

# Effect of Various Sizes of TiO<sub>2</sub> Nano-Particulate Composites on Mechanical and Tribological Properties Synthesized by Bottom Pouring Stir Casting Method

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# Abstract

Nanocomposites are a combination of matrix and nanoparticle reinforcement. The general idea behind adding reinforcements at the nanoscale is to create synergies between the various components, allowing for new features that meet or exceed design expectations. The properties of nanocomposites is governed by a range of variables, particularly reinforcement materials that have nanoscale dimensions, dispersal, size, shape, and orientation of the reinforcements with secondary phase matrix materials. Recent researchers has focused on Nano Metal Matrix Composites (NMMC) because of their unique properties of high strength and lightness. However, because of the high cost of raw materials and processing technology, it is difficult to mix the nanoparticles with the matrix. Therefore, the practical application of nano composites in industry will differ from other Metal Matrix Composites (MMC), has not been realized. Nano-fillers such as particles, fibers, rods, etc. are too expensive to produce, even complex roots. The nature of NMMC as well as mechanical, tribology, electrical and thermal property will be significantly different from those of MMC. NMMC is different from micro- particle and macro-particle sized MMC because the surface area to surface ratio of the nanoparticles is very high. The sorts of nano reinforcements includes Nanoparticles, Nano-flakes, Nanotubes, Nano-rods, Nano-sheets etc. Due to the higher surface area of the nanoparticles, which consecutively increases in the interface surface area of the matrix and particles. The material properties of the NMMC are significantly superior to those of the traditional MMC.

Nanocomposites are materials that integrate nanoparticles into a matrix of standard materials and alloys. The addition of nanoparticles results in a significant enhancement of performance, which may include strength, durability in addition to electrical or thermal conductivity. The efficiency of nanoparticles is such that the extent of added material generally represents merely 0.5 to 15% by weight.

In MMC, Aluminium Metal Matrix Composites (AMMCs) have acknowledged specific consideration over the last three eras because of high specific strength, stiffness and their excellent wear resistance. However, their application is limited since of its low wear resistance. Currently Particle-reinforced aluminium matrix composites is well-thought-out to have higher mechanical and tribological properties than conventional alloys. As such, these composites are widely finds applications in the automotive and aerospace industries. The focus is on developing aluminium NMMC at a reasonable price, with numerous hard and soft reinforcements, such as SiC, Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, B<sub>4</sub>C, Zircon, Tungsten Carbide, Graphite and mica.

There are generally two types of manufacturing techniques accessible: (i) Solid phase manufacturing route comprises diffusion bonding, extrusion, drawing, hot rolling, PM route ii) Liquid phase manufacturing system comprises liquid metal infiltration, squeeze casting, compo casting, pressure casting. Bottom pouring Stir casting is generally considered to be a promising approach in various manufacturing processes that can be used for short-fiber metal matrix composites because of its simplicity, its flexibility and its ability for mass production.

Nanoparticles of  $\text{TiO}_2$  in the form of fibers or particles have long been considered as high-strength materials. Nano  $\text{TiO}_2$  particle reinforced with Aluminium matrix produced by the solidification technique represent an expensive class of custom made materials for various engineering applications such as automotive parts, bushes and bearings. The use of nano- $\text{TiO}_2$  reinforcing materials in metal matrix can exhibit outstanding properties at high temperatures. But, the lack of wettability amongst the aluminium and the reinforcing material leads to manufacturing complications and cavitation of the material at high temperatures.

Much work has not been carried out for analysing the effects of various sizes of nano  $\text{TiO}_2$  Particles reinforced with Al Alloy matrix, so it is necessary to evaluate the various properties of aluminium metal matrix nano composites to know the effect of various nano sized  $\text{TiO}_2$  reinforcement on base matrix. Mechanical properties like ultimate tensile strength, yield strength, percentage elongation, creep, fatigue and hardness, are assessed by very few researchers. During working numerous components are exposed to sliding or rolling contacts. Subsequently it is crucial to study the mechanical and wear behavior of AMNCs.

In the current work an effort has been made to review the effect of different nano sized  $\text{TiO}_2$  particles reinforced with LM0 Al alloy synthesised by using bottom pour stir casting technique and Microstructure, EDS, XRD, Mechanical and Tribological behaviour have been evaluated.

# 1. Introduction And Literature Review

## 1. Definition of Nano Composites.

According to Cammarata R [1], a nanocomposite is a multiphase hard/soft solid material wherein one of the phases has one, two, or three dimensions that are less than 100 nanometers (nm), or structures that demonstrate nano-scale repetition distances between the various phases that make up the material. The nanocomposite will have significantly different mechanical, electrical, thermal, optical, electrochemical, and catalytic properties from its component materials.

## 1. Metal Matrix Nano-Composites (MMNC).

MMNCs is combination of a metal matrix and dispersed nano ceramic particles or a metal phase. SiC, Boron,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ , SiN,  $\text{B}_4\text{C}$ ,  $\text{B}_4\text{N}$  are widely used nano ceramic reinforcement [2]. MMNC is used in space shuttles, Aeroplanes, electronic media, bicycles, automobiles, golf clubs, mechanical and wear application. Compared with aluminium-based microscopic and macroscopic composites and unreinforced aluminium alloys, MMNCs show significant improvements in physical and mechanical properties such as strength, modulus of elasticity, fatigue strength and resistance to wear rate [3].

## 1. Nano Particulate Reinforced Metal Matrix Composites (NPMNC).

NPMNCs show substantial improvements in tribological properties, particularly with respect to sliding plus abrasion wear resistance and seizure resistance [4]. Therefore, due to the good wear resistance, extraordinary load capability, plus light weight of NPMNCs, there has been much research into NPMNCs for tribological applications. Some studies have shown that these nanocomposites have potential applications in case of wear. Because of the ceramic particle content, the nanoparticles develop the wear resistance beside protect the metal matrix from wear. The foremost problems of aluminium alloys is that limited tribological properties because of its moderately low resistance to seizures compared to cast iron in dry sliding conditions. Compared to the micron reinforcements, the addition of nano-reinforced materials makes it possible to maintain the ductility of the matrix materials upto certain extent for the same weight or volume percent [5].

### 1. Aluminium Matrix Nano-Particulate Composites (AMNC).

Nanoparticle reinforced aluminium based composites, also known as aluminium based nanocomposites (AMNCs), have been researched globally in modern era because of their favorable properties for a comprehensive kind of applications like functional plus structural applications along with weight reduction. Reducing the size of the nanoscale reinforcement phase makes the interaction of particles and dislocations very important and, when compared with other common reinforcing effects in conventional AMC systems, results in significantly improved properties like mechanical and wear resistance [6].

Production techniques is divided into two broad categories namely ex-situ and in-situ. The first production techniques involves the addition of nano-reinforcements to liquid or powdered metals, whereas in-situ method refers to the processes that lead towards the production of nano- ceramic compounds via reactions during processing, such as the use of reactive gases. Several ex-situ synthesis methods of MMNC have been developed.

## 1.5 Material Selection

### a) Why Al Matrix Selection?

Currently, researchers around the world are concentrating on aluminium since it is having excellent mechanical and corrosion properties along with low density. Aluminium composites have excellent thermal properties, so it is extensively used in aerospace, automotive and avionics fields. Titanium finds extensive applications in aerospace engines because of its high temperature resistance, primarily for blades and compressor discs [7]. The literature work carried out shows that maximum published work has focused on aluminium-based composites thru the following advantages: extensive variety of alloys, heat treatment capacity, low density, and processing flexibility.

#### 1. Reinforcement.

When nano-TiO<sub>2</sub> material reinforced with metal matrix composites, has the potential to produce materials suitable for high temperature applications because of its high thermal conductivity, outstanding

mechanical properties plus attractive damping properties.  $\text{TiO}_2$  is extensively used as a reinforcing phase because it can enhance the hardness, tensile strength and wear resistance of aluminium composites [8–9].

Titanium dioxide or Titania ( $\text{TiO}_2$ ) occurs in many crystalline forms, the utmost significant being Anatase and Rutile. Unadulterated titanium dioxide will not exist in nature but comes after Ilmenite or Leucocene ore. Besides it is easy to exploit from one of the purest forms, rutile beaches. Most of the Anatase form of titanium dioxide is produced in the form of a white powder and the different qualities of Rutile are usually off-white and may even have a slight color reliant on the physical form that disturbs the reflection of the light. Alumina and silica are used as coating material for Titanium dioxide to increase the technical performance. Rutile can be a thermodynamically stable form of titanium dioxide, Anatase will rapidly converted to Rutile at temperatures above  $700^\circ\text{C}$  and Rutile melts between  $1830$  and  $1850^\circ\text{C}$  [10].

### **1. A Review on Metal Matrix Nano Particulate Composites.**

Zapata [11] showed that the manufacturing of nanocomposites can be divided in to three types, namely "solution mixing, the liquefied state and in-situ polymerization". He also showed that the latex consisted in employing the monomer plus the reagent used amongst the clays. During the polymerization process, the spacing between the layers of clay gradually increased and the dispersion state of the clay increased from intercalated to exfoliate. Benefits of this technique includes i) single-phase casting of the metallocene polymer nanocomposite, ii) enhanced compatibility of the clay with the polymer matrix, iii) the improvement of the dispersibility of the clay.

Zhang Z et al [12] demonstrated in their study that the Orowan equation disclosed that nano-size reinforcement was very beneficial for tribological applications compared to macroscopic size reinforcement. Merely a slight amount of nano-sized reinforcing material is sufficient to provide the base alloy superior wear resistance.

Ray et al [13] have shown that due to the presence of particles at the bottommost portion, a high stirring speed is essential to lift the particles for dispersion, the stirring speed being greater than the speed agitation required to dispense the added particles from above. The benefit of removing the trapped air formed around the particles during the infiltration process is that the porosity of the cast composite is minimized, which can be compensated for the necessity of a high stirring speed, causing a bubble inhalation rate higher in the vortex, causing more porosity in the synthesized composites.

K. R. Padmavathi et al [14] compared the properties of nano and micron sized SiC reinforced with Al6061 with wt. % of 5, 10 & 15 for micron sized SiC and 0.5, 1 & 1.5 for nano sized SiC by stir casting methods. The findings indicate that produced nano composites outperformed micron-sized composites in terms of hardness and wear resistance. Considering every factor shown that nano-sized SiC reinforcement added to an aluminum-based composite at a weight-based ratio of 1.0% had superior wear resistance characteristics compared to micron-sized SiC reinforcement added to an aluminum-based metal matrix composite. In comparison to Al6061-micron sized SiC metal matrix composites, Al6061-nano SiC exhibits

lower wear rates and friction coefficients. Comparing wear rates of Al6061-10% micro SiC and Al6061-1.0% nano SiC composites to those of other reinforcing weight percentages, these composites have a low wear rate. Al6061-nano SiC composites have harder values than Al6061-micron SiC composites; the VHN values for micron-sized SiC and nano-sized SiC particles, respectively, are 55, 57, and 59.

Iman S. El-Mahallawi et al [15] studied the microstructure of a cast aluminium alloy by accumulating nanoparticles of  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  and  $\text{ZrO}_2$  of 40 nm particle size to A356 aluminium by stir casting methods. Nano powders are stirred in an A356 matrix at variable speeds of 270, 800, 1500, 2150 rpm at  $600^\circ$  and  $700^\circ\text{C}$  which means at both semi-solid and liquid state, with 0–5% by weight, with a stirring time of one minute. The microstructure of the alloy comprises a primary aluminium matrix and an Al-Ti eutectic. The microstructure of as cast displays an even distribution particles in the phases. In the base part of the matrix, the microstructure is dendritic, while in other rheological samples, the main dendritic structure is broken by mechanical stirring. But with continuous agitation, the plastic deformation in the fragmented grains will be substantially reduced. The UTS were 155, 158, 164, 185 and 163 MPa, respectively, with respective elongations of 57%, 64%, 72%, 77% and 49% for 0,1,2,3 and 4 wt. % respectively. The results show, when the weight percentage of  $\text{TiO}_2$  nanoparticles increased to 3%, the UTS also increased to 185 MPa. Above this % by weight, the UTS decreases as the weight % of  $\text{TiO}_2$  nanoparticles increases. The ductility reached its maximum for 3% by weight of  $\text{TiO}_2$  nanoparticles, and then reduced for 4% by weight of  $\text{TiO}_2$  nanoparticles. Since the hard nature of the  $\text{TiO}_2$  nanoparticles results in an increase in the position of the local stress concentration, there is an embrittlement effect. These  $\text{TiO}_2$  particles will restrict the passage of dispersion by creating a stress field in the matrix or causing large differences in elastic behavior between the matrix and the dispersion. Since the intercalated nano  $\text{TiO}_2$  particles do not react with the matrix, it can be expected that the embrittlement effect of  $\text{TiO}_2$  is mechanical in nature.

By adopting bottom pouring stir casting technique by in-situ method, H M Nanjundaswamy et al. [16] demonstrated the impact of forged and unforged magnesium-based composites and demonstrated how the BHN and Tensile characteristics will differ in the forged and unforged composites. Due to reduced porosity and intermetallic particle, the BHN and Tensile values for the forged composites were higher than those for the unforged composites.

Amal E. et al. [17] investigated the mechanical performance of pure aluminium reinforced with nano titanium di-oxide particle composites that had a volume percentage of 0.5, 1.5, 2.5, 3.5, and 4.5 and an average diameter of 50 nm. 52, 65, 71, 76, 81, and 85 BHN are the corresponding BHN values. This might be owing to the presence of nanoparticles, which delayed the movement of matrix disruption which may be the principal reasons for improved strength and hardness and due to (a) the existence of relatively harder ceramic particles in the matrix, (b) higher restriction in the localized matrix distortion through the indentation due to their existence, and (c) reduced grain size to nanometer.

1. S. Krishnaraj et al [18] studied the hardness behaviour of  $\text{TiO}_2$ -SiC reinforced metal matrix composites made thru powder metallurgy method. The test specimens of metal matrix composites

were prepared by varying reinforcement ratio as 5%, 10%, 15% and 20% correspondingly. The BHN values observed were recorded as 15, 26, 30 and 35 respectively, the hardness of the specimen varies because of adding silicon carbide and titanium di oxide to the matrix material. The addition of 10 Percentage of Titanium di-Oxide also has the higher hardness value compared with 10 percentage addition of Silicon Carbide particles.

1. Ganesh raja et al [19] synthesized and compared the properties of composites which is produced by stir casting method via Aluminium as base Matrix,  $TiO_2$  and SiC as reinforcements in the wt. % of 5 & 10. For  $TiO_2$  Particulate composites the UTS observed were 153 & 182 Mpa, the YS were 142 & 149 Mpa respectively and for SiC Particulates composites UTS are 145 and 171 Mpa and YS are 117 and 123 Mpa. He reported that the YS and UTS of  $TiO_2$  Particulate composites were greater than that of reinforced with SiC Particulate composite for the same wt. % mainly because of grain refinement in the matrix.
1. M. Karbalaee Akbari et al [20] observed changes in weight loss and wear rate, in nano particulate composites. The resistance to wear of the composite is greater than that of the unreinforced alloy. The existence of ceramic nanoparticles protects the matrix and the  $TiO_2$  phase from the direct experience of the applied loads. In addition, as the nanoparticles are on the surface, this will lighten the partial shear stress accumulated to get relieved. The outcomes of the wear test exhibited that a substantial development in the wear resistance of the sample was eminent at 1.5 volume % of nanoparticles. An additional rise of nano particle content shows a decrease in wear resistance may be due to clustering phenomena. The laminate layer is clearly visible on the worn surface, which seems to protect the  $TiO_2$  surface from direct contact with the abrasive during the wear of these hard ceramic nanoparticles on the matrix interface and  $TiO_2$  particle.

Vinaykumar S Shet et al [21] showed that as  $TiO_2$  content increases in Al 6063 a marginal improvement of about 6.25%, 18.75%, 37.5%, 43.75, in wear resistance is observed in composites of Al6063 with 2, 4, 6 and 8% wt. and compared with Al-6063 matrix alloy. Absence of porosity in composite material will increase the contact between the sliding surfaces which may limit the surface harshness, along these lines it limits the contact weight and diminishes the chances of molecule separation during sliding. The impact connected to typical load on the wear rate of Al-6063 combination and the composites under sliding speed of 0.3141m/s is delineated. It is seen that with increment in typical load there is increment in wear rate of both the Al-6063 and Al-6063- $TiO_2$  composites. Wear rate can be increments with the expansion in stack in both Al6063 amalgam and Al6063- $TiO_2$  composites which can be credited to higher degree of plastic disfigurement.

The wear behaviour of an Al-2Mg alloy spray produced and the wear behaviour of Al-2Mg-11 $TiO_2$  composites made by stir casting were compared by S K Chaudhury [22] and his colleagues. Due to the sample's adhesion characteristics to the sliding disc, which has a high coefficient of friction value at the beginning phases of wear, a higher wear rate is observed at the initial stage. The wear rate of the

composite as a function of load shows that it is significantly lower than that of the matrix alloy (spraying and stirring), suggesting that the presence of a harder phase (in this case,  $\text{TiO}_2$  particles) will result in a lower wear rate. Additionally, under identical test conditions, the wear rate of the spray-formed composite under various loads was lower than that of the stir-cast composite. This difference can be attributed to the lack of a uniform distribution of  $\text{TiO}_2$  in the matrix phases, which leads to high porosity and inadequate interfacial bonding.

Ganesh Khandoori [23] and his collaborators have studied the wear behaviour of aluminium with  $\text{TiO}_2$  reinforcement in the ratios of 5%, 10% and 15%, processed by stir casting. The test clearly shows that as the load value increases, the mass loss of all three samples increases, but the rate is different. It is clear that as the  $\text{TiO}_2$  composition increases, the loss of mass decreases which convey that the mass loss of the composite decreases as the percentage of  $\text{TiO}_2$  increases. The relationship between the volumetric wear rate and the applied load at a constant sliding speed (2.0106 m/s) shows that the volumetric wear has a lower value for greater percentages by weight of  $\text{TiO}_2$  particles. The porosity causes a temporary increase in the composite's specific wear rate. Due to the relatively moderate effect on porosity, the wear rate of in situ cast composites with strong reinforcing particles increases slightly as the volume percentage of porosity increases. The link between the mass loss and the sliding distance between the constant load (25 N) and the sliding speed is stronger as the sliding distance rises.

## 1.7 Problem Formulation

Incorporating nano ceramic, intermetallic, or metallic particle reinforcement into a ductile matrix has produced a promising class of materials known as Nano Particulate Metal Matrix Composites (NPMNCs), according to a critical evaluation of the available literature. Aluminium based NMMC's can be used for lightweight structural and functional components for automotive and aerospace applications to further decrease cost of transportation, environmental pollution by lowering fuel consumption. Aluminium based MMNCs provide better tribological performances, better castability and dimensional stability, and superior machinability compared with aluminium alloys and aluminium based MMCs with micron sized particles (M Karbalaei Akbari et al [20]).

However, Nano  $\text{TiO}_2$  particles increases ductility of aluminium matrix composites compared with the same wt. % of micron sized particulate composites. In recent years, the nano-particles of  $\text{B}_4\text{C}$ ,  $\text{SiC}$  and  $\text{Al}_2\text{O}_3$  have also received attention as particle reinforcement for Aluminium based composites. The present study explores the possibility of reinforcing various nano particles with the sizes of 200nm, 60nm, 25nm and 15nm of  $\text{TiO}_2$  with LM0 aluminium alloy by using bottom pouring stir casting furnace, which possess outstanding features as mentioned in the following paragraphs.

Stir casting, a method of synthesising metal matrix composites (MMNCs), is less expensive for mass production than other manufacturing processes for generating particle-reinforced aluminum-based MMNCs (Ray [13]), hence it has received a lot of attention. Additionally, using traditional foundry procedures, composites made of aluminium may be manufactured into nearly-net shapes.



The industry and scientific community are paying more attention to aluminum-based nano composites and its alloys as there is a growing need for lightweight materials in automotive and aerospace applications (Ambreen Lateef [24], Charles Chikwendu Okpala [25], and Reddy BS [3]). Typical examples of the components made out of aluminium and its alloys for automotive application include piston, fuel injection pipe, cylinder head, suspension arms and steering systems, car doors etc., (M. Azeem Dafedar [26]).

If the size of the particle decreases the mechanical properties will also vary for the same volume or weight percent of reinforcement (K. R. Padmavathi [14]). So by considering the different factors, the choice of reinforcement in our current investigation has been narrowed down to nano  $\text{TiO}_2$  with particle size of 200nm, 60nm, 25nm and 15nm with Rutile grade, and 5 wt. % of magnesium is added in order to improve the wettability of the composites which are expected to remain stable in aluminium alloy matrix at elevated service temperatures. The reinforcement phases are expected to serve the interest of automobile industries in the contest of their need for new materials to be employed in various structural and tribological applications.

By varying the particles size the mechanical properties can be varied. Hall-Petch theory states that by reducing the particle size it is seen that the strength as well the ductility properties will also enhances (Sanaty-Zadeh [27], Luo P [28]). M. S. Islam [31] showed that the mechanical properties will increase with decrease of particle sizes until 10 nm. The decrease in 5 nm system was attributed to poor dispersions of nano-particles in the composites which results in decreased properties because of stress concentration effect of agglomerated particles.

## 1. OBJECTIVE OF THE PRESENT WORK.

In the present work an attempt has been made to Prepare, Characterize, Evaluation of Mechanical Properties and Tribological Behavior of LM0 alloy reinforced with various Nano Particle size of  $\text{TiO}_2$  particulates by stir casting route. Some of the specific objectives are:

- Preparation of LM0 with 200nm, 60nm, 25nm and 15nm  $\text{TiO}_2$  nano particulate composites to know the effect of reinforcements in steps of 0, 4, 8 and 12 wt. % by using bottom pouring casting furnace.
- Microstructural characterization of prepared composites by using Scanning Electron Microscope, Energy Dispersive Spectroscope and X-Ray Diffractometer to know the uniform distribution of nano  $\text{TiO}_2$  particles in the LM0 alloy matrix and to know the presence of Titanium and Magnesium in the various sized nano  $\text{TiO}_2$  particulate composites.
- Further, prepared samples are tested for mechanical properties like hardness, ultimate tensile strength, yield strength and percentage elongation as per ASTM standards of various nano sized  $\text{TiO}_2$  particulate composites.
- Studies on wear behaviour of various Nano sized  $\text{TiO}_2$  particulates reinforced LM0 alloy by considering varying parameters like applied load and sliding velocity by keeping sliding distance

constant at room temperature by using pin-on-disc wear testing machine.

## 2.0 Experimental Work

### 2.1 Experimental Set-up

The muffle furnace which is used for the melting of LM0 alloy is as shown in Fig. 1. The thick mild steel are used for outside surface of the furnace, and is wrapped by glass wool and hysil slabs for insulation. A control box is placed adjacent of the furnace which comprises of indicating lamps, On/Off switch, contactor and digital temperature along thru a thermocouple sensor. Insulation Bricks, Hard bricks, Ceramic Fiber blankets, accosite – 50 clay, silica solution, special type white Cement, Alumina brick etc. are used to construct the bottom pouring stir casting muffle furnace. At the bottom of the furnace a locking mechanism is adopted to allow the flow of composite to the mould.

#### Fig 1

Schematic diagram showing experimental set-up of bottom pouring stir casting furnace used for solidification processing of cast composites.

This experiment used a batch type stir casting furnace with a bottom pouring setup and a maximum melting capacity of 750 gram for the solidification procedure of all the different nano sized TiO<sub>2</sub>-Aluminum based nano composites. The following components make up the majority of the experimental setup:

#### 1. Power Supply Unit and Melting Unit.

The melting unit comprises of an electrical resistance heating vertical muffle furnace designed for a maximum input power of 2.5 kW and a maximum temperature of 1000°C. The furnace was constructed by winding an electrical resistance heating element, Kanthal A-1 rod with muffle of size 150 mm × 150 mm × 200 mm, having a total resistance of 21 ohms. One end of the muffle was kept open and the other end was closed with a hole at its centre. For melting Aluminium, a cylindrical graphite crucible having a diameter of 130 mm and 150 mm height with conical bottom with hole of 12 mm diameter was fabricated. The hole was closed by inserting a graphite plug in it. A lever arrangement was made for unplugging the crucible to pour the composite slurry into the mould, which is kept in the bottom of the furnace.

#### 1. Selection of Stirrer.

If an axial stirrer were used to stir molten metal or alloy, both axial and radial flows would result, with their relative magnitudes controllable by the stirrer's design to fit a desired condition of mixing. Additionally, the viscosity of the melt-particle slurry and the mixing method was crucial. For the solidification process of composites by stir casting, three types of stirrers, including flat blade stirrers, turbine blade stirrers, and

pitched blade stirrers, were frequently utilised. In the current experiment, zirconium-coated mild steel stirrers with pitched blades were chosen and made. A stirrer entailing four pitched blades (45° pitch angle), as shown in Fig. 2 was used in an effort to retain maximum amount of oxide particles and to result in uniform distribution of particles in the melt-particle slurry to be casted.

### 1. Matrix Material and Reinforcements.

## 2.2.1 Aluminium (LM0) Alloy

Al alloy is used as a matrix for obtaining composites, which has superior wear resistance, satisfactory mechanical properties at room temperature and at elevated temperatures. Al (LM0) alloy was chosen as it is having good castability and good resistance to corrosion. The main areas are automotive, aerospace, electrical applications, food and chemical industries, including plastic injection nozzles, as well as jewellery. Aluminium ingot supplied by FENFEE Metallurgical Company Limited. Bangalore, India, was having a purity and density of 99.65% and 2.73 g/cm<sup>3</sup> respectively.

## 2.2.2 Reinforcement Materials

Rutile grade Nano sized Titanium dioxide (TiO<sub>2</sub>) powder with the particle sizes of 200nm, 60nm, 25nm, and 15nm was selected as reinforcement to know the effect of particle size on aluminium based nano composites. TiO<sub>2</sub> powder supplied by ANHUI ELITE Industrial Company Limited. HEFEI, China, was having a purity and density of 99% and 4.23 g/cm<sup>3</sup> respectively.

## 2.2.3 Magnesium

Magnesium is used to improve wettability of composites and to reduce the coating film defects by ejecting the unstable components present in the melt at the time of casting. Magnesium ingot supplied by FENFEE Metallurgical Company Limited, Bangalore India, was having a purity and density of 99.5% and 1.74 g/cm<sup>3</sup> respectively. The magnesium ingot is machined and added to the melt at the time of synthesizing the aluminium composites.

### 1. Al-Nano TiO<sub>2</sub> Composites Processing.

About 750 grams of commercial pure aluminium was melted to a required processing temperature in a clay-graphite crucible kept inside the muffle furnace. Prior to any accumulation, the surface of the melt was cleaned by skimming, the weighed elemental nano-TiO<sub>2</sub> particles were preheated to about 400° C and the rate of addition of the particles was controlled at a rate of about 0.3 to 0.5 g/s. The TiO<sub>2</sub> nanoparticles were dispersed in the melt using a steel pitched blade stirrer having four pitched blades (45° tilt angle). The speed of the stirrer was kept constant at 500 rpm and the stirring speed was measured using a non-contact speed sensor tachometer.

The temperature of the melt was measured using a digital temperature indicator connected to a chromel-alumel thermocouple at a depth of 15 to 20 mm inside the melt. The temperature of the slurry was

maintained at  $\pm 10^\circ \text{C}$  of the processing temperature throughout the stirring process. Prior to the addition of the  $\text{TiO}_2$  nanoparticles, 5% by weight of magnesium chips were wrapped in aluminium foil and inserted into a slurry of molten particles. When the desired stirring time had elapsed, the stirrer is stopped and removed from the crucible. Then, the graphite plug at the bottom of the furnace was removed and the slurry of melt-particle was poured into a pre-heated permanent type mold of split type, having a size of 40 x 40 x 150 mm kept below the plug. No degassing of the melt or slurry is carried out at any stage of the process.

The cast nanocomposite ingot was immediately cooled by immersion in to water bath. The nano composite has been designated on the basis of its constituents. The first letter indicates the base metal, Al in the present case for aluminium followed by NP which indicates the nano particle size of  $\text{TiO}_2$  and 4, 8 & 12 numbers indicate amount of nano  $\text{TiO}_2$  added to the aluminium based composite along with its amount in wt. %. The scheme of variation of the constituents, their nominal amounts of different cast Al-based nano composites are shown in Table 1.

Table 1

The constituents and their nominal amounts used for solidification processing of different cast Al-Nano  $\text{TiO}_2$  Composites.

Al-Nano $\text{TiO}_2$ Composites with designation	Aluminium (wt. %)	Magnesium (wt. %)	Particle Size in Nano- Meter	Titanium Di-Oxide ( $\text{TiO}_2$ ) (wt. %)
Al-0NP	95	5	---	---
Al-4NP1	91	5	200	4
Al-8NP1	87	5	200	8
Al-12NP1	83	5	200	12
Al-4NP2	91	5	60	4
Al-8NP2	87	5	60	8
Al-12NP2	83	5	60	12
Al-4NP3	91	5	25	4
Al-8NP3	87	5	25	8
Al-12NP3	83	5	25	12
Al-4NP4	91	5	15	4
Al-8NP4	87	5	15	8
Al-12NP4	83	5	15	12

## 2.4 Microstructural Studies

## 1. Scanning Electron Microscopy (SEM).

The study of a material's microstructure is called metallography. Analysis of the microstructure of the material determines whether the material has been properly processed and the mechanical properties depend on how the nanoparticles are distributed in the composite and therefore constitute a crucial step in determining the reliability of the product and determining the reason for failure of material. Field Emission Scanning Electron Microscopy (FE-SEM), Carl Zeiss, German model: Neon 40. The SEM study was carried out with an acceleration potential of 2 to 10 kV, with a magnification of 100K. Basic steps in the preparation of suitable metallographic samples include: segmenting and cutting, mounting, plane grinding, rough polishing, final polishing and microscopic analysis.

### 1. X-Ray Diffraction Analysis.

A NDT technique used to examine the crystalline structure of crystalline materials is called X-Ray Diffraction (XRD). The materials utilised for the microstructure investigation were analysed using an X-ray diffractometer (XRD) with a D8 Advance, Bruker axs, Karlsruhe, Germany, in the two theta range of 20°-100° utilising a CuK radiation target and nickel filter.

### C) Energy Dispersive X-Ray Spectroscopy (EDS).

The metallographic samples of different cast composites of different Nano particle sizes of TiO<sub>2</sub> reinforcements for 8 Wt. %, counter parts are already prepared for SEM were also examined under FE-SEM with EDS attachment, Carl Zeiss, Germany Model: Neon 40 at an accelerating potential of 15 kV. The intermetallic phases in the microstructure of the different particle sized cast composites were recognized and quantitative chemical analysis was carried out by EDS. During EDS investigation, the beam was selectively focused on the phase constituents of relatively larger size in order to obtain reliable results by avoiding any significant influence of matrix alloy.

## 2.5 Studies on Mechanical Properties

### 1. Hardness Testing.

As stated in ASTM E10, the Brinell hardness test method is utilised to estimate BHN. Using a ball indenter with a 10 mm diameter and a 500 kg applied stress, produced nano composites' hardness is evaluated. A sample was subjected to a load for around 180 seconds using a ball indenter, and the diameter of the indentation was then determined with the aid of a travelling microscope. The equivalent hardness was determined for each depression by taking the average of two diameters that were measured perpendicular to one another. Each sample had at least thirty indentations made for hardness measurement at various points, and the material's hardness value was determined by averaging these measurements.

### b) Tensile Testing

With the use of a universal testing machine, the UTS, YS, and % elongation of the unreinforced alloy and for various nano-sized  $\text{TiO}_2$  particles are measured. An extensive tensile test record can yield information on a number of characteristics. You may get crucial details about the material's elasticity, plastic deformation properties, yield strength, tensile strength, and toughness. The fracture surfaces were subsequently examined using FE-SEM, and the distinctive characteristics of each fracture surface were photographed. The dimensions of Tensile Specimen is shown in Fig. 3 used for research work.

### Figure 3

Dimension of specimen used for evaluating tensile properties.

## 2.6 Wear Testing

A cylindrical pin with a polished and flat end was moved against the counterface of the hardened steel plate to conduct a dry sliding wear test in accordance with ASTM-G99 standard as shown in Fig. 4 under ambient conditions (relative humidity = 35–60%, temperature = 21–30° C). The Pin on Disc wear testing device is a TT-10 type made by DUCON, Bangalore, India. According to Fig. 4, the test pin has a diameter of 10 mm and a length of 30 mm. The counterface disc is constructed of EN-32 steel and has a 62–65 HRC hardness. Different loads of 9.81 N, 19.62 N, and 29.43 N were applied to the pins perpendicular to the sliding contact during the wear test of each cast composite. The track's radius stays constant at 40 mm, while the disc's rotational speed was held constant at 200, 300, and 400 rpm, translating to a linear speed of 1.0 m/s. With a total sliding distance of about 3000 m, a wear test was conducted. The cumulative volume loss is measured by stopping the wear test at regular sliding distance intervals.

After the wear test, the tip of the test pin is cut in a plane parallel to its wear surface. SEM was used to examine the characteristics of all worn surface samples. Wear debris generated during the wear test of the various nano sized  $\text{TiO}_2$  particulate composite and the unreinforced alloy were carefully collected and examined under an SEM.

All dimensions are in mm.

### Figure 4

Shape and size of the wear and friction test specimen.

## Results And Discussion.

### 3.0 Microstructure Analysis

#### 1. Structural Characterization of Various Sized Nano Particulate Composites.

- Ray Diffraction Studies.

Typical XRD patterns of the 1 cm<sup>3</sup> specimen of cast composite, Al-8NP1, Al-8NP2, Al-8NP3 and Al-8NP4 are taken in a Diffractometer with Cu-K $\alpha$  radiation in copper medium with an angle ranging from 100 to 1000 at gynometer speed 20/min as shown in Fig. 5 (a), (b), (c) and (d) respectively. The peaks in the XRD pattern of cast various sized nano composites as shown in the figure reveals the presence of primary solid solution of aluminium phase. The XRD pattern of the cast nano composite marked in the Fig. 5, shows presence of three peaks one at about  $2\theta = 38^\circ, 46^\circ, 66^\circ$  and  $78^\circ$ , second at  $2\theta = 26^\circ, 28^\circ$  and  $74^\circ$  and the third one at  $2\theta = 34^\circ$  and  $42^\circ$  respectively, the strongest peak belonging to aluminium and the other two peaks belonging to TiO<sub>2</sub> and MgO respectively.

### 1. Microstructure Study of Various Sized Nano TiO<sub>2</sub> Particulate Composites.

The typical microstructure consists of primary Aluminium matrix (dark gray) and nano TiO<sub>2</sub> particles (whitish color). The microstructure clearly shows the uniform dispersal of the TiO<sub>2</sub> particles in the LM0 Aluminium alloy matrix and, because of the greater friction amongst the particles and the matrix Al alloy the vortex created during the stirring process breaks the solid dendrites because of these very low agglomeration and particle segregation are observed. The SEM image shows that the Al 12 wt. % nanocomposite has more clustering and agglomeration than the 4 and 8 wt. % composites. Because of the strong wettability between the matrix and reinforcement particles that can be seen in the SEM pictures, the produced composites had less porosity. Figure 6 display the SEM image of 200nm particle size with uniform particle distribution.

Because of the high surface energy of the particles and the non-uniform distribution of temperature and mass flow in the combustion flame, it is evident from the SEM image that the Nano TiO<sub>2</sub> particles do not have a uniform shape. A reasonably uniform distribution of TiO<sub>2</sub> particles, as seen in the micrograph, may result in strong interfacial bonding between the matrix of LM0 Al alloy and TiO<sub>2</sub> particle.

As the particle size decreases in the designated composites the clustering and agglomeration is more which is observed especially in the 15nm and 25nm particles of TiO<sub>2</sub> nano composites which is evident in Figs. 7, 8 and 9. In order to avoid clustering and agglomeration of TiO<sub>2</sub> particles in the nano composites, the TiO<sub>2</sub> nano particles has to be preheated and the continuous stirring is done while adding the particles to the melt which avoids the settling of particles at the bottom of the matrix.

[

(a) (b) (c)

The EDS study for the 200nm with 8 Wt. % of TiO<sub>2</sub> particulate composites, which reveals the presence of TiO<sub>2</sub> particles in the cast nano composites. The major elements which are present in the composite are Al, TiO<sub>2</sub>, Mg and MgO respectively. The EDS test have to be carried out at 40,000 magnification with 15 KV voltage, because of high magnification and higher voltage we can observe the dark patches between the particles and the matrix. The reason may be because of high voltage concentrates at focused point

which leads to burn sample surface and forms a black patches and also drifting of the specimen is seen because of high electrical conductivity property of the pure aluminium as shown in Fig. 10.

## Figure 10

EDS point analysis of the matrix and different types of particles in cast Al – TiO<sub>2</sub> Nanocomposites.

### 1. Mechanical Properties.

## 4.1 Hardness

Figure 11 shows the average BHN for the various particle sizes of 200nm, 60nm, 25nm & 15nm TiO<sub>2</sub> of 0, 4, 8 & 12 wt. %. From the results we can see that as the size of the nano particle decreases there is an increase in BHN value, due to the small spacing between the nano particles, and higher rate of work hardening. Reduction of particle size to nano-meter increases the direct strengthening and indirect strengthening effect compared to micron sized particle composites. The interfacial area between the matrix and the nano TiO<sub>2</sub> particles reduces as the size of the TiO<sub>2</sub> nanoparticles decreases, allowing more load to be transmitted from the matrix to the nano TiO<sub>2</sub> particles. A smaller interfacial area can also help the matrix generate fewer dislocations, improving the composite's mechanical characteristics.

The 200nm TiO<sub>2</sub> particles are considered as reference for comparison of BHN values of 60nm, 25nm & 15nm TiO<sub>2</sub> particulate composites. For the 4 wt. % the percentage increases in BHN are 13.1, 25.9 & 35.2% respectively. Similarly for 8 wt. % the BHN value increases to 7.2, 18.5 & 26.2%, and for 12 wt. % the observed value of BHN increases to 8.4, 18 & 22.2% respectively.

Tremendous increase in BHN value is observed for the 15nm & 25nm TiO<sub>2</sub> particulate composites compared with 200nm and 60nm, this is because of hardness nature of TiO<sub>2</sub> reinforcement which reduces the inherent property of hardness to the matrix material, thus increasing its resistance to deformation. The hardness of the ductile aluminium matrix material can be enhanced by accumulating nano TiO<sub>2</sub> particles. Particle size, the fine and equal distribution of nano reinforced particles, and the grain refinement of the aluminium matrix alloy in the composites all have an adverse effect on the hardness performance. The hardness of composite materials is continuously improved by the reduction of nanoparticle size; the smaller the particle, the more obstacles there will be for dislocation motion, which strengthens the resistance to plastic deformation and raises the hardness value for smaller nanosized TiO<sub>2</sub> particles.

## 4.2 Tensile Properties

The Fig. 12 (a), (b) and (c) shows the tensile properties – YS (yield strength), UTS (ultimate tensile strength) and percentage elongation of cast nano composites of different particle sizes of 200nm, 60nm, 25nm & 15nm TiO<sub>2</sub> particles of 0, 4, 8 & 12 wt. %. As the particle size decreases there is an increase in



overall Tensile properties, because it is expected that the bigger- sized particles fracture more easily than the smaller ones in tensile testing. The homogeneous dispersion of TiO<sub>2</sub> nanoparticles and their robust adhesion to the aluminium matrix are the key contributors to the increase in tensile strength.

The LM0 alloy reinforced with 15 nm particle size of TiO<sub>2</sub> composites show highest Tensile Properties when compared with 200nm, 60nm & 25nm particle size, the increase in Tensile properties is because of Orowan mechanism, the 15nm TiO<sub>2</sub> particles act as barriers to obstruct the motion of dislocations near the particles in the matrix. The tremendous interfacial bond amongst the matrix and the reinforcing material plays a vital role in enhancing the tensile properties of the composite. The fine size and shape of the nano reinforcement correspondingly contributed to enhance the UTS and YS of the composite.

The below Table 2, 3 and 4 displays the increase in Tensile properties as the TiO<sub>2</sub> nano particle size decreases for the 4, 8 & 12 wt. %, the 200nm TiO<sub>2</sub> particulate composite is used as the base for comparing the tensile properties of other three nano sized particulate composites. From the table we can see that the YS increases drastically for 25nm & 15nm particles compared to 60nm particle size which is mainly due to dispersion strengthening increases as the particle size decreases. For the 12 wt. % much difference is not seen for 25nm & 15nm particles of TiO<sub>2</sub> because of clustering and agglomeration increases with decreasing particle size and increasing wt. % of TiO<sub>2</sub> particles.

Table 2  
Comparison of YS of 15nm, 25nm & 60nm WRT 200nm Particles of TiO<sub>2</sub>.

Sl. No	Wt. % of Reinforcement	Percentage of increase in YS of different nano particle size of TiO <sub>2</sub> in percentage w.r.t 200nm TiO <sub>2</sub> particle size.		
		60nm	25nm	15nm
1	4	12.8	36.6	47
2	8	17.8	45.3	55.1
3	12	9.6	43.3	48.9

Table 3  
Comparison of UTS of 15nm, 25nm & 60nm WRT 200nm Particles of TiO<sub>2</sub>.

Sl. No	Wt. % of Reinforcement	Percentage of increase in UTS of different nano particle size of TiO <sub>2</sub> in percentage w.r.t 200nm TiO <sub>2</sub> particle size.		
		60nm	25nm	15nm
1	4	7.9	30.11	42.9
2	8	7.5	33.66	44.43
3	12	6.9	39.63	49.83

Table 4  
Comparison of Ductility of 15nm, 25nm & 60nm WRT 200nm Particles of TiO<sub>2</sub>.

Sl. No	Wt. % of Reinforcement	Percentage of increase in Ductility of different nano particle size of TiO <sub>2</sub> in percentage w.r.t 200nm TiO <sub>2</sub> particle size.		
		60nm	25nm	15nm
1	4	1.75	4.67	7.79
2	8	2.54	4.33	7.29
3	12	2.47	3.07	5.88

The UTS also increases with decreasing nano particle size of TiO<sub>2</sub>, because of strain hardening rate of composites is low for 15nm & 25nm particles of TiO<sub>2</sub> compared with 200nm & 60nm particles. The smaller nano sized particles will act as barriers which prevent plastic deformation of the matrix. The 15nm & 25nm TiO<sub>2</sub> particles only have capability of elastic deformation, although, the aluminium matrix has capability of plastic deformation. It seems that there is a strong interface between the nanoparticles and matrix which would ban the plastic deformation that lead to strain hardening phenomenon.

Al-12NP4 TiO<sub>2</sub> composites have a higher tensile strength due to two factors: first, each particle has a tiny interfacial area with the matrix, allowing it to withstand larger stress concentrations. Second, the inherent defects in the particle regulate the fracture strength of the particle. Larger particles will fracture more readily because they have a higher statistical likelihood of containing a fault that is larger than the critical size because the size and quantity of flaws are constrained by the particle size. The composites with larger TiO<sub>2</sub> particle sizes demonstrated worse mechanical characteristics in comparison to those with smaller particle sizes because the broken particles cannot withstand any further load and instead act as favoured failure sites.

From the above Table 2, 3 and 4 we can see that the ductility decreases with increase in both wt. %, and size of nano TiO<sub>2</sub> particle. The ductility of 15nm TiO<sub>2</sub> is more when compared with other particle size and for 12 wt. % the ductility decreases mainly due to agglomeration and clustering of TiO<sub>2</sub> particles. The Ductile-Brittle mode of failure is seen in the cast particulate nano composites.

The fractured surfaces of tensile specimens of cast various sized nano TiO<sub>2</sub> particulate composites concludes that as the size of the nano particle decreases and wt. % of nano TiO<sub>2</sub> increases in cast particulate composite, the size of the void decreases due to increased number of particles and limited the growth of void due to reduced ductility. As the nano particle size of TiO<sub>2</sub> decreases to 25nm and 15nm the ductility of the developed composites increases for the same wt. % of 200nm and 60nm TiO<sub>2</sub> particulate composites. It may be recognized as the particle size decreases the ductility increases but care has to be taken to avoid agglomeration and clustering of particles. The fractured surfaces of 15nm

and 25nm TiO<sub>2</sub> particulate composites shows slightly more dimples compared to those observed in 200nm and 60nm TiO<sub>2</sub> particulates composites which confirms the presence of ductility.

## 1. Tribological Behaviour of Various Nano Sized TiO<sub>2</sub> Particulate Reinforced Composites.

### 1. Effect of Load on Wear Rate.

The Wear test was conducted under dry condition on a pin-on-disc apparatus under normal room temperature according to ASTM standard by varying load in terms of 9.81, 19.82 and 29.43N respectively by keeping speed and sliding distance constant that is 400rpm and 3000m. The tribological behavior of nano particulate composites depends on the processing technique and parameters followed at the time of synthesizing, along with suitable reinforcement choice, particle size and shape, nature of the reinforcing phases, and ceramic particle spatial distribution in the aluminium matrix. To achieve better characteristics, the interface between the nano-TiO<sub>2</sub> particle and the aluminium matrix must be clean and devoid of porosity and reaction products.

The load effects on 200nm, 60nm, 25nm, and 15nm TiO<sub>2</sub> particle composites are shown in Fig. 13 (a), (b), and (c), respectively, for loads of 9.81N, 19.82N, and 29.43N. As can be seen in Fig. 13, the wear rate increases as the load does, but it reduces as the weight does. The percentage of nano TiO<sub>2</sub> particles rises as the size of the nano TiO<sub>2</sub> particles falls. Nano particle composites have greater wear resistance than non-reinforced alloys. Ceramic TiO<sub>2</sub> nanoparticles may act as a barrier between the matrix and TiO<sub>2</sub> phases and the applied load from the counter face. Subsequently the nanoparticles are on the surface, some of the shear strains that have accumulated in the subsurface are relieved as a result. An important role in the wear process is played by the bonding of the nano-sized TiO<sub>2</sub> particles to the matrix LM0 Al alloy. The wear rate falls in direct proportion to the increase in bond strength caused by increasing nano reinforcing.

From the Fig. 13a significant increase in wear rate can be seen in the early phases, which stabilises as it progresses. Due to the nano particle composites adhesive properties toward the sliding disc, which exhibits a high value of wear rate, the initial wear rates are higher. Because of the Orowan principle, which states that smaller nanoparticles will fill more of the available space in nanoparticle composites, the wear rate lowers as the particle size increases.

The Table 5 shows the increase in wear behaviour as the TiO<sub>2</sub> nano particle size decreases for the 4, 8 & 12 wt. %, the 200nm TiO<sub>2</sub> particulate composite is used as the base for comparing the wear behaviour with varying load of other three nano sized particulate composites. From the table it can be seen that as the size of the nano TiO<sub>2</sub> particle decreases the resistance to wear rate increases and tremendous increase to wear rate resistance has been seen for 25nm and 15nm TiO<sub>2</sub> particles compared to 60nm particulate composites.

Table 5  
Comparison of wear rate by varying load of 15nm, 25nm & 60nm WRT 200nm Particles of TiO<sub>2</sub>.

Sl. No	Load Applied in N	Wt. % of Reinforcement	Percentage of increase in wear rate of different nano particle size of TiO <sub>2</sub> in percentage w.r.t 200nm TiO <sub>2</sub> particle size.		
			60nm	25nm	15nm
1	9.81	4	15.1	33.46	43.84
		8	25.86	34.9	46.98
		12	31.06	40.7	47.57
2	19.62	4	10.8	23.64	35.78
		8	17.19	32.6	45.96
		12	17.28	32.92	41.56
3	29.43	4	11.57	30.00	39.73
		8	14.58	31.54	44.04
		12	12.72	30.38	42.04

### 1. Effect of Speed on Wear Rate.

The Fig. 14 (a), (b) and (c) shows the effect of speed on 200nm, 60nm, 25nm and 15nm TiO<sub>2</sub> particulate composites respectively for 200, 300 and 400 rpm speed. From the Fig. 14 it is observed that as the speed increases the wear rate also increases but correspondingly the wear rate decreases as the wt. % of nano TiO<sub>2</sub> particle increases and the particle size of nano TiO<sub>2</sub> decreases. The effect of speed on wear rate is more compared to the effect of load because at higher sliding speed there will be increase in surface temperature between pin and disc in dry sliding so that the subsurface layer may totally peel off.

(b)

#### 1. (b)

The results show that the samples wear resistance have significantly improved for 12 wt. % of composites with nano TiO<sub>2</sub> particles measuring 15 nm and 25 nm, respectively. It is clear that 15nm and 25nm nanoparticles have favourably improved the wear resistance of composites compared to 200nm and 60nm since the depth of the deformed zone in these composites is noticeably lower than that of the 60nm and 200nm particulate composites.

The Table 6 shows the increase in wear behaviour as the TiO<sub>2</sub> nano particle size decreases for the 4, 8 & 12 wt. %, the 200nm TiO<sub>2</sub> particulate composite is used as the base for comparing the wear behaviour with varying speed of other three nano sized particulate composites. From the result we can see that at lower speed the wear rate is less because of mild wear as speed increases the wear rate also increases

irrespective of wt. % and particle size because at higher speed the mild mode will be converted to severe mode mechanism of wear.

Table 6  
Comparison of wear rate by varying speed of 15nm, 25nm & 60nm WRT 200nm Particles of TiO<sub>2</sub>.

Sl. No	Speed in RPM	Wt. % of Reinforcement	Percentage of increase in wear rate of different nano particle size of TiO <sub>2</sub> in percentage w.r.t 200nm TiO <sub>2</sub> particle size.		
			60nm	25nm	15nm
1	200	4	12.98	30.17	39.29
		8	16.73	28.68	40.63
		12	23.78	37.44	46.69
2	300	4	13.41	21.72	33.22
		8	16.84	27.95	39.78
		12	22.13	30.43	39.52
3	400	4	13.82	28.45	40.92
		8	13.93	30.03	40.86
		12	21.10	32.52	46.02

**1.11 Worn Surfaces and Wear Debris.**

As the nano TiO<sub>2</sub> content increases the depth, grooves and delamination decreases gradually because of the presence of hard nano TiO<sub>2</sub> which will protect the surface from wear. The depth and number of grooves in the worn surface decreases as the particle size decreases from 200nm to 15nm particulate composites which is evident from the Figs. 15, 16, 17 and 18. The delamination has been reduced on the worn surface of the nano particulate composites compared with 200nm TiO<sub>2</sub> particulate composites.

(a) Pure Al (b) 4% (c) 8% and (d) 12.

(a) Pure Al (b) 4% (c) 8% and (d) 12.

(a) Pure Al (b) 4% (c) 8% and (d) 12.

It is observed that the collected debris is continuous for Al-NP0 composites (without reinforcement) and severe plastic deformation has been observed and for 8wt. debris collected is not continuous and glossy metallic wear with irregular shaped platelet has been seen for all the sizes of the TiO<sub>2</sub> Nano Particulates Composites as shown in Fig. 19.

## 6.0 Conclusions

1. X-Ray Diffraction (XRD) shows the existence of Al, TiO<sub>2</sub> and MgO for all the nano particle sizes of TiO<sub>2</sub> composites but the intensity of the generated peak increases with decreasing TiO<sub>2</sub> particle sizes.
2. The cast microstructure of 200nm, 60nm, 25nm and 15nm TiO<sub>2</sub> particulate shows the uniform distribution of TiO<sub>2</sub> nano particles as the wt. % of TiO<sub>2</sub> increases and as the size of nano TiO<sub>2</sub> particles decreases the clustering and agglomeration phenomenon has been observed in the developed nano particulate composites.
3. EDS studies carried out for phase identification in the microstructure of cast composite of 8 wt. % on the basis of chemical composition of 200nm, 60nm, 25nm and 15nm confirm the presence of Al, TiO<sub>2</sub>, Mg and MgO respectively but because of the electrical conductivity property of the pure Al used and because of higher magnification and higher voltage caused the drifting of specimen at the time of testing for lower particle sized nano composites.
4. EDS colour mapping shows the confirmation of Aluminium, Oxygen, Magnesium and Titanium are evenly distributed in the developed nano composites.
5. The Brinell hardness of 200nm, 60nm, 25nm and 15nm particulate composites increases with increasing wt. % and decreasing size of TiO<sub>2</sub> nano particle. Tremendous increase in BHN value is witnessed for the 15 & 25nm TiO<sub>2</sub> particulate composites compared with 200nm and 60nm, this is because the smaller the grain size, the greater will be the obstacle of the dislocation movement, thus improving the resistance to plastic deformation resulting in increased hardness value.
6. The yield strength of various nano sized TiO<sub>2</sub> particulate composite increases with increasing particle content, The LM0 alloy reinforced with 15nm and 25nm particle size of TiO<sub>2</sub> composites show highest yield strength when compared with 200nm and 60nm particle size, the increase in yield strength is because of Orowan mechanism and mainly due to dispersion strengthening increases as the particle size decreases.
7. UTS is observed to increase at relatively for lower particle sizes of nano TiO<sub>2</sub> that is for 15nm and 25nm particulate composites which may be attributed to progressive bonding of particles to matrix during plastic deformation and the smaller nano sized particles will act as barriers which prevent plastic deformation of the matrix.
8. The ductility decreases as the wt. % of TiO<sub>2</sub> increases, but interestingly as the size of the nano TiO<sub>2</sub> particle decreases, the ductility increases and as the wt. % of nano TiO<sub>2</sub> increases in cast composite, the size of the void decreases. This is may be due to increased number of particles and limited the growth of void.
9. During dry sliding wear against hardened steel counter face, the wear rate in the alloys and nano composites investigated is primarily following the order of hardness observed in these materials and so, it is primarily the function of real area of contact.
10. As the load increases the wear rate also increases but correspondingly the wear rate decreases as the wt. % of nano TiO<sub>2</sub> particle increases and the particle size of nano TiO<sub>2</sub> decreases. The existence of ceramic TiO<sub>2</sub> nanoparticles might protect the matrix and TiO<sub>2</sub> phases from direct experience of applied load from the counter face.

11. As the speed increases the wear rate also increases but correspondingly the wear rate decreases as the wt. % of nano TiO<sub>2</sub> particle increases and the particle size of nano TiO<sub>2</sub> decreases. The effect of speed on wear rate is more compared to the effect of load because at higher sliding speed there will be increase in surface temperature between pin and disc in dry sliding so that the subsurface layer may totally peel off which may result in severe wear mechanism.
12. It was observed that the deformation zone depth of the 15nm and 25nm nanocomposites was significantly lower than that of the 60nm and 200nm particulate composites, which indicates that 15nm and 25nm particles have favourably enhanced the wear resistance of composites compared to 200nm and 60nm.
13. The depth and number of grooves in the worn surface decreases as the particle size decreases from 200nm to 15nm TiO<sub>2</sub> particulate composites. The delamination has been reduced on the worn surface of the nano particulate composites compared with 200nm TiO<sub>2</sub> particulate composites.
14. Over all we can see that as the nano particle size of TiO<sub>2</sub> decreases attributed to tremendous increase in mechanical properties and wear resistance.
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## **2. Conflict of interest:**

3. **There is no conflict of interest from all the authors.**

## **4. Author's Contribution:**

1. **Raghu S: Conceptualization, Methodology, experimentation and writing original paper.**
2. **H M Nanjundaswamy: Guiding in Experimentation and Results obtained.**
3. **Jayasheel I Harti: Editing of paper.**
4. **Manjunatha B: Data Collection and Tabulation work.**
5. **Sreenivasa M: Graph plotting for the Obtained results.**
6. **Sujana N: Formatting and Grammar Corrections in the paper.**

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## Unsectioned Paragraphs

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