Materials Science and Engineering A 413–414 (2005) 408–411

MATERIALS SCIENCE & ENGINEERING A

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Grain-refining mechanisms of superheat-treatment of and carbon addition to Mg–Al–Zn alloys

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Received in revised form 30 June 2005

Abstract

Experiments were performed to clarify the grain-refining mechanisms of Mg–Al–Zn alloys by superheat treatment and addition of a new grain refiner. Numerous Al_4C_3 nucleants were formed in the commercial AZ91E alloy by superheat treatment. Carbon is an important element for grain refining of commercial AZ91E alloy instead of harmful C_2Cl_6. Numerous Al_4C_3 nucleants were also formed in the molten alloy.

Keywords: Magnesium alloy; Casting; Grain refinement; Superheat treatment; Solidification; Nucleation

1. Introduction

Grain refining of cast magnesium alloys containing Al is achieved by superheating or by adding carbon materials. Both techniques are well established, but the grain refining mechanisms have not been clarified yet. However, some theories have been proposed. One is heterogeneous nucleation due to Al–Mn or Al–Mn–Fe compounds that are formed during superheat treatment [1,2]. Another is the formation of Al_4C_3 in the molten magnesium by addition of carbon [3]. Two sets of experiments were conducted. The first set was to clarify the grain refinement by superheat treatment. The second one was performed to study the grain refining effect of adding carbon powder.

2. Experimental procedures

2.1. Preparation of crucible and superheat treatment of molten alloy

A crucible for melting the alloy was made from Fe to 18% Cr stainless steel that has corrosion resistance at elevated temperatures. It was 61 mm in diameter, 120 mm in height and 2 mm in wall thickness. The crucible was also lined with pure aluminum by dipping it into a molten aluminum bath. It was then lined inside with magnesia to prevent interaction with impurities from the crucible. An AZ91E alloy was used in the experiment. Table 1 lists the chemical composition of the AZ91E magnesium alloy. The molten alloy was superheat-treated at 1123 K for 900 s. It was then cooled at 2.5 K/s and poured at the desired temperature for obtaining quenched samples. To prevent the molten alloy from burning during heating and pouring into the mold, a mixture of SF_6 and CO_2 gas was used.

2.2. Experiments for quenched samples during pouring

The alloy was melted and superheated at 1123 K for 900 s then cooled at 2.5 K/s. The casting temperatures were 1023, 973, 923 and 873 K. During pouring, the molten alloy was quenched by using two chilled copper blocks.

2.3. Addition of carbon powder as new grain refiner

High-purity carbon powder was added to the molten alloy by using a magnesium capsule. Five micrometers size carbon powder was charged into the magnesium capsule. The optimum additive mass% for grain refinement and examination were 0, 0.02, 0.04, 0.06, 0.08 and 0.10%. The additive temperature for grain refinement was also examined. In this experiment, carbon powder was added at 973, 993, 1013, 1023 and 1073 K for 600 s.

2.4. Microscopic observation and EPMA analysis

Samples obtained were observed by optical microscopy, and analysis was performed by EPMA. Grain sizes of

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0921-5093/$ – see front matter
doi:10.1016/j.msea.2005.08.214

Table 1
Chemical composition of AZ91E magnesium alloy (mass%)

Al	9.01
Zn	0.82
Mn	0.22
Si	0.01
Cu	0.001
Ni	0.0002
Fe	0.0017
Mg	Balanced

the samples that were cast between the chill block were measured.

3. Results and discussion

3.1. Structures of quenched samples with and without superheat treatment

Fig. 1 depicts the microstructures of superheat-treated and untreated AZ91E alloy samples obtained by quenching from 873 K. The superheat-treated samples reveal that foreign substances existed in the center of each dendrite crystal as shown in Fig. 2. In contrast, foreign substances existing in non-superheat treated samples were independent of the dendrite growth. The foreign substances consisted of Al, C, and O or Al, C, Mn, Si, and O. C, Mn, and Si were impurities in the AZ91E alloy. An Al_4C_3 or Al_2CO compound probably formed during superheat treatment. However, O in the Al_2CO might have been introduced by polishing the sample because water was used for polishing and the following chemical reaction could have occurred.

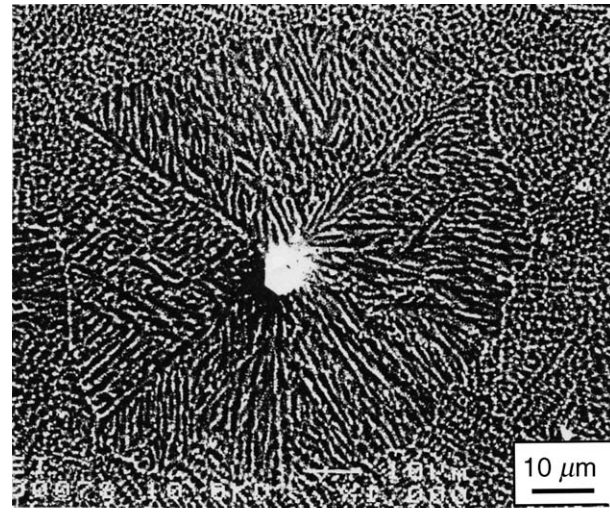

Fig. 2. Al_4C_3 compounds appeared by quenching from 1023 K.

$$Al_4C_3 + 12H_2O = 3CH_4 + 4Al(OH)_3$$

Samples quenched from 873, 923, and 973 K revealed that Al, Mn, C, O and Si elements coexisted. Mn-free and Si-free Al–C–O compounds appeared only in the sample from 1073 K. Al, Mn, and Si were probably solute in the molten magnesium during superheat-treating. Mn and Si may have crystallized on the Al_4C_3 compounds or only on Mn compounds formed below 973 K. In contrast, compounds consisting of Al, Mn, and Si also existed in non superheat-treated alloy, but they did not cause the magnesium dendrite crystals to nucleate.

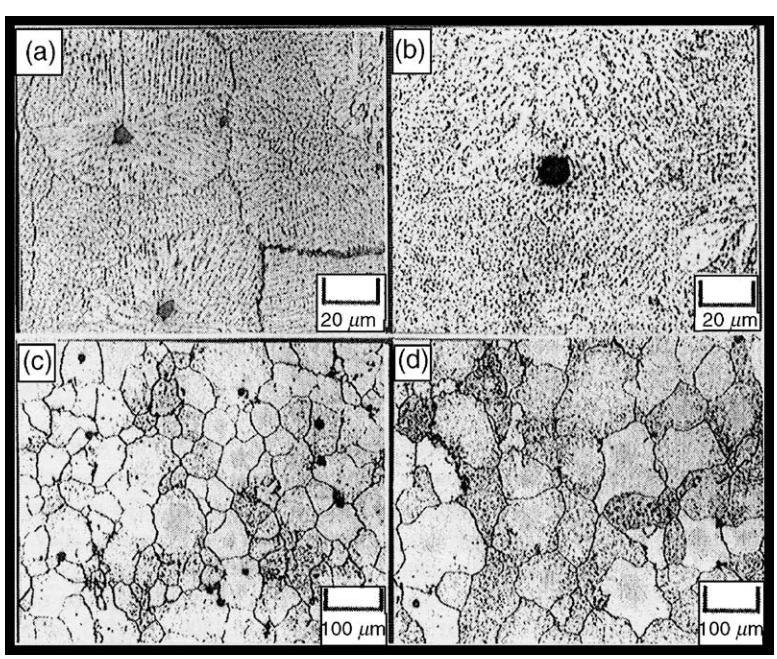

Fig. 1. Mn compound appeared by quenching or casting from 873 K. (a and b) Quenched sample, (c and d) cast sample, (a and c) superheat treated, (b and d) non-superheat treated.