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Grain Refinement of AZ31 and AZ91 Mg Alloys by Nucleation Ultrasonic Melt Treatment

Jeong IL Youn[1‡], Kee Joo Jung[1], Young Ki Lee[1], Bong Jae Choi[1], and Young Jig Kim[1]
([1] School of Advanced Materials Science and Engineering, Sungkyunkwan Univ. Korea)

1. Introduction

Magnesium is the lightest metal of structural metals, widely used in various field for its excellent properties such as specific strength, castability, electromagnetic shielding, and machinability. Especially, grain refinement in magnesium casting products is required to improve the mechanical properties and homogeneity, also to reduce the segregation and casting defects. Grain refinement method is generally divided into chemical and physical process. The chemical process is mainly depends on the selection of nucleating agent which must have the versatile properties to apply the various casting processes. However, it is very difficult to find the suitable nucleants, and its effectiveness is limited. The physical process is very simple and it only has been focused the break of dendrites. Ultrasound has also been studied for the grain refinement based on these mechanisms. The first is cavitation-enhanced nucleation, and the other is cavitation-induced dendrite-fragmentation. This suggests that the fragmented dendrites by physical destruction can be act as a nucleant. However, the application of the ultrasound for the grain refinement couldn't be an effective process because of the limited using condition that have to be solid and liquid co-existence state of alloy. In this study, the progressive process named NUMT (Nucleation Ultrasonic Melt Treatment) for grain refinement by ultrasound is suggested to overcome these problems. NUMT is based on the mechanism on grain refinement hypothesis, Solute paradigm, which proposes the role of solutes on grain refinement. The sonotrode made by the special metals which are able to make grain refine is eroded by the cavitation effect in melt very uniformly. In this study, very popular magnesium alloys, AZ91 and AZ31 were selected for the grain refinement by NUMT, and the important experimental parameters were established for the process.

2. Experimental procedure

AZ31and AZ91 alloy ingots were melted in an electric furnace using a mild steel crucible having a diameter of 40 mm under a protective cover gas

younj1@skku.edu

Table Ⅰ. Solute parameters of various alloying elements in Mg (m: slope of the liquidus line, k: equilibrium distribution coefficient, m(k-1): growth restriction factor)

Element	m	k	m(k-1)	system
Ti	500	120	5.95×10^4	peritectic
Fe	-55.56	0.054	52.56	eutectic
Zr	6.9	6.55	38.29	peritectic
Ca	-12.67	0.06	11.94	eutectic
Si	-9.25	~0.00	9.25	eutectic
Ni	-6.13	~0.00	6.13	eutectic
Al	-6.87	0.37	4.32	eutectic

mixture of SF6 and air to protect it from the atmosphere and prevent its urgent oxidation. The about 530 g alloy ingot was melted at 700 ^{o}C, the sonotrode of 40 mm diameter was pre-heated for 5 minutes at just above the melt before dipping it into the melt. And the sonotrode was dipped into melt in the depth of 20 mm for 5 minutes to maintain the melt temperature constantly before NUMT. The 20 kHz ultrasonic system was used and NUMT was carried out for 5 minutes in the furnace sustained at 700 ^{o}C. The melt after NUMT was poured into the steel permanent mold (120 × 130 × 12 mm) preheated to 100 ^{o}C. Samples were sectioned at the corner of the bottom place, and its cross section area was polished. The macro structure was observed after etched by a reagent (Picric acid 0.42 g + Acetic acid 1 ml + Ethanol 8 ml + water 1 ml). Optical Microscope and Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) are used to evaluate the grain size and the contents of sonotrode material.

3. Results and discussions

In this study, Ti and Fe were selected as a sonotrode material to induce the grain refinement by NUMT process based on GRF (Growth restriction factor) listed in Table Ⅰ. GRF means the capability to restrain the growth of grain, and promote the constitutional undercooling for grain refinement. The most powerful elements on m(k-1) value

Table Ⅱ. Grain size with Ti and Fe contents in various Mg alloys after NUMT

Alloy Type	Sonotrode Type	Ti content	Fe content	GRF(Ti)	GRF(Fe)	Total GRF	Grain Size
Pure Mg	No Injection	<1 ppm	10 ppm	5.95	0.05	6	>2000 μm
	Ti sonotrode	10 ppm	10 ppm	59.50	0.05	60	>2000 μm
	Fe sonotrode	<1 ppm	20 ppm	5.95	0.11	6	>2000 μm
Mg-3Al	No Injection	< 1 ppm	30 ppm	5.95	0.16	19	140 μm
	Ti sonotrode	30 ppm	30 ppm	178.5	0.16	192	100 μm
	Fe sonotrode	< 1 ppm	50 ppm	5.95	0.26	19	100 μm
Mg-1Zn	No Injection	< 1 ppm	40 ppm	5.95	0.21	10	510 μm
	Ti sonotrode	40 ppm	40 ppm	238	0.21	242	150 μm
	Fe sonotrode	< 1 ppm	60 ppm	5.95	0.32	10	100 μm
AZ31	No Injection	10 ppm	20 ppm	59.50	0.11	76	470 μm
	Ti sonotrode	20 ppm	20 ppm	119	0.11	136	120 μm
	Fe sonotrode	10 ppm	30 ppm	59.50	0.16	76	280 μm

converted directly to GRF, Ti and Fe shown in the table were made as the sonotrode. However, because Ti and Fe have very low solid solubility in Mg, it is difficult to alloy these elements uniformly. Thus, it was supposed that ultrasound injection by the sonotrode made by these metals into melt could be one way of achieving this goal. The macrostructure of AZ31 and AZ91 alloy with and without NUMT are shown in Fig. 2. The effect of NUMT on grain refinement was very clear and effective.

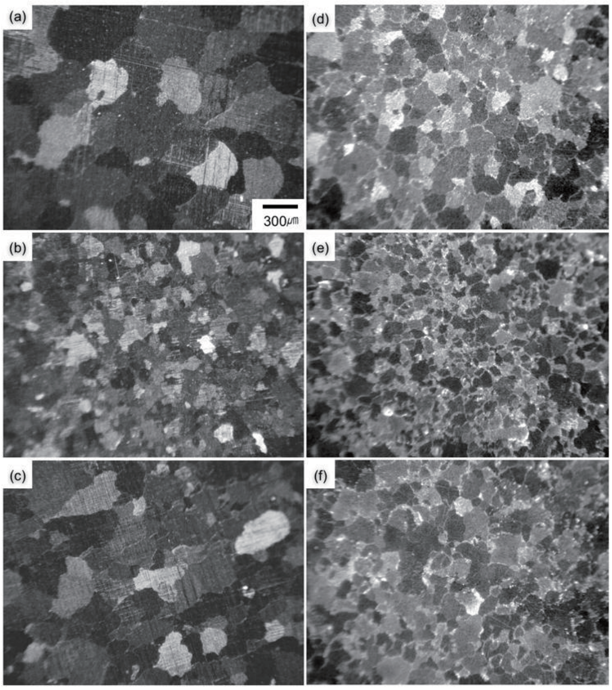

Fig. 2. Macrostructure of Mg alloys;
(a)~(c): AZ31, (d)~(f): AZ91
(a), (d): without NUMT
(b), (e): after NUMT with Ti sonotrode
(c), (f): after NUMT with Fe sonotrode

The grain size of AZ31 without NUMT is 470 μm, and it is rapidly decreased to 120 μm and 280 μm after NUMT with Ti and Fe sonotrode respectively. The Ti sonotrode is more effective to make grain refine, and this is supposed that the formation of Mn-Fe intermetallics reduces the ability of Fe solute injected into melt after NUMT. The variation of the grain size of AZ91 shows also same result. The grain size after NUMT is decreased, however, the degree of the variation is different with the sonotrode material. The mechanism of grain refinement of NUMT is based on ‘Solute paradigm’ mentioned in previous paragraph. NUMT was carried out at pure Mg, Mg-3wt%Al, and Mg-1wt%Zn alloys to investigate the effect of solutes in Mg alloys on the grain refinement by NUMT, and these results are listed in Table Ⅲ. As shown in table, there is no effect of NUMT on the grain refinement in pure Mg only. However, the grain size of Mg-3wt%Al, and Mg-1wt%Zn was decreased after NUMT. This result means that the effect of NUMT on the grain refinement could not be achieved without the other solute element, the Ti and Fe atoms injected into melt by NUMT must be amplified by the other elements in melt for the restriction of grain growth.

4. Conclusion

NUMT, Nucleation Ultrasonic Melt Treatment, is suggested for the grain refinement by ultrasound. In previous study, the grain refinement must be carried out in special conditions, however, NUMT can make grain refine by using the sonotrode which can supply the special solutes in melt, and this results in the effective grain refinement in Mg alloys.