

## **Tensile and Wear Characterization of Aluminum Alloy Reinforced with Nano –ZrO<sub>2</sub> Metal Matrix Composites (NMMCs)**

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### **Abstract**

In the present research nano –ZrO<sub>2</sub> particles were dispersed in aluminum alloy (LM 13) by melt deposition technique followed by hot extrusion. The size of particulates dispersed varies from 80 to 100nm and the amount of addition varies from 3 to 12wt% in steps of 3%. Microstructural analysis of the developed nano-composite reveals the uniform distribution of the reinforcement in the matrix alloy with significant grain refinement with minimum porosity. The mechanical properties indicates the presence of nano – ZrO<sub>2</sub> particulates has improved the strength and hardness significantly with slight reduction in ductility as compared against the matrix alloy. This paper also explores wear characteristics of the developed NMMCs in pin-on-disc dry sliding tests against hardened tool steel under varying loads. The wear resistance of the composites improved with increasing amounts of reinforcement, which are particularly effective under higher sliding speeds. However, specific wear rate decreased with sliding distance for all composites, with no significant effect of ZrO<sub>2</sub> content, in contrast to the substantial increases observed for the monolith. The wear mechanism was studied through worn surfaces and microscopic examination of the developed wear tracks.

**Keywords:** Nanocomposites, ZrO<sub>2</sub>, mechanical properties, wear, microstructure.

## 1. Introduction

The current high prices of oil have pushed the economics of transportation to the forefront of the general public's consciousness. There is renewed urgency to address the issue of weight reduction in aerospace and automotive sectors. A significant improvement in the properties of Al alloys, reduced fuel consumption because of light weight has made huge demands from automobile industry. This growing requirements of materials with high specific mechanical properties with weight savings has accelerated significant research activities in recent times targeted primarily for further development of Al based composites. It is also predicted that for Al alloys demand increased globally at an average rate of 20% every year [1-6]. Aluminum matrix composites reinforced with hard ceramic particles have emerged as a potential material especially for wear resistant and weight critical applications such as brake drums cylinder liners, pistons, cylinder block, connecting rods, etc.[7,8]. These components are subjected to sliding type of wear with their counter bodies. Extensive research reported in the direction of sliding wear behavior of aluminum alloy based MMCs [9-18]. MMCs provide high wear and seizure resistance as compared to the alloy irrespective of applied pressure and sliding speed. This is primarily due to the fact that the hard dispersoid makes the matrix alloy plastically constrained and improves the high temperature strength of the virgin alloy [19,20]. Also the hard dispersoids, present on the surface of the composite as protrusions, protect the matrix from severe contact with the counter surfaces and thus resulting in less wear, lower coefficient friction and temperature rise in composite as compared to that in the alloy[21,22]. Other exploration reveals higher values of coefficient of friction of aluminum based MMCs as compared to the alloy [23-26]. This may be due to: (i) greater depth of penetration of hard dispersoids onto the counter surface and (ii) fracture/fragmentation of the dispersoids, which in due course gets entrapped into the contact surface. Limitation of composites with hard rigid ceramic reinforcements is the tendency of these phases to act as rigid abrasive particle, which are causing more abrasive action to the counter surface [27]. These reinforcing particles act as wear debris roll over the contacting surfaces which causes more wear on both the contacting surface. The degree of this situation depends on sliding speed, applied load and frictional heating. In fact during sliding wear of metals and alloys a mechanically mixed layer its formed over the specimen surface, which strongly dictates the wear behavior of the materials[28-31]. It is believed that these layers are formed due to formation of wear debris, transfer of materials from the counter surfaces, mixing and compaction of these materials on the contact surfaces under applied load and higher frictional heating. However limited attempt as been made to examine the wear behavior of hot extruded aluminum alloy based NMMCs reinforced with nano  $\text{ZrO}_2$  particulates.

## 2. Experimental

In this research, nano- $\text{ZrO}_2$  particulates were dispersed in Al alloy. (The chemical composition of the matrix material is shown in Table 1) by vortex method followed by hot extrusion. The size of the nano- $\text{ZrO}_2$  particulates dispersed varies from 80 to 100 nm and the amount of addition varies from 3 to 12 Wt.% in steps of 3%. The

primary process consists of synthesis of monolithic and nano-sized  $ZrO_2$  reinforced Aluminum composites containing four different weight percentage of  $ZrO_2$  was carried out using DMD technique. The process involved heating of Aluminum alloy in a graphite crucible up to  $750^\circ\text{C}$  using resistance furnace to which the preheated reinforcement (upto  $400^\circ\text{C}$ ) was added and stirred well by a mechanical impeller rotated at 400rpm to create vortex in order to get uniform distribution of the reinforcement. In secondary process, the developed NMMCs containing nano sized  $ZrO_2$  in Aluminum matrix were hot extruded in hydraulic press at  $260^\circ\text{C}$ .

Properties of reinforcement ( $ZrO_2$ ) are as follows. Density :  $8.1 \text{ gm / cm}^3$ , melting point:  $1860^\circ\text{C}$ , UTS: 425 MPa , VHN : 150 , young's modulus: 98GPa ( $ZrO_2$  is supplied by nano Structured and Amorphous Materials, Inc., USA)

Micro structural characterization studies were conducted on polished NMMC specimens using OLYMPUS metallographic microscope to investigate morphological characteristics of grains, reinforcement distribution and interfacial integrity between the matrix and enforcement. Vickers micro hardness measurements were made on the polished specimens in accordance with ASTM E18-94 standard. Tension test were performed on AFS standard tensometer specimens in accordance with ASTM E8M-01 standard using Instron 8516 machine. Dry sliding wear tests were conducted on computerized pin-on-disc wear testing apparatus (Wear and Friction Monitor, Ducom Make, Bangalore, India) under varying applied load at a fixed sliding speed- against EN32 steel disc of hardness 500HV. The pin samples were 30mm in length and 6mm in diameter. The surface of the pin sample and the steel disc were ground using emery paper (grit size 240) prior to each test. In order to ensure effective contact of fresh surface with the steel disc, the fresh samples were subjected to sliding on steel disc. During sliding the load is applied on the specimen through cantilever mechanism and the specimens brought in intimate contact with the rotating disc at a tract radius of 90mm for 10 minutes. The wear rate was calculated from the height loss method and expressed in terms of volume loss per unit sliding distance.

**Table 1.** Chemical Composition of Matrix alloy

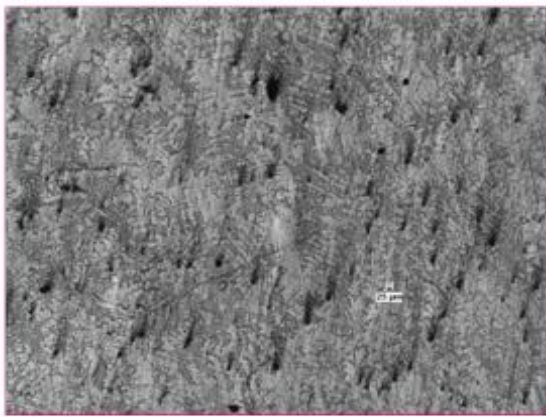
Elements	Zn	Mg	Si	Ni	Fe	Mn	Al
Wt.%	0.5	1.4	12	1.5	1.0	0.5	Bal

### 3. Results and Discussion

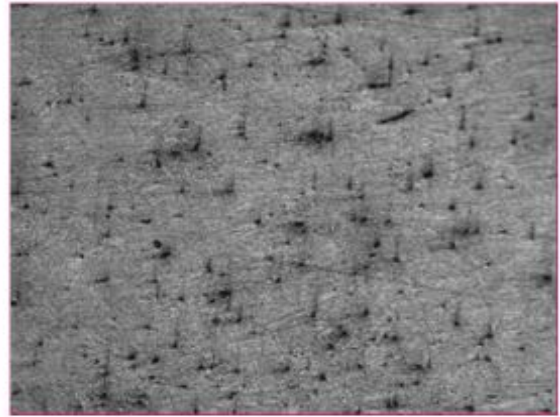
Synthesis of Al-alloy based NMMCs were successfully accomplished by DMD process followed by hot extrusion. The conditions established (as mentioned in sec 2) during melt, dispersion of reinforcement, pouring and solidification was instrumental in the prevention agglomeration of reinforcement due to the effect of its gravity. The results, in essence has indicated judicious selection of stirring parameters, solidification conditions during pouring into the green sand molds to achieve sound castings. Thus realizing the feasibility of DMD process as a potential fabrication technique for nanocomposites.

### 3.1 Micro Structural Characterizations

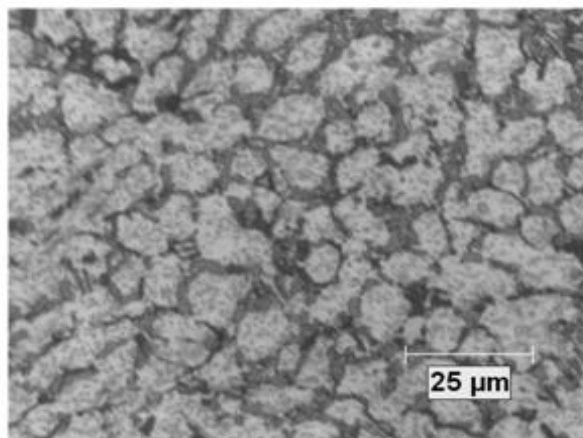
Microstructural characterization of hot extruded NMMCs is discussed in terms of distribution of reinforcement and reinforcement matrix interfacial bonding. Microstructural studies conducted on hot extruded NMMCs revealed uniform distribution of reinforcement (fig 1 & 2), good reinforcement matrix interfacial integrity, and significant grain refinement with minimal porosity. This is due to the gravity of  $\text{ZrO}_2$  associated with judicious selection of stirring parameters (vortex route), good wetting of preheated reinforcement by the matrix melt. Metallography studies of the extruded samples revealed that, the matrix is recrystallized completely. Grain refinement in the NMMCs can primarily be attributed to capability of nano  $\text{ZrO}_2$  particulates to nucleate Al grains during solidification and restricted growth of recrystallized Al grains because of presence of nano dispersoid. It also revealed that a strong bond exists between the interface as expected from metal/oxide system[32] Fig 1 & 2 shows of microstructure of hot extruded NMMC with 9 and 12 Wt.% of reinforcement and fig 3 shows microstructure of matrix alloy (LM 13).



**Fig.1.** Optical microstructure of extruded NMMC containing 9Wt. % at 500 X magnification



**Fig.2.** Optical microstructure of extruded NMMC containing 12Wt.% at 500 X magnification



**Fig.3.** Optical microstructure of matrix alloy at 500 X magnification

### 3.2 Mechanical Properties (hardness and strength)

The mechanical properties of NMMCs reveals with addition of reinforcement content up to 9 Wt.% enhances mechanical properties and addition above this limit deteriorates mechanical properties. The Vickers hardness test results conducted on extruded NMMCs samples revealed an increasing trend in matrix hardness with increase reinforcement content up to 12% (see tab 2). This increase in trend of hardness in the matrix alloy can be attributed primarily to the presence of harder  $ZrO_2$  ceramic particulates in the matrix and higher constraint to the localized deformation during indentation due to their presence and reduced grain size.

The ambient temperature tensile test results revealed significant improvement in 0.2% YS and UTS (ref tab 2) for NMMCs developed with little adverse effect on ductility. Increase in tensile strength (reinforcement up to 9 Wt.%) is attributed to incoherence and a high density of dislocations near the interface between the dispersoid and the matrix. Precipitation reactions are accelerated because incoherence and the high density of dislocations act as heterogeneous nucleation sites for precipitation. Increase in grain boundary area due to grain refinement, built of thermal stresses at the interface due to difference co-efficient of thermal expansion (CTE) and effective transfer of applied tensile load (since the aspect ratio of the particles are large) to the uniformly distributed well bonded reinforcement (strength of ceramic materials lies much higher than metallic materials [33] as the UTS of composite developed is 15% higher than that of un-reinforced alloy. Under the applied stress, increasing the amount of grain boundaries acts as obstacle to the dislocation movement and end up with dislocation pileup at the grain boundary region. [34]. multi-directional thermal stresses induced during processing easily starts multi-sliding system under applied stress so that, dislocations were found developing and moving in several directions [35]. These multi-slip planes agglomerate under the applied stress forms grain boundary ledges. As the applied load increases these ledges acts as obstacles to dislocation movement resulting in pile-ups. The coupled effects of these two obstacles lead to increase in the strength of the composite.

**Table 2.** Mechanical properties of NMMCs and Matrix alloy (LM 13 )

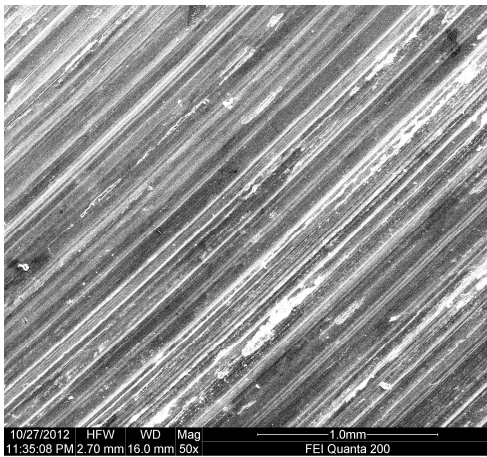
ZrO <sub>2</sub> (wt.%)	Property			
	HV	0.2% YS(MPa)	UTS (MPa)	Ductility %
<b>Matrix alloy(LM13)</b>	<b>62</b>	<b>55.62</b>	<b>72.15</b>	<b>11</b>
<b>3</b>	<b>68</b>	<b>65.32</b>	<b>73.41</b>	<b>8.4</b>
<b>6</b>	<b>70</b>	<b>70.23</b>	<b>79.21</b>	<b>8.0</b>
<b>9</b>	<b>71</b>	<b>107.24</b>	<b>83.52</b>	<b>5.3</b>
<b>12</b>	<b>74</b>	<b>75.24</b>	<b>78.41</b>	<b>3.2</b>

### 3.4. Sliding wear behavior

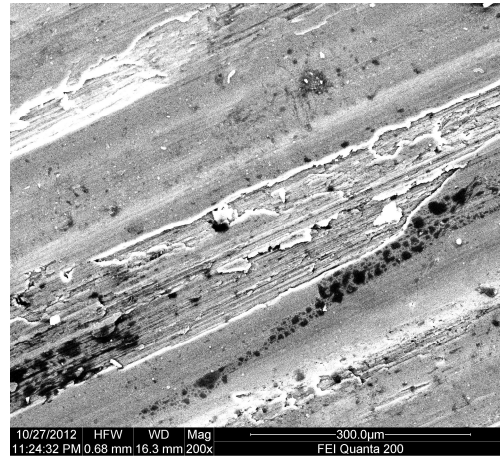
Fig 4-6 shows the variation in volume loss of the samples as a function of sliding distance tested at loads of 10 N, 30 N and 50 N respectively. Un-reinforced matrix alloy and NMMCs with lower Wt.% of  $\text{ZrO}_2$  underwent large volume loss during the initial stages of test. After sliding for some distance, the volume loss increased approximately linearly with sliding distance. It can be seen that the volume loss of both the matrix alloy and the NMMCs specimens increases with the increase of the sliding distance and applied load. It is also noted that the NMMCs possess significantly lower wear rates than the matrix alloy. The wear volume loss of each developed NMMCs reduces with increase in Wt.% of  $\text{ZrO}_2$  content. The figures show that the wear rate in all the samples increases marginally with applied load.

### 3.5. Wear mechanism

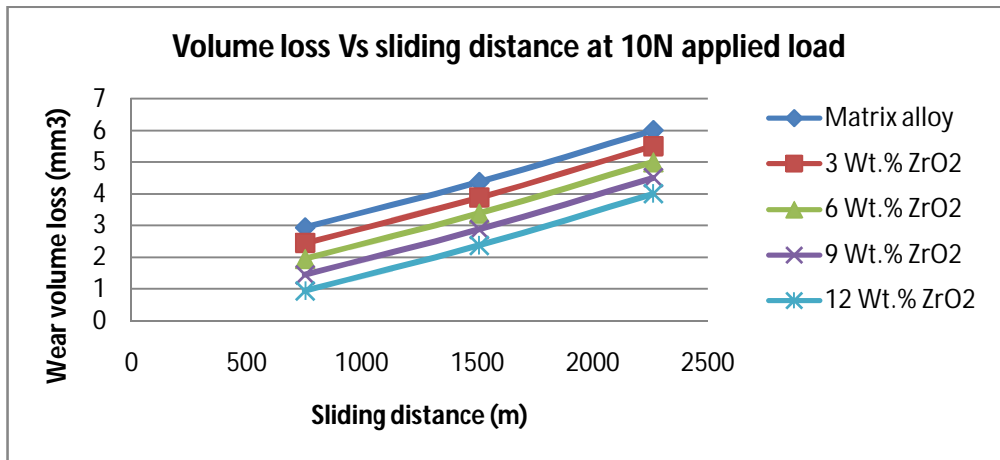
Figs. 7 and 8 shows the SEM photograph of the worn surface of the matrix alloy as well as NMMCs indicating the wear mechanism. It is observed that the worn surface of the matrix alloy is completely smooth and flat and containing wear tracks in the direction of sliding. In addition, the signature of intense plastic flow is seen in the substance. In contrast, the worn surface of NMMCs with 9 Wt. %  $\text{ZrO}_2$  was found to be rough. Microscopic examination of its subsurface shows fragmentation of the dispersoid and their uniform dispersion on the entire worn surface and, as a result, plastic flow of the matrix is limited. Further, the worn surface of NMMCs with dispersoid content of 9 Wt.%  $\text{ZrO}_2$  was found to be very rough with a characteristic brittle fracture. The subsurface region consisted of fragments of crushed dispersoids (refer Fig. 8). A very thin tribo-induced layer was also formed on the entire surface and in addition some dispersoids projected onto the surface.



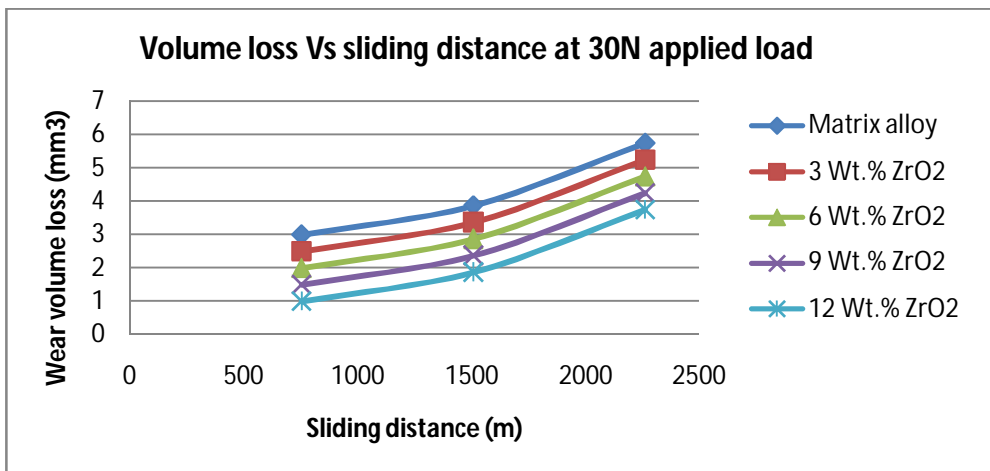
**Fig.5.** SEM Photomicrograph of worn surface of matrix alloy (LM 13)



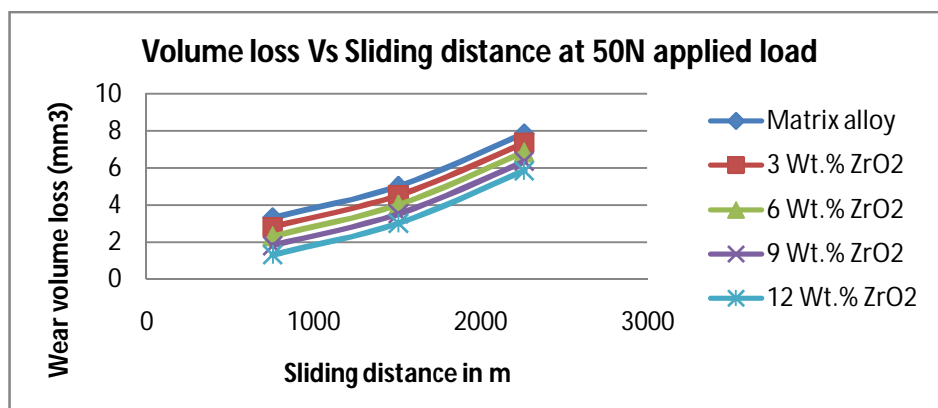
**Fig. 6.** SEM Photomicrograph of worn surface of NMMCs (9 Wt. %  $\text{ZrO}_2$ )



**Fig 1.** Plot showing volume loss of matrix alloy and MMC ( $\text{mm}^3$ ) with various Wt. % of  $\text{ZrO}_2$  as a function of sliding at 10N applied load



**Fig 2.** Plot showing volume loss of matrix alloy and MMC ( $\text{mm}^3$ ) with various Wt. % of  $\text{ZrO}_2$  as a function of sliding at 30N applied load



**Fig 3.** Plot showing volume loss of matrix alloy and MMC ( $\text{mm}^3$ ) with various Wt. % of  $\text{ZrO}_2$  as a function of sliding at 50N applied load

#### 4. Conclusions

Synthesis of NMMCs was achieved through DMD technique coupled with hot extrusion. Microstructural analysis reveals fine grain refinement with uniform distribution of dispersoid. Mechanical characterization exhibits significant improvement in the hardness and UTS due to the presence of nano ZrO<sub>2</sub> particulates and its uniform distribution in the matrix alloy. Pin-on-disc dry sliding wear characterization of developed NMMCs reveals improved wear resistance with increasing Wt. % of ZrO<sub>2</sub>. Nano ZrO<sub>2</sub> particulates has an excellent load bearing capacity under applied load in sliding process, thus enhancing the wear resistance of NMMCs. The SEM photographs of worn surfaces of NMMCs with 9 Wt. % ZrO<sub>2</sub> was found to be rough, in contrast to the worn surface of matrix alloy, which is completely smooth and flat.

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