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In-situ synthesis of AlN particles in Mg-Al alloy by Mg_3N_2 addition

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Abstract

This communication reports an experimental study on the in-situ formation of hard AlN particles in an Mg-10wt.%Al alloy at 700°C by externally adding Mg_3N_2 powders.

Keywords: Magnesium, Composite Materials, Mg_3N_2 , AlN, In-situ, Casting

Introduction

Magnesium alloys are attractive engineering materials because of their low density, along with other beneficial properties such as good damping capacity, excellent castability, and superior machinability. However, compared with other metallic materials, magnesium alloys have a lower strength, especially at elevated temperatures. Magnesium matrix composites reinforced with various hard particles have been widely studied to further improve the mechanical properties of magnesium alloys. Although these composites are most commonly prepared by stir casting, squeeze casting, or powder metallurgy [1-3], the more recently developed in-situ synthesis process is considered an attractive alternative to the existing processes. The attractiveness of this alternative lies in that the fine particles produced are more compatible with the matrix and their distribution throughout the matrix is more uniform. The method has been used for the fabrication of aluminum matrix composites [4-7]. For example, AlN particles were formed in-situ successfully in aluminum alloys by the reaction of

aluminum with Mg_3N_2 [7]. The formation of AlN in this system is favored by the high concentration of Al in the molten system, and was assisted by the existence of magnesium [7]. Even though reported results indicated the possibility of in-situ formation of AlN particles in the Mg-Al alloys as well, this possibility has not yet been substantiated by experimental research. This communication presents the results of a recent investigation on in-situ forming hard AlN particles in a magnesium alloy by addition of Mg_3N_2 into molten magnesium during the melting process.

Experimental procedure

An AM60B magnesium alloy (Al 5.6-6.4wt.%, Mn 0.26-0.5wt.%, Zn<0.2wt.%, Si<0.05wt%, Cu<0.008wt.%, Ni<0.001wt.%, Fe<0.004wt.%, Mg balance) was employed as the base material. Additional aluminum was added so that the total aluminum content in the melt was increased to 10 wt.%. 10 vol.% of Mg_3N_2 powder supplied by Alfa Aesar was added to the alloy. The powder had a cubic lattice structure and a mesh size of -325 (<44 μm). Melting was conducted in a vacuum induction furnace using a steel crucible. The experimental set up is schematically shown in Fig.1. After melting the AM60B alloy together with an Al block, the steel tubing for gas and thermocouple was lowered, followed by the bubbling of Ar gas (industrial purity) into the melt at a pressure of 15 psi for 1 minute at about 700°C. The molten alloy was then poured into a steel crucible and the solidified ingot was subsequently cut into specimens for microstructural analysis.

The microstructure of the samples was investigated using a Hitachi S-3500 scanning electron microscope (SEM) with an energy dispersive spectrometer (EDS) attachment and Philips

Xpert X-ray diffractometer. The microhardness was measured using a Buehler Micromet II hardness tester.

Results and Discussion

Figure 2 shows a SEM micrograph of the as cast specimen. A significant amount of fine bright particles were uniformly distributed in the matrix, with a large amount of coarse particles (labeled as *A*) existing in a relatively segregated manner. The large type *A* particles are of irregular shape with sizes of up to about 100 μm .

An EDS spectrum of a type *A* particle and the distribution of element Mg, Al, and N around the analyzed particle in the sample are demonstrated in Fig. 3. The EDS spectrum shows mainly Al, Mg, and N peaks. The average composition of the type *A* particles was 18.6-wt.% N, 23.7-wt.% Mg, and 57.7-wt.% Al. The matrix had an average composition of 92.8-wt.% Mg and 6.5-wt.% Al, with minor amount of other alloying elements.

In order to characterize the composition and microstructure of the particles formed, the particles were extracted from the samples by chemically dissolving the matrix in an ethanol solution of 10-vol.% acetic acid. The extracted particles were then subjected to SEM and X-ray diffraction examinations. The SEM images of the raw Mg_3N_2 powders and the extracted particles are shown in Fig. 4 and Fig. 5, respectively. The composition of the extracted particles obtained by EDS analysis was 28.7- wt.% N, 67.9-wt.% Al, and 3.4-wt.% Mg, corresponding to an atomic ratio of 44-at.% N, 53-at.% Al, and 3-at.% Mg. The XRD diffraction pattern of the extracted particles is shown in Fig. 6. These results clearly revealed that the extracted particles were predominantly AlN. It should be noted that the deviation of

the atomic ratio of Al/N from stoichiometry could be caused by a number of factors, such as the analysis error of EDS for light elements and/or a deficiency of nitrogen in the AlN lattice. On the other hand, the small amount of magnesium detected in the extracted particles is believed to be from residues in element or other compound forms, since magnesium is not known to replace Al in the AlN lattice.

The experimental results indicate that 10 wt.% Al in the magnesium alloy melt is sufficient to form AlN through the reaction with externally added Mg_3N_2 powder. Mg_3N_2 , AlN, and Mg have high coherency between their lattice structures. The Mg_3N_2 has a cubic structure with a lattice parameter a being 0.99657 nm [8]. AlN is characterized of a hexagonal structure with lattice parameters of 0.3113 nm and 0.4981 nm for a and c , respectively [9]. Mg has a hexagonal structure and the corresponding lattice parameters are 0.3209 nm and 0.5211 nm for a and c , respectively [10]. The lattice discrepancies between Mg_3N_2 and AlN phases are 6.7% in the $\langle 1210 \rangle \{0001\}_{\text{AlN}} // \langle 001 \rangle \{100\}_{\text{Mg}_3\text{N}_2}$ orientation and 0.03% along the $\langle 0001 \rangle \{1010\}_{\text{AlN}} // \langle 001 \rangle \{100\}_{\text{Mg}_3\text{N}_2}$ orientation. Those between AlN and Mg along these two directions are 3.7% and 4.6% respectively. These discrepancy numbers, except for one, are smaller than the coherency criterion of 5%, indicating that Mg_3N_2 and/or Mg may serve as an effective nucleation site for the formation of AlN.

The results of microhardness measurements are demonstrated in Fig. 7. The average Vickers hardness of the matrix was 59, while that of AlN was 623, over 10 times that of the matrix. This high hardness also shows the high concentration of AlN in the extracted particles, and is a desirable property for reinforcing the matrix.

Summary

AlN particles were successfully produced, in situ, in a magnesium alloy containing 10 wt.% aluminum by adding Mg_3N_2 powders into the melt. The in-situ reaction occurred readily at 700 °C. However, the AlN particles produced were relatively large and unevenly distributed. Further optimization of the process parameters is required for uniform distribution and refining of the particles.

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Figure caption

Fig. 1. The setup of melting process.

Fig. 2. The SEM microstructure of the sample.

Fig. 3. EDS spectrum and mappings of element distribution in the sample.

Fig. 4 . The SEM image of the parent Mg_3N_2 powders.

Fig. 5. The SEM image of the extracted particles.

Fig.6. The (Cu-K α) XRD pattern of the extracted particles.

Fig. 7. The microhardness of matrix and particles in the samples.

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