofluids

Cu-Zn-Ag nanofluid was prepared by dispersing the estimated amount of milled nanopowders in vegetable oil by ultrasonication using an ultrasonic probe sonicator for 2 hours each. Figure 3.39 (a,b) shows the prepared Cu-Zn-Ag nanofluids containing 0.1, 0.2, 0.3, 0.4 and 0.5% volume fraction in 100 ml of the base fluid (vegetable oil) from left to right.



(a)



(b)

Figure 3.39: Cu-Zn-Ag Vegetable oil nanofluid, (a) Initial, (b) After 4 days

3.5.3 Measurement of Thermophysical properties of Cu-Zn-Ag hybrid nanofluids

3.5.3.1 Thermal conductivity of nanofluids

Figure 3.40 shows the thermal conductivity enhancement in of Cu-Zn-Ag/vegetable oil nanofluids with different volume fractions. It is observed that thermal conductivity increases with increase in volume fraction and temperature. The thermal conductivity enhancement of Cu-Zn-Ag vegetable oil nanofluid at 0.5% of volume concentration was 96.77% ($k_{eff} = 1.9677$) when compared to basefluid at 30°C. The increase in thermal conductivity of the nanofluid is due to the increased area to heat transfer which increases as the particle size decreases, the high thermal conductivity of Ag nanoparticle dispersed in it.

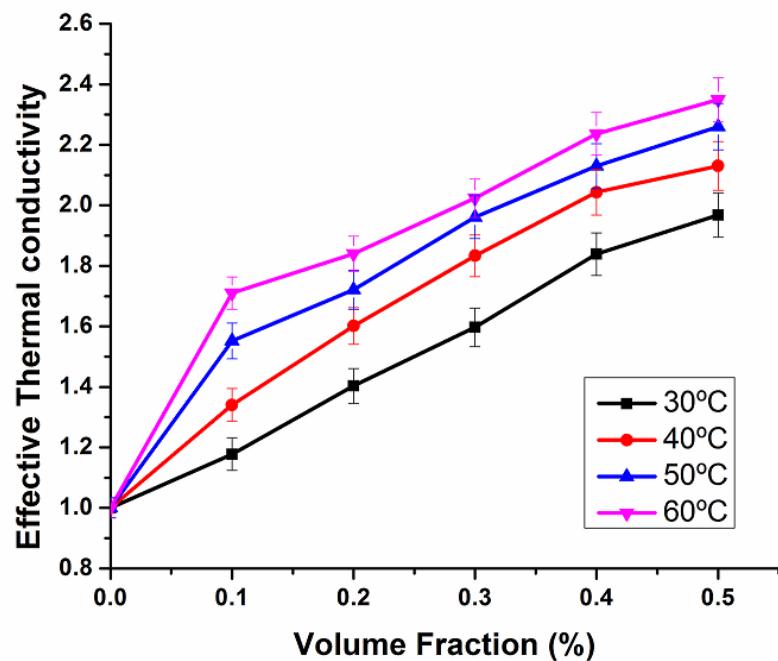


Figure 3.40: Thermal conductivity results.

3.5.3.2 Viscosity of nanofluids

From the Figure 3.41it is also observed that, as the temperature increases from 30 °C to 60 °C the slope of the curve reduces, this indicates that there is reduce in viscosity with increase in temperature. The viscosity is affected by nanoparticle size, volume concentration, nanoparticle material and due to the agglomeration of the nanoparticles dispersed in the base fluid. The viscosity enhancement for hybrid Cu-Zn-Ag/vegetable oil nanofluid was maximum at a volume concentration of 0.5% with an increase of 13.7% at 30°C. As the volume fraction

of nanoparticles increases, the contact between particles and base fluid also increases; which in turn increases the total contact area as well as the resistance to movement of the nanoparticle in the base fluid, which results in increase in viscosity of the hybrid nanofluids.

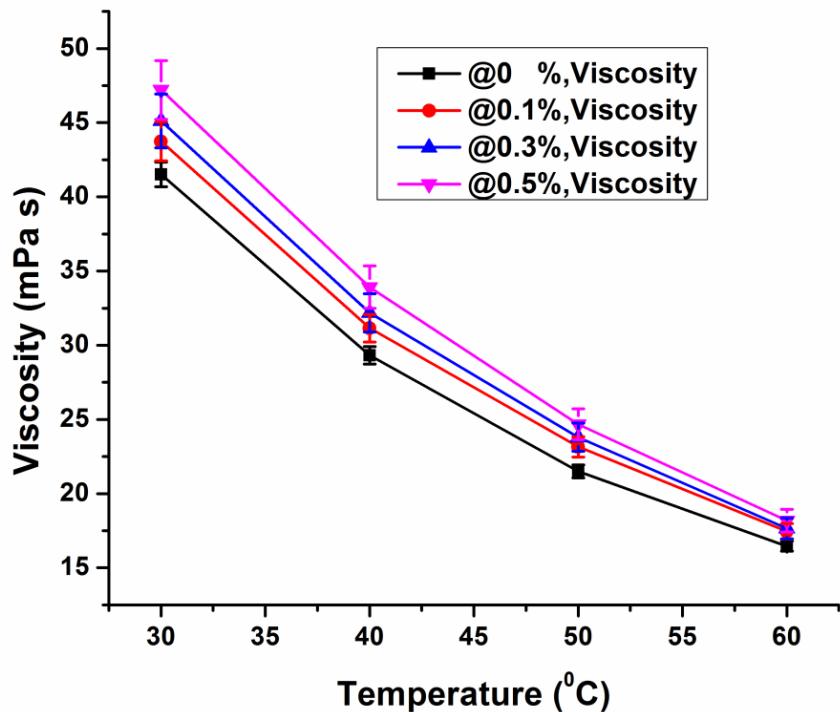


Figure 3.41: Viscosity results

3.5.3.3 Comparison of effective thermal conductivity all prepared hybrid alloys of Cu-Zn (50:50, 75:25, 25:75) and Cu-Zn-Ag (40:50:10)

Figure 3.42 shows the comparison of effective thermal conductivity of all prepared hybrid alloys of Cu-Zn (50:50, 75:25, 25:75) and Cu-Zn-Ag (40:50:10). In phase 3, the results illustrated that Cu-Zn (50:50) yielded better result compared to Cu-Zn (75:25, 25:75). With addition of Ag to Cu-Zn (50:50) the thermal conductivity of the hybrid nanofluids increases by 9%. This enhancement comes at higher cost. Considering the sustainability criteria hybrid nanofluid with Cu-Zn (50:50) can be adopted as cutting fluids.

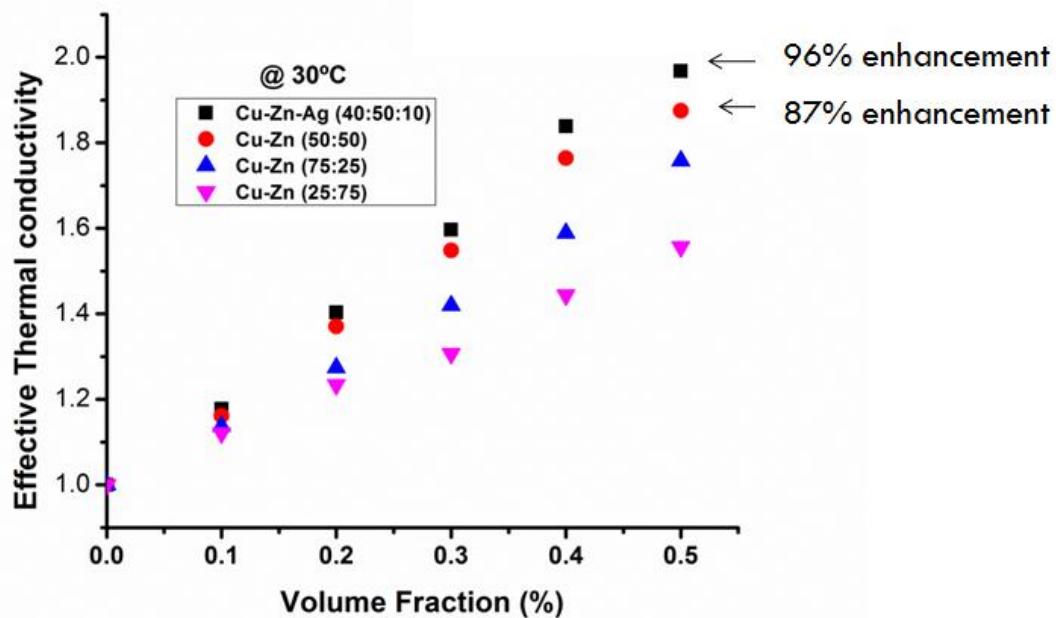


Figure 3.42: Effective thermal conductivity of all prepared hybrid alloys of Cu-Zn (50:50, 75:25, 25:75) and Cu-Zn-Ag (40:50:10)

3.5.4 Summary

The thermal conductivity and viscosity of Cu-Zn-Ag hybrid nanofluid were determined at various temperatures intervals (30, 40, 50 and 60°C) and for five nanoparticle weight concentrations (0.1, 0.2, 0.3, 0.4 and 0.5 % volume). The enhancement in thermal conductivity was found to be 96.77% ($k_{eff} = 1.9677$) and enhancement in viscosity was 13.7% when compared to the basefluid.

CHAPTER-4

MACHINING OF INCONEL 718 USING HYBRID Cu-Zn (50:50) NANOFUIDS AS CUTTING FLUIDS

Chapter Synopsis

Nickel based alloys are extensively used in aerospace industries, marine equipments, nuclear reactors etc. as they exhibit higher strength, resistance to corrosion, fatigue, creep and erosion. In nickel based alloys Inconel 718 is the most frequently used metal due to its mechanical properties and thermal properties. Inconel 718 does not have good machinability when compared to aluminium and steels. High temperatures are generated while machining Inconel 718 and increase further with increase in CSs, this reduce the tool life and deteriorate the surface quality etc. The machining industry has been using conventional cutting fluids like oil based and water based fluids, to lubricate and cool the cutting zone. These conventional cutting fluids create environmental and health problems. The increasing attention to the environmental and health impacts of industry activities by governmental regulation and by the growing awareness in society is forcing manufacturers to reduce the use of lubricants. An alternate to flood coolant and conventional lubricants is minimum quantity lubrication (MQL) with biodegradable oils. In this chapter an attempt was made to make nano lubricant based MQL cutting fluids for near dry machining.

From chapter 3, we have seen that the thermal conductivity of the basefluids can be enhanced by using hybrid nanofuids. A combination of Cu-Zn with 50:50 wt percentage hybrid nano particles showed better results than the other combination of Cu-Zn (25:75; 75:25). The vegetable oil represented better thermophysical properties when compared to conventional oils. Having narrow down to the type of base fluid and the particles to be incorporated in it, the testing of the fluid is done by using the developed fluids as cutting fluid for machining of Inconel 718. In this chapter turning of Inconel 718 is carried out and a comparative study is done with various lubricating conditions. Various lubricating conditions

are used and compared for output responses such as CFs (CF), surface roughness (SR) and residual stresses.

This chapter mainly consist of following sections.

4.1 Material and methods:

In this section materials and methods used for synthesis, characterization, preparation and measurement of thermophysical properties of nanofluids is discussed.

4.2 Phase 6 - Turning of Inconel 718 Using Hybrid Cu-Zn Nanofluids:

The effect of in-situ developed hybrid nanofluid (MQL/Nanofluid) is compared with dry and MQL/vegetable oil cutting conditions. Experiments were carried out according to L9 (dry) and L18 (MQL/vegetable and MQL/Nanofluid) array and the ANOVA is used to analyse the effect of cutting medium on (1) CFs and (2) Surface Roughness.

4.3 Phase 7 - Residual Stress Analysis of Inconel 718:

In this phase, study was carried out to study the effect of lubricating conditions on residual stresses. The residual stresses were measured before and after machining.

4.1 MATERIALS AND METHODS

4.1.1 Machine tool

The cutting tests performed in this study were conducted using magnum precision lathe. A photograph of the lathe along with details of its capability is shown in Figure 4.1 and Table 4.1.

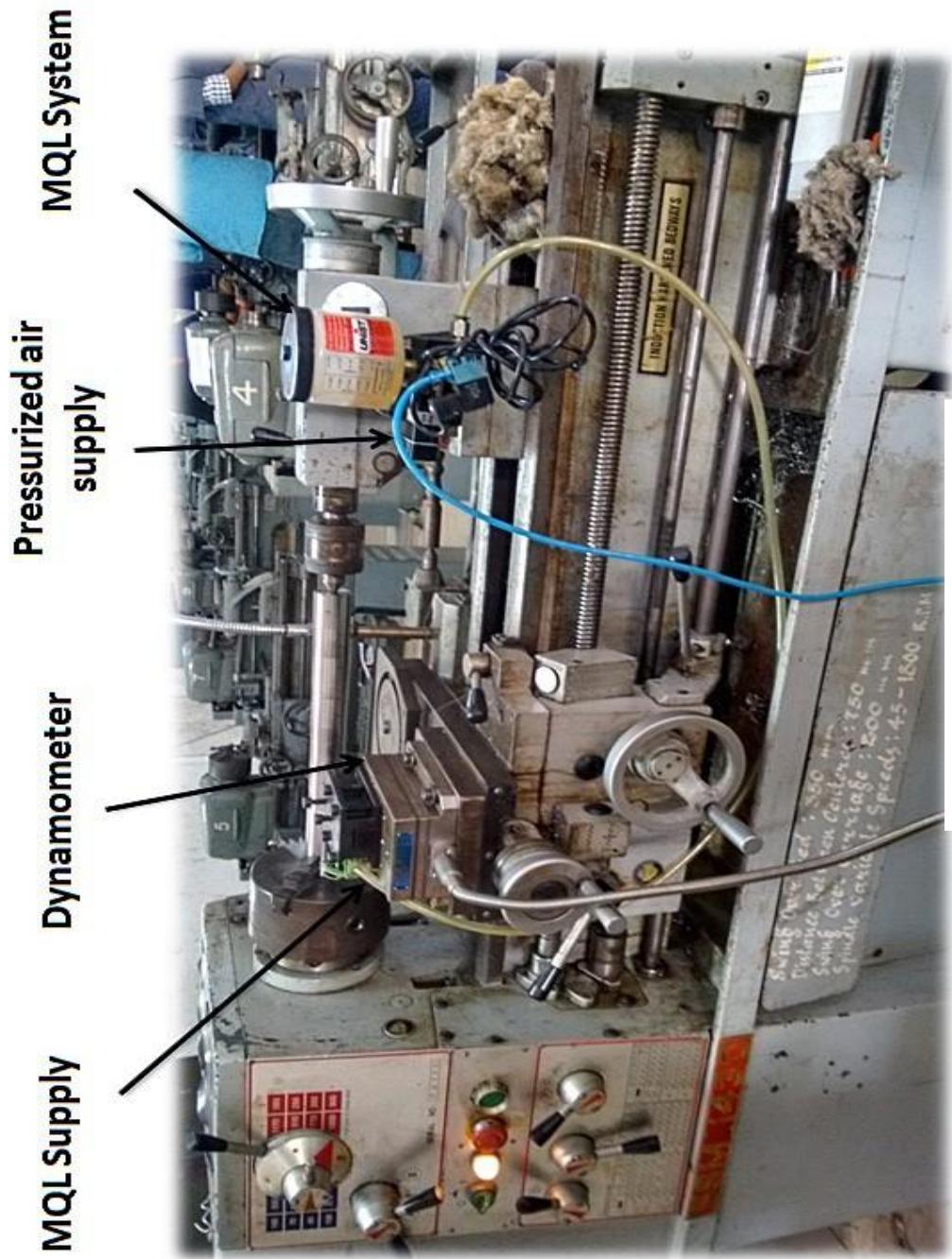


Figure 4.1: Machine tool setup

Table 4.1: Specification of Machine tool

Make	Magnum
Spindle variable Speed	45 - 1800 rpm
Swing over bed	350 mm
Distance between centers	750 mm
Swing over carriage	200 mm
Induction Hardened bedways	

The schematic diagram of the experimental setup is shown in figure 4.2, showing the MQL setup, tool and insert used.

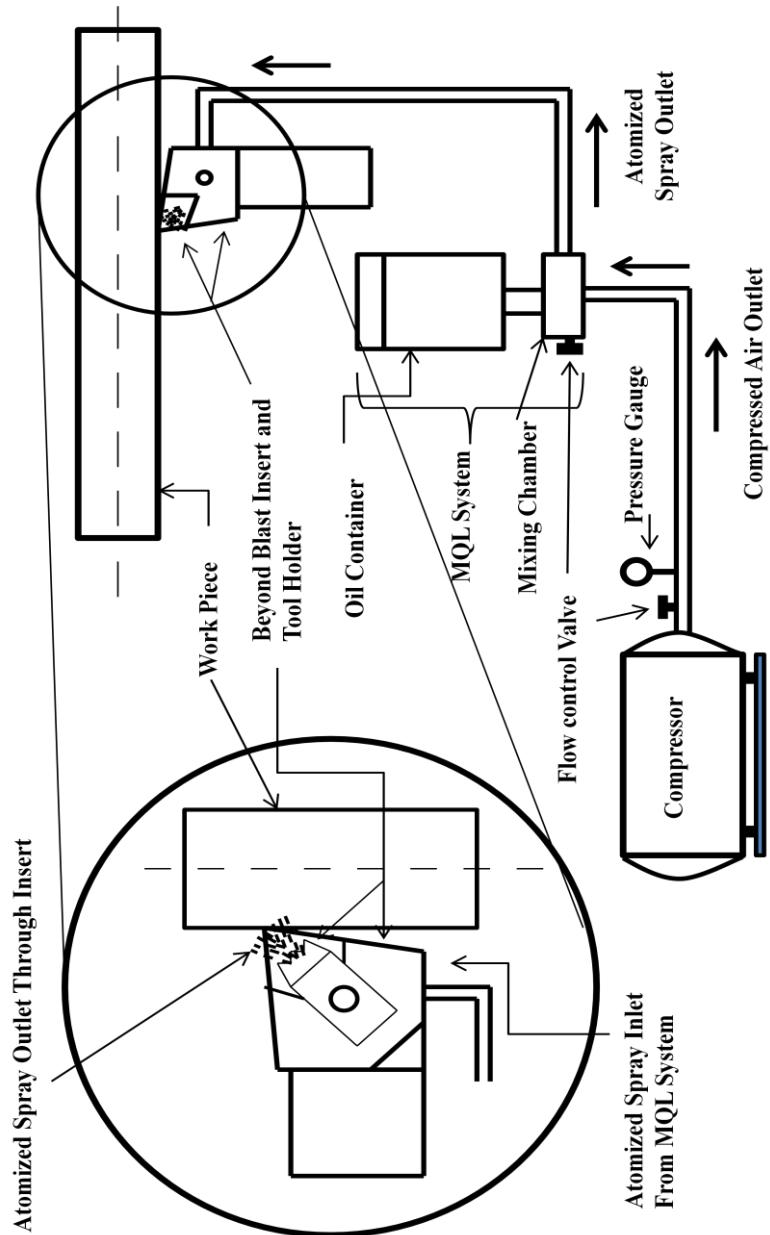


Figure 4.2: Schematic diagram of experimental tool setup

4.1.2 Workpiece material:

The work material used in the study is Inconel 718, as received material was prepared under ASTM B637 standard with \varnothing 40 mm and length 300 mm. The EDAX and microstructure of the as received material is as shown in figure 4.3 (a and b).

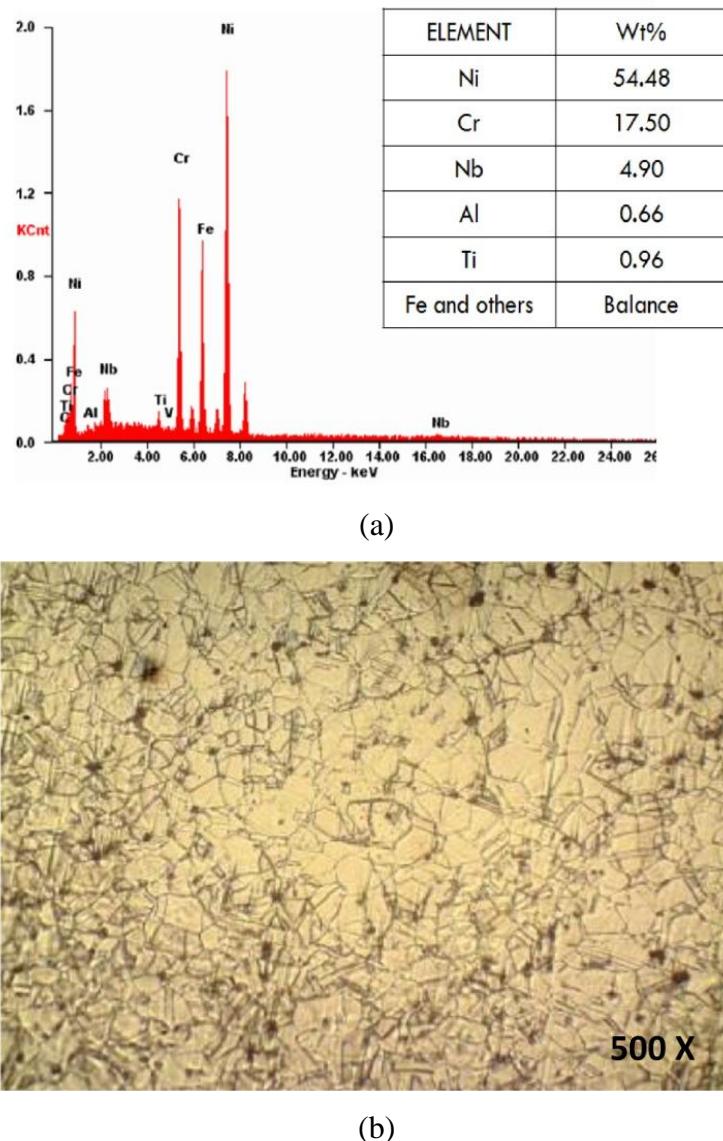


Figure 4.3: EDAX and microstructure of Inconel 718

4.1.3 Insert and holders:

The inserts used were TiAlN coated beyond blast insert from kennametal with ISO designation CNGG 120408. The insert holder used was from kennametal beyond blast with ISO designation MCLNL 2525 M12BB are shown in figure 4.4. Beyond blast tool holder from kennametal is a new class of through insert delivery system, designed to machine

titanium and other high temperature alloys systems. Beyond blast system delivers coolant directly and precisely to the cutting edge, significantly reducing temperatures at the point of the cut.

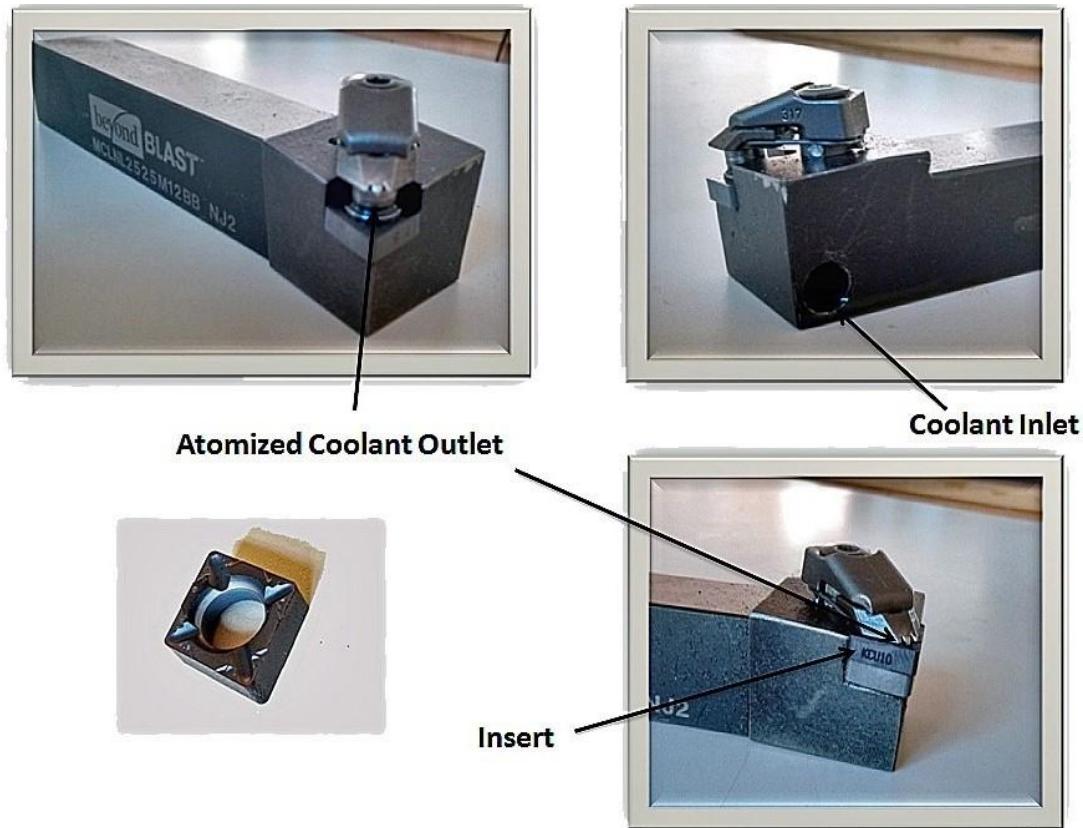


Figure 4.4: Tool holder and inserts used for turning Inconel 718

4.1.4 MQL setup:

Unist coolubricator JR was used to create air/oil mist which continuously produces precisely metered lubricant. The figure 4.5 shows the image of MQL system. The system operates when the adjustable pulse generator automatically cycles the metering pump. It has provision to adjust outputs from 4-200 injection cycles per minute with 0.1 to 3.0 drops of lubricant per cycle. A durable, vented 474 ml. reservoir holds more than 14,400 drops of lubricant. Lubricant is combined with air in the patented Co-Axial Nozzle tip. It maintains atomization and distribution of the liquid consistent regardless of the hose length. A heavy-duty magnetic base at the hose/nozzle connection makes it easier to relocate and position the nozzle at the friction points.

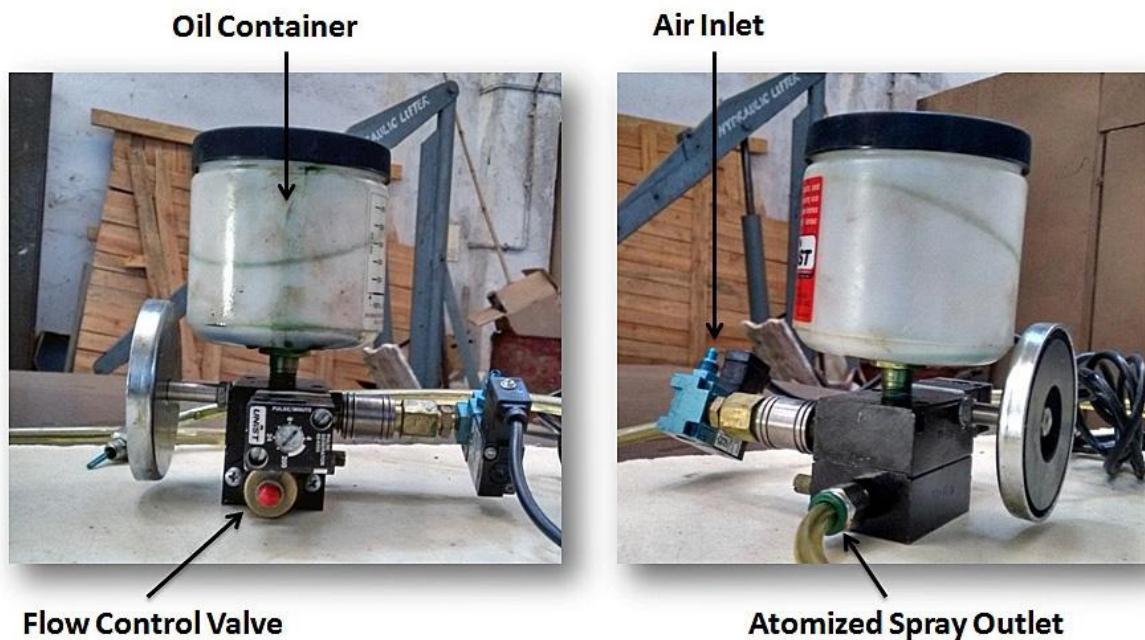


Figure 4.5: Nano lubrication MQL Setup

4.1.5 Compressed air supply:

Compressed air supply to the MQL system is given with the use of compressor. Figure 4.6 shows the reciprocating type compressor used for experimental purpose. There is a provision to control the output air pressure using a flow control valve.

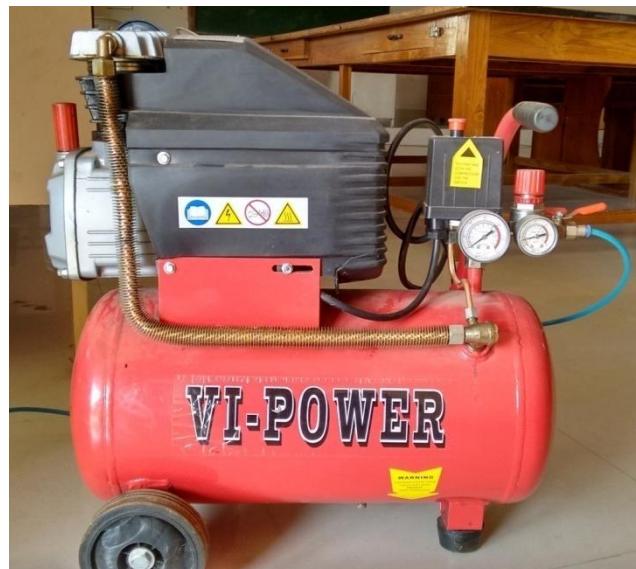


Figure 4.6: Compressor used for supply of pressurized air

4.1.6 Cutting Force measurement:

CFs are a function of various factors, including tool geometry, work material, work piece geometry cutting conditions and the sense of machining with respect to the surface. Forces vary greatly with the machining strategy, both in magnitude and direction. The CFs were measured using a 6 component dynamometer (Kistler make, model 9257B).

4.1.6.1 The Measuring System

The reliability of the system is also extremely important when measuring CFs. The arrangement for the measuring system is as shown in figure 4.7. In order to be able to guarantee this, KISTLER places great emphasis on coordinating components properly at the planning stage. In the measuring system for CFs, particular attention is given to the stability and sealing of individual components against cutting fluid and other contamination. All dynamometers and cables are ground-isolated and guarantee interference-free operation.

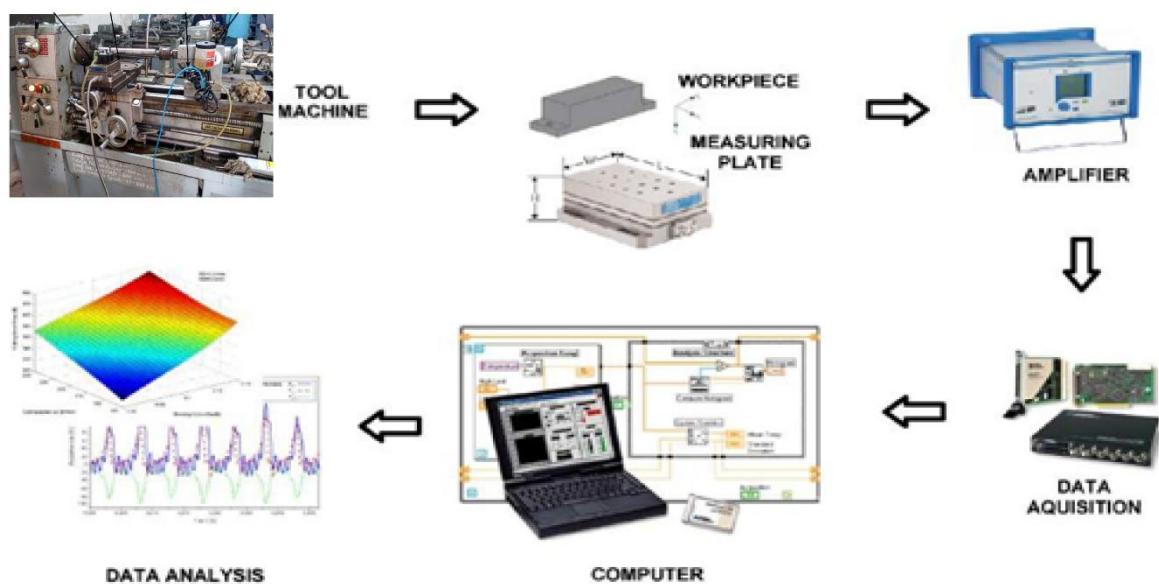


Figure 4.7: The CF measuring system using 6 component dynamometer

4.1.7 Surface Roughness Measurement:

To measure the surface roughness on the machined surface of the Inconel 718 workpiece material, a Taylor Hobson Talysurf Surtronic S100 was used as shown in Figure 4.8. The specification of equipment used is shown in table 4.2.

Table 4.2: Specification of Talysurf Surtronic S100

Measurement	Best capability
Roughness standards	$\pm (2\% + 0.004 \mu\text{m})$
Workpiece surface texture (Ra)	$\pm 3\%$ of measured value per track

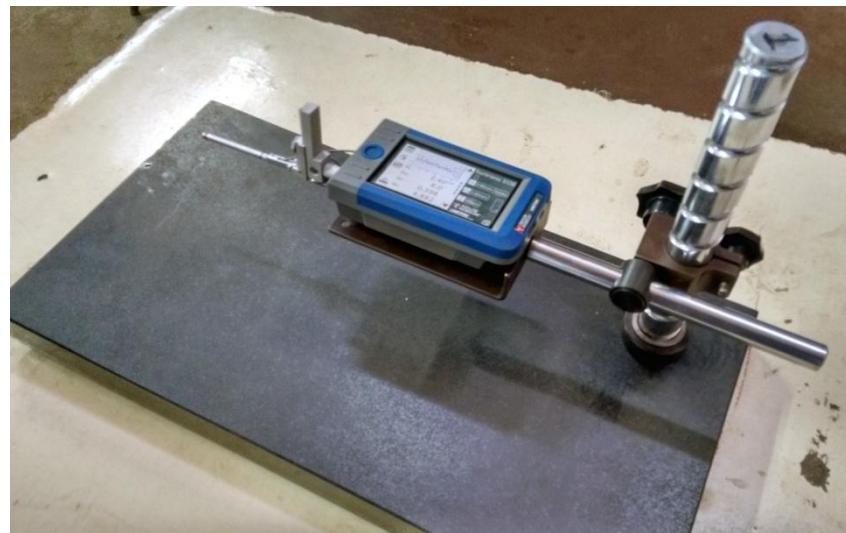


Figure 4.8: Taylor Hobson Surface roughness tester

4.1.8 Furnaces:

Two types of furnaces were used for heat treatment of Inconel 718 workpiece.

4.1.8.1 Box furnace:

It's the simplest type of furnace, which has only one stage of operation, either on or off. The picture of the box furnace used is shown in figure 4.9.



Figure 4.9: Box Furnace

Table 4.3: Specifications of Box furnace

Make	Swan equip
Model	1151
Temperature range	1200°C
Temperature control	Electronic PID based ON/Off
Heating type	Standard
Safety feature	Broken thermocouple and temperature over shoot problem

4.1.8.2 Muffle Furnace:

It's an advance type of furnace, which features intelligent controller integrated design. The figure 4.10 shows the muffle furnace used. It uses advanced lightweight materials (0.3 density) molding, compared with the traditional furnace 1/2 lighter heating rate doubled, saving energy. With a new digital instrument, intelligent temperature control, reducing visual reading and human operator error, greatly improving the work efficiency; with a variety of protection features, improved security and reliability. Programming facility is also available, which can be set according to requirements of different experimental heating process, with over-temperature, with a variety of settings and adjust the interface to suit all require different environmental conditions. The specification of muffle furnace is shown in table 4.3.



Figure 4.10: Muffle Furnace

Table 4.4: Specification of the Muffle Furnace

Model	BST/AF/1400
Working Temperature	1300 °C
Maximum Temperature	1400 °C
Thermocouple	S Type
Heating Element	SiC Heater
Temperature Control	30 steps programmable and PID automatic control
Temperature Accuracy	± 1 °C
Outer Construction	Powder Coated Mild Steel
Inner Chamber	Fiber Alumina Refractory Liner

4.1.9 Residual stress measurement:

Residual stress measurement for the workpiece was carried out using iXRD portable type equipment, manufactured by PROTO. The picture of the residual stress measuring equipment is as shown in figure 4.11 and its specification in table 4.4.

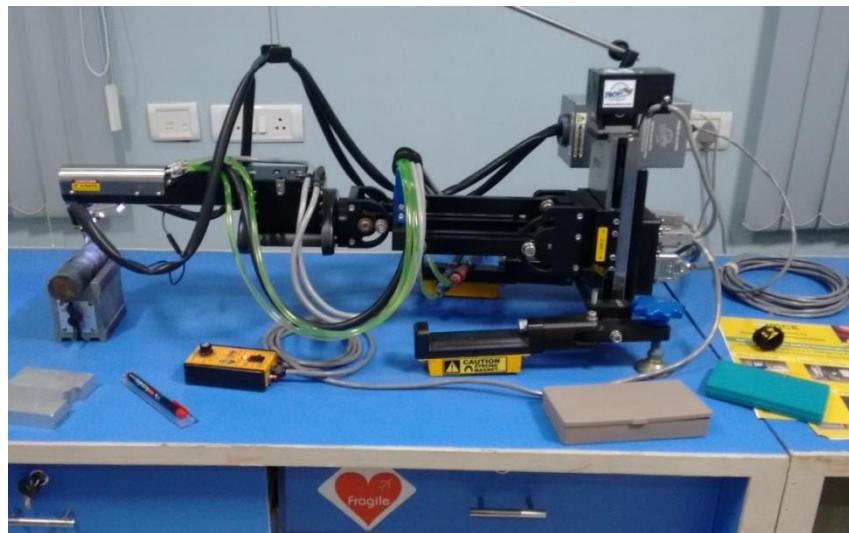


Figure 4.11: iXRD portable type Residual stress measurement system

Table 4.5: Specification of iXRD portable type Residual stress measurement system

Dimensions (L*B*H)	40*30*20 mm
Recommended Part Size	Unlimited
Goniometers	MG40, MGR40, MG30, MG15

Field Stands	FS2, FS4
Mapping Stages (X,Y)	FS4: 100*100mm
Focusing Axis (Z)	FS2: 100 mm
Geometry	iso (omega)
X-ray Tubes	Fine focus 30 mm diameter metal ceramic
X-ray Tube cooling	Integrated recirculating liquid-to-air heat exchanger
X-ray Detectors	Proprietary dual position sensitive scintillation detectors
Detectors Width (2θ)	Standard 18.4°, wide 29.5°
2θ Range	Residual stress: 123°-171°
Power Requirements	90-240 VAC, 50/60 Hz, single phase
System Compliance	ASTM E915, ANSI N43.2, CE

4.2 Phase 6: TURNING OF INCONEL 718 USING HYBRID Cu-Zn NANOFUIDS

Turning test was carried out on magnum precision lathe (Fig 4.1). The aim of this present study is to haul out the influence of the cutting fluids medium on CFs and SR. The percentage contribution of the process parameters on the responses with relation to the cutting fluid medium is also studied. The process parameters for machining Inconel 718 is shown in table 4.5. Design of experiment layout is used to carry out experiments with process parameters and there levels are as shown in table 4.6. L9 orthogonal array was considered for conducting experiments in dry condition, with process parameters- speed, feed and doc. L18 orthogonal array was used for MQL/vegetable oil and MQL /nanofuid based experiments, with process parameters- speed, feed, doc, volume of fluid and air pressure. SR of the machined surface was measured using surftronic duo from taylor hobson.

Table 4.6: Process parameters for machining of Inconel-718

Factors	level 1	level 2	level 3
DOC (mm)	0.5	0.75	1
Feed (mm/rev)	0.082	0.137	0.178
Speed (m/min)	80	120	160
Volume of fluid (ml/hr)	40	80	120
Air pressure (bar)	3	5	7
Cutting fluid	Dry	MQL/Veg	MQL/NF

Table 4.7: Experimental results for Inconel-718 (For Dry turning (L9))

Run order	DOC (mm)	Feed (mm/rev)	Speed (m/min)	CF (N)	SR (μm)
1	1	2	2	249	1.8
2	3	3	2	601	2.51
3	3	1	3	358	1.41
4	1	3	3	314	2.1
5	1	1	1	195	1.28
6	3	2	1	556	1.61
7	2	2	3	398	1.52
8	2	3	1	480	1.96
9	2	1	2	269	1.73

L18 table For MQL/ Vegetable oil and MQL/ Nanofluid

Run Order	CF	DOC (mm)	Feed (mm/rev)	Speed (m/min)	Volume of fluid (ml/hr)	Air Pressure (bar)	Cutting Force (N)	Surface Roughness (µm)
1	1	2	1	2	1	2	275	1.45
2	2	2	3	3	1	2	415	1.60
3	1	3	1	3	2	1	332	0.83
4	2	2	1	1	2	3	255	1.3
5	2	1	2	3	1	1	208	1.14
6	2	2	2	2	3	1	339	0.9
7	2	1	3	1	2	2	310	1.8
8	2	3	1	3	3	2	302	0.78
9	1	1	1	1	1	1	195	1.07
10	1	3	3	2	1	3	596	1.42
11	1	2	2	3	2	3	356	1.38
12	2	1	1	2	3	3	175	0.74
13	1	1	2	2	2	2	227	1.39
14	1	2	3	1	3	1	467	1.66
15	2	3	2	1	1	3	532	1.11
16	1	1	3	3	3	3	299	1.69
17	2	3	3	2	2	1	546	1.3
18	1	3	2	1	3	2	511	1.22

4.2.1 Cutting Forces:

CF's are the primary indicator of the energy consumption in machining processes. CF are influenced by many parameters tool-chip friction, properties of work material, tool material and the coatings, and the geometry of the cutting tool, cutting conditions, cooling/lubricating, etc. The current work presents the effects of in-situ developed hybrid nanofluid on CF, compared with dry cutting and MQL/vegetable oil. Experiments were carried out according to L9 and L18 array and the ANOVA is used to analyse the effect of cutting medium on CF. ANOVA values of the CF experiments are shown in table 4.3. PCR values were taken in to consideration to identify the significance of the variable on the response. From the table 4.7 it is seen that, when machining with the MQL/vegetable oil and MQL/nanofluids as cutting fluids it is seen that the PCR of speed, feed and depth of cut reduces, when compared to dry machining.

Table 4.8: ANOVA for CF results

	Source	Sum of Squares	df	Mean Square	F value	p-value	Percentage Contribution
L9	Doc	95532.667	2	47766.33	50.05204	0.0196	60.20156954
	Feed	56706	2	28353	29.70975	0.0326	35.73427102
	Speed	4540.6667	2	2270.333	2.378973	0.2959	2.861379983
	Error	1908.6667	2	954.3333			1.202779458
		158688	8				100
	R-Squared	0.9880, Adj R-Squared	0.9519				
L18	Cutting fluid	10054.941	1	1720.889	4.147831	0.0879	3.498817678
	Doc	162356.75	2	82256.06	198.2604	< 0.0001	56.49527653
	Feed	96079.87	2	50770.06	122.3702	< 0.0001	33.43291075
	Speed	6814.5384	2	5589.556	13.47242	0.0060	2.3712548
	Vol of fluid	5134.0196	2	1636.056	3.943358	0.0807	1.7864847
	Air pr	3812.0665	2	1333.722	3.214649	0.1125	1.3264847
	Error	2478.8288	6	414.8889			0.862558
		287381.11	17				100
	R-Squared	0.9913, Adj R-Squared	0.9755				

The effect of cutting medium on cutting parameters is further discussed in figure 4.12-4.14. Figure 4.12 shows the effect of cutting fluid on CF, at different levels of DOC. In this figure 4.12 (a) and (b) shows the variation of CF with respect to various level of DOC, when machined under different kinds of lubricating conditions. Figure 4.12 (b) shows the percentage variation in CF when machined with different lubricating conditions. The results indicate 5.744% and 11.75% reduction in CF with use of MQL/ vegetable oil and MQL/nanofluid with respect to DOC. Figure 4.13 (a) and (b) show the effect of cutting fluid on CF, at different levels of feed. A reduction of 2.9-3.45% was found with use of MQL/ vegetable oil and 10.58-11.61% in case of MQL/nanofluid, when compared to dry machining. Figure 4.14 (a) and (b) shows the influence of CS (CS) on CF with respect to speed. A

reduction of 5.3-6.8% was found with MQL/ vegetable oil and 10.46-17.36 % with MQL/nanofluid.

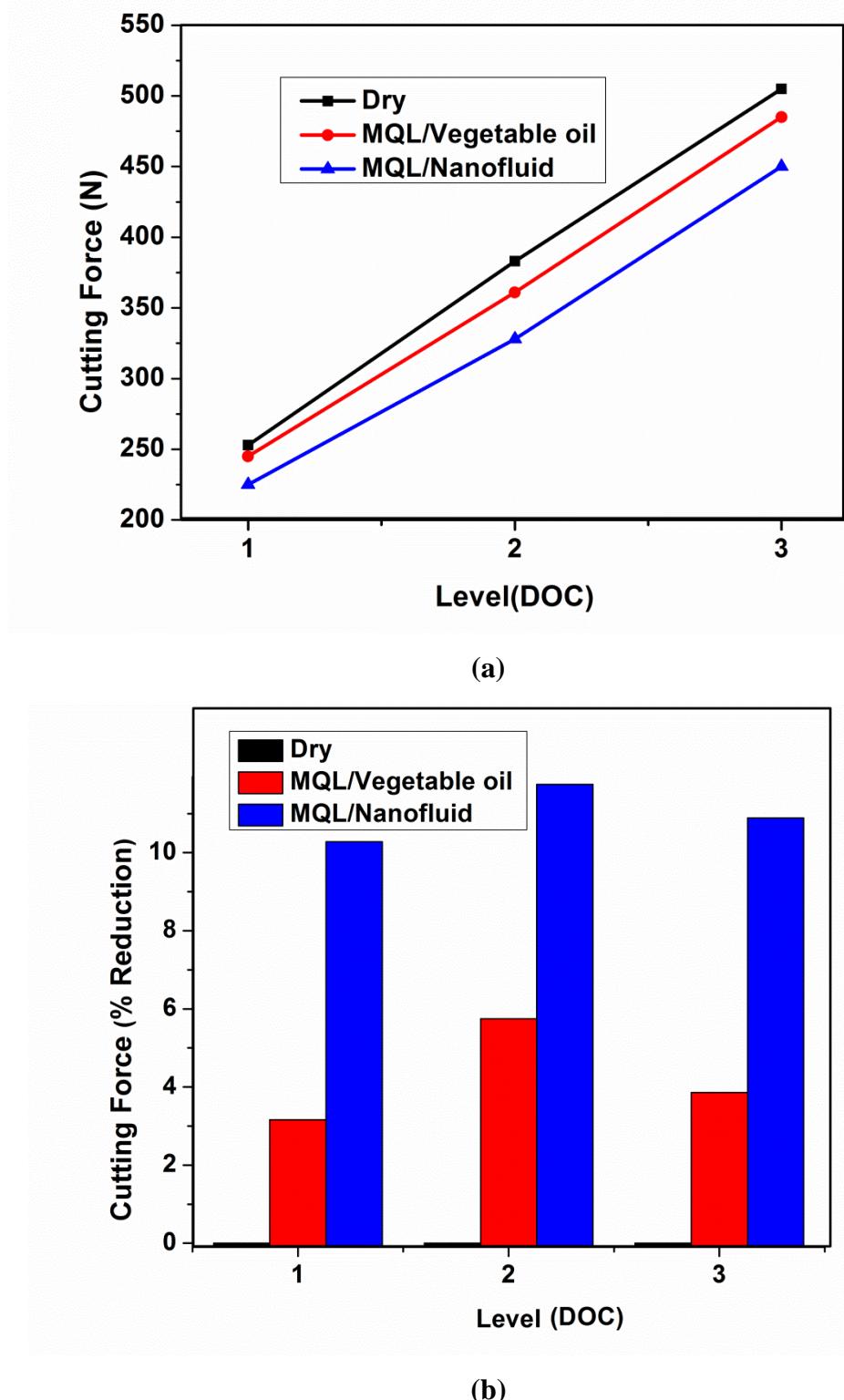
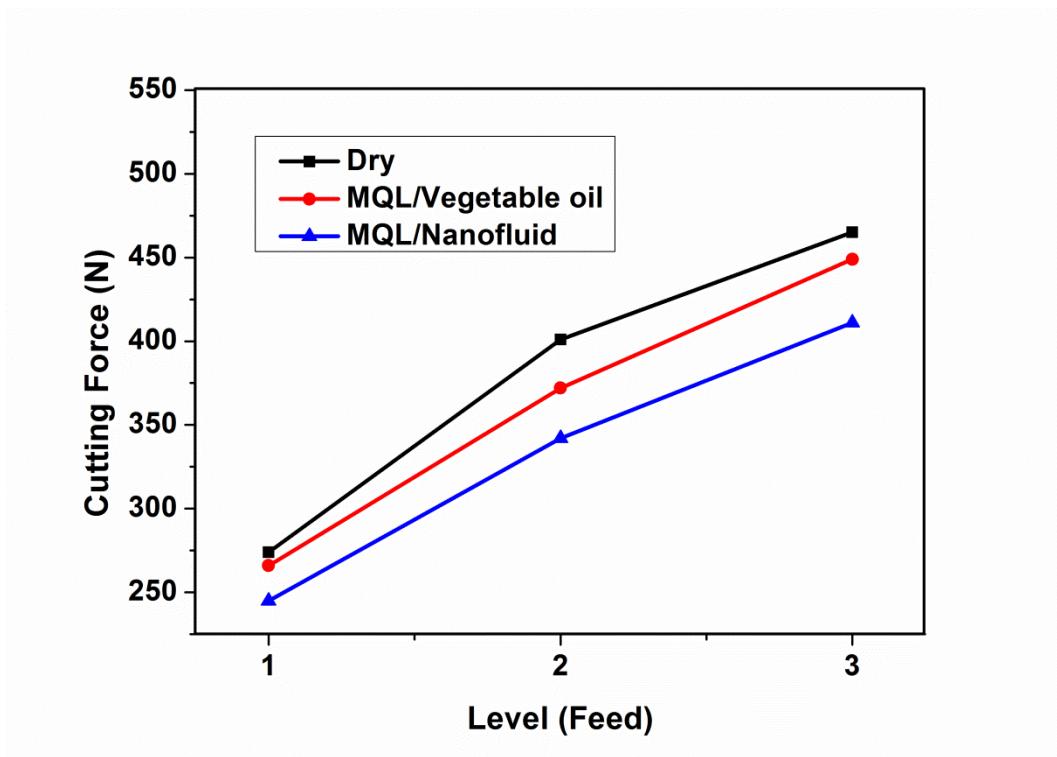
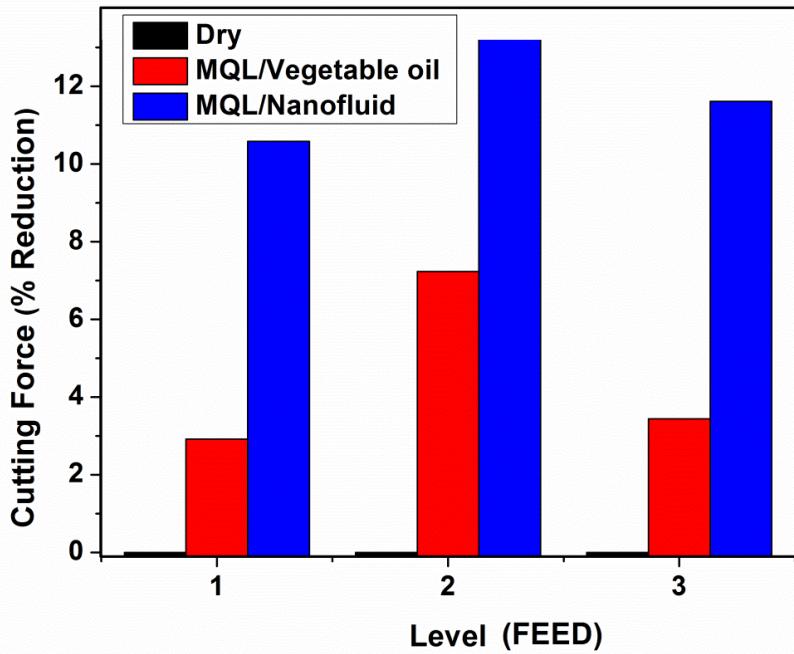


Fig 4.12: Effect of cutting fluid with DOC on CF (a) CF at various depth of cuts (b) percentage reduction in CF – compared to dry machining

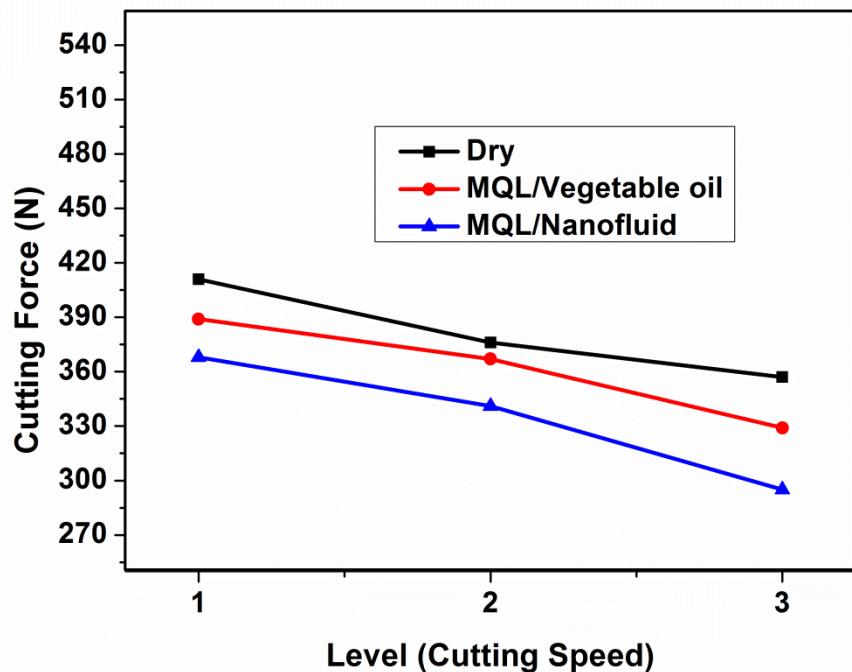


(a)

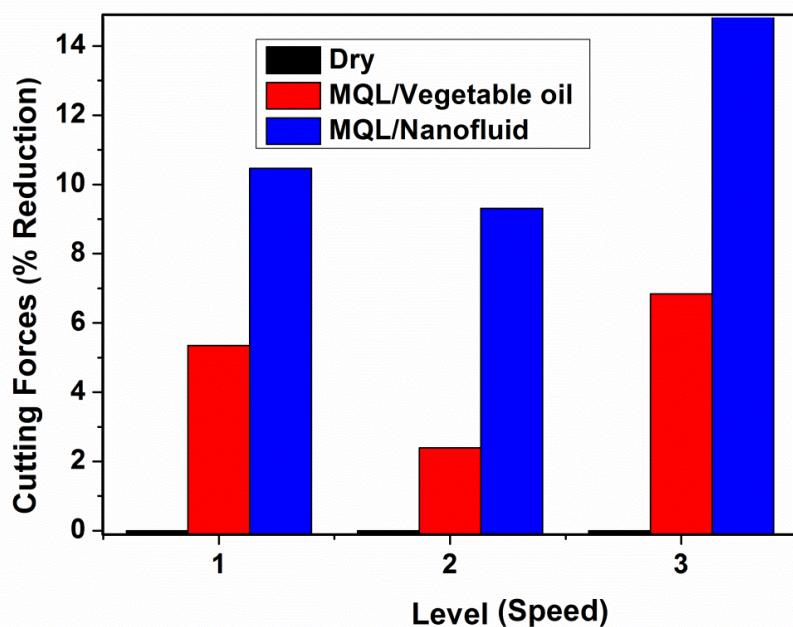


(b)

Fig 4.13: Effect of cutting fluid with feeds on CF (a) CF at various depth of cuts (b) percentage reduction in CF – compared to dry machining



(a)



(b)

Fig 4.14: Effect of cutting fluid with speed on CF (a) CF at various CS (b) percentage reduction in CF – compared to dry machining

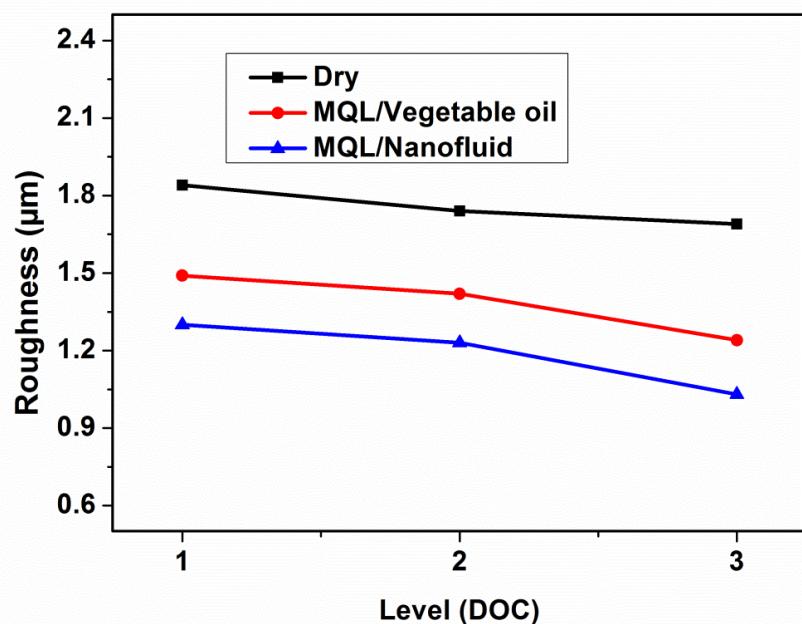
4.2.2 Surface roughness:

SR is critical for the components machined from Inconel-718, because these alloys are termed as difficult to cut materials. Experiments were carried out according to L9 and L18 array and the ANOVA is used to analyse the effect of cutting medium on SR. ANOVA values of the CF experiments are shown in table 4.8. PCR values were taken into consideration to identify the significance of the variable on the response. From the table it is seen that, when machining with the MQL/vegetable oil and MQL/nanofluids as cutting fluids it is seen that the PCR of speed, feed and depth of cut reduces, when compared to dry machining. From table 4.5 it can be seen that feed is the major influencing factor for SR, with contribution of 69.9 % in dry machining. Figure 4.15- 4.17 represents the SR obtained for different cooling strategies. Figure 4.15 shows the influence of cooling strategies on roughness with respect to DOC. With the aid of MQL the SR values reduce, when compared to dry machining. The percentage reduction in SR values is 19-24 % in case of MQL/vegetable oil and 30-39% in case of MQL/nanofluids compare to dry machining. Figure 4.16 shows the influence of CS on SR with respect to feed. With increase of feed level SR increases for all cooling strategies. A reduction of 20-23% and 35-37% respectively for MQL/vegetable oil and MQL/nanofluids is obtained, compare to dry machining. Speed is the second most influencing factor on roughness and with the CS, reduction of 19-27 % for MQL/vegetable and 35-38% (Figure 4.17) reduction in SR for MQL/nanofluids is obtained. Improved performance can be attributed to the combination of lubrication and cooling at same time.

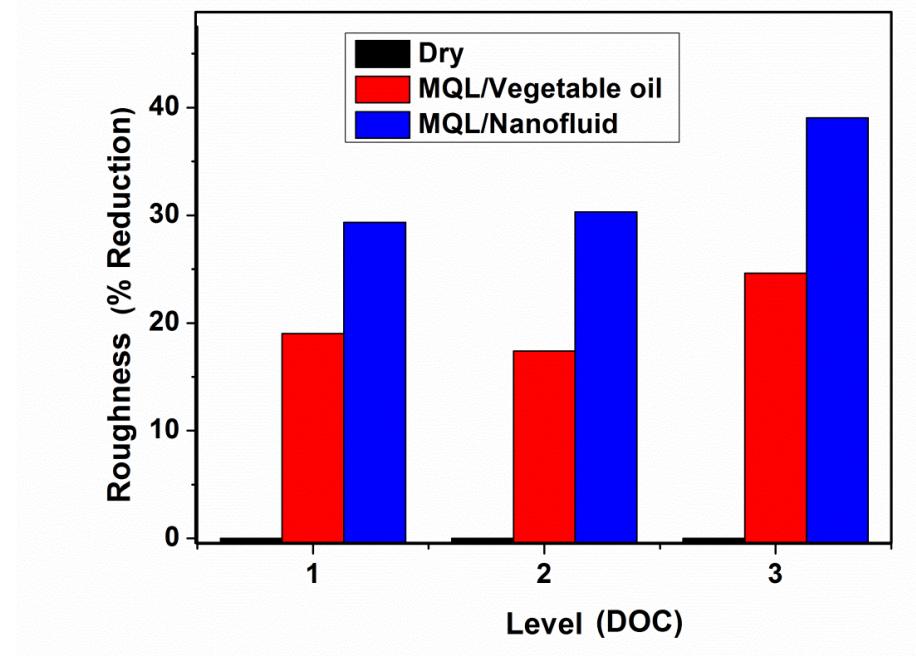
Table 4.9: ANOVA for Surface roughness results

	Source	Sum of Squares	df	Mean Square	F value	p-value	Percentage Contribution
L9	Doc	0.082733	2	0.012544	2.063985	0.3264	7.176176
	Feed	0.806769	2	0.420678	69.21572	0.0142	69.97802
	Speed	0.251231	2	0.137144	22.5649	0.0424	21.79144
	Error	0.012156	2	0.006078			1.054356
		1.152889	8				100
	R-Squared	0.9895, Adj R-Squared	0.9578				

L18	Cutting fluid	0.1152	1	0.1152	6.982602	0.0384	6.629666
	Doc	0.113776	2	0.117706	7.134471	0.0259	6.54772
	Feed	0.993631	2	0.479439	29.06016	0.0008	57.18264
	Speed	0.171193	2	0.042156	2.555169	0.1575	9.852036
	Vol of fluid	0.150178	2	0.047339	2.869346	0.1335	8.642609
	Air pr	0.094678	2	0.075089	4.551353	0.0627	5.448642
	Error	0.098989	6	0.016498			5.69673
		1.737644	17				100
	R-Squared	0.9430, Adj R-Squared		0.8386			

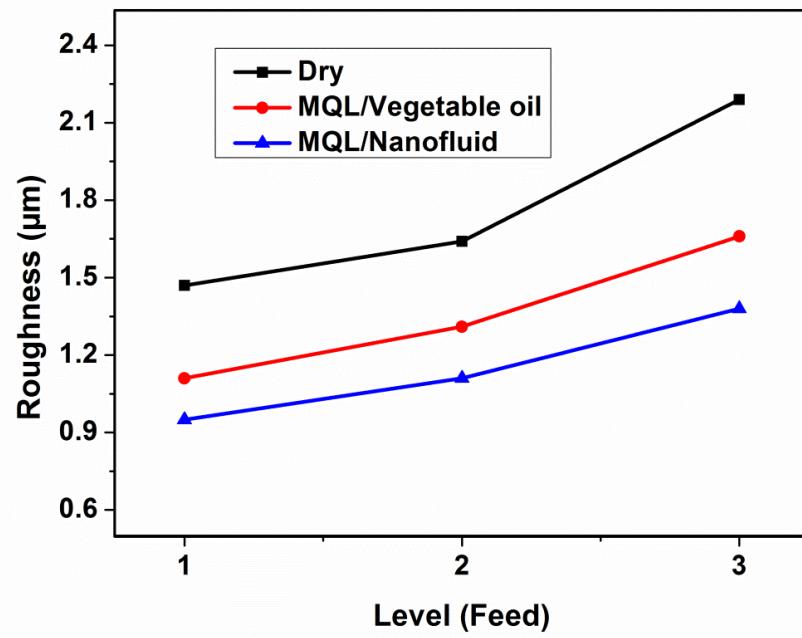


(a)

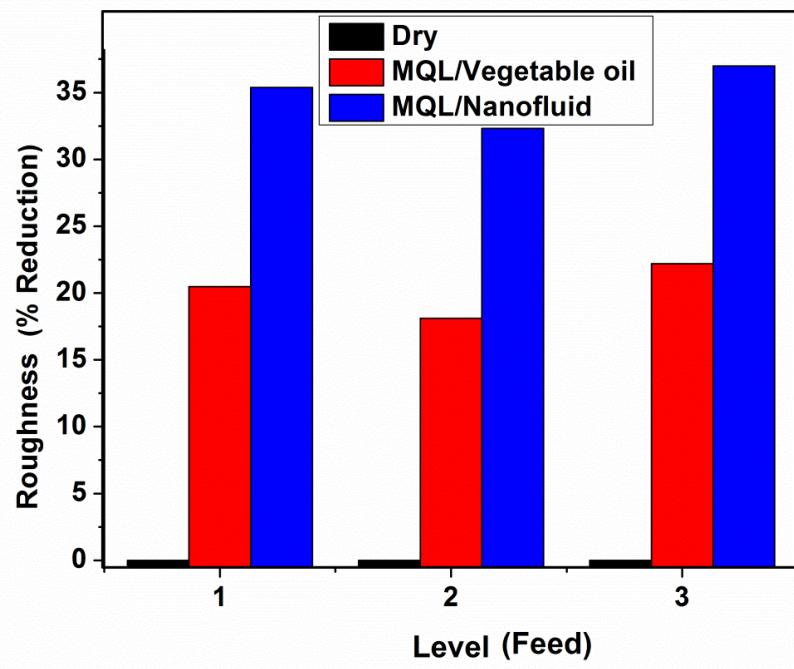


(b)

Fig 4.15: Effect of cutting fluid with DOC on SR (a) SR at various depth of cuts (b) percentage reduction in SR – compared to dry machining

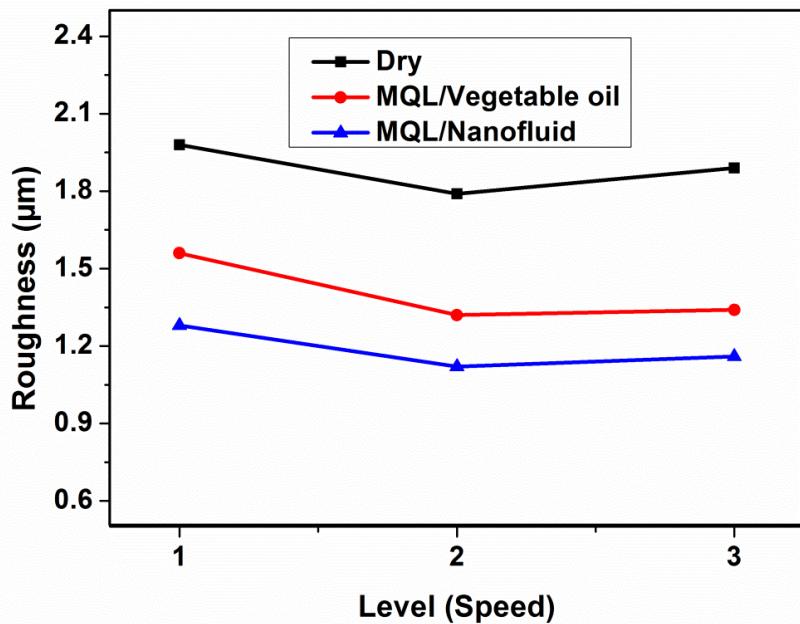


(a)

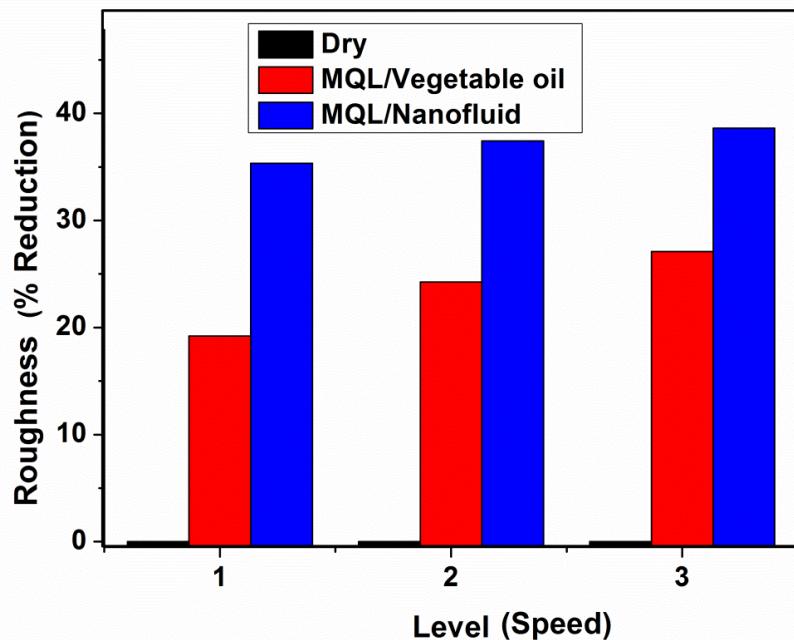


(b)

Fig 4.16: Effect of cutting fluid with feed on SR (a) SR at various feeds (b) percentage reduction in SR – compared to dry machining



(a)



(b)

Fig 4.17: Effect of cutting fluid with speed on SR (a) SR at various CS's (b) percentage reduction in SR – compared to dry machining

4.2.3 Summary

This work demonstrated machining of Inconel-718 with hybrid nanofluid (MQL/Nanofluid) as cutting fluid, and compared its performance with dry and MQL/ vegetable oil conditions. MQL/ nanofluid and MQL/ vegetable oil in general demonstrated contribution in improving performance if used with suitable cutting parameters. Doc is the major contributor for CFs followed by feed and speed. With the use of MQL the contribution of these parameters is reduced.

4.3 Phase 7: RESIDUAL STRESS ANALYSIS OF INCONEL 718

When machining is carried out on workpiece, residual stresses are induced in the workpiece due to non uniform thermal and mechanical loads. These stresses play a vital role in surface integrity of the final product or the output. Inconel 718 is commonly used in structural critical components of aircraft engines due to its properties at high temperatures. Therefore it is important to keep down the stresses induced due to machining. This can be achieved through proper lubricating conditions. As discussed in previous phase-5, MQL/Nanofluids showed better results in terms of reducing cutting forces and surface roughness when compared to MQL/Vegetable oil and dry machining. In this phase, experiments will be carried out to check the influence of the developed nanofluid as cutting fluids on residual stresses developed during the course of machining.

Below given is the experimental plan carried out to test the influence of developed nanofluid as cutting fluid and compared to other lubricating conditions.

4.3.1 Experimental plan:

1. Heat treatment of Inconel 718 (workpiece) for 980°C for 1 hour.
2. Aging the workpiece at 720 °C for 8 hours and furnace cooling.
3. Residual stresses were measured for heat treated workpiece.
4. Machining carried out at 3 lubricating conditions (Dry, MQL/Vegetable oil and MQL/Nanofluid).
5. Results will be compared with 3 lubricating conditions.

4.3.2 Heat treatment of Inconel 718:

The heat treatment of Inconel 718 was carried out according to AMS 5662, in two stages. In stage one the workpiece is heated in a box furnace for one hour at 980 °C and was air cooled. In second stage, the aging of workpiece was carried out in a high temperature muffle furnace. In this stage the workpiece is heated to 720 °C and held at this temperature for 8 hours. The workpiece is then furnace cooled.

4.3.3 Machining:

Turning of Inconel 718 was carried out on the lathe setup as shown in previous phase. The process parameter on which the turning of workpiece was carried is as shown in table 4.6.

From the table it can be seen that the speed, feed and DOC are taken on the higher level to find the effectiveness of the developed nanofluid. The volume of fluid and air pressure level is taken considering the outcomes of cutting forces in phase 5. Turning was carried out using these parameters on the heat treated workpiece with three lubricating conditions. The process parameters used are as stated in table 4.9. Once the turning was carried out, the residual stress was measured for all three lubricating conditions.

Table 4.10: Process parameters for finding residual stresses

Factors	Values
Cutting speed (m/min)	160
Feed (mm/rev)	0.178
DOC (mm)	1
Volume of fluid (ml/hr)	80
Air pressure (bar)	5

4.3.4 Residual Stress Measurement:

When a polycrystalline piece of metal is deformed elastically in such a manner that the strain is uniform over relatively large distances, the lattice plane spacings in the constituent grains change from their stress-free value to some new value corresponding to the magnitude of the applied stress, this new spacing being essentially constant from one grain to another for any particular set of planes similarly oriented with respect to the stress. This uniform macrostrain, causes a shift of the diffraction lines to new 2θ positions. On the other hand, if the metal is deformed plastically, the lattice planes usually become distorted in such a way that the spacing of any particular (hkl) set varies from one grain to another or from one part of a grain to another. This nonuniform microstrain causes a broadening of the corresponding diffraction line.

The residual stresses were measured before and after machining. Figure 4.18- 4.21 show the residual stress plot for heat treated, dry, MQL/vegetable oil and MQL/nanofluid lubricating conditions. Table 4.10 displays the value of the residual stress induced of all lubricating conditions. The results show reduction in residual stresses with use of MQL/Vegetable oil and MQL/Nanofluid when compared to dry machining. Between MQL/Vegetable oil and MQL/Nanofluid, MQL/Nanofluid showed better results, as not only

it lubricates the cutting zone, but is capable of carry away heat from the cutting zone with the presence of hybrid nanoparticle in the fluid.

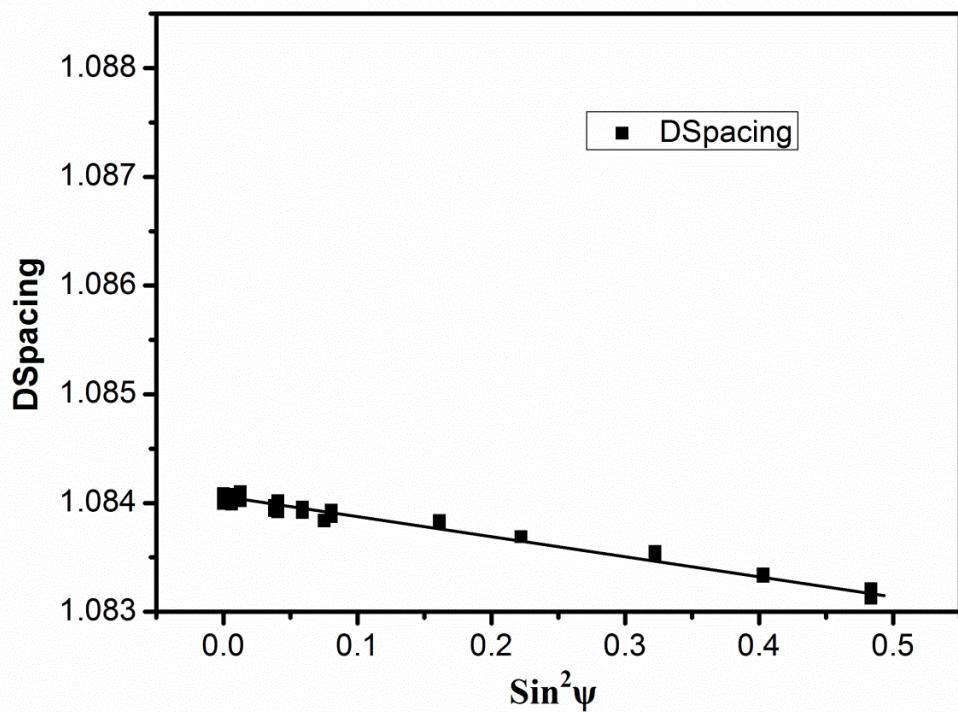


Figure 4.18: Residual stress after annealing

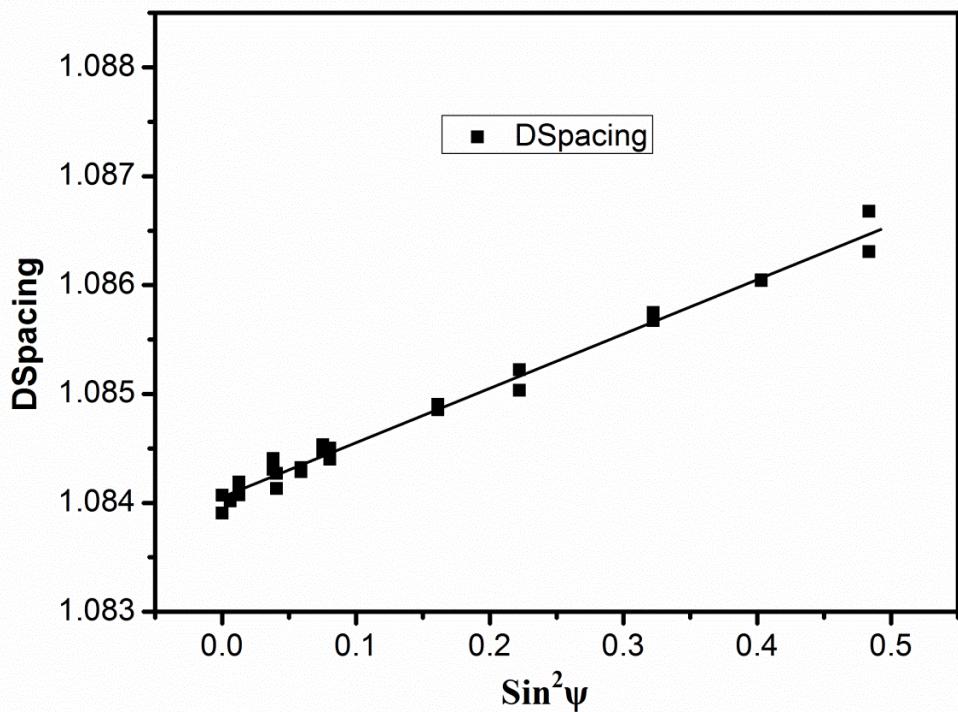


Figure 4.19: Residual stress after dry machining

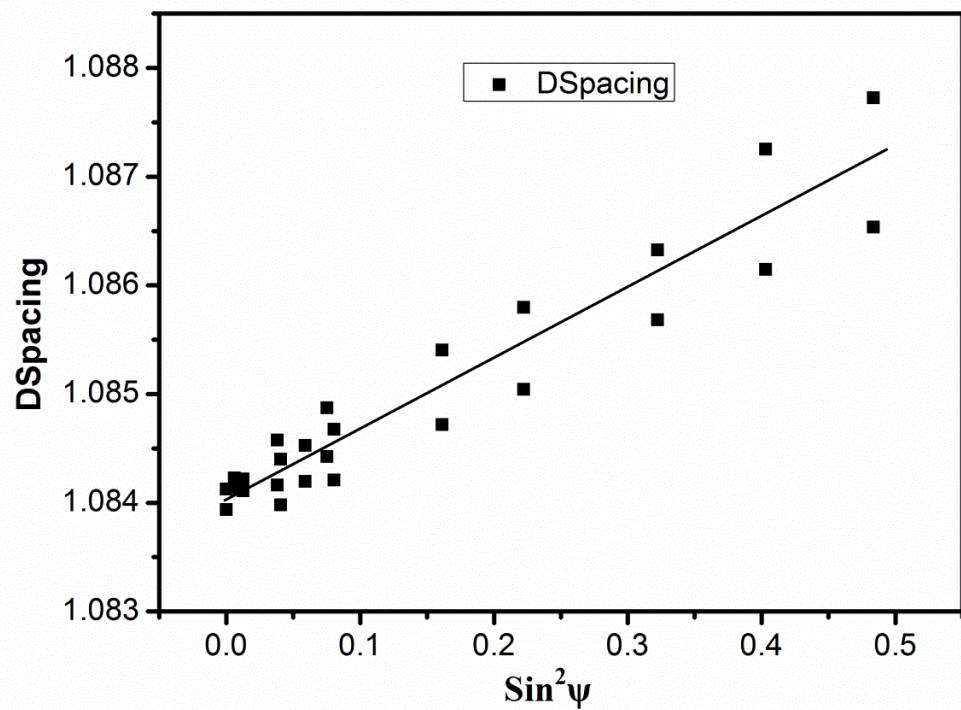


Figure 4.20: Residual stress after machining with MQL/Vegetable oil

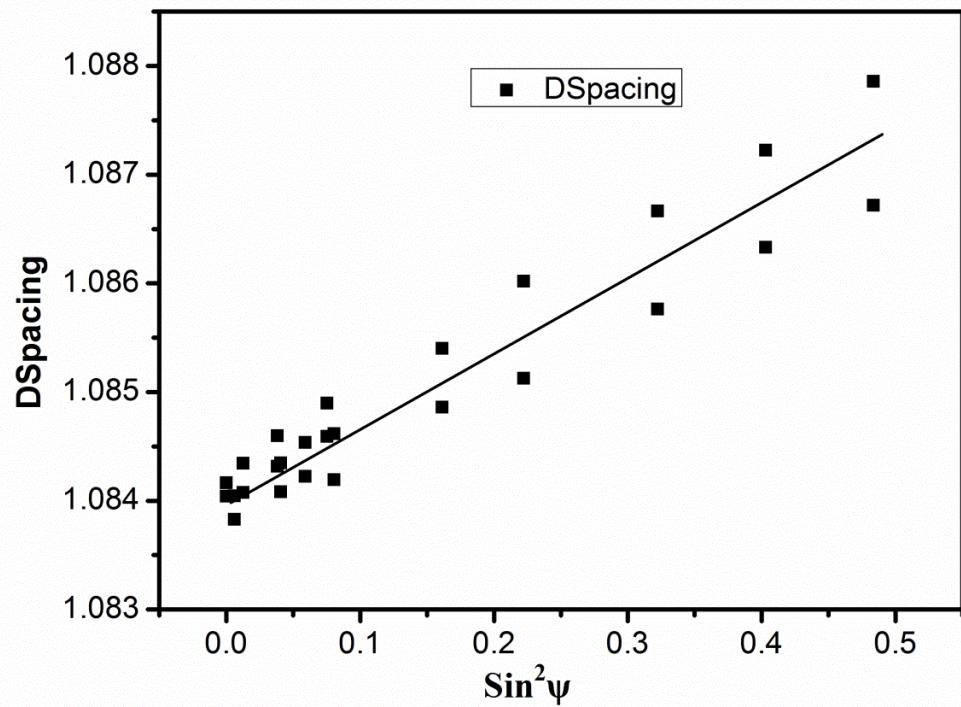


Figure 4.21: Residual stress after machining with MQL/Nanofluids

Table 4.11: Residual stress results when compared to heat treated workpiece

Type Of Lubrication	Residual Stress (Mpa)	Total (Mpa)
After Heat Treatment Before Machining	-223.2	
Dry	+871.2	1094.4
Mql/Vegetable Oil	+820.9	1044.1
Mql/Nanofluid	+652.3	875.5

4.3.5 Summary

This work demonstrated machining of Inconel-718 under dry, MQL/Vegetable oil and MQL/Nanofluid lubricating conditions. MQL/ nanofluid and MQL/ vegetable oil in general demonstrated contribution in improving performance if used with suitable cutting parameters.

Chapter-5

CONCLUSIONS AND SCOPE FOR FUTURE WORK

5.1 Conclusions

This chapter summarizes the main success of this research work and discusses an about future research work to achieve.

“Sustainable manufacturing” is defined as the creation of manufactured products that use processes that are nonpolluting, conserve energy and natural resources, and are economically sound and safe for employees, communities and consumers. Sustainability in manufacturing can be achieved by reducing the use of cutting fluids and adopting ecofriendly manufacturing techniques [171-172]. Vegetable oils based cutting fluids is a proven alternate to conventional cutting fluids and acts as a good lubricator, but lack in heat transfer abilities. The heat transfer properties can be enhanced by incorporating nanoparticles. In this work, noval hyrid nanoparticles are developed considering the economical terms, which possess higher thermal conductivity than commercially available particles. Vegetable oil based cutting fluids are tested and compared with conventionally used cutting fluids. Hybrid nanofluids combined with advances in lubricating techniques (MQL) is used to reduce the drawbacks associated with flood cooling and dry machining of difficult to cut material. The following sections address the individual phase conclusions.

Hybrid Nanofluids: Synthesis, Characterization and Thermophysical Properties

Phase – I: Synthesis and Characterization of Single and Hybrid Cu and Zn Nanofluid

An alloy of Cu and Zn could be formed through mechanical alloying which has a composition of 1:1. The increase in thermal conductivity and viscosity in the case of Cu-Zn hybrid nanofluid is more compared to that of the Zn and Cu nanofluids. The increase in thermal conductivity of the nanofluid is due to the increased area to transfer heat which increases as the particle size decreases, the high thermal conductivity of the nanopowders dispersed in it and due to the Brownian motion of the suspended particles which is inversely proportional to the density of the particles. Cu-Zn hybrid particles have the advantage of high

thermal conductivity and high Brownian motion. Therefore the thermal conductivity of the Cu-Zn-vegetable oil nanofluid is the highest. The viscosity of the nanofluid is directly proportional to the viscosity of the base fluid (vegetable oil). The viscosity enhancement is tormented by particle size, volume concentration, material and agglomeration of the ultrafine particles dispersed in the base fluid. Therefore the viscosity enhancement is utmost in the case of Cu-Zn nanofluid

Phase – 2: Selection of Basefluids

The thermophysical properties of hybrid nanofluids were studied, with incorporation of hybrid nanoparticles. The results illustrates that the hybrid nanofluids exhibited stability for 72 hours and then the agglomeration rate of particles increases and by 168 hrs almost all the particles tend to settle down. Hybrid nanofluids with vegetable oil showed marginal less stability. The flash point results illustrates, increase in flash point value with addition of hybrid nanoparticles, with all fluids. Vegetable oil as basefluid showed better increment in flash point. The thermal conductivity test indicate higher enhancement with vegetable oil. Vegetable oil behaved as Newtonian fluid and the change in viscosity with application of shear was less noticed compared with other two fluids. On the basis of integrated study of thermal conductivity and viscosity, in the point of cost and higher relative thermal conductivity to relative viscosity for effective heat transfer vegetable oil based nanofluid showed 53% better results. Hence, for machining process eco-friendly vegetable oil based Cu-Zn (50:50) hybrid nanofluids are best.

Phase – 3: Effect of Various Combinations of Hybrid Cu And Zn (50:50, 75:25, 25:75)

Mechanical milling is used to synthesize Cu-Zn hybrid nanoparticles alloy with 50:50; 75:25; 25:75 combinations using a planetary ball milling. Enhancement in thermal conductivity is observed with increase in volume concentrations and enhancement in viscosity is also observed with increase in volume concentration. It is observed that when the shape and size of the hybrid nanoparticles are utmost identical the viscosity of the hybrid nanofluid is more or less the same at same volume concentrations. With minimal difference in viscosity the motion of the particle is dependent on the densities and heat transfer properties of the hybrid nanofluid are dependent on the thermal conductivity of the hybrid nanoparticles. Cu-Zn with 50:50 combination results in better enhancement in thermal conductivity due to the Brownian motion of the particles.

Phase – 4: Effect of Ag Particle Addition

The thermal conductivity and viscosity of Cu-Zn-Ag hybrid nanofluid were determined at various temperatures intervals (30, 40, 50 and 60°C) and for five nanoparticle weight concentrations (0.1, 0.2, 0.3, 0.4 and 0.5 % volume).

The results showed that the thermal conductivity of nanofluids increases with an increase in concentration and temperature. The enhancement in thermal conductivity was found to be 96 % ($k_{eff} = 1.9677$) with addition of Ag when compared to 87% with Cu-Zn (50:50). This enhancement of thermal conductivity with addition of Ag comes at a higher cost and owing to the sustainability of cutting fluids, using Cu-Zn (50:50) in place of Cu-Zn-Ag. Enhancement in viscosity was 13.7% when compared to the basefluid.

Phase 5: Modelling of Nanofluids

Modelling Technique for Thermal Conductivity of Hybrid Nanofluid:

A relationship between the predicted values and experimental values were established by developing the regression models for the experimental data. The regression models were established using ANN using Matlab nntool box and the predicted output has 0.999% confident levels. The output of this function is also compared with the theoretical models and showed better results.

Modelling Technique for Viscosity of Hybrid Nanofluid:

With ANN the predicted output has 0.999% confident levels. The output of this function was compared with the theoretical models and showed better results. The theoretical models couldn't account all the parameters affecting the viscosity of nanofluids. Einstein and brinkman models accounted for volume concentrations and Nguyen model accounted temperature. These models either over predict or under predict the viscosity of nanofluids. Therefore a well-trained ANN model can be used to study the rheological behaviour of hybrid Cu-Zn for all combinations (0:100, 75:25, 50:50, 25:75, and 100:0), at various temperatures, volume fractions, diameter of particles, viscosity of nanofluids and basefluids.

Comparative study of thermal conductivity enhancement with past studies:

A comparative study was carried out to find out the enhancement in thermal conductivity. Data from the past studies was collected. Figure 5.1 shows the percentage thermal conductivity enhancement with respect to various nanofluids reported in the literature and present work. Cu-Zn hybrid nanofluids with 0.5% volume fraction shows better enhancement than other nanofluids.

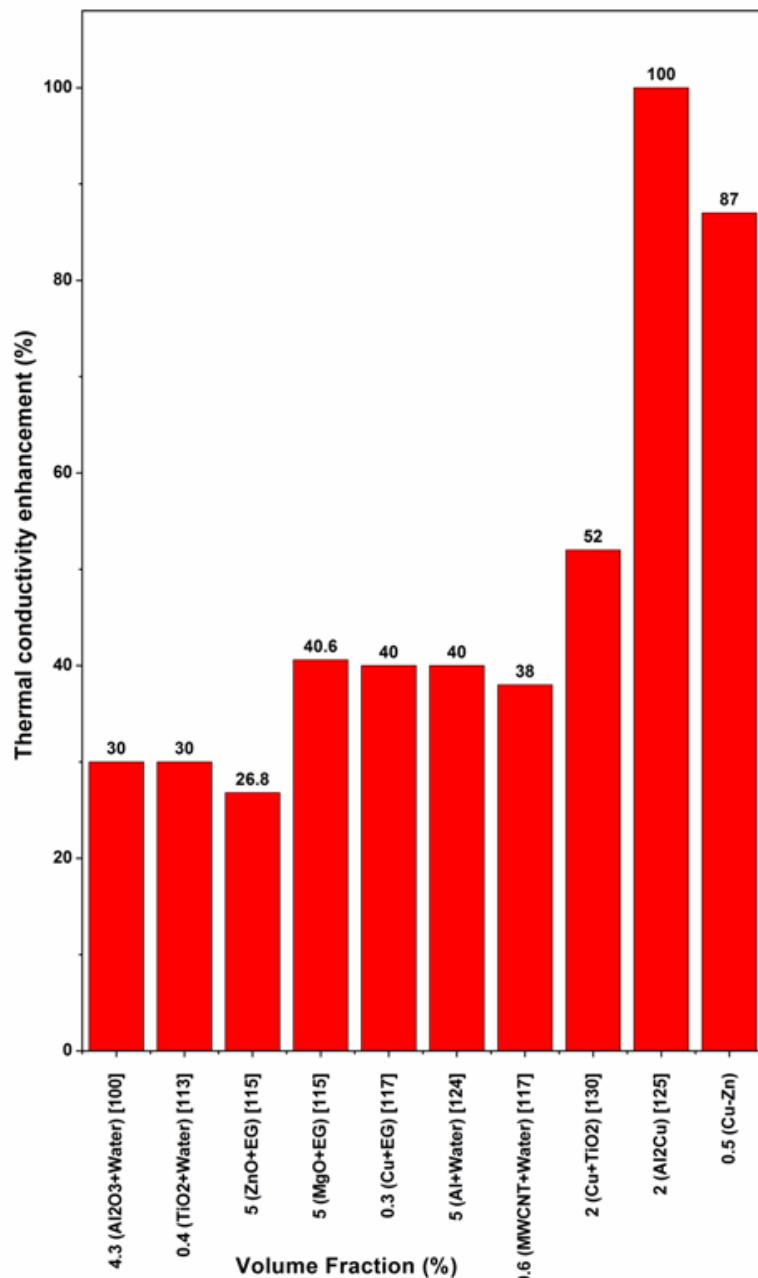


Figure 5.1: Comparative study on enhancement in thermal conductivity

Machining of Inconel 718 using Hybrid Cu-Zn (50:50) Nanofluids as Cutting Fluids

Phase 6: Turning of Inconel 718 Using Hybrid Cu-Zn Nanofluids

This work demonstrated machining of Inconel-718 with hybrid nanofluid (MQL/Nanofluid) as cutting fluid, and compared its performance with dry and MQL/ vegetable oil conditions. MQL/ nanofluid and MQL/ vegetable oil in general demonstrated contribution in improving performance if used with suitable cutting parameters. Doc is the major contributor for cutting forces followed by feed and speed. With the use of MQL the contribution of these parameters is reduced.

Phase 7: Residual Stress Analysis of Inconel 718

This work demonstrated machining of Inconel-718 under dry, MQL/Vegetable oil and MQL/Nanofluid lubricating conditions. MQL/ nanofluid and MQL/ vegetable oil in general demonstrated contribution in improving performance if used with suitable cutting parameters. The results show reduction in residual stresses with use of MQL/Vegetable oil and MQL/Nanofluid when compared to dry machining. MQL/Nanofluid showed better results, as not only it lubricates the cutting zone, but is capable of carry away heat from the cutting zone with the presence of hybrid nanoparticle in the fluid.

Comparative study of affect of MQL on Machining:

A comparative study was carried out to find out the effect of MQL on turning. Data from the past studies was collected. Figure 5.2 shows the comparison of percentage reduction in cutting forces for turning under dry and MQL Lubricating conditions. The lubricating conditions used in the present work shows better reduction in cutting forces compared to the lubricating conditions used in the past. A similar trend can be seen in figure 5.3 where percentage reduction in surface roughness is studied. This results show that the developed hybrid nanofluid developed contributes both in lubricating and carrying away the heat generate in the cutting zone, resulting in reduction in cutting forces and surface roughness.

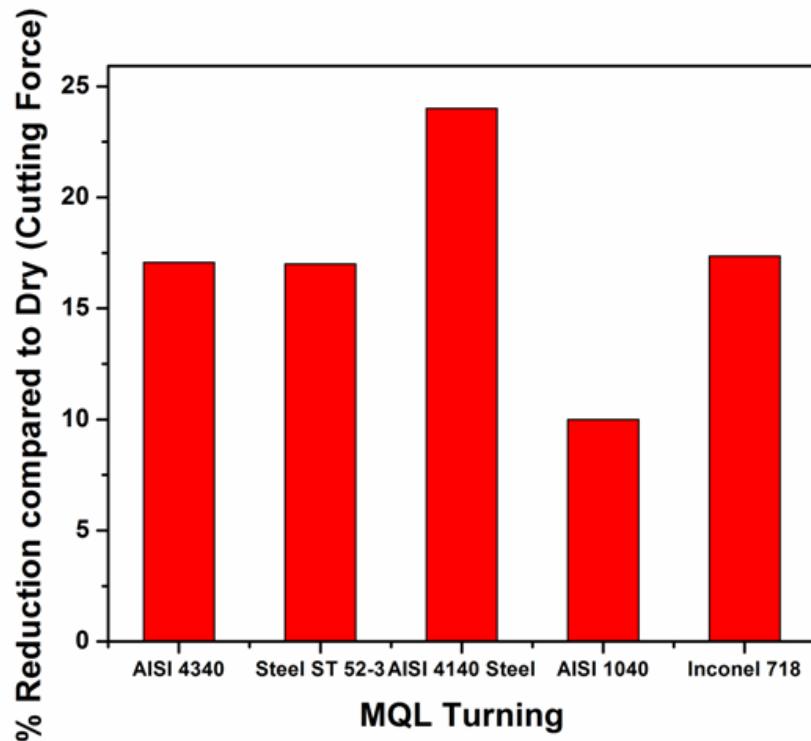


Figure 5.2: Comparative study on Percentage reduction in cutting forces [173-176]

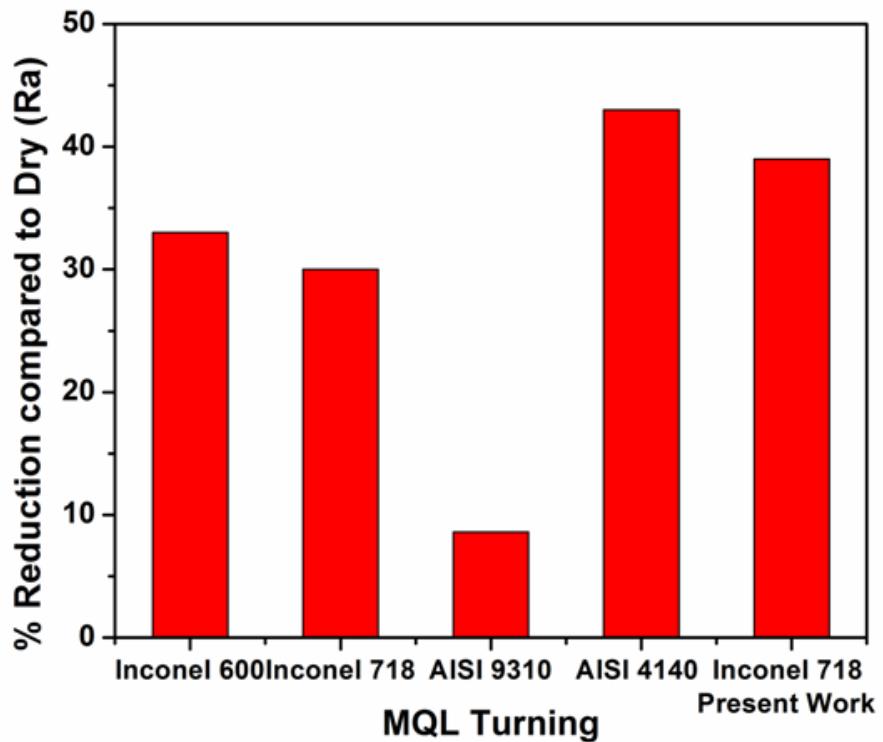


Figure 5.3: Comparative study on Percentage reduction in surface roughness [92, 177-179]

5.2 Scope for future work

Up to now, not many research efforts have been devoted to hybrid nanoparticles and its use as cutting fluids. This thesis provides initial work in this respect. As a very new research area there are several interesting and important future directions are as follows.

1. Different hybrid nanoparticles were synthesised and used throughout this work. Uniform size distributions of hybrid nanoparticles were not obtained. The work can be extended to identify an appropriate synthesis method.
2. The developed hybrid nanofluids were used as cutting fluids and only cutting force and surface roughness parameters were studied. Further work can be carried out on temperature and tool life study.
3. The effect of developed hybrid nanofluids on surface residual stresses were studied and compared with other lubricating conditions. Study can be carried out on residual stress induced in the depth direction.
4. The variation of the three components of force can be studied with respect to the various parameters.

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Visible Research Outcomes

Publications in Journals:

1. “Thermal Conductivity and Viscosity Of Vegetable Oil Based Cu, Zn And Cu-Zn Hybrid Nanofluids”, Journal of Testing and Evaluation, 2014, 44 (3), 1077-1083 (DOI: 10.1520/JTE20140286), Impact factor 0.423 (SCI journal).
2. “Thermal Conductivity and Rheological Studies for Cu-Zn Hybrid Nanofluids with Various Basefluids”, Journal of the Taiwan Institute of Chemical Engineers, 2016, 66, 321-327 (DOI 10.1016/j.jtice.2016.05.033), Impact factor 2.848 (SCI journal).
3. “Investigation of Thermal conductivity and Rheological Properties of Vegetable Oil Based Hybrid Nanofluids Containing Cu-Zn Hybrid Nanoparticles” Experimental Heat Transfer, 2016, 1-17 (DOI 10.1080/08916152.2016.123317), Impact factor 1.288 (SCI journal).
4. “Thermal Conductivity of Cu-Zn Hybrid Newtonian Nanofluids: Experimental data and modelling using neural network”, Procedia Engineering, 2015, 127, 561 – 567 (DOI:10.1016/j.proeng.2015.11.345) (Scopus journal).

Publications in International Conferences:

1. (“Thermal Conductivity of Cu-Zn Hybrid Newtonian Nanofluids: Experimental data and modelling using neural network”), International Conference on Computational Heat and Mass Transfer (ICCHMT)-2015
2. (“Rheological study of Cu-Zn Hybrid Newtonian Nanofluids: Experimental data and modelling using neural network”), International Conference On material processing and characterization (ICMPC)-2016
3. (“Investigation on Influence of Hybrid Nanofluid/MQL on Cutting Forces in Turning Inconel-718”), 6th International & 27th All India Manufacturing Technology, Design and Research Conference (AIMTDR-2016), December 16-18, 2016 at College of Engineering, Pune, Maharashtra, INDIA