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Influence of calcium hexaboride reinforced magnesium composite for the mechanical and tribological behviour

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ABSTRACT

The effect of calcium hexaboride $(CaB₆)$ reinforced magnesium metal matrix composite (Mg-MMC) for mechanical and tribological behavior have been investigated. Fabrication of the Mg-MMC was done by the squeeze casting process using the bottom pouring stir casting furnace. Mechanical properties such as tensile strength and hardness of the composites were analyzed. The dry sliding wear behavior of the pure Mg and the composites was evaluated in the pin-on-disk tribo-meter using SAE52100 bearing steel disk at room temperature. Improvement in the mechanical property was found in $CaB₆$ reinforced Mg-MMC as compared with pure Mg. A significant fact was that, addition of $CaB₆$ enhanced the wear resistance of Mg-MMC, revealing the delamination wear mechanism is dominant from other wear mechanisms.

1. Introduction

In recent years, extensive investigations have been done on lightweight materials in the automobile domain for increasing fuel economy [\[1,2\].](#page--1-0) In general, Magnesium, Aluminum and Titanium are considered as lightweight materials for structural applications. Among them, Mg is considered as an essential candidate because of its high specific strength and easy recyclability [\[3\]](#page--1-1). But, the utilization of pure Mg has certain limitations such as poor ductility, thermal stability, wear resistance, creep and corrosion resistance etc. Such limitations can be overcome through use of various methods such as alloying and composite processes, which form new precipitates and the beneficial effect on hard reinforced particle [4–[10\].](#page--1-2) However, studies in the Mg based alloys and composites for development of tribological behavior have not been much to be talked. In recent years, many reports have been published to explain the tribological behavior of Mg based alloys and composites. Particularly, the hard particles present in Mg-based composite provide improved wear resistance compared to pure Mg [11–[18\].](#page--1-3)

 $CaB₆$ belongs to the 2 A group alkaline earth boride [\[19\].](#page--1-4) It has favorable properties such as low density (2.45 g cm^{-3}) , high melting point (2235 °C), low coefficient of thermal expansion $(6.2\times10^{-6}$ °C⁻¹), high hardness (27 GPa), chemical stability and good wear resistance in a corrosive environment [20–[22\].](#page--1-5) Previous studies have proved that the addition of $CaB₆$ to metals and non-metals provides improved mechanical properties [\[23,24\]](#page--1-6). However, there are not many reports as

the subject of the $CaB₆$ reinforced composites for mechanical and tribological behavior.

On the other hand, the fabrication processes also influence the mechanical and tribological behavior. Squeeze casting is one of the fabrication technique to promote the solidification under the high pressure. The application of squeeze pressure has the possibility of recrystallization, grain refinement and porosity reduction, resulting in improvement of mechanical and tribological behavior [25–[27\].](#page--1-7)

Here, an attempt has been made to prepare Mg based composites by reinforcing 2 wt\% of CaB_6 using squeeze casting process. The prepared samples were mechanically polished and tribological tests were conducted under different loads (10, 20 and 30 N) and velocities (0.4, 0.6 and 0.8 m s⁻¹) for a constant sliding distance of 2000 m. Optical microscope (OM) was used for revealing the grain structure of the etched surface. Mechanical tests were conducted and enhancement of mechanical property was estimated as a function of hardness and tensile strength. Essentially, the wear mechanisms were investigated as a function of wear rate and co-efficient of friction using worn-out surface morphology. Fractography and surface morphology of the worn out surfaces were observed using Scanning electron microscope (SEM) analysis. Energy dispersive X-ray (EDX) analysis was used for analyzing the worn out debris to confirm wear mechanism.

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2. Experimental details

2.1. Materials and processing

A pure Mg ingot with 99% purity was purchased from Vaishnavi castings Ltd, Chennai, India. This is the matrix material, it was cut into 50 mm x 50 mm x 20 mm billets using a band saw. The $CaB₆$ with a mean particle size of 50 mesh and purity of 99.5%, was supplied by Alfa aeser, USA. A bottom pouring stir casting furnace (made by swam equipment Ltd, Chennai, India) was used for the composite fabrication process. The Mg billets were melted in a steel crucible at 700 °C in an electrical resistance furnace. A productive gas atmosphere of argon and sulfur hexafluoride in the ratio of 5:1 was maintained. The matrix molten slurry was stirred at 450 rpm for 2 min using a twin blade impeller inclined at 45°. The mild steel blade was coated by zirtex to avoid sticking. After stirring, the slags were removed from the matrix molten slurry. In order to remove the moisture and increase wettability, 2 wt% of reinforced particles $(CaB₆)$ was preheated in the box furnace at 400 °C for 2 h.

The preheated reinforced CaB_6 particles were then mixed in to the matrix molten slurry and stirred at 450 rpm for 15 min for achieving uniform distribution. The molten composite was then poured into the mold of 50 mm diameter and 250 mm height by the bottom pouring technique. Before pouring, the mold was preheated to 250 °C for 1 h. Squeeze casting which is similar to the above technique together with squeeze pressure (120 MPa) was applied at 300 °C for 2 min during solidification. The squeeze pressure was applied using a hydraulic press with a capacity of 50 t.

2.2. Density and porosity

The densities of pure Mg and its composites were determined using the Archimedes principle under ASTM standard D3800. Eq. [\(1\)](#page-1-0) was used for calculating the density of the materials.

$$
D_a = \frac{W_a X D_w}{W_a - W_w} \tag{1}
$$

where, D_a - Actual density of the specimen, W_a - weight of the specimen in air, D_w - water density, W_w - weight of the specimen in water.

Eq. [\(2\)](#page-1-1) was used to calculate the porosity of the composite material,

Percentage of porosity =
$$
\frac{D_t - D_a}{D_t} \, X \, 100\%
$$
 (2)

where, D_t - Density of pure Mg.

The weight of the samples was obtained using an analytical weighing balance (wensar, India, accuracy of ± 0.00001 g). The theoretical density of the composites was calculated on the basis of the assumption of full density. All the calculations were done using the rule of mixture.

2.3. Microstructure observation

Microstructural observations such as grain structure and interfacial integrity between Mg and $CaB₆$ were analyzed by using the metallographic polished samples. The polishing was carried out with various grid ranges (240, 600 grid SiC sand paper, 6 µm, 1 µm diamond suspension and finally colloidal silica) by using mechanical polisher. For every grid range, the samples were initially washed with water and ultra-sonication was then performed in ethanol for 2 min. The polished samples were immersed in acetic - picral etchant (6 g picric acid, 10 ml acetic acid, 70 ml ethanol and 20 ml water) for etching until the formation of brown film on the surface. It was subsequently washed with ethanol to get clear grain structures. Olympus DP −10 microscope was used for analyzing the grain structure.

2.4. X -Ray diffraction Studies

The crystalline structure of pure Mg and Mg-MMCs were analyzed by using XRD (Shimadzu, Japan). The X-ray wavelength was λ =1.5405 Å with a scan speed of 2° min⁻¹. The joint committee on powder diffraction standards (JCPDS) was used for analyzing the XRD results of both pure Mg and Mg-MMC samples.

2.5. Hardness test

The hardness of pure Mg and Mg-MMCs were evaluated to determine the effect of reinforced particles at room temperature. Vickers micro-hardness test (Buehler) was used with a load of 50 gf for 15 s dwell time. Ten measurements were processed at ten different locations on the same sample to avoid the effect of indenture resting on hard reinforced particles.

2.6. Tensile test

The universal testing machine (Instron UTM – 50 kN, model: 3369) was used for studying the tensile behavior of pure Mg and Mg-MMC materials at room temperature with a minimum cross head speed of 1 mm. min−¹ . Prior to taking the tensile test, the composite specimens were machined as per the ASTM: E8 Standard. Three samples were used for each composite to ensure a reproducibility. The surface morphology of tensile fractured surface was observed by using a Hitachi scanning electron microscope for analyzing fracture behavior.

3. Tribological test

The tribological test was carried out at room temperature by using a DUCOM pin-on-disk tribo-meter, as schematically shown in [Fig. 1.](#page-1-2) Each pin sample has a diameter of 12 mm and length of 50 mm. The pin sample slides on the SAE 52100 bearing steel (63 HRC) disk with a diameter of 55 mm and a thickness of 10 mm. The same surface roughness was maintained on the disk and the pin samples. The tribological experiments were conducted at room atmospheric condition with various loads (10, 20 and 30 N) and sliding velocities (0.4, 0.6 and 0.8 m. s−¹) for a constant sliding distance of 2000 m. The wear rate was calculated by mass loss method (Eq. ([3\)](#page-1-3)), and coefficient of friction was measured by the software which is configured with DUCOM pinon-disk tribo-meter.

mass of the pin before the wear test – mass of the pin
wear rate =
$$
\frac{after\text{ the wear test}}{\text{total sliding distance}}
$$
gm⁻¹

Fig. 1. Schematic representation of pin- on-disk tribometer.

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