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SYNTHESIS AND CHARACTERIZATION OF TRIBOLOGICAL PROPERTIES OF ALUMINIUM ALLOY REINFORCED WITH ZIRCONIUM DIOXIDE COMPOSITE

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Abstract

The properties of Metal Matrix Composites (MMC) are inevitably a compromise between the properties of the matrix and reinforcement phase. It is clear that the composition and properties of the matrix and reinforcement phase affect the properties of the composite directly, by normal strengthening mechanisms, and indirectly, by chemical interactions at the reinforcement/matrix interface. Aluminium is the most abundant metal, in the earth's crust so aluminium is used as the most popular matrix for the MMC. The aluminium alloys are quite attractive due to their greater strength, improved stiffness, reduced density, improved high temperature properties, controlled thermal expansion coefficient. In the present study, Cast composites have been synthesized using stir casting technique, by addition of 0, 3, 6, 9 and 12 wt% of ZrO_2 powder to molten Al1100-Mg alloy. The influence of increasing amount of ZrO_2 powder addition on the evolution of cast microstructure is studied under SEM and their impact on the tribological properties of the resulting composites has been investigated.

Keywords: MMC, ZrO_2, Al1100-Mg, SEM, Tribological properties

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1 INTRODUCTION

The benefit of using Aluminium MMC (AMMC) is the advantage of attaining improved property combinations compared to monolithic materials that can result in a number of service benefits. Among these is increased strength, decreased weight, higher elastic modulus and improved wear resistance [1]. The excellent mechanical properties of these materials, together with the material saving through weight reduction and the relative low cost in production, makes them very attractive for a variety of engineering applications in the automotive and aerospace industries [2-3].

In AMMC one of the constituent is aluminium/aluminium alloy, which forms percolating network and is termed as matrix phase. The other constituent is embedded in this matrix phase and serves as reinforcement, which is usually non-metallic and commonly ceramic such as ZrO_2, SiC, Al_2O_3, TiO_2 etc [4-6]. The major methods to produce AMMC are stir casting, vortex mixing method, powder metallurgy, liquid metal infiltration, squeeze casting, rheo casting and spray deposition technique [7-9]. Stir casting method is widely used because of its simplicity, low cost and also suitable for batch production. In a stir casting process, the reinforcing phases are distributed in to molten aluminium by mechanical stirring. In order to achieve the optimum properties of the MMC, the distribution of the reinforcement material in the matrix alloy must be uniform and the wettability or bonding between these substances should be optimized [10-11]. The porosity levels need to be minimized and chemical reactions between the reinforcement materials and the matrix alloy must be avoided [12]. The word tribology is derived from Greek word 'Tribos' that means rubbing, which deals with the study of interface between two or more bodies in relative motion. Tribology at present deals with all aspects of transmission and dissipation of energy and materials in mechanical equipment's including the various topics of friction, wear and lubrication. In the present work, an attempt has been made to investigate the hardness and tribological behavior of particulate reinforced composites prepared by stir casting technique. The wear-rate is estimated taking into consideration the effect of frictional heat on the wear properties at contact surfaces, the effect of reinforcement, mechanical-load, sliding distance, sliding velocities on wear-rates, coefficient of friction and transition wear [13-14].

2 EXPERIMENTAL DETAILS

2.1 Test materials

A11100 of 99.674% purity and commercial magnesium of 99.92% purity is used as the matrix and Zirconium dioxide (ZrO_2) of 99.87% purity and average particle size of 25 µm was used as reinforcement for the synthesis of particle reinforced MMC. Chemical composition of the commercial A11100 and Magnesium (Mg) used in the present investigation are shown table 1.

Table 1: Chemical composition of A11100 and Mg used in present study

	Chemical Composition (wt %)					
Material	**Cu**	**Mg**	**Si**	**Fe**	**Mn**	**Al**
Al-ingot	0.1 %	0.05 %	0.3 %	0.6 %	0.05 %	BAL
Mg-ingot	0.016	BAL	0.006	0.020	0.002	0.023

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2.2 Methodology

About 740 g of commercially pure A11100 was melted and superheated to a desired processing temperature in a clay-graphite crucible inside the muffle furnace. Before any addition, the surface of the melt was cleaned by skimming. The weighed amount of powder was added in to molten A11100 at a processing temperature of 900^oC. A coated pitched blade stirrer was used to disperse the ZrO_2 particles in the melt. The speed of the stirrer was kept constant at 300 rpm. A non-contact type speed sensor was used to measure the stirring speed. The temperature of the melt was measured by using a digital temperature indicator connected to a chromel-alumel thermocouple. During stirring, the temperature of the slurry was maintained within 10^oC of the processing temperature. A magnesium lump of 3 wt% was wrapped by aluminium foil and plunged into the melt-particle slurry after the addition of ZrO_2 particles. When the desired time of the stirring elapsed, reduce the stirrer speed. After completion of processing steps, the graphite stopper at the bottom of the crucible is removed by using the lever to pour the melt- particle slurry into split type graphite coated and preheated permanent steel mould of size three cavities of 12 mm and one cavities of 36 mm diameter and length of 80 mm cavities provided in the mould. No degassing practice of the melt or the slurry was carried out at any stage of processing.
The composite has been designated on the basis of its constituent and the first letter **A** indicates the base metal of A11100 and the next letter **M** indicates alloying element of magnesium, which was kept at 3 wt%. **AM** is followed by a letter **P** indicates the ZrO_2 powder followed by the number indicating the wt% of ZrO_2 powder added. Different composites synthesized by solidification processing and their designation are given in the table 2.

Table 2: Nominal composition of alloy and composites

Designation of composites	Magnesium (wt %)	Particle (wt %)
AM	3	0
AMP3	3	3
AMP6	3	6
AMP9	3	9
AMP12	3	12

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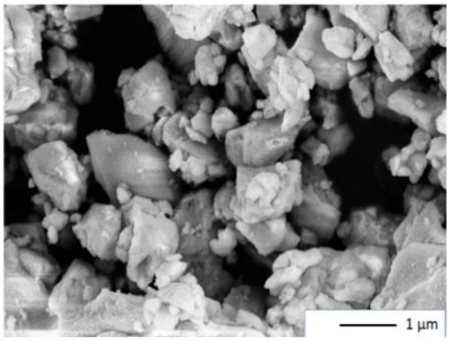
3 RESULTS AND DISCUSSIONS

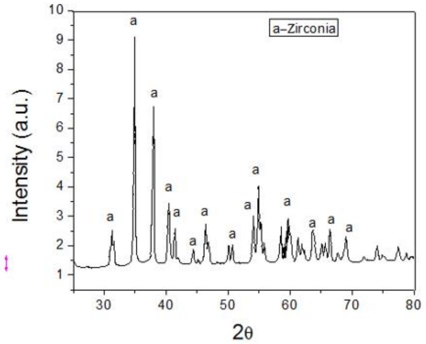
3.1 SEM Microstructure and X-ray diffraction analysis of ZrO_2 particles

The size and particle shape of the ZrO_2 particles in the powder has been observed under SEM and the results are shown in fig 1. The size is in the range between 0.2 µm to 2 µm and it is observed that some particles are in spherical shape and some particles have irregular shape.

Fig.1: SEM micrographs showing size and particle shape of the Zirconia powder

Fig. 2: XRD pattern of ZrO_2 particles used in the synthesis of Al1100 (Mg)-ZrO_2 composites





The powder has been examined for its X-ray diffraction (XRD) pattern using X-ray diffractometer in the two theta range of 5-80^0 using CuK_α radiation target and nickel filter, step size and dwell time were suitably adjusted, which was used for identification of various phases with the help of inorganic JCPDS (Joint committee on powder diffraction standards) x-ray diffraction data card which shows the ZrO_2 particles are fairly pure. The fig 2 shows the XRD pattern of ZrO_2 particles used in the synthesis of Al1100 (Mg)-ZrO_2 composites.

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3.2 SEM Microstructure studies on Cast composites

The SEM micrographs of the composites synthesized by addition of increasing amounts of powder are shown in fig 3. There are bright particles of oxide which are of a few microns which are visible at a 250X magnification. The number of such particles increases with increasing addition of powder mix as evident by comparison of micro structures shown in fig 3(a), (b), (c), (d) and (e). During processing at a high temperature, it is expected that these oxides will react with molten aluminium and get reduced to metallic Zirconium, which will alloy with molten aluminium. The reaction will also produce alumina by oxidizing aluminium. After reaction, the resulting slurry may be solidified to get a composite based on Al-Zr alloy containing generated alumina. The resulting composite is Al (Mg, Zr)-Al_2O_3 (ZrO_2) indicating a matrix of Al-Mg-Zr alloy reinforced with oxide particles containing of un-reacted ZrO_2 and Al_2O_3 resulting from reduction of ZrO_2 by molten aluminium. Four composites have been developed by addition of different amounts of ZrO_2 following the same processing route. ZrO_2 oxides will react with molten aluminium and following phases expected that alumina, ZrO_2, $MgAl_2O_4$, MgO, $MgZrO_4$ and Al_2ZrO_2.

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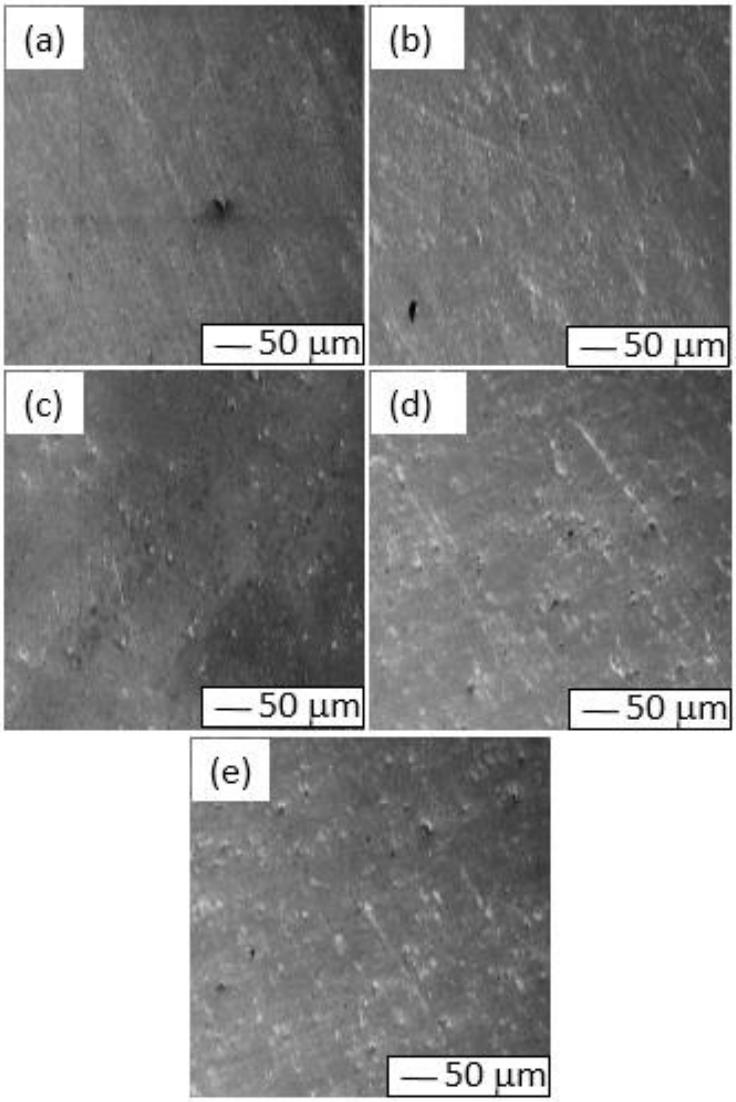

Fig. 3: SEM micrographs of different cast composites developed by increasing amounts of ZrO_2 powder of resolution 250X designated as (a) AM (b) AMP3 (c) AMP6 (d) AMP9 (e) AMP12 AMP6 (d) AMP9 (e) AMP12

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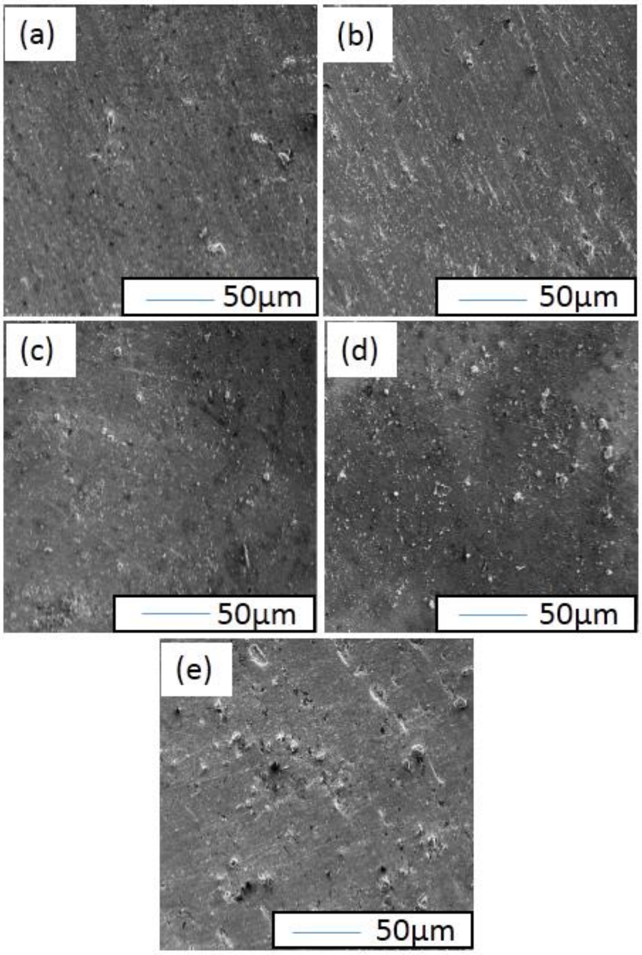

Fig. 4: SEM micrographs of different cast composites developed by increasing amounts of ZrO_2 powder of resolution 500X designated as (a) AM (b) AMP3 (c) AMP6 (d) AMP9 and (e) AMP12

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All the four SEM microstructures in fig 4 (a) AM (b) AMP3, (c) AMP6, (d) AMP9 and (e) AMP12 respectively contain similar phases but their weight fraction varies depending upon the amount of Zirconium dioxide addition. The composite, AMP12, has more distributed phase than that of the composite AMP3. It is observed that the porosity (dark spot) in the composite increases with increasing addition of ZrO_2 particles. This is often attributed to attachment of particles with bubble during processing. This attachment takes place during particle transfer by stirring as mentioned earlier. It may also happen during solidification as the dissolved gases start nucleating on the heterogeneous surfaces of particles. Often these bubbles are not able to float out rapidly due to increase density because of attached particles and get entrapped during solidification, enhancing the porosity in the composite. Thus, porosity increases with increasing addition of ZrO_2 powder in cast composites.

3.3 Mechanical properties of Al1100 (Mg)-ZrO_2 composites

3.3.1 Rockwell hardness

Rockwell hardness has been measured for the unreinforced alloy and cast composites developed by addition of ZrO_2 with 1 mm hardened steel ball indenter under 10 to 150 kg load. Eight indentations have been taken for each sample; the distance of 0.8 cm has been maintained from one indentation to another indentation. The hardness of the cast composites increases with increasing addition of ZrO_2 particles compared to AM base alloys as observed in fig 5, the composites developed by addition of 12 wt% of powder have higher hardness than the composites developed by addition of 3, 6 and 9 wt% of ZrO_2 powder. However, the alloy without any powder added has the lowest hardness. As the reinforcement contents increases in the matrix material, the hardness of the composites also increases. Al (Mg) alloy is a soft material and the reinforced ZrO_2 and generated Al_2O_3particles being hard, contributes positively to the hardness of the composites. The presence of stiffer and harder ZrO_2 reinforcement leads to increase in constraint to plastic deformation of the matrix during the hardness test. Increase in hardness of composites not only depends on wt% of ZrO_2 but also depends on structure of the composite and condition of interface bonding between matrix and reinforcement particles.

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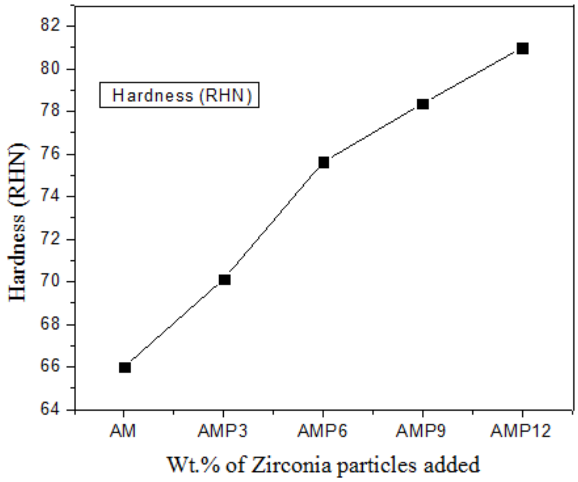

Fig. 5: Variation of average hardness of unreinforced base alloy and cast composites developed by addition of ZrO_2

3.3.2 Tribological properties of Al1100 (Mg)-ZrO_2 composites

A pin-on-disc test apparatus was used to investigate the dry sliding wear characteristics of the aluminium alloy (Al1100-Mg) and Al1100 (Mg)-ZrO_2 composites as per ASTM G99-95 standards. Wear specimen 10 mm in diameter and 27 mm high were cut from as-cast samples, machined, and then polished metalographically. Wear tests were conducted with loads ranging from 10, 20 and 30N and sliding speeds of 1.50 m/s for a sliding distance of 1500 m. All tests were conducted at room temperature. The initial weight of the specimens was measured using a single pan electronic weighing machine with an accuracy of 0.0001g.

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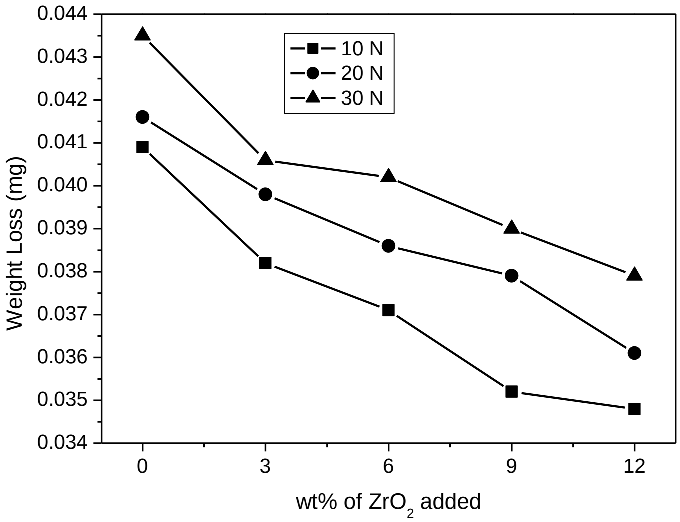

Fig. 6: Variation of differences in weight loss between tests carried out at different loads for the cast composites developed by increasing amounts of ZrO_2 powder designated as (a) AM (b) AMP3 (c) AMP6 (d) AMP9 (e) AMP12 AMP6 (d) AMP9 (e) AMP12

During the test, the pin was pressed against the counterpart rotating against an EN32 steel disc (hardness 65 HRC) by applying the load. All the specimens followed a single-track, 35mm in diameter, with a tangential force. After running through a fixed sliding distance, the specimens were removed, cleaned with acetone, dried, and weighed to determine the weight loss due to wear. The differences in weight measured before and after tests give the wear of the composite specimen. The wear of the composite specimens were studied as a function of the weight percentage of reinforcement, sliding distance, applied load, and sliding velocity.
The wear rate have been analyzed by plotting graphs with wt% of ZrO_2 along the X-axis and weight loss of the cast composites along Y-axis. The wear resistance of the cast composites increases with increasing addition of ZrO_2 particles; compared to AM base alloys as observed in fig. 6. The composite developed by addition of 12 wt% of powder has higher wear resistance than the composites developed by addition of 3, 6 and 9 wt% of ZrO_2 powder. However, the alloy without any powder added has the highest wear rate. As the reinforcement contents increases in the matrix material, the wear resistance of the composites also increases, it also observed that as the load increases wear rate also increases by increasing the wt% addition of Zirconia.

4 CONCLUSIONS

1. The liquid metallurgy route (stir casting technique) was successfully adopted in the preparation of Al1100-Mg-ZrO_2 MMCs alloy and composites containing 0, 3, 6, 9 and 12 wt% of ZrO_2 powder reinforcement.
2. XRD analysis shows the ZrO_2 particles are fairly pure.
3. Porosity (dark spot) in cast composites increases with increasing addition of ZrO_2 powder
4. The hardness of the composites is found to increase with increase in reinforcement content and the higher hardness noticed for the 12 wt% of ZrO_2 powder addition.
5. The 12 wt% of ZrO_2 powder addition composite exhibit minimum wear rate which means wear resistance found to increase with increase in percentage of reinforcement.
6. The wear resistance of the cast composites increases with increasing addition of ZrO_2 particles as compared to AM base alloys and also the wear rate of both reinforced and unreinforced specimen increases as the load increases

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