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SYNTHESIS AND MECHANICAL CHARACTERIZATION OFALUMINIUM REINFORCED WITH VARIOUS NANO-SIZED TIO_2 PARTICULATE COMPOSITE

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ABSTRACT

The Aluminium metal matrix Nano composites can be used in numerous applications because of their Excellent Mechanical, Tribological Properties and Light Weight. To enhancethe performance of composite matrix in aluminium alloy metal is reinforced with Nano particles in the matrix of alloy uniformly, which ameliorates composite properties without affecting limit of ductility. The paper presents the results of experimental investigation on mechanical properties of Various Nano sizes of Particles of 25 and 60 Nano meter Titanium-di-oxide (TiO_2-p) particles reinforced with LM0 aluminium metal matrix. The influence of reinforced ratios of 0, 4, 8 and 12 wt. % of TiO_2-p on mechanical properties was examined. It is commonly accepted that decreasing the reinforcement size improves mechanical properties of the composites for a given particle volume fraction, because of the smaller inter-particle spacing and larger work hardening rate. The decrease in the particle size increases both the effects of direct strengthening and indirect strengthening. The composites were synthesized by Stir Casting method by using bottom pouring muffle furnace and Magnesium was added to the melt in order to improve the wettability of the composites. Care has to be taken while synthesis of composites because as the Particle size decreases the clustering and agglomeration is seen in the composite.

Keywords:*Nano TiO_2, LM0alloy, Nano Composites, Stir Casting, Hardness and Tensile Properties.*

INTRODUCTION:

Metal matrix composites (MMCs) reinforced with nano-particles, also called Metal Matrix nano-Composites (MMNCs), and are being investigated worldwide in recent years, owing to their potential properties suitable for a large number of functional and structural applications. The reduced size of the reinforcement phase down to the nano-scale is such that interaction of particles with dislocations becomes the significant importance and, when added to other strengthening effects typically found in conventional MMCs, results in a remarkable improvement of mechanical properties [1–4].

The main issue to be faced in the production of MMNCs is the low wettability of ceramic nano-particles with the molten metal matrix, which do not allow the production of MMNCs by conventional casting processes. Small powder aggregates are in fact prone to form clusters, losing their capability to be homogeneously dispersed throughout the matrix for an optimal exploitation of the strengthening potential. For this reason, several alternative methods have been proposed in order to overcome this problem.

The production methods can be categorized into two major groups: ex situ and in situ. The first synthesis route consists of adding nano-reinforcements to the liquid or powdered metal, while in situ processes refer to those methods leading to the generation of ceramic nano-compounds by reaction during processing, for example by using reactive gases. Several methods have been developed for ex situ synthesis of MMNCs. In particular, different powder metallurgy techniques were successfully employed. Moreover, ultrasound-assisted casting plays a particularly promising role for its high potential productivity. Alternative methods are listed and discussed in a following section.

In recent years ceramic particle reinforced metal matrix composites (MMCs) have gained wide acceptance because of their attractive properties. Nano MMCs offer high strength to weight ratio, high stiffness and good wear resistance resulting in an ever increasing use in the aerospace, automotive and bio-medical industries [5].

R. A Sagar et al [6] in his study showed that TiO_2 has been used widely for the preparation of different types of Nano materials, including nanoparticles, Nano rods, Nano wires, Nano tubes, and meso-porous and Nano porous TiO_2 – containing materials.

Cammarata R [7] defined nanocomposite as a multiphase solid material where one of the phases has one, two or three dimensions of less than 100 nanometers (nm), or structures having Nano-scale repeat distances between the different phases that make up the material. The mechanical, electrical, thermal, optical, electrochemical, catalytic properties of the nanocomposite will differ markedly from that of the component materials.

The general idea behind the addition of the nanoscale reinforcement is to create a synergy between the various constituents, such that novel properties capable of meeting or exceeding design expectations can be achieved. The properties of nanocomposites rely on a range of variables, particularly the matrix material, which can exhibit nanoscale dimensions, loading, degree of dispersion, size, shape, and orientation of the nanoscale second phase and interactions between the matrix and the second phase. Recently research work is focused on Nano metal matrix composites (NMCs) due to its unique properties such as, high strength with light weight [8].

Nano particles has tendency to agglomerate under vendors walls forces. So inclusion of these nano particles reduces the interfacial interaction between resin and reinforcement. Dispersion of these nano particles in matrix phase also has influence on mechanical and thermal behavior of composites. Many fabrication methods were tried for uniform distribution of these nano particles into the matrix phase of polymer composites [9].

A. Chennakesava Reddy[10]Synthesised Titanium oxide nanoparticle reinforced with AA6061 metal matrix composite specimens by high pressure die casting technique by using bottom pouring stir casting technique. The effect of porosity and clustering content and their influence on micromechanical properties were evaluated in as-cast condition by finite element method and experimental procedure. Porosity and clustering were measured with different volume percent's of 10, 20 and 30% titanium oxide nanoparticles reinforced to AA6061 metal matrix composites. Results showed that the Porosity content was significantly linear with increasing percentage of titanium oxide nanoparticles in the clusters. Increase in volume fraction of TiO_2 nanoparticles has reduced the tensile strength and stiffness of AA6061/TiO_2 metal matrix composites.

K. Yoganandam et al [11] in his study showed that when Al6082 is reinforced with Titanium Oxide (TiO_2) particles. Aluminum-TiO_2 composites reinforced with various weight percentages (0, 3, 6 and 9 wt. %) were produced by semi-solid state compo casting route. The microstructure of fabricated composites and monolithic alloys were examined by Optical Microscope (OP). The test results show that the mechanical behaviours of the fabricated composites are enhanced by increasing the Titanium Oxide content. The UTS and hardness of the produced composite enhanced with the addition of higher percentage of TiO_2.

Amal E. Nassar, et.al. [12] Fabricated Pure aluminium Nano composite reinforced with Nano titanium dioxide by powder metallurgy route. Measurements of tensile strength, hardness, and density showed that the porosity and the tensile strength of composites increased with an increase in volume fraction of nanoparticles; however ductility of aluminum was decreased. Wear test revealed that composites offer superior wear resistance compared to alloy.

R. D. Snahnican Frantisek et.al [13], created Al–TiO_2 Nano composites with various amounts of titanium di oxide, using powder metallurgy technique. The obtained composites were examined for structural, wear and mechanical properties. Hardness of Al–TiO_2 increases with an increase in the amount of TiO_2 nanoparticles, maximum hardness was 82 BHN and it resulted from the sample containing 4.5 vol. % of TiO_2 nanoparticles.

N. A.Thangarasua et.al [14],investigated that the tensile test result with the addition of Nano TiO_2 particles to Al matrix composites which increases the tensile strength of the alloy at room temperature along with an increase in the TiO_2 particles. The measured maximum and minimum tensile strength of the composite samples were 250 and 198 MPa respectively.

A. Nazaruddinet.al [15] showed that Aluminium matrix nanocomposites (AMNC) can have a wider application area because of their low density and high specific stiffness. Experimental analysis was conducted on aluminium matrix nanocomposite samples using TiO_2 nanoparticles as the reinforcement by using powered stir casting method. The mechanical properties such as tensile strength and hardness are determined and compared with that of Aluminium. Microstructural characterization and X-ray diffraction studies were conducted on these prepared nanocomposite samples. The study reveals that the presence of TiO_2 nanoparticles in the aluminium matrix led to a significant improvement in the mechanical properties of the prepared nanocomposite samples when compared with that of the commercially pure aluminum. Microstructural evaluation and X-ray diffraction studies showed a positive response to the addition of TiO_2 nanoparticles into the aluminium matrix.

X. H. Chen et al[16],synthesised aluminium metal matrix composites (AMMCs) with different weight percentages of $TiAl_3$ particles by in situ stir casting process. A heterogeneous nucleation phenomenon in the cast AMMCs was studied. Microstructural characterisation was investigated by optical microscopy, X-ray diffraction and scanning electron microscopy. Tensile test was carried out in order to identify the mechanical properties of composites after aging. The primary aluminium phase which nucleated heterogeneously on $TiAl_3$particles has been identified with a small lattice disregistry while their crystallographic orientation relationship was (001)$TiAl_3$ // (100) Al. The microstructure analysis revealed uniform distribution of reinforcements, grain refinement and clean $TiAl_3$/Al interface in specimens. The mechanical results showed that the addition of up to 1 wt%Ti led to an improvement in the tensile strength and ductility as compared against matrix. Fractography of the specimens showed that the fracture surfaces of as cast composite exhibited mixed rupture characteristics of quasi-cleavage and tough.

Manjunath M Narwateet al[17],conducted studies onGraphene and its derivatives as an innovative material for various engineering applications. These materials have excellent mechanical properties such as high strength to weight ratio, stiffness, and modulus of elasticity. Attempt were made to incorporate the graphene oxide in various compositions (0.5. 0.75 and 1.0 wt. %) to newly developed AA7075 - TiO_2 reinforced aluminum metal matrix composites by Stir casting technique. The newly prepared composite specimens were subjected to microstructure, hardness, tensile, and wear examinations. Microstructure examination reveals uniform distribution of reinforcements. Hardness, ultimate tensile strength and wear resistance of prepared composites increased with increase in reinforcement percentage in matrix.

Iman S. El-Mahallawi et al. [18] observed the properties of cast aluminum alloys by adding nanoparticles. In his work, the effect of adding alumina (Al_2O_3), titanium dioxide (TiO_2) and zirconia (ZrO_2) Nano-particles (40 nanometre) to the aluminum cast alloy A356 as a base metal matrix was investigated. Alumina, titanium dioxide and zirconia Nano-powders were stirred in the A356 matrix with different fraction ratios ranging from (0%–5%) by weight at variable stirring speeds ranging from (270, 800, 1500, 2150 rpm) in both the semisolid (600 °C) and liquid (700 °C) state using a constant stirring time of one minute. The cast microstructure exhibited change of grains from dendritic to spherical shape with increasing stirring speed. The fracture surface showed the presence of nanoparticles at the interdendritic spacing of the fracture surface and was confirmed with EDX analysis of these particles. The results of the study showed that the mechanical properties (strength, elongation and hardness) for the Nano-reinforced castings using Al_2O_3, TiO_2 and ZrO_2 were enhanced for the castings made in the semi-solid state (600 °C) with 2 weight% Al_2O_3 and 3 weight% TiO_2 or ZrO_2 at 1500 rpm stirring speed.

Ganesh Khandooriet. al [19] studied about aluminium reinforced with 5%, 10%, 15% TiO_2particulate fabricated by stir casting process and properties are investigated. Addition of Mg improves the wettability in aluminium melt and thus increases the amount of reinforcing phase in the composite. The wear resistance and frictional properties of Aluminium composite are studied by performing dry sliding wear test using a pin on disc wear tester. The wear rate decreases with increasing TiO_2 volume content of the reinforcement.

Padmavathi.K.R et.al [20], Developed composites of Al 6061- micro TiO_2 (5, 10 and 15 wt. %) and Al 6061-nano TiO_2 (0.5, 1.0 and 1.5 wt. %) were produced by stir casting technique. Hardness and wear tests were performed on the micro and nano composite specimens. The fabricated nano composites showed improvement in hardness and wear resistance over the micro composites. The microstructure of the worn out specimen was examined by scanning electron microscope. Considering all the factors, it can be concluded that aluminium based composite with 1.0% by weight nano TiO_2 reinforcement possess better wear resistance properties as compared to micro TiO_2 reinforced aluminium metal matrix composites.

S K Chaudhury& A K Singh et.al [21] studied about Aluminum alloy reinforced with 2% volume fraction of Magnesium (Mg) and 11 % volume fraction of Titanium di oxide(TiO_2) produced by spray forming and stir casting method were studied. The composite were subjected to microstructure analysis, micro hardness test, grain size studies and wear test. Microstructure shows fairly uniform distribution of TiO_2 particles in matrix alloy. The hardness measurement showed that hardness values of Al-2Mg alloy, spray formed and stir cast Al-2Mg-11TiO_2 composites are 20.66, 28.69, and 23.3 KgF/mm2. The hardness in spray formed composites has higher value than stir cast composites is due to the micro-structural refinement. Wear properties of the spray formed composites are significantly improved with incorporation of TiO_2 particles. The coefficient of friction of both as-sprays and stir cast composites are lower than the base alloy and decreases with increase in load.

G. Elangoa B.K.Raghunath et.al [22], studied about aluminium alloy LM25 reinforced with Sic and TiO_2 particulate fabricated by stir casting process. The wear resistance and frictional properties of hybrid metal matrix composite are studied by performing dry sliding wear test using a pin on disc wear tester. The wear rate decreases with increasing TiO_2 volume content of the reinforcement. The result also proves that lubricating nature of reinforcement material enhances the wear resistance and this property can be considered as a factor in design of new material for different application.

S.HanishAnandet al [24] studied about the mechanical behavior of aluminum composite reinforced with Titanium di-oxide and Sodium hexa-fluoro-aluminate with 5, 10, & 15 wt. % of Na3AlF6 and 10, 15 & 20 Wt. % of TiO_2 produced by stir casting route. Na_3AlF_6, which improves the ductility of TiO_2 with the Aluminium melt, is lost from the melt by oxidation, during melting and stirring of the alloy. Measurements of hardness and density is carried out test showed that the hardness is increased with an increase in volume fraction of TiO_2& Na_3AlF_6 particles.

2. EXPERIMENTAL STUDIES:

Al (LM0) alloy was chosen as matrix because it is having good castability and good resistance to corrosion.

Table 1: Chemical composition of LM0 alloy

Element	Si	Cu	Mg	Mn	Fe	Zn	Ni	Ti	Al
Wt. (%)	0.3	0.03	0.03	0.03	0.4	0.07	0.03	0.03	Balance

Table 2: Physical Property of LM0 alloy and Nano-TiO_2.

Material	Density(g/cm^3)	Melting Point °C.	Yield Strength (Mpa)	Thermal Conductivity (W/mK)
LM0 Alloy	**2.7**	**650**	**60**	**209.2**
Nano TiO_2	**3.97**	**1843**	**2000C**	**11.8**

In Nature, titanium dioxide exists in three primary phases – Anatase, Rutile, and Brookite – with different sizes of crystal cells in each case. The popularity of titanium dioxide in materials sciences began with the first photo catalytic splitting of water in 1972[25]. In the present work Anatase grade Nano-TiO_2 is used as reinforcement.

In stir casting method before the casting the reinforcements, stirrer, and permanent mould are preheated to 350^{o}C to remove moisture and gases from the surface of the reinforcements, and equipments before casting. Now the essential amount of LM0 is weighed and placed in the graphite crucible and heated to 730-750^{0}C using bottom pouring muffle furnace subsequently the magnesium is added to minimize the coating film defects by expelling the volatile components present in the melt during casting. Then the matrix LM0 is reinforced with Nano particle size of TiO_2 particulates with different weight percentages. The Nano particle of TiO_2 was added at the temperature of 730-750^{o}C and a constant rigorous stirring was done for 3- 4 minutes at a speed of 500-550 rpm until a clear vortex is formed with the help of stirrer having four pitched blades (45° pitch angle).At the pouring temperature of 730^{0}C the molten mixture was poured by releasing the locking nut in the bottom of the furnace to the cast iron mould and immediately after filling the mould is water quenched.

Figure 1: Bottom Pouring Muffle Furnace

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The bottom portions of the casting were taken for Microstructural characterization to check the distribution of second phase particulates. Analysis of a materials microstructure aids in determining whether the material has been processed correctly and to check whether the reinforcement are distributed evenly and is therefore a critical step for determining product reliability and for determining why a material failed.

The Brinell hardness testing method is used to determine Brinell hardnessnumber of the compositeas per ASTM E10 standards. The Brinell hardness analysis is a simple indentation test for determining the hardness of a wide variety of materials and it is principally preferred for particle reinforced composites as it could provide a better average hardness over a larger area containing several fine particles in the matrix. The testing was carried out at a load of 500 Kg by using a steel ball indentor of diameter 10 mm, the load was applied for about 180 seconds on a sample and then the diameter of indentation was measured with the help of travelling microscope. For each indentation, an average of two diameters measured perpendicular to each other was used to find the corresponding hardness. On each sample, at least thirty (30) indentations for hardness measurement were made at different locations and the average of these readings is reported as the hardness value of the material.

Among the various mechanical testing, tensile testing is most important and widely used one. Many parameters can be derived from the complete tensile test record. One can obtain important information concerning the materials elastic properties, the character and extant of plastic deformation, yield strength, tensile strength and toughness [18]. The response of materials to other type of loading can sometimes be explained or predicted on the basis of their behaviour under simple tension.

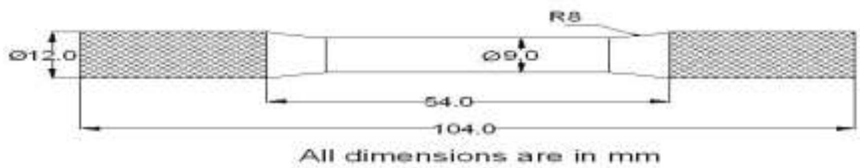

Figure 2: Dimensions of ASTM E8-M88 Standard for Tensile Strength.

Testing specimen was prepared according to the American Standard Testing Materials ((ASTM) E8-M88 standard) [18] as shown in figure 2.

3. RESULTS AND DISCUSSION.

3.1 Microstructure

Fig. 3 (a),(b) and (c) shows the SEM micrographs of LM0 reinforced with 25nm TiO_2 particles with 4, 8 and 12 wt.%, and Fig. 3 (d),(e) and (f) shows the SEM micrographs of LM0 reinforced with 60nm TiO_2 particles with 4, 8 and 12 wt.%.From the SEM Micrographs it can be seen that as the particle size decreases the agglomeration and clustering of the particles will be more in composites. The vortex generated in the stirring process breaks solid dendrites due to higher friction between particles and Al matrix alloy, which further induces a uniform distribution of particles. The agglomeration is more in higher weight percentage of TiO_2 (12 wt.%), but agglomeration is more in composites prepared with 25 nm particles of TiO_2 compared to composites of 60 nm

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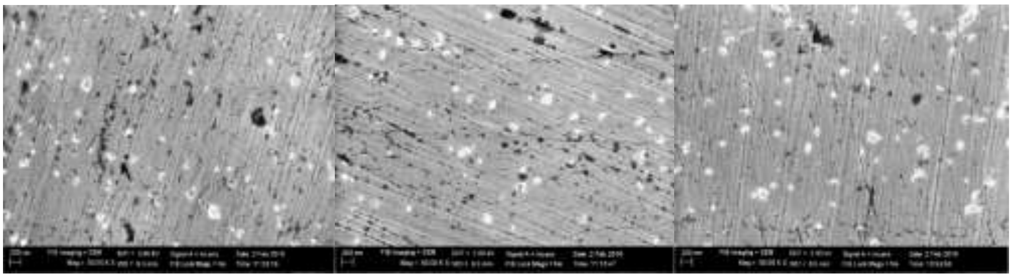
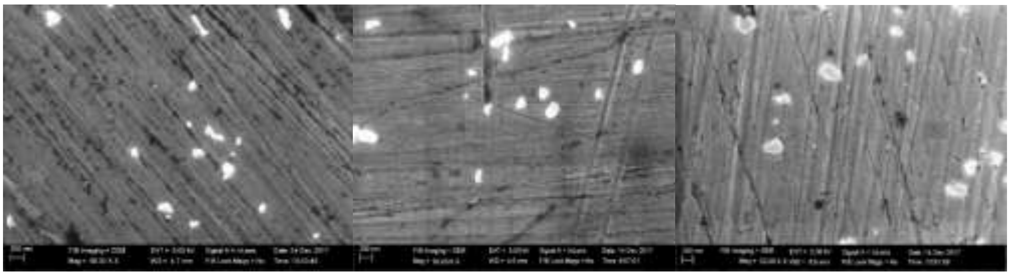
TiO_2 particles. The microstructure of these aluminium based composites contains intermetallic Al_3Ti and oxide Al_2O_3 particles and their amount increases with increasing addition of TiO_2 particles.



(a) **(b)** **(c)**



(d) (e) **(f)**

Fig. 3: Scanning Electron Micrographs of (a) LM0-4 % 25 nmTiO_2 and (b) LM0-8 % 25nmTiO_2 (c) LM0-12% 25nmTiO_2, (d) LM0-4 % 60 nmTiO_2 (e) LM0-8 % 60nm$TiO_2$and (f) LM0-12 % 60nmTiO_2 composites.

3.2 Hardness Measurements.

The hardness of composite depends on the hardness of the reinforcement and the matrix. So, an enormous amount of dislocations are generated at the particle-matrix interface during solidification process, which further increases the matrix hardness. The higher the amount of particle-matrix interface, the more is the hardening due to dislocations [18].

The hardness of as cast composites synthesis with 60 and 25 nm particle size of $TiO_2$with various weight percentage of 0, 4, 8 and 12wt.%are evaluated using ball indenter of diameter 10mm at an applied load of 500kg for each sample at different locations. Variation of Hardness of Aluminium alloy 60nm TiO_2 composites and 25nm TiO_2 composites are shown in figure 5 (b) respectively.The hardness is increases with increasing particle content in the composite and lower the parcel size higher the hardness that is 25nm TiO_2 particle composites shows higher hardness compare to 60nm TiO_2 particle composites. The increase in hardness is mainly due to the coefficient of thermal expansion (CTE) of ceramic particles is less than that of aluminium alloy and because of sudden water quenching.

From the testit is observed that with increase in weight % of TiO_2 particle and decrease in particles size, the hardness of composites increases. The BHN of the composites is approximately 39.3% for 60nm TiO_2 particle and 45.5% for 25nm TiO_2 particle which ishigher than that of the un-reinforced alloy. Also, the hardness of

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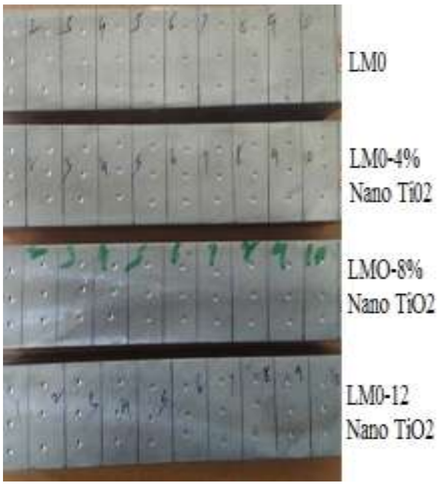
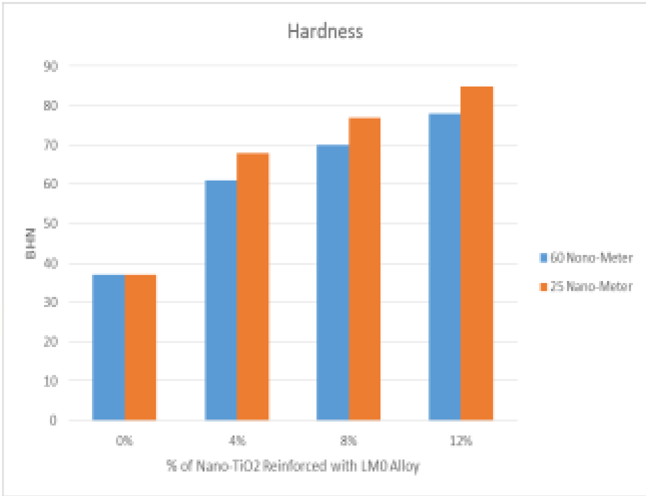
Nano-composites was greater than that of micron composites because of the more influence of Nano particles on the strengthening mechanism (Orowan mechanism).

From the graph it is observed that for 4% the BHN of 25 nm TiO_2p is 11.47% more when compared with 60 nm TiO_2p composites. Similarly for 8% and 12% the BHN of 25 nm TiO_2p is 10.1% and 8.9% more than 60 nm TiO_2p composites respectively. This is because of clustering and agglomeration is more as the percentage of reinforcement increases.





(a) (b)

Fig.4. (a) Indentations on the Composites of LM0-60 nm $TiO_2$with different wt. % of reinforcements,and (b) Comparison of hardness of LM0-60nm and 25nm $TiO_2$with different wt. %of reinforcement.

3.3 Tensile Properties.

Figure 5 shows the effect of particle sizes of Nano TiO_2 particulates on the tensile behaviour of LM0 alloy. From the graphs, it is noted that the composites prepared with 25nm TiO_2particles showshigher ultimate and yield strength as compared to 60nm TiO_2 reinforced composites. The increase in tensile strength is higher in case of 12% Nano TiO_2 composites compare to un-reinforced alloy. It may be due to the presence of moreAl_3Tiand Al_2O_3particles present in the composites.The UTS of the composites is approximately 21.7% for 60nm TiO_2 particle and 31.8% for 25nm TiO_2 particle which is higher than that of the un-reinforced alloy.

For the same(4wt. %) weight % of Nano TiO_2 particles the ultimate tensile strength of 25 nm and 60nm composite are 107.1MPa and 129.2MPa respectively i.e. 25 nm TiO_2p approximately 19.6% more when compared to 60 nm TiO_2p Composites. Similarly for 8 and 12% ultimate tensile strength of 25 nm and 60nm composite are 118.7Mpa, 147.6MPaand 127.8 Mpa, 164.8Mpa respectively i.e. 25 nm TiO_2P approximately 18.2% and 21.8% more when compared to 60 nm TiO_2P Composites.

Tensile strength of 25nmTiO_2p composites is higher because of two reasons- First; each larger-sized particle has larger interface area with the matrix, and thus endures higher stress concentration. Second, the particle fracture strength is controlled by the intrinsic flaws within the particle. Since the size and number of flaws is limited by

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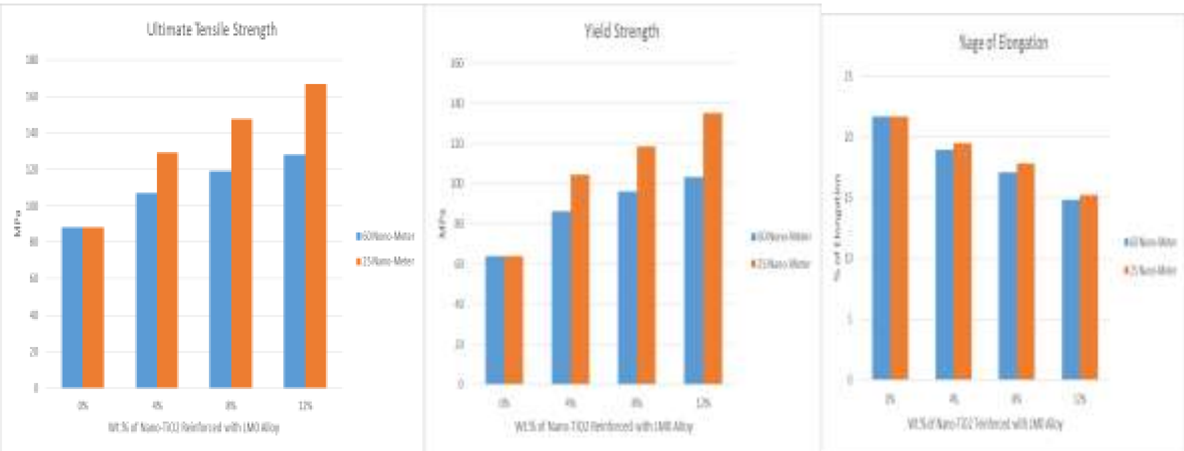
the size of the particle, larger particles are more likely to fracture because they have a greater statistical probability of containing a flaw that is greater than the critical size [26].

The Yield Strength of the composites is approximately 25.9% for 60nm TiO_2 particle and 36.8% for 25nm TiO_2 particle which is higher than that of the un-reinforced alloy.For the same(4wt. %) weight % of Nano TiO_2 particles the yield strength of 25 nm and 60nm composite are 86.4MPa and 104.6MPa respectively i.e. 25 nm TiO_2P approximately 17.4% more when compared to 60 nm TiO_2P Composites. Similarly for 8 and 12% yield strength of 25 nm and 60nm composite are 96Mpa, 118.4MPa and 103.8 Mpa, 134.8Mpa respectively i.e. 25 nm TiO_2P approximately 18.9% and 21.3% more when compared to 60 nm TiO_2P Composites.

Since the fractured particles cannot withstand any load, but act as privileged failure sites, the composites with larger TiO_2 Nano particle size show lower mechanical properties as compared to that with smaller Nano particle size.This increase in UTS and YS is mainly due to the presence of $Al_3Ti$and Al_2O_3particles present in composites. These particulates act as the barrier for dislocations.Theductility decreases with increasing in wt. % percentage of Nano TiO_2particles in the composites butinterestingly, the ductility of all the 25nm composites with differentweight % shows higher than that of respective weight % of 60nm composites.



(a) (b) (c)

Fig 5: Comparison of LM0-60 and 25Nano-TiO_2 composites (a) Ultimate Tensile Strength, (b) Yield Strength, c) %age of Elongation.

4. CONCLUSION.

Nano Composites are successfully fabricated by Bottom Pouring Stir Casting Method and Effect of various Nano Particle Sizes of TiO_2 was evaluated. The following conclusion ware made after testing and characterization.

1. The micrographs shows the uniform distribution of $Al_3Ti$and Al_2O_3reinforcements with low amalgamation but at higherweight % of TiO_2addition in composites, clustering is more in Nano size composites of 25nm particles when compared with 60nm Particles.

2. The Brinell hardness and UTS of the Nano-composites increases with increase in Nano TiO_2particles content, and these are increases with decrease in particles size of Nano TiO_2.
3. The percentage of elongation decreases with increase in Nano TiO_2particlescontent, but interestingly 25nm size particles composites shows higher ductility than 60nm size particles composites.
4. From the results it can be concluded that as the particle size decreases the mechanical properties will enhance i.e. mechanical properties of composites depends on Particle size and weight % of the particulates.

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