Microstructure and Wear Behavior of Graphene Nanoplatelets Reinforced Magnesium Matrix Composites Fabricated by Solidification Processing

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Abstract

Magnesium matrix nanocomposites, in which magnesium is strengthened with nano-sized reinforcements, have recently emerged as attractive materials for engineering applications due to their promising mechanical and tribological properties. In this study, graphene nanoplatelets (GNP) reinforced magnesium matrix composites were fabricated by means of a solidification processing that combines conventional mechanical stirring and ultrasonic dispersion of reinforcements in liquid matrix. The nanocomposites were fabricated with various contents of GNPs with an average thickness of 50-100 nm and diameter of 5 μm, i.e. 0, 0.25 and 0.5 wt.%. A pin-on-disc apparatus with a steel disc as counterface was utilized to determine the dry sliding wear behavior of nanocomposites. The pin-on-disc tests were carried out under a constant sliding velocity of 0.3 m/s and distance of 1000 m with loads of 5, 10 and 15N. The microstructure of fabricated nanocomposites and worn surfaces of pins were investigated under optical and scanning electron microscopy (SEM), respectively. It was found that the Mg/0.25wt.%GNP nanocomposite exhibited better wear resistance compared to the reference matrix and the Mg/0.5wt.%GNP nanocomposite at loads higher than 5N. It was suggested that increasing GNP content, i.e. 0.5 wt.% was more likely to result in agglomeration, and hence led to reduction in wear resistance. Abrasive wear and delamination were considered to be dominant wear mechanisms for magnesium and its nanocomposites.

1. Introduction

Magnesium (Mg) which is one of the lightest engineering materials has a density about two-thirds of aluminum, good machinability, high damping and recycling capacity, good castability and high specific strength[1-4]. These properties make Mg ideal for to its use in engineering applications about to provide energy efficiency. However, Mg and its alloys suffer from lower mechanical properties and wear resistance due to their hexagonal close-packed crystal structure with restricted number of operative slip systems [4-6]. In order to develop the mechanical and tribological performance, a wide range of reinforcements including micron/nano-sized ceramic and carbonaceous fillers are conventionally incorporated into Mg matrices [6,7]. Among these reinforcements, carbonaceous fillers such as carbon nanotubes (CNT) and GNPs have received a great deal of attention not only for their outstanding mechanical strength but also for their self-lubricating effect that may reduce coefficient of friction (COF), and hence contribute to wear resistance [8].

In the literature, several attempts were made to incorporate GNPs consisting of a few graphene layers into Mg matrices [2,9]. There are a number of methods to fabricate GNPs reinforced metal matrix composites, e.g. powder metallurgy and solidification processes. Solidification processes seem suitable for large scale production. However, it is difficult to uniformly disperse GNPs through liquid matrices due to large surface area and poor wettability of GNPs. It has been shown that the introduction of high intensity ultrasonic waves into molten Mg, namely ultrasonic cavitation-based dispersion method, led to homogeneous dispersion of nano-sized reinforcement into the matrix [10,11].

Although there are a number of studies on the determination of wear behavior of Mg and its ceramic reinforced composites in the literature, the number of works to document the self-lubricating effect of GNPs on the wear performance of Mg [12,13]. Therefore, the aim of the present study is to fabricate GNP reinforced magnesium nanocomposites by a solidification processing that combines conventional mechanical stirring and ultrasonic method, and to investigate their microstructures and wear performance.

2. Experimental Procedure

Mg ingots with 99.9% purity and commercially available GNPs with an average thickness of 50-100 nm and x, y dimension of 5 μm were used as the matrix and reinforcement, respectively. In order to feed the GNPs into the matrix without floating on the melt surface, the pellets, in which the GNPs are encapsulated with pure Mg powders, were prepared. For the preparation of pellets, appropriate amount of
Mg powders and GNP s were first ball-milled in stainless steel vials with 10 mm diameter stainless steel balls for 2h at 350 rpm. The ball-milled Mg powders and GNP s were then cold pressed under 250 MPa to form pellets in 30 mm in diameter.

For the fabrication of nanocomposites, approximately 130 g pure Mg was melted in a steel crucible by using an electric resistance furnace under the protective gas of 99%CO₂ + 1%SF₆ at 700 °C. Mechanical stirring was applied by a graphite stirrer at 1000 rpm for 15 min and the pellets were introduced into the melt during the stirring. After the removal of mechanical stirrer, the 12.7 mm diameter titanium alloy ultrasonic probe which is a part of the ultrasonic system was dipped into the melt and the melt was ultrasonically processed for 15 min at 675 °C. Then, the molten composite reheated to 700 °C was cast into a steel mold preheated to 400 °C. The Mg matrix composites were fabricated with approximately 0.25 and 0.5 wt.% GNP. For comparison, the reference Mg was also cast using the pellets in the absence of GNP s under similar conditions.

For the metallographic studies, the samples were traditionally ground and polished down to 0.5 μm finish with diamond suspension. The samples which were etched with acetic picral were characterized with optical (Leica DM 2500M) and SEM (FEI Quanta FEG 250 equipped with an energy dispersive x-ray spectroscopy, EDX, system) microscopy. The dry sliding wear behavior of fabricated nanocomposites was determined via pin-on-disc tests according to the ASTM G99-95a standards. Prior to the wear tests, the surface of pins in 10 mm diameter was also ground and polished to 1 μm. The pin-on-disc tests were conducted against counterface AISI 52100 (62 HRC) disc under a constant sliding velocity of 0.3 m/s and distance of 1000 m with loads of 5, 10 and 15N. COF data was recorded during the wear test. Volume loss and wear rate were calculated after the tests. At least 5 discs were tested for each set of samples and the average data was reported.

3. Results and Discussion

The optical microscopy images of reference Mg and nanocomposites are shown in Fig.1(a-c). The average grain sizes of these samples were measured based on the linear intercept method in the ASTM E112 standards. While the grain size of reference Mg was 95 μm, it decreased to 86 and 45 μm with the addition of 0.25 and 0.5 wt.% GNP s, respectively. This shows that the grain size was reduced with increasing GNP concentration into the matrix.

Fig.2 shows a representative SEM image from the Mg/0.25wt.%GNP nanocomposite. It is seen that the GNP s were embedded into the matrix. In order to ensure the presence of GNP s in the matrix, the EDX analysis was performed on the potential GNP site in Fig.2. The distinct carbon peak is more likely to confirm the presence of carbonaceous GNP s.

Figure 1. Optical images of pure Mg with GNP s: (a) 0, (b) 0.25 and (c) 0.5 wt.%. The COF values of reference Mg and nanocomposites were presented in Fig.3. It can be seen that the composites had lower COF values compared to the reference Mg at 5N, indicating self-lubricating effect of GNP s. However, at higher loads the composites exhibited similar or higher COF values than that of unreinforced pin. This unexpected trend could be attributed to the lack of GNP film on
the surface of composite pins or agglomeration of GNP sites into the matrix.

**Figure 2.** SEM image and EDX analysis for a potential GNP site into the Mg/0.25wt.%GNP nanocomposite.

**Figure 3.** COF values of reference Mg and nanocomposites under various loads.

Fig. 4 shows that the wear rates of pure Mg and its GNP reinforced composites. It is clear that Mg/0.25wt.%GNP nanocomposite exhibited better wear resistance compared to the pure Mg and the composite with 0.5 wt.% GNP at higher loads, i.e. 10 and 15 N. This enhancement may suggest that finer microstructure were obtained for the Mg/0.25wt.%GNP nanocomposite due to relatively uniform dispersion of harder GNPs into the matrix. Besides, a sharp decrease in the wear resistance of Mg/0.5 wt.%GNP nanocomposite compared to the reference Mg can be attributed to the agglomeration tendency of increased GNP content. It is also clear from Fig. 4 that the wear rates gradually increased with increasing loads for each set of reference and nanocomposite samples. On the other hand, there were some inconsistent results in the wear tests. The number of pins worn in this study is small, therefore further investigation is required.

**Figure 4.** Wear rates of reference Mg and nanocomposites under various loads.

**Figure 5.**

a) pure Mg, b) 0.25% GNPs, c) 0.5% GNPs SEM images after tribology test.
Fig. 5 presents the SEM images of worn pin surfaces of pure Mg and nanocomposites at 10N. Abrasive wear mechanism was observed for all samples. However, Mg/0.5wt.% GNP nanocomposite in Fig. 5c did not reveal significant delamination due to the hard nature and self-lubricating effect of GNPs.

4. Conclusion

The following conclusions can be drawn from the experimental results:

1) Commercially pure Mg composites with 0.25 and 0.5 wt.% GNPs were fabricated by a combination of mechanical stirring and ultrasonic method.
2) The grain size reduced with increasing GNP content in Mg matrix.
3) The SEM investigations with the EDX analysis showed that the GNPs were incorporated into the matrix.
4) The composites had lower COF values compared to the reference Mg at 5N, indicating self-lubricating effect of GNPs.
5) Mg/0.25wt.% GNP nanocomposite exhibited better wear resistance compared to the reference matrix and the Mg/0.5wt.% GNP nanocomposite at higher loads, i.e. 10 and 15N. It was suggested that increasing GNP content was more likely to result in agglomeration, and hence reduction in wear resistance.
6) Abrasion and delamination were found to be the main wear mechanism for Mg and its nanocomposites under 10N.

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References