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MICROSTRUCTURE AND WEAR PROPERTIES OF ZIRCONIUM NANO METAL MATRIX COMPOSITES

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ABSTRACT
A356.1 Aluminium alloy reinforced with Nano-sized ZrO_2 particle are widely used for high performance applications such as automotive, military, aerospace, and electric industries because of their improved physical and mechanical properties. In this research, Zirconium Oxide (ZrO_2) Nano particle were synthesized by Solution Combustion Synthesis process. Prepared Nano particles were characterized by Powder X-ray diffraction (PXRD). Nano sized Zirconium Particle were reinforced with A356.1 Aluminium alloy with varying Wt. % such as 2.0, 2.5, 3.0 and 3.5 via stir casting Technique at a constant stirring speed of 150 rpm. The composites were then characterized by scanning electron microscopy (SEM). Wear tests were carried out at Varying Wt. % ratios with varying Conditions of Speed, Load and Time. The results reveal that the Nano Metal Matrix Composite (NMMC)'s containing 3.5 Wt.% reinforcement particle has improved wear properties.

KEYWORDS: Nano Metal Matrix Composite, PXRD, SEM.

INTRODUCTION
The attention of material scientists and engineers has shifted from monolithic materials to composite materials for the development of light weight, environment friendly and high performance appliances.As aerospace technology continues to advance, there is a rapidly increasing demand for advanced materials with high mechanical and thermal capabilities for such ultrahigh applications [1]. Its application also stretched to automobile, electronic and computer industries to replace the existing materials including plastics [2].The early 1990s are considered to be the renaissance for Aluminium as structural material due to environmental concerns, increasing safety and comfort levels .A significant improvement in the properties of Aluminium alloys, reduced fuel consumption because of light weight has made huge demand from automobile industry [3,4]. This growing requirements of materials with high specific mechanical properties with weight savings has fueled significant research activities in recent times targeted primarily for further development of Aluminium based composites[5–7]. A recent industrial review revealed that there are hundreds of components from structural to engine in which Aluminum alloy is being developed for variety of applications [8]. It is also predicted that for Aluminium alloys demand increased globally attain average rate of 20% every year [9]. It is noticed that the limited mechanical properties (strength and hardness) of Aluminium and its alloys adversely affect its applications in automobile and aerospace industries [10,11]. This remains one of the major concerns in its fabrication to suit its application in recent days. Search of open literature indicates that for number of

Aluminium based MMCs(Metal Matrix Composites) including chilled MMCs [12–15] are being developed but no work has been done in this field. Hence the present research is undertaken to fill the void and to investigate the integrated properties of A356.1alloy/ZrO2 NMMCs(Nano Metal Matrix Composite). Among all the reinforcements used in Aluminium based composites only Nano-size particulates has shown their potential superiority in improving mechanical properties, such as wear and microstructure with noticeable weight savings [16].

Liquid metallurgy technique is one of the most economical of all the available routes for Nano metal–matrix composite production and generally can be classified into four categories: pressure infiltration, stir casting, spray deposition and in situ processing [17]. Compared to other routes, melt stirring process has some important advantages ,e.g., the wide selection of materials, better matrix– reinforcement bonding, easier control of matrix structure, simple and inexpensive processing, flexibility and applicability to large quantity production and excellent productivity for near-net shaped components[18]. However, there are some problems associated with stir casting of AMCs such as: poor wettability and heterogeneous distribution of the reinforcement material. Poor wettability of reinforcement in the melt means that the molten matrix cannot wet the surface of reinforcement particles. Therefore, when the reinforcement particles are added into the molten matrix, they float on the melt surface. This is due to the surface tension, very large specific surface area and high interfacial energy of reinforcement particles, presence of oxide films on the melt surface and presence of a gas layer on the ceramic particle surface. Mechanical stirring can usually be applied in order to mix the particles into the melt, but when stirring stops, the particles tend to return to the surface. There are some methods to improve the wettability of the reinforcement particles within the molten matrix alloy, for example heat treatment of the particles before dispersion into the melt caused removal of the adsorbed gases from the particle surface [19]. Another problem is distributing of reinforcement particles uniformly in molten matrix. When the particles were wetted in the metal melt, the particles will tend to sink or float to the molten melt due to the density differences between the reinforcement particles and the matrix alloy melt, so that the dispersion of the Zirconium Nano particles is not uniform and the particles have high tendency for agglomeration and clustering. Mechanical Stirring is preferred. In addition to mechanical stirring, there are some other techniques for introducing particles into the matrix. One of them is injection of the particles with an inert carrier gas into the melt. It has been reported that the technique is helpful in improving the distribution of the reinforcement particles within the melt [20]. Wettability and distribution of reinforcement particles becomes more difficult when the particle size decreases to the Nano scales. This is due to the increasing surface area and surface energy of Nano particles which cause an increasing tendency for agglomeration of reinforcement particles. Moreover, several structural defects such as porosity, particle clusters, oxide inclusions and interfacial reactions arise from the unsatisfactory casting technology [21]. In this study zirconium oxide Nano particles were prepared using combustion synthesis method at a temperature of 850 ± 5º C using stir casting technique at a speed of 100 rpm, the Nano metal matrix composites thus obtained were casted using Die cast technique and characterized for their micro structure, wear properties and the Nano particles is characterized by PXRD and SEM. Zirconium Nano material is reinforced with A356.1 Aluminium alloy in the ratio of 2.0, 2.5, 3.0 and 3.5Wt%.

EXPERIMENTAL PROCEDURE

Synthesis of ZrO_2 powder:

The ZrO_2 Nano powder was prepared by dissolving zirconyle nitrate ($ZrO\ (NO_3)_2$) and Crystal sugar ($C_6H_{12}O_6$) as fuel in a minimum quantity of double distilled water is taken in a ceramic crucible. The crucible containing the solution was placed in a preheated muffle furnace maintained at 850 ± 5º C. The solution initially boils and undergoes dehydration followed by decomposition with the evolution of large amount of gases resulting in a transparent gel. The gel then formed in to white foam, which expanded to fill the vessel. Shortly thereafter, the reaction was initiated somewhere in the interior and a flame appeared on the surface of the foam and proceeded rapidly throughout the entire volume, leaving a white powder with an extremely porous structure. The entire combustion process for producing ZrO_2 powder takes place. The reaction for combustion synthesis in the present case can be written in equation (1)

$$ZrO(NO_3)_2 + C_6H_{12}O_6 + H_2O \rightarrow ZrO_2 + N_2 + 6CO + 7H_2O \quad \ldots (1)$$

Table 1. Chemical composition of Aluminium alloy A356.1

Elements	Al	Si	Fe	Cu	Mg	Mn	Zn	Ni
Wt.%	91.7	7.2	0.32	0.18	0.38	0.02	0.05	0.05

Fabrication of Zirconium Nano Metal matrixZrO_2 Nano composites

Aluminium alloy (A356.1) and Nano sized ZrO_2 were used to fabrication of composites. The composition of A356.1 is shown in Table1 [1]

The samples were prepared using a resistance furnace, equipped with a stirring system. After smelting of aluminum ingots, ZrO_2 Nano powder was added to the molten metal and stirring was carried out at constant rate of 150 rpm for 20 min. According to the results of literature and previous works [31, 32]. Nano-powder ZrO_2 (2.0, 2.5, 3.0, and 3.5Wt %) added to the molten metal during stirring. The casting was performed at 850^0C. Steel circular die was used for casting of specimens. Finally, specimens fabricated in five various conditions were prepared for subsequent Microstructural and wear analyses.

RESULTS AND DISCUSSION

X-ray Diffractometer (XRD) Studies

The powder X-ray diffraction studies were carried out using Phillips X-ray diffractometer (model PW 3710) with Cu Kλ radiation (λ = 1.5405 A˚) The X-ray diffraction pattern of Nano-ZrO_2 powder confirms the crystalline phase and mean crystal size determined was around 40 nm. In the XRD observations three Strongest peaks shown in Fig. 2 were detected with Miller indices (223), (054), (126), and (082) corresponding to Bragg angles 30°, 36°, 51°and 59°respectively. The characteristic peaks are higher in intensity which indicates that the products are of good crystalline nature. No peaks corresponding to impurities are detected, showing that the final product is purely ZrO_2 Nano powder. It is observed that intensity of the peaks increases with thermal treatment due to Agglomeration, which means that the crystalline has been improved. The full width at half maxima of major peaks decrease and confirms the grain size growth.

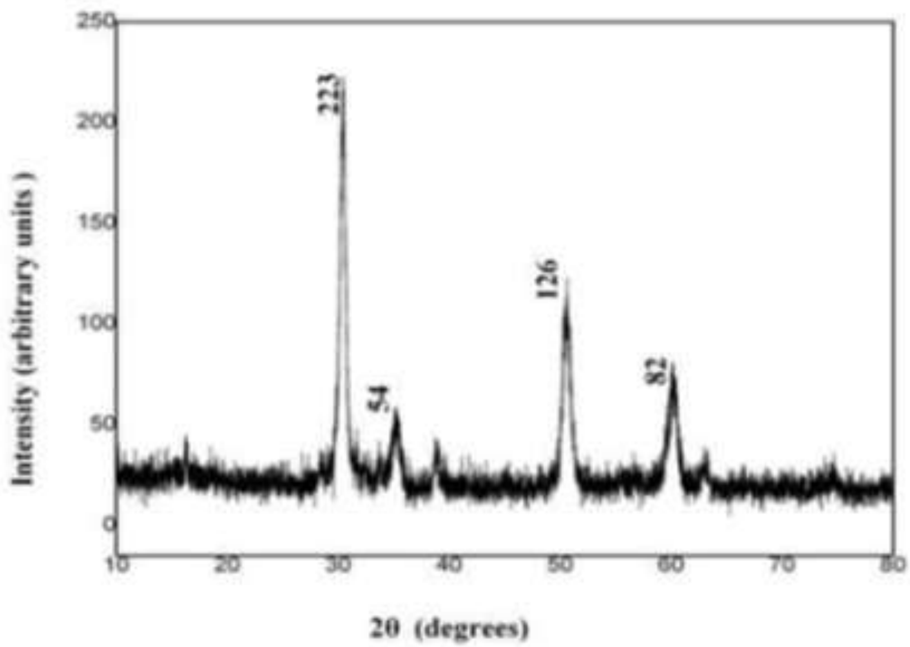

Fig.1:PXRD pattern of Nano particles fabricated at 940^{0}C

SEM analysis:

The size and morphology of the ZrO_2 Nanoparticles have been determined using scanning electron microscopy. Fig.2 shows the image random distribution of the ZrO_2 Nanoparticles having non- spherical shape and diameter in the range of Nanometer

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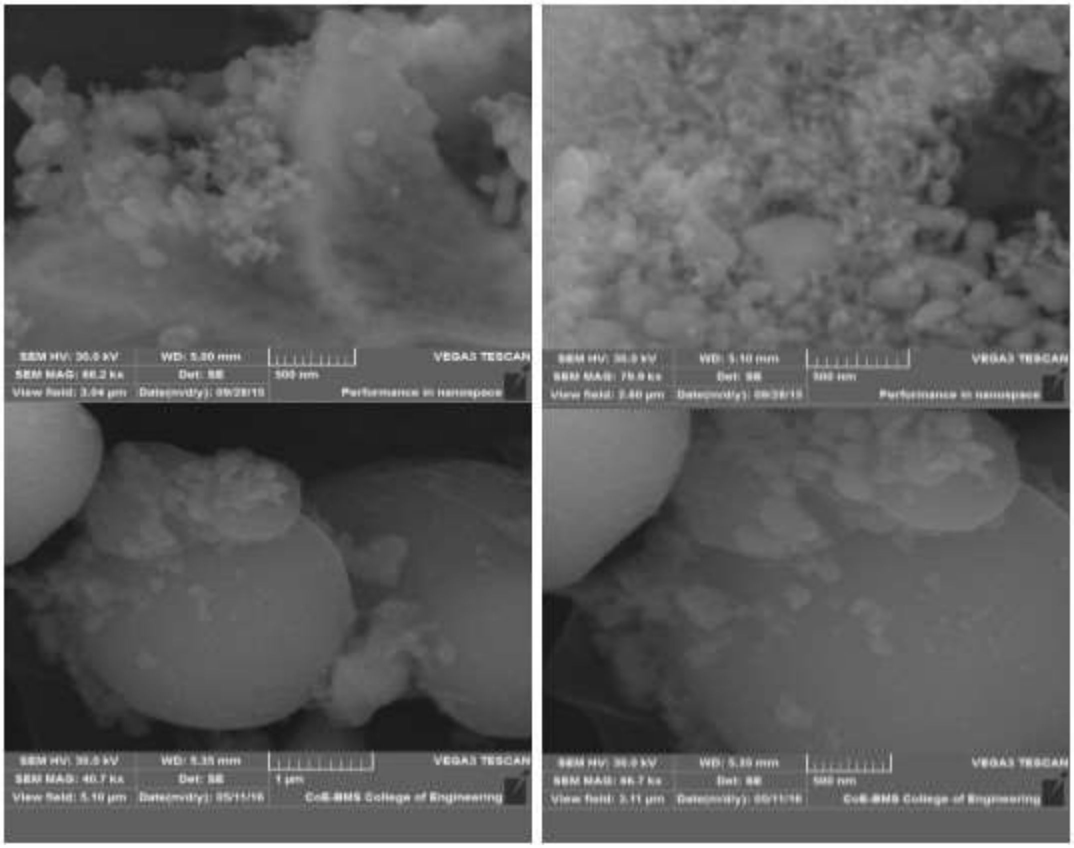

Fig.2:SEM Micrographs of ZrO_2 Nanoparticle

The Nano composites were found to be agglomerated when analyzed by scanning electron microscopy (SEM: JEOL, Japan, JSM 840A) studies shown in Fig.3.It can be observed that the ZrO_2 crystallites have no uniform shape. This believed to be related to the non-uniform distribution of temperature and mass flow in the combustion flame, due to the high surface energy of the particles and from the SEM there is such difference was observed for different Wt % of ZrO_2 dispersed aluminum powder. In micrographs also observing that the big particles are aluminum powder and the very small particles surrounded by that are ZrO_2.

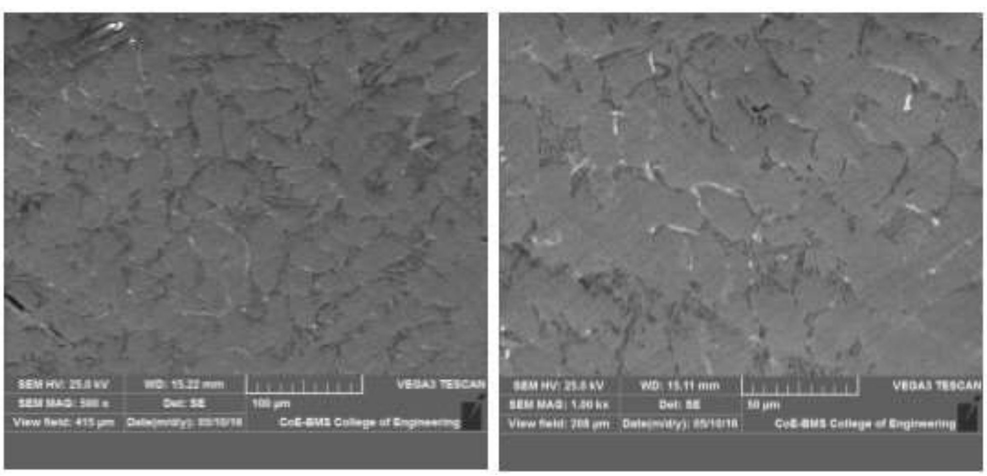

Fig. 3: SEM micrographs of As cast A356.1 Aluminium alloy

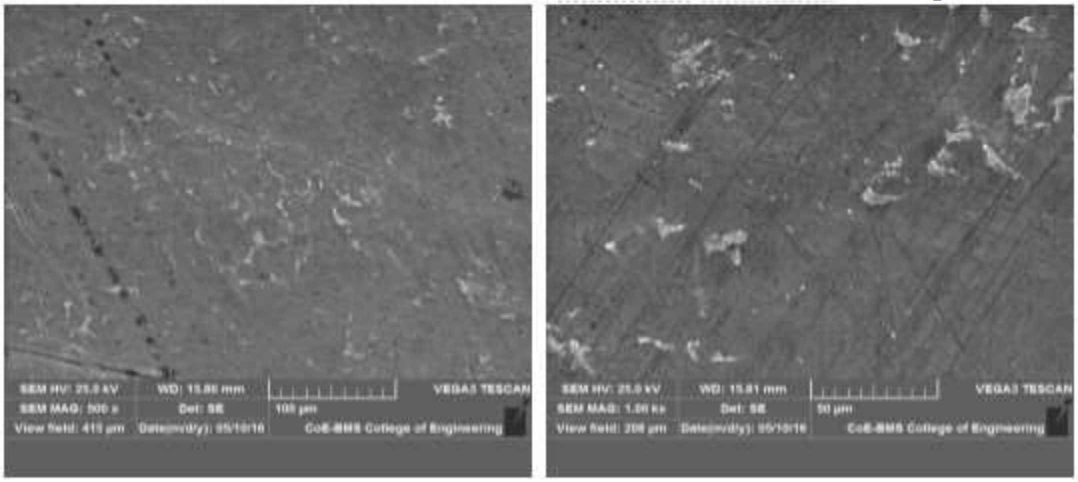

Fig. 4: SEM micrographs of 2.0Wt % Zirconium Nano Particle reinforcement

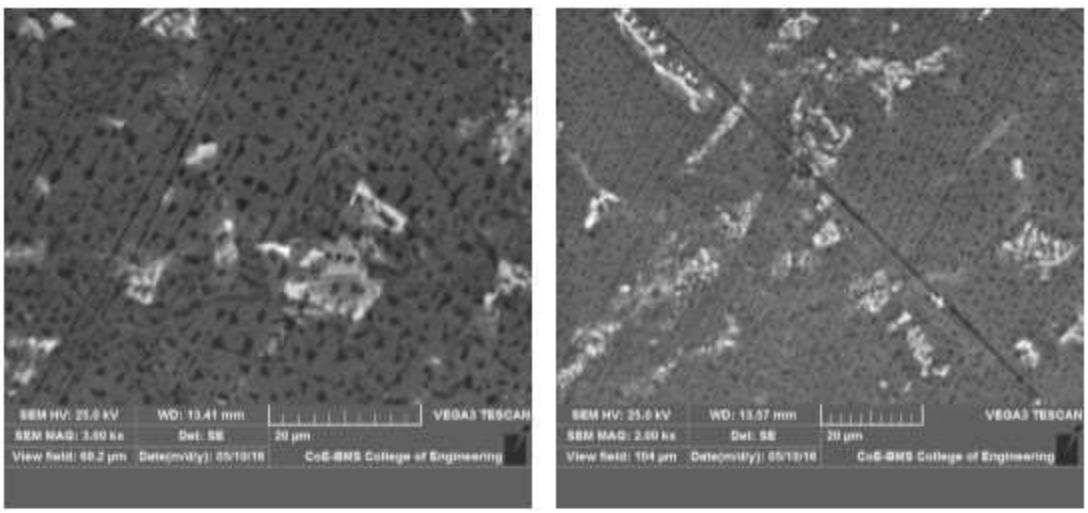

Fig. 5: SEM micrographs of 2.5Wt % Zirconium Nano Particle reinforcement

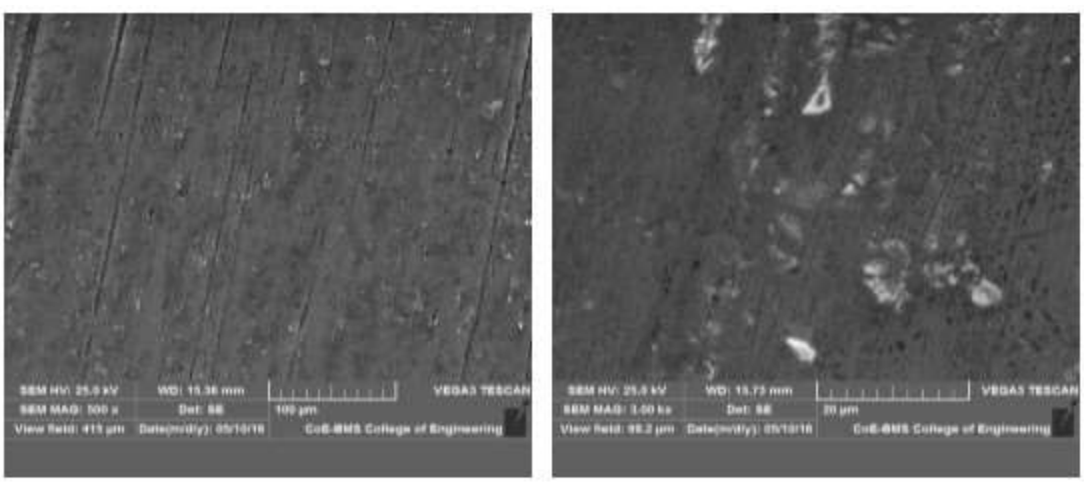

Fig.6: SEM micrographs of 3.0Wt % Zirconium Nano Particle reinforcement

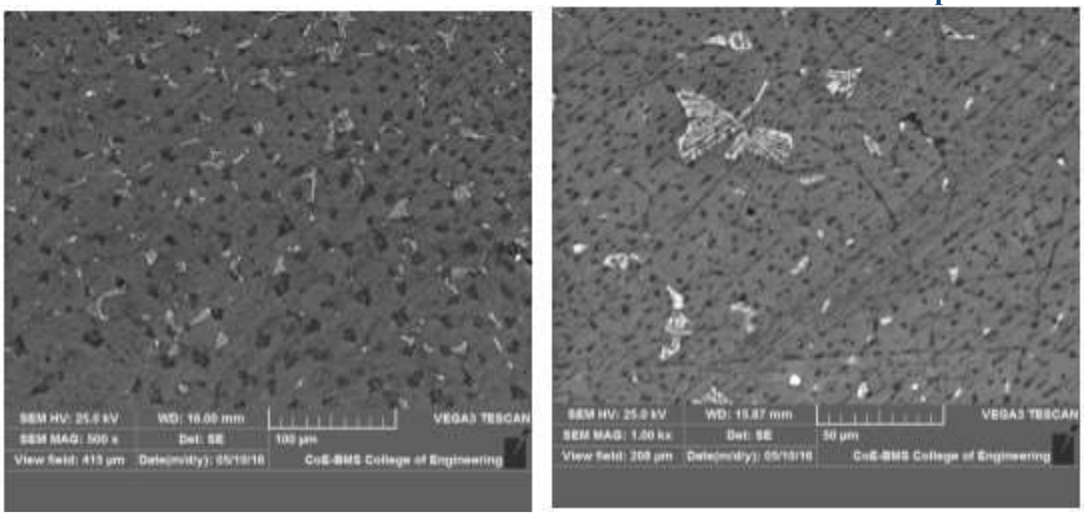

Fig.7: SEM micrographs of 3.5Wt % Zirconium Nano Particle reinforcement

Wear properties

Wear test were performed, ZrO_2 Nano made up of A356.1 alloy incorporating 2.0, 2.5, 3.0 and 3.5 Wt% respectively. Test were performed using Pin on Disc machine using samples in the form of 28 X 8 mm cylindrical block.One side of sample were put in contact with rotating disc at different loading conditions. The Fig.8 depicts of as cast and different varying Wt. % of ZrO_2 Nano metal matrix composites(NMMC) .The wear rate of 3.5Wt% of ZrO_2 Nano particles shows better wear rate compared to as cast and other Wt. % NMMC's . The graph shows when load at 10N the wear rate is $47.74X10^{-6}$ g/m for as cast A356.1 alloy, for the same load 3.5Wt% of ZrO_2 Nano particle reinforced alloy shows a decrease of wear rate consistently by adding different percentages of Nano particles. The Fig.8 reveals that when a varying Wt% of ZrO_2 Nano particle is added, it increases wear rate with increase of loads.

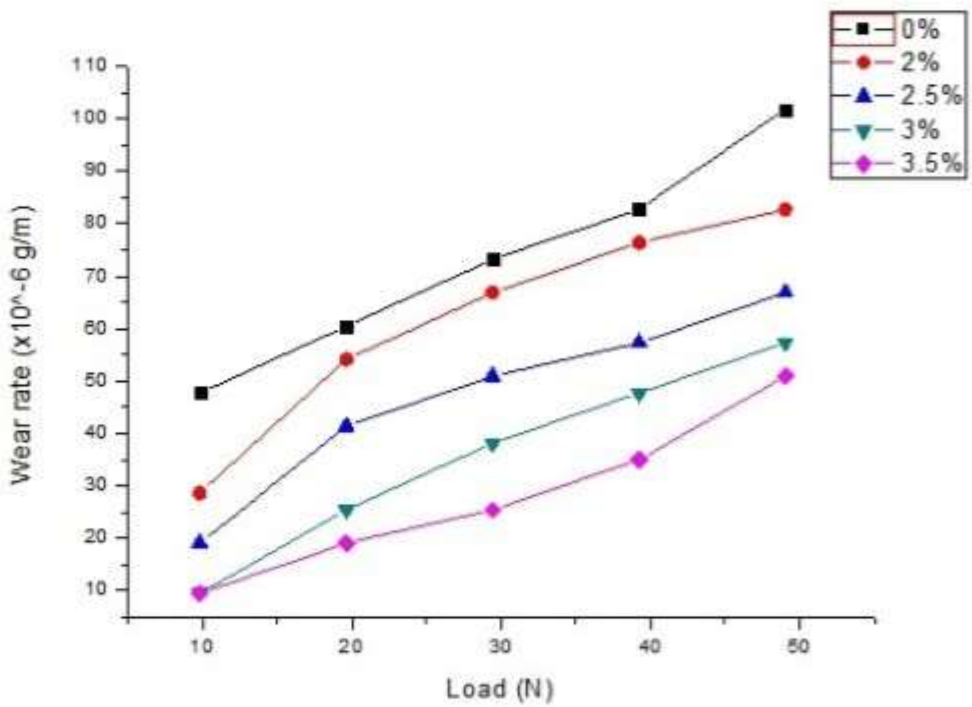

Fig.8: Wear Rate Vs Load

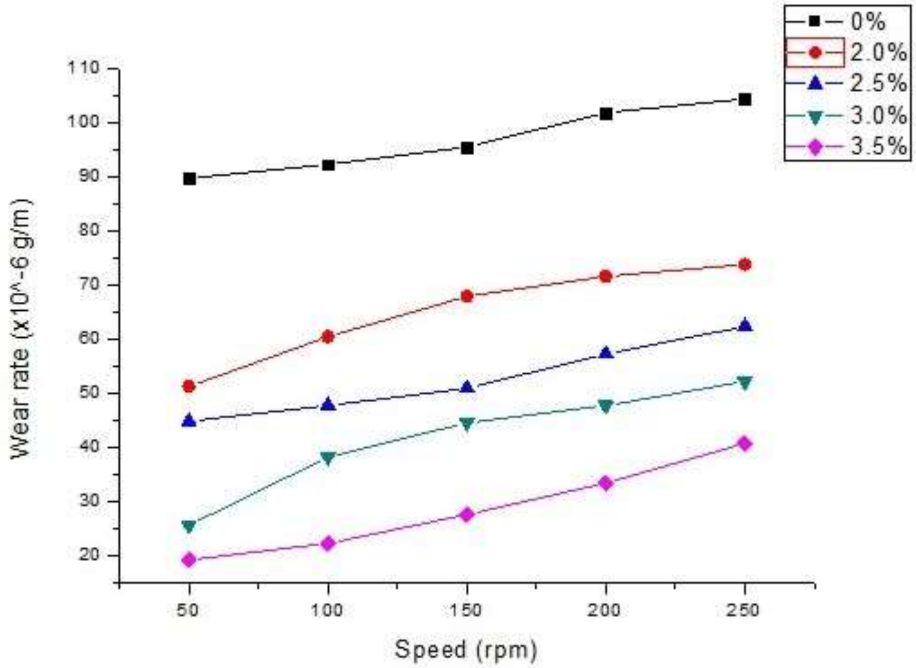

Fig.9: Wear Rate Vs Speed

To investigate the speed effect of reinforcing particulates with ZrO_2 Nano is added to the A356.1 alloy at varying percentages, at different speed rates. It is generally believed contribution of Nano particles to A356.1 alloy results in improvement of base alloy to great extent [33, 34]. Based on the results from Fig.9, when speed increases,wear rate increases as cast to Wt. 3.5% added. For as cast alloy wear rate is $89.74X10^{-6}$g/m at 50rpm and for Wt.3.5% wear rate is $19.23X10^{-6}$g/m..And for as cast alloy wear rate is $104.4X10^{-6}$g/m at 250rpm and for Wt.3.5% wear rate is $40.74X10^{-6}$g/m at 250 rpm.

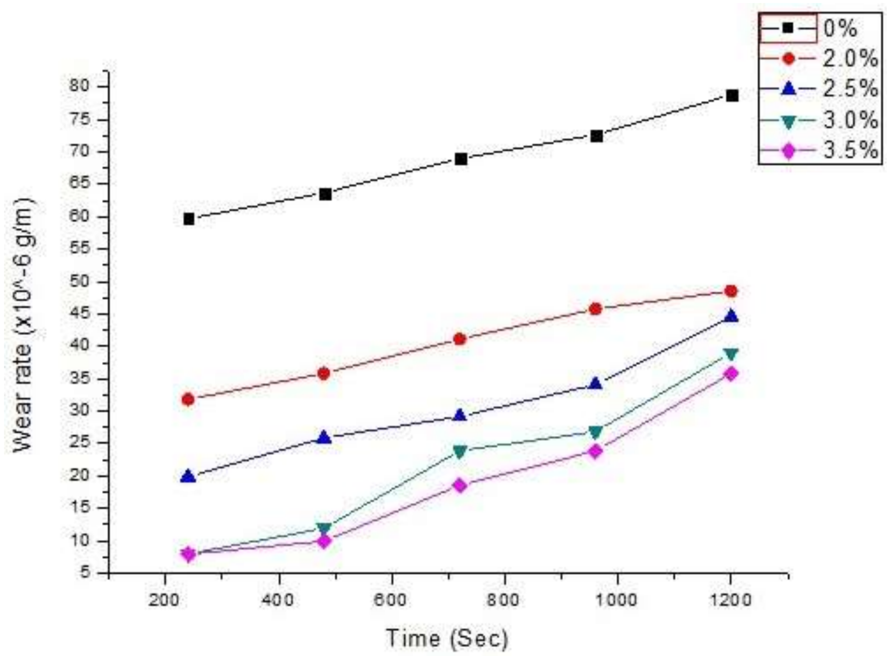

Fig.10: Wear rate Vs Time

Operating Time is proportional to Wear rate, as shown in the Fig.10.Based on the this graph, for As Cast alloy at the Operating time of 240sec,Wear rate is $59.69x10^{-6}$g/m and for the same time with 3.5 Wt. % reinforcements of Nano particle to base alloy the wear rate decreases to $7.96x10^{-6}$g/m. For As Cast alloy at Operating time of 1200 Sec, Wear rate is 78.8 $x10^{-6}$ g/m and for the same operating time with 3.5 Wt. % reinforcements of Nano particle to base alloy, the wear rate considerably decreases to $35.81x10^{-6}$g/m.

CONCLUSION
A356.1 Aluminium alloy reinforced with Nano sized ZrO_2 was successfully fabricated via stir casting method. Reinforcement particles were well distributed in the matrix of composites. However, particle agglomeration was observed in composites with high content of ZrO_2. Therefore stir casting was found as suitable method for fabrication of this kind of composites.SEM micrographs revealed that the presence of Nano-ZrO_2 particulates in A356.1 alloy. Graphs shows improved wear properties of NMMC with a reinforcement content of 3.5 Wt% of Zirconium Nano particle compared to the base metal.

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