Journal of Materials Processing Technology 214 (2014) 551–555

Contents lists available at ScienceDirect

Journal of Materials Processing Technology

journal homepage: www.elsevier.com/locate/jmatprotec

Distribution of TiB_2 reinforcements in magnesium matrix composites by a multi-physical coupling field

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ARTICLE INFO

Article history:
Received 24 June 2013
Received in revised form 23 October 2013
Accepted 25 October 2013
Available online 4 November 2013

Keywords:
Microstructure
Composite materials
Multi-physical coupling field
Self-propagating high-temperature synthesis (SHS)
TiB_2

ABSTRACT

TiB_2/AZ31 magnesium matrix composites were prepared under the separate effects of an electromagnetic field, ultrasound and of both in combination. The electromagnetic field appeared to expand the zone of ultrasonic action, resulting in fine grained and more uniform microstructure with a more homogeneous distribution of the reinforcing TiB_2 particle clusters in the magnesium alloy matrix. Subsequent hot-rolling further improved the microstructural homogeneity. The resulting TiB_2/AZ31 composite sheets exhibited excellent overall mechanical properties, with an ultimate tensile strength of 350 MPa and a tensile ductility approaching 8%.

1. Introduction

In particle reinforced magnesium matrix composite materials, the reduced particle size is believed to be one of the predominant factors that lead to greatly improved material performances (Rahimian et al., 2010), while the uniform distribution of such fine particles within the matrix has long been a challenge (Hashim et al., 2002). As pointed by Wang et al. (2010), fine particles are easily agglomerated into large particle clusters, driven by the large excess surface free energy in the fine particles. As stressed by Nie et al. (2012), when the volume fraction of the reinforcing particles is considerable, the large particle clusters would cause stress concentration and the mechanical performances of the composites get deteriorated. In the previous study by Liu et al. (2012), high-intensity ultrasonic treatment has demonstrated an effective way of breaking the large particle clusters, while the range of the ultrasonic action is limited, i.e. the ultrasonic effects are localized around the probe region and a global homogeneity throughout the entire volume of the composite material is far from reached. In this present work, we introduce a so-called multi-physical coupling field method to resolve this problem. The effects of the multi-physical coupling field on breaking and dispersing particle clusters are shown in a TiB_2/AZ31 composite, in which the applied power of ultrasonic intensity is as low as 330 W. The effects of hot-rolling processing on the dispersion of particle clusters are also investigated, along with the account of the improved mechanical properties of the TiB_2/AZ31 composite.

2. Experimental procedures

2.1. Preparation of in situ TiB_2/AZ31 composite

The material used in the present study is an in situ TiB_2/AZ31 magnesium matrix composite. The Al-Ti-B preform was made with the commercial powders of aluminum, titanium and boracium in a weight ratio (wt.%) of 50:34:16, in which the atomic ratio of titanium and boracium approximates to the stoichiometry of TiB_2. The mean diameters of aluminum, titanium and boracium powders were 29, 30 and 2 μm, respectively. Commercially pure magnesium (ingot, >99.8 wt.%), aluminum (ingot, >99.8 wt.%) and zinc (ingot, >99.9 wt.%) were used to prepare the AZ31 magnesium matrix. The AZ31 magnesium alloy was first melt in a steel crucible covered by an anti-oxidizing flux. At 1073 K, the preform was added into the AZ31 alloy melt and the addition amount is about 3 wt.% of the AZ31 melt. Following the method of Wang et al. (2004), micron and submicron TiB_2 particles were in situ synthesized via the self-propagating high-temperature synthesis reaction (SHS) in molten magnesium. Conventional mechanical stirring was carried out within the semi-solid temperature interval for about 20 min to preliminarily break up the SHS products. Subsequently, the composite alloy melt was reheated to about 923 K. While the alloy melt is stabilized at this temperature, a helical

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http://dx.doi.org/10.1016/j.jmatprotec.2013.10.018

electromagnetic, a low-power ultrasonic field and the coupling field of these two were applied to modify particle dispersion in the composite melt, respectively. The current and frequency of the helical electromagnetic field were 150 A and 10 Hz, respectively, and the power and frequency of the ultrasonic field were 330 W and 20 kHz, respectively. In particular, after having been ultrasonically processed for different times, a portion of high-temperature composite melt close to ultrasonic probe bottom was extracted using a quartz tube (7 mm diameter) and then water-cooled. These samples thus obtained are named as as-extracted samples. Finally, the remaining composite melt was reheated up to 953 K and cast into a steel mold. For comparison, a AZ31 magnesium alloy and a TiB_2/AZ31 composite were also made by means of conventional casting without any external physical field. The ingot samples for hot-rolling were solution-treated at 688 K for 20 h. Before hot-rolling, the samples were annealed at 653 K for 1 h for temperature homogenization. Cross-rolling was conducted at 653 K to reach a total plastic deformation of 70% in the samples.

2.2. Instrumentation

The microstructures and fracture surfaces were observed using a MEF-3 optical microscope (OM) and a JSM-5600LV scanning electron microscope (SEM). In order to make the matrix grains and the TiB_2 particles to be clearly distinguishable, the samples for OM observation were annealed at 688 K for 20 h. Room temperature tensile test was carried out on a WD-10 electronic universal tester with a strain rate of $1 \times 10^{-3}\ s^{-1}$.

3. Results and discussion

3.1. Distribution of TiB_2 particles in composite melts under ultrasonic field

Fig. 1 shows the optical micrographs of as-extracted TiB_2/AZ31 magnesium matrix composites undergone different ultrasonic processing times. Assuming that rapid quenching can well preserve the microstructure of a composite melt, these as-extracted samples can be used to evaluate the distribution of TiB_2 particles in the composites melt. It is seen that TiB_2 particles were mainly distributed along grain boundaries in the case that only the mechanical stirring is applied (Fig. 1a). The agglomerated TiB_2 particles were rejected by the quick-growing liquid-solid interface, and precipitated along grain boundaries. As shown in Fig. 1b, the size of TiB_2 particle clusters evidently reduces in the sample treated by ultrasonic field for 15 min. This indicates that a low-power ultrasonic field (~330 W) is capable of breaking particle clusters. With increasing ultrasonic processing time, the size of particle clusters further decreases and TiB_2 particles are distributed uniformly in the matrix (Fig. 1c). Liu et al. (2012) have attributed the cluster size reduction effect to the alternating shear stress created by nonlinear effects of high-power ultrasonic field, such as cavitation and acoustic streaming. Cao et al. (2008) stated that the strong impact coupling with high local temperatures on particle clusters is the main reason for size reduction. A small amount of clusters around 15 μm size appear after 35 min ultrasonic treatment (Fig. 1d), which implies that prolonged processing enhances the chance of collision and agglomeration among particles, and the process of forming large particle clusters takes over. The competition between breaking and re-agglomeration results in an optimum ultrasonic treatment duration of 25 min. The comparison of the microstructure in Fig. 1a with those in Figs. 1b, c and d reveals that the amount of TiB_2 particles is much smaller in the samples treated by ultrasonic field than in the untreated. It is very likely that a large portion of TiB_2 particles have sank to the bottom of the crucible due to the fairly localized stirring effect of acoustic streaming and the density difference between the molten AZ31 metal and TiB_2 particles.

3.2. Distribution of TiB_2 particles in composite ingots under various external physical fields

The AZ31 and TiB_2/AZ31 melts with and without various physical field treatments have been prepared for a comparative study. The AZ31 reference alloy is composed of a primary α-Mg phase and a secondary phase β-$Mg_{17}Al_{12}$. The β-$Mg_{17}Al_{12}$ particles are mainly formed along the grain boundaries of the α-phase. After annealing at 688 K for 20 h, the secondary phase dissolved almost completely into the matrix (Fig. 2a). The mean grain size of α-Mg is about 195 μm.

The microstructure of TiB_2/AZ31 composite prepared by conventional casting is shown in Fig. 2b. Particle clusters composed of TiB_2 and Al_3Ti particles are seen in the matrix. The great detail of particle distribution is shown by the SEM image in Fig. 2c. In our experiment, it was found that a large portion of in situ formed particles deposited on the bottom of the crucible during melting. The presence of TiB_2 particles is scarce in the interior of grains, and most of the residual TiB_2 particles agglomerated into large clusters and located at grain boundaries. Comparing with the AZ31 alloy (Fig. 2a), the microstructure of the composite alloy is refined to a mean grain size of 130 μm. Though individual TiB_2 particles can act as heterogeneous sites of the α-phase (Wang et al., 2006), the microstructure refinement of TiB_2/AZ31 composite does not seem to follow the heterogeneous nucleation mechanism. The products of SHS reaction mainly consists of Al, TiB_2 and Al_3Ti (Wang et al., 2004). The Al solute coming from the SHS reaction facilitates grain refinement because of its relatively larger growth restriction factor (GRF) (Lee et al., 2000).

The ultrasonic treatment effect on microstructure is shown in Fig. 2d. TiB_2 particles are seen to uniformly distributed in the matrix, which is similar to that of the as-extracted sample (Fig. 1c). The TiB_2 particles are mainly distributed in grain interiors. The volume fraction of the reinforcing particles is small, which is indicative of a low efficient of in situ particle utilization. This phenomenon implies that individual particles or very fine particle clusters are readily engulfed by liquid-solid interface. It should be noted that in the present case the ultrasonic field did not result in a significant grain size refinement of the Mg matrix. Moreover, the microstructure is rather inhomogeneous. The introduced ultrasonic energy would heat the melt and reduce the solidification rate and, hence lead to coarsening. Noticing the large GRF of Al, another reason for coarsening may be attributed to the consumption of a large portion of Al by the precipitation of β-$Mg_{17}Al_{12}$.

The introduction of electromagnetic field can provide contactless stirring in the composite melt, which has resulted in a much more refined grain size (~91 μm) and homogeneous microstructure (Fig. 2e). A couple of mechanisms should be involved in the microstructure refinement consideration, for instances, the blocking of particles and their clusters on grain growth, the heterogeneous nucleation of some TiB_2 particles, and the large GRF of Al, and so forth. Some large particle clusters are observed in the samples, which suggests that the electromagnetic field alone is not very effective in breaking particle clusters. Due to electromagnetic stirring, most of the in situ formed TiB_2 particles are preserved in the flowing melt, and thus serves as the reinforcement phase in the final composites.

Qian et al. (2009) have found that the significant effects induced by ultrasonic treatment occurred exclusively below the radiating face of the probe. In addition to that, the weak acoustic streaming effect only produce stirring phenomenon in a small area. Notice the results obtained from the independent use of the ultrasonic and electromagnetic field, it is expected that particle distribution