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Effect of high-intensity ultrasonic treatment on microstructure, hardness and wear behaviour of the hypereutectic Mg-5Si alloy

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Abstract. The effect of high-intensity ultrasonic treatment (HIUST) on microstructure, hardness and wear behavior in Mg–5wt.%Si hypereutectic alloy has been investigated. The results showed clearly that without HIUST, most of primary Mg2Si appeared as coarse dendritic morphology with average size of about 200 μm. With HIUST, the average size of primary Mg2Si decreased significantly to about 33 μm and their morphologies changed to polyhedral shape. The modification mechanism is mainly attributed conjugation of two mechanisms: cavitation-enhanced heterogeneous nucleation and cavitation-induced dendrite fragmentation. The alloy treated with HIUST has higher hardness and wear resistance than that untreated with HIUST. The wear mechanism of investigated alloys at low applied load (10 N) and low sliding speed (0.3 m/s) is a mild abrasive oxidative wear with little adhesion. However, the wear mechanism due to the applied high loads (30, 50 N) at low sliding speed (0.3 m/s) and/or to the applied high sliding speeds (0.6, 0.9 m/s) under low load (10 N), could be described as delamination mechanism. The microstructures of the specimens were analyzed by optical microscope (OM) (model OPTIKA M–790, Italy). Energy dispersion spectrum (EDS) affiliated to field emission scanning electron microscopy (FESEM) (model Quanta FEG, The Netherlands) were performed to reveal the concentration of alloying elements in selected areas of the microstructure.

1. Introduction

Magnesium alloys are widely applied in the fields which are strongly driven towards weight-reduction, such as automotive, aeronautic and astronautic industries [1]. Meanwhile, improving the elevated temperature properties of magnesium alloys has become a critical issue for the further application [2]. In recent years, the fascinating properties and promising application of hypereutectic Mg–Si alloys have attracted particular interest due to the formation of thermally stable Mg2Si phase [3]. It is known that the intermetallic compound of Mg2Si exhibits an excellent combination of superior properties, such as high melting temperature (1085 °C), low density (1.99 × 10³kg m⁻³), high hardness (4.5 ×10⁹ Nm⁻²), low thermal expansion coefficient (7.5 ×10⁻⁶ K⁻¹) and reasonably high elastic modulus (120 GPa). Furthermore, the Mg2Si phase is exceptionally stable and therefore could effectively impede grain boundary sliding at elevated temperatures. However, the hypereutectic Mg–Si alloys prepared by ordinary ingot metallurgy process showed very low ductility and strength due to the large primary Mg2Si particle size and the brittle eutectic phase [3, 4].
Much work has been focused on the modification effects of Ca [1], P [2], Sr–Sb [3], Y [4], KBF₄ [5] and Bi [6] on the primary and eutectic Mg₂Si in magnesium alloys. However, certain internal defects could be existed. For example, although KBF₄ has a good effect on the modification of Mg₂Si phase in Mg–5Si alloys, a large quantity of smoke and sputter is produced, which is harmful to the surrounding environment [5]. Y [4] and Bi [6] can modify and refine the primary Mg₂Si, but the Mg₂Si phase becomes coarse again when the adding amount exceeds a certain limit. Therefore, an alternative method for the development of hypereutectic Mg–Si alloys with modification of Mg₂Si phase and high mechanical properties is desired.

HIUST is a simple and effective physical method for solidification control. Several investigations were carried out between 1960 and 1990 [7, 8], mainly in the former Soviet Union countries. They clearly demonstrated that the grain–refinement effect on magnesium alloys improvement of mechanical properties. The renewed interest in magnesium materials in recent years has resulted in increased attention in the grain-refinement potential of HIUST for magnesium alloys [9–11], particularly for Mg–Al based alloys.

Recent research [12, 13] has shown that the primary Mg₂Si could be effectively refined and modified by the application of HIUST on the hypereutectic Mg–5 wt.%Si alloy during solidification process. However, the effect of HIUST on wear behavior of hypereutectic Mg–Si alloys has not been reported. Therefore, the main aims of this work are to investigate the effect of HIUST on hardness and wear behavior at different applied loads (10, 30, 50 N) and different sliding speeds (0.3, 0.6, 0.9 m/s) of Mg–5wt.%Si hypereutectic alloy and to explore the wear mechanisms of resultant samples.

2. Experimental procedures

2.1. Materials and processing

Commercial pure Mg (99.8 wt.% purity) and Si (99.9 wt.% purity) were used as starting materials. Charges of about 20 kg with the nominal composition of hypereutectic Mg–5wt.%Si alloy were prepared as the base material in the present study. The charge was melted in a graphite crucible by a 200 kW medium frequency induction furnace under the mixed gas protection consists of tetrafluoroethane (CF₃CH₂F, HFC-134a, 1 vol.%) and carbon dioxide (CO₂, Bal.). Firstly commercial Mg ingots were melted to above 650 °C, and then silicon was added into Mg melt. After that the melt were heated to above 800 °C and kept about 30 min to ensure Mg and Si fully reacted and formed Mg₂Si. Finally the melt was poured into a cast iron mold. The hypereutectic Mg–5wt.%Si alloy ingots were sliced for subsequent experiments.

The ultrasonic treated alloy were prepared as following. The hypereutectic Mg–5 wt.%Si alloy of about 1 kg was remelted at 800 °C in a mild steel crucible using an electric resistance furnace under the mixed gas protection consists of tetrafluoroethane (CF₃CH₂F, HFC-134a, 1 vol.%) and carbon dioxide (CO₂, Bal.). The melt was manually stirred for 2 min using a stainless steel rod, and then was held for additional 10 min in order to get full homogenization. After that the slag was removed, and then the melt was poured into a cylindrical resin-bonded sand mold with dimensions of outer diameter (Ø 100 mm), inner diameter (Ø 42 mm) and length (250 mm) which mounted on the ultrasonic sonotrode of diameter (Ø 40 mm) as shown in Figure 1. The reason for using a cylindrical resin-bonded sand mold is to reduce the cooling rate effect on the resulted microstructures of the prepared samples. Therefore, the difference in the morphology and size of primary Mg₂Si in the microstructures of investigated samples were obtained mainly as a result of the difference in the application of HIUST conditions. HIUST which was generated by using ultrasonic generator (model TS6MD1, Russia) and magnetostrict transducer (model PMS-15-22, Russia) with the maximum output power of 5 KW and the fixed frequency of 21.4 KHz was applied right before pouring the melt at pre-determined optimum temperature of about 800 °C for the pre-determined optimum vibration time of about 90 s based on recorded data [20]. At the end of planed HIUST application time, the ultrasonic source was switched off and the melt was left to room temperature. Casting temperature was controlled within an accuracy of ±2 °C. The ultrasonic waves emitted from the transducer and passed through the acoustic sonotrode were propagated directly into the melt during solidification. The poured melt became a part of acoustic sonotrode, so the action of ultrasonic energy on the melt was raised remarkably. For comparison, alloy without the application of HIUST was prepared at pouring temperature of 800 °C. The chemical composition of the prepared alloy without and with HIUST was measured with X-ray fluorescence analyzer (XRF) (model Axios advanced-PANALYTICAL, The Netherlands) as shown in Table 1.

Table 1: The chemical composition of prepared alloys (wt.%).

| Prepared alloys | Mg  | Si  | Fe  | Cu  |
|-----------------|-----|-----|-----|-----|
| Mg-5Si          | Bal.| 5.02| 0.033| 0.041|
2.2. Materials characterization

All metallographic specimens were cut at the same position of 10 mm from the bottom of castings and prepared according to usual procedures developed for magnesium alloys. Grinding of specimens is carried out on silicon carbide papers wet with water, down to grit size 1200 then followed by fine polishing with diamond, particle size from 6 to 1 μm, on nylon cloth, rinsing with water and ethyl alcohol. For best results, the polished surface must be mirror like, i.e., free from any residual tarnishing layer. After that, they are etched by solution with 10 ml nitric acid, 30 ml acetic acid, 40 ml water, and 120 ml ethanol for 2–3 min. They are then rinsed in anhydrous ethyl alcohol and dried in a blast of dried air.

The microstructures of the specimens were analyzed by optical microscope (OM) (model OPTIKA M–790, Italy). In the present study, the average length of primary Mg2Si was measured as the size of Mg2Si. Six OM micrographs were taken for each sample from the optical observed area at magnification of 100x. The average size of the primary Mg2Si was measured by ImageJ1.44 software. All Mg2Si existed in one picture taken from the observed area were measured. Energy dispersion spectrum (EDS) affiliated to field emission scanning electron microscopy (FESEM) (model Quanta FEG, The Netherlands) were performed to reveal the concentration of alloying elements in selected areas of the microstructure.

Phase constituents of samples were analyzed by X–ray diffraction (XRD) (model X'PERT PRO, The Netherlands) using Cu Kα radiation in step scan of 20 from 20° to 120° with an increment of 0.02° and a scanning speed of 4°/min.

2.3. Hardness and wear tests

The Vickers hardness test of the prepared alloys using the metallographic specimens, at room temperature, was carried out in a Vickers hardness tester (model INSTRON WOLPERT GMBH–930/250, England) according to ASTM E92-82 with a normal load of 3 Kgf (designated as HV). The mean of ten successful measurements was taken to establish the hardness values.

Dry sliding wear test without lubricant was conducted using a pin–on–disc type apparatus (model TNO TRIBOMETER, The Netherlands) in accordance to the ASTM G99-05 standard. The cylindrical pin specimens having diameter (Ø7 mm) and length (12 mm) machined out from the same position of the prepared castings were used as test samples. Hardened ball bearing steel disc (HRC 63) of outer diameter (Ø73 mm), inner diameter (Ø65 mm) and thickness (25 mm) was used as the counterpart surface. Specimens and counterpart surfaces were ground with different emery papers up to 1200 grit and cleaned ultrasonically in acetone to avoid the presence of humidity and non–desirable deposits. During testing, a jet of compressed air was pointed at the edge of the disc to avoid accumulation of wearing particles on the disc. All tests were performed under ambient atmosphere. The wear test conditions were divided into three groups. In the first group, the wear test of samples was carried out with three different normal loads (10, 30, 50 N) at a constant sliding speed of 0.3 m/s for 10 min. In the second group, the wear test was carried out with two different sliding speeds (0.6 and 0.9 m/s) at a constant load of 10 N for 10 min. The weights were measured before and after the experiment using electronic scales with 0.1 mg accuracy, after which the results of the experiment were evaluated according to the loss in
weight. Worn surfaces and wear debris of the specimens were examined and analyzed using FESEM equipped with EDS in order to determine the post-experimental wear mechanisms.

3. Results

3.1. Materials investigation

XRD results reveal that the constituents of the obtained microstructures for both conditions without and with HIUST are only Mg2Si and Mg phases, as shown in Figure 2. Therefore, no change of phase constituents obtained due to HIUST.

![Figure 2: XRD patterns of the investigated hypereutectic Mg–5wt.%Si alloys (a) without and (b) with HIUST.](image)

Figure 3 shows the optical images of the investigated alloy without and with HIUST. Furthermore, Figure 4 shows the FESEM micrographs with EDS line scan across primary Mg2Si and EDS elemental mappings for the elements Mg, Si and Ca for the investigated alloy without and with HIUST. The as-cast microstructures of the prepared Mg–5wt.%Si hypereutectic alloy reveal the presence of primary Mg2Si, Mg halos and eutectic Mg–Mg2Si (Figure 3). Moreover, all the primary Mg2Si are surrounded by Mg halos, followed by eutectic structure. Without any treatment, most of primary Mg2Si are coarse dendritic morphology (Figure 3a) with average size of about 200±2 µm. Also, it can be seen that the coarse primary Mg2Si is formed in the preferred growth direction that occurs at the tips of branches (Figure 4a), resulting in a complex regular with sharp-angled shapes and dendritic morphologies with a nonuniform distribution of Mg in the interdendritic regions (Figure 4b, c). With HIUST, most of primary Mg2Si become polyhedral shape (Figures 3b, 4d) containing a network of segregated Mg along the grain boundaries (Figure 4e, f) and their average size are reduced to about 33±1µm.

![Figure 3: Optical images of the as-cast Mg–5wt.%Si alloy: (a) Without treatment and with (b) HIUST.](image)

![Figure 4: The FESEM micrographs with EDS line scan across primary Mg2Si and EDS elemental mappings of Mg, Si and Ca for the prepared Mg–5 wt.%Si alloy: (a), (b) and (c) without HIUST and (d), (e) and (f) with HIUST.](image)

3.2. Hardness and wear behavior

The average hardness values of the investigated alloy significantly increases from about 50±5.5 HV1 without HIUST to about 69±1 HV1 with HIUST (i.e. a 38% increase), implying that HIUST can improve the hardness of the investigated alloy.
Figures 5 and 6 show the variation of weight loss of the investigated alloy without and with HIUST for various loads (10, 30, 50 N) at constant sliding speed (0.3 m/s) and for various sliding speeds (0.3, 0.6, 0.9 m/s) at constant load (10 N). The investigated alloy with HIUST has better wear resistance than without HIUST due to its lowest weight loss at the same load level (Figure 5) and at the same load sliding speed level (Figure 6). However, with the application of high normal loads or high sliding speeds for the same process, the weight loss increases drastically. However, the investigated alloy due to HIUST still has better wear resistance than without HIUST.

![Figure 5: The variation of weight loss for various loads at constant sliding speed (0.3 m/s) of investigated hypereutectic Mg-5wt%Si alloy without and with HIUST.](image)

![Figure 6: The variation of weight loss for various sliding speeds at constant load (10 N) of investigated hypereutectic Mg-5wt%Si alloy without and with HIUST.](image)

Figure 7 shows FESEM micrographs of the worn surfaces and their wear debris with their EDX analysis at different wear conditions of the investigated alloy without and with HIUST. It is found that the worn surfaces after wear test exhibit different morphologies. Traces of parallel grooves and ridges can be observed on the surface of the sample without HIUST (Figure 7a, g, m). Moreover, there are both delamination of large plateaus and also some cavities or craters on the worn surfaces of alloy at wear conditions of high load (50 N) (Figure 7g, j) or high sliding speed (0.9 m/s) (Figure 7m, p) as compared with that at wear conditions of low load (10N) and low sliding speed (0.3 m/s) (Figure 7a, d). Furthermore, the surface of sample without HIUST is rougher than that of HIUST-sample as shown in Figure 7a, d, g, j, m, p. The wear debris of sample without HIUST are large size as shown in Figure 7b, h, n which gives rise to a relatively high weight loss. The wear debris of HIUST-sample are smaller in size as shown in Figure 7e, k, q which gives rise to a relatively low weight loss. The size of wear debris in worn surfaces under wear conditions of high load (50 N) (Figure 7h, k) or high sliding speed (0.9 m/s) (Figure 7n, q) reveal numerous large flakes or sheets as compared with that of low load (10N) and low sliding speed (0.3 m/s) (Figure 7b, e) give rise to a relatively high weight loss with increasing applied load or sliding speed. For the samples without and with HIUST, analysis on the debris confirms the presence of Mg, Si, Fe and O as shown in Figure 7c, f, i, l, o, r.

4. Discussion

4.1. Material investigation

Under the present experimental conditions, the effect of the cooling rate on the modification of primary Mg2Si in hypereutectic Mg–5 wt.%Si could be neglected. Therefore, the difference in the morphology and size of primary Mg2Si were result almost exclusively from the difference in the application of HIUST. Usually, the microstructure of materials depends on the nucleation process and growth conditions. For the Mg2Si, its structure belongs to face centered cube (FCC) and its dendrite arm should grow along the preferential [100] crystallographic directions [12]. As a result, the morphologies of primary Mg2Si in the sample without HIUST are mainly characterized by dendrites with complex morphologies, as shown in Figures 3a and 4a. The refinement and modification of primary Mg2Si (Figures 3b, 4d) can be mainly attributed to HIUST during the solidification process. This can be explained as conjugation of two mechanisms: cavitation-enhanced heterogeneous nucleation and cavitation-induced dendrite fragmentation.
During the first stage of solidification between pouring temperatures and the liquidus temperature of the investigated alloy, HIUST can only be attributed to cavitation-enhanced heterogeneous nucleation. This because dendrite fragmentation will not be possible at those temperatures since solidification has not started yet. The cavitation-enhanced heterogeneous nucleation is further explained by three different mechanisms. The first is based on the pressure pulse-melting point ($T_m$) mechanism [12, 13], where the pressure pulse arising from the collapse of bubbles alters $T_m$ according to the Clausius–Clapeyron equation as $dT_m/dP = T_m(V_{L} - V_{S})/ΔH$, where $T_m$ is the freezing point in K, $P$ is the pressure in MPa, $V_L$ and $V_S$ are the specific volume of the liquid and the solid phase in cm$^3$/g, respectively, and $ΔH$ is the latent heat of freezing in J. An increase in $T_m$ is equivalent to increasing the undercooling so that an enhanced nucleation event is expected. The second is based on cavitation-enhanced wetting [8], which assumes that cavities and cracks on the substrate surfaces and insoluble non-metallic inclusions that either pre-exist in the melt or form on cooling during solidification, can be wetted by the melt under the pressure pulse from the collapse of the bubbles. Consequently, this enables these substrates to act as effective nucleation sites. The third mechanism [12, 13] assumes that rapid adiabatic expansion of gas inside the bubbles created during cavitation undercools the liquid at the bubble–liquid interfaces resulting in
The formation and removal of this layer determines the overall wear rate of the material [19]. At higher load, to higher temperature rise, gets oxidized. The oxidized layer may cover the surface and increase the weight loss. Numerous grooves and shallow scratches running parallel to the sliding direction generally characterize abrasive wear. In the present work, abrasive wear is favor at wear conditions of a low sliding speed (0.3 m/s) under the low applied load of 10 N. The presence of grooves and ridges on the surfaces of the sample should be homogeneous. Furthermore, the fine Mg2Si also reduces the extent of abrasion wear. Moreover, the alloy with HIUST has higher hardness than that without HIUST which leads to the highest wear resistance. As cited in [16], the wear law states that the materials with higher hardness will exhibit better resistance to wear. Thus, all of these lead to the lowest weight loss (Figure 5), the smooth surface (Figure 7d) and the small wear debris (Figure 7e) of the alloy with HIUST and therefore its high wear resistance. However, it can be found that with the application of wear conditions at higher loads or higher sliding speeds for the same process, the weight loss increases drastically (Figures 5, 6). This is obvious that at higher loads and/or sliding speeds, the soft Mg matrix smears off leading to higher wear in spite of the presence of hard Mg2Si particles [17]. However, the sample due to HIUST still has the best wear resistance.

The presence of Fe in wear debris of investigated alloy (Figure 7c, f, i, o, r) is due to the ploughing by hard primary Mg2Si on the rotating steel disc [15, 17]. Also, the presence of O2 was considered to have arisen in reactions with the environment, indicating that the mode of wear is mildly oxidative [15]. At very low load and/or sliding speed, an oxidative mechanism controls the wear process, generating debris comprising predominantly oxides. The oxidation was considered to be taken place due to the ability of the metal to oxidize under ambient conditions. On the other hand, material loss under abrasive wear conditions was removed by ploughing and micro-cutting. These mechanisms required penetration by hard abrasive particles which in turn are controlled by the hardness of material [15]. It has also reported [16] presence of abrasive wear in the low speed regime. Numerous grooves and shallow scratches running parallel to the sliding direction generally characterize abrasive wear. In the present work, abrasive wear is favor at wear conditions of a low sliding speed of 0.3 m/s under the low applied load of 10 N. The presence of grooves and ridges on the surfaces of the sample without HIUST (Figure 7a) characterize abrasive wear. Adhesion is a relevant wear mechanism in many lightweight alloys [18]. The adhesion wear mechanism appears due to the formation of micro-joints between the pin and the disc. As a consequence of their relative movement, the softer material breaks, leaving a small void in the Mg alloy and transferring some material to the steel disc. Figure 7a, d present some evidence of the existence of adhesion wear mechanism in the worn surface of investigated alloy. Therefore, it can be concluded that these as–cast alloys had experienced a mild abrasive oxidative wear with little adhesion.

However, examinations of the wear debris due to the applied high load (50 N) (Figure 7h, k) at low sliding speed (0.3 m/s) and/or the applied high sliding speed (0.9 m/s) (Figure 7n, q) at low load (10 N) reveal numerous flakes or sheets linking to the process of delamination. Since, at relatively slower speed 0.3m/s, if the load increases considerably (50 N), mixing of wear debris and counterface material could take place which due to higher temperature rise, gets oxidized. The oxidized layer may cover the surface and increase the weight loss. The formation and removal of this layer determines the overall wear rate of the material [19]. At higher load,
removal the oxide film through sliding become greater that its rate of formation, and a transition to metallic wear occurred that caused delaminating in the bulk material and increased the wear rate [17, 19]. This is termed as delamination mechanism. Delamination is observed more extensively under the high loads [17]. Furthermore, at higher sliding speed (0.9 m/s), relatively at lower applied load of 10 N, delamination mechanism is also prevailing. This may be due to more adiabatic type of heating and cause more adhesive action between the two surfaces. At a critical applied load and sliding velocity, temperature rises so high and because of severe degree of delamination, mixed layer becomes discontinuous and naked material comes in contact with counter surface. This also leads to a rise in surface temperature to a critical value i.e. flashing temperature at which strong adhesion between counter surface and specimen takes place.

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