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Combined effect of high-intensity ultrasonic treatment and Ca addition on modification of primary Mg_2Si and wear resistance in hypereutectic Mg–Si alloys

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ABSTRACT

The combined effect of high-intensity ultrasonic treatment (HIUST) and 0.3 wt.%Ca addition on modification of primary Mg_2Si and wear resistance in the hypereutectic Mg–5 wt.%Si alloy has been investigated. The results show that without treatment, the dendrites of primary Mg_2Si are coarse and non-uniform in size. With HIUST or 0.3 wt.%Ca addition, nearly fine uniform and polyhedral shape of primary Mg_2Si are achieved. Interestingly, modification and refinement of primary Mg_2Si arise from the combined effect of HIUST and 0.3 wt.%Ca addition. The sample due to combined effect of HIUST and 0.3 wt.%Ca addition has the best wear resistance due to the lowest weight loss among all the other samples for various loads (10, 30 and 50 N) at constant sliding speed (0.3 m/s). Modification mechanisms resulting in the development of microstructures are also investigated.

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 has become a critical issue for possible application of magnesium alloys in hot components [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 Mg_2Si [2,3]. However, the hypereutectic Mg–Si alloys prepared by ordinary ingot metallurgy process showed a very low ductility and strength, due to the presence of coarse primary Mg_2Si and the brittle eutectic Mg_2Si [2–4].

Ca could modify and refine both the primary and eutectic Mg_2Si in Mg–6Zn–4Si alloy [4] as well as eutectic Mg_2Si in Mg–5Al–1Zn–1Si alloy [5]. Recent research [2] has shown that the primary Mg_2Si could be effectively refined and modified by the application of HIUST on the hypereutectic Mg–5 wt.%Si alloy during solidification process. However, combined effect of HIUST and Ca addition on modification of primary Mg_2Si and wear resistance in hypereutectic Mg–Si alloys has not been reported. Therefore, the main aims of this work are to investigate the effect of HIUST on modification of primary Mg_2Si in Mg–5 wt.%Si hypereutectic alloy in combination with addition of 0.3 wt.%Ca, to explore the modification mechanisms and to investigate the wear resistant of resultant samples.

2. Experimental procedures

2.1. Materials and processing

The hypereutectic Mg–5 wt.%Si alloy was melted in a mild steel crucible in an electric resistance furnace under protective gases mixture of tetrafluoroethane (CF_3CH_2F, HFC-134a, 1 vol.%) and carbon dioxide (CO_2, Bal.). Nominal amount of 0.3 wt.%Ca in the form of high purity Ca (99.999 wt.%) was added into the melted alloy at about 800 °C. The melt was manually stirred for 3 min and then was held for additional 15 min in order to get a full homogenization. After that the slag was removed, and then the melt was poured at about 800 °C into a cylindrical resin-bonded sand mold with dimensions of outer diameter (⌀100 mm), inner diameter (⌀42 mm) and length (250 mm) which mounted on the ultrasonic sonotrode of diameter (⌀40 mm) as shown in Fig. 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.

The solidification characteristic of the prepared Mg–5 wt.%Si and Mg–5 wt.%Si–0.3 wt.%Ca alloys was confirmed using thermal analysis. The thermal analysis test sample was obtained by pouring the quantity of the melt at about 830 °C into standard Qiuk–Cup resin-bonded sand cup with dimensions described in Fig. 2. A high sensitivity type K thermocouple (chromel–alumel) located vertically at the center of the cup, facilitated the capturing of the temperature during solidification. The data for thermal analysis were collected using a data logger and transferred to a personal computer for analysis. Thermal analysis trial was repeated three times to ensure reproducibility and the accuracy of the results.

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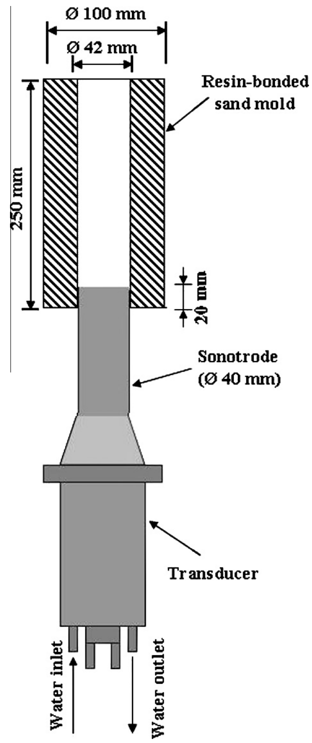

Fig. 1. Schematic of the experimental setup used in this study.

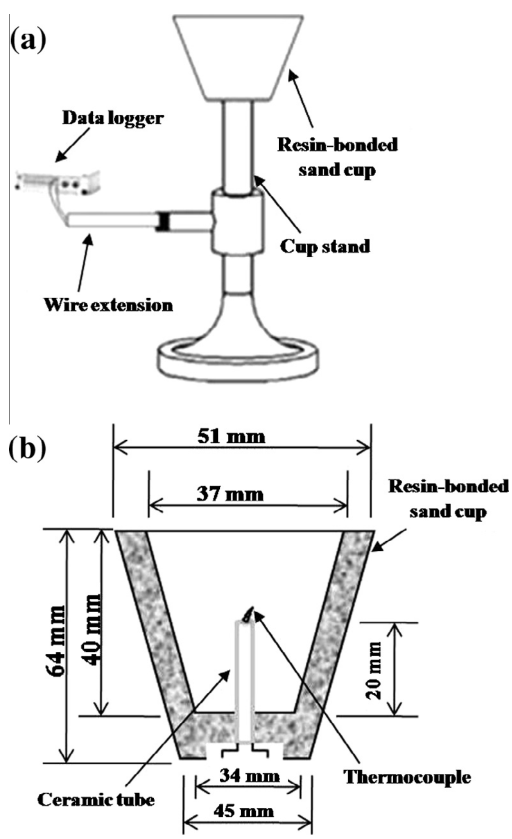

Fig. 2. Schematic of (a) experimental setup for thermal analysis and (b) resin-bonded sand cup with type K thermocouple.

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 [2]. 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, a sample without the application of any treatment, a sample treated only by HIUST for 90 s and a sample treated only with 0.3 wt.%Ca addition were prepared at pouring temperature of 800 °C. The chemical composition of the prepared alloys was measured with X-ray fluorescence analyzer (XRF) (model Axios advanced-PANALYTCAL, The Netherlands) as shown in Table 1.

2.2. Materials characterization

All metallographic specimens were cut at the same position of 10 mm from the bottom of castings. The samples were grinded, polished and etched by solution of 10 ml nitric acid, 30 ml acetic acid, 40 ml water, and 120 ml ethanol for 2–3 min, then analyzed by optical microscope (OM) (model OPTIKA M-790, Italy) and the field-emission scanning electron microscope (FESEM) (model Quanta FEG, The Netherland) equipped with an energy dispersion spectrum (EDS). Six OM micrographs were taken for each sample. The average size of the primary Mg_2Si was measured by ImageJ1.44 software. 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 2θ from 20° to 80° with an increment of 0.02° and a scanning speed of 4°/min.

Dry sliding wear tests without lubricant were 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 7 mm diameter and 12 mm length machined out from the castings were used as test samples. Hardened ball bearing steel disc (HRC 63) of 73 mm outer diameter, 65 mm inner diameter and 25 mm thickness 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. The wear tests were carried out under ambient temperature with three different normal loads (10, 30 and 50 N) at a constant sliding speed of 0.3 m/s for 10 min. The weights of the samples 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.

3. Results and discussion

According to the Mg–Si binary phase diagram [6], Mg–5 wt.%Si alloy is a typical hypereutectic alloy. From the thermal analysis data, the cooling curves with their first derivative curves of the prepared Mg–5 wt.%Si and Mg–5 wt.%Si–0.3 wt.%Ca alloys were plotted as shown in Fig. 3. It is found from Fig. 3a that the primary Mg_2Si began to precipitate from the prepared Mg–5 wt.%Si alloy melt at about 767 °C as refereed to liquidus temperature (T_L). Then, along with the temperature decrease, the eutectic reaction (T_E) occurs at about 633 °C. After adding 0.3 wt.%Ca, the liquidus temperature (T_L) becomes about 756 °C and eutectic temperature (T_E) becomes about 632 °C as shown in Fig. 3b. Therefore, addition of Ca has very obvious effect on decreasing the liquidus temperature of investigated Mg–5 wt.%Si hypereutectic alloy but slight effect on eutectic temperature of investigated alloy.

XRD results reveal that the constituents of the obtained microstructures without and with different treatments are only Mg_2Si and Mg phases, as shown in Fig. 4. Therefore, no change of the phase constituents obtained due to different treatments. The absence of the new phase in alloy with the additions of 0.3 wt.%Ca is presumably ascribed to the relatively small amount of Ca.

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

Prepared alloy	Mg	Si	Ca	Fe	Cu
Mg–5Si	Bal.	5.02	–	0.033	0.041
Mg–5Si–0.3Ca	Bal.	5.059	0.342	0.031	0.043