Article ID: 1003 - 6326(2000)02 - 0235 - 05

Effects of RE on microstructure and properties of AZ91 magnesium alloy[①]

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Abstract: AZ91 magnesium alloy was adopted as master alloy and rare earths (RE) of 1%, 2% and 3% additions were added, respectively. The influence of RE on the microstructure was investigated. By casting fluidity spiral specimens, effect of RE on fluidity was achieved. The microhardness of the alloys was tested. By casting specimens in permanent mold, tensile properties of the alloys with different RE addition at ambient and elevated temperatures were studied. The fracture mechanisms of the alloys were studied by SEM. RE additions cause the formation of $Al_{11}RE_3$ precipitation besides phase change in the alloys. RE firstly decreases and then increases the fluidity. RE has little influence on ambient temperature tensile properties but greatly improves high temperature tensile properties at 150 ℃. Tensile failure of the alloys are mainly brittle cleavage and/or quasi-cleavage fracture.

Key words: rare earth alloys; tensile property; fractures **Document code:** A

1 INTRODUCTION

Magnesium alloys are one of the lightest structural alloys, which have incomparable ratio of strength to mass. In recent years, research and development of magnesium alloys have been greatly promoted by the lightweight requirement in automobile industry. However their commercial applications have been limited because of poor high temperature properties[1~3]. Rare earths are important alloying elements to magnesium alloys, which can improve castability, high temperature properties and corrosion resistance[4~7] without affecting the electrical conductivity of the base alloys[1]. The commonly used systems of magnesium alloys containing rare earths are Mg-Zn-RE-Zr(ZE, EZ), Mg-Ag-RE-Zr(QE), Mg-Y-RE-Zr (WE) and Mg-Al-RE(AE) etc.

Rare earths have been used to magnesium alloys for many years, whereas the alloys of Mg-Al-RE system have been developed recently. Some investigations have been done on the precipitating, morphology, structure and thermal stability of intermetallic phases and strengthening mechanism at elevated temperatures have been studied when rare earths were added to Mg-Al alloys[6~11]. For the cost reason, rare earths were generally added to magnesium alloys as misch metal (MM), but there are still some studies that were based on single rare earth element[8,12]. However no systematic research on the effects of rare earths on casting characteristics, as-cast microstructure and mechanical properties has been reported.

In the present work, rare earths were added to AZ91 magnesium alloy, which is the most widely used die casting magnesium alloy, to study their effects on tensile properties at ambient and elevated temperatures, casting characteristics and microhardness. The fracture mechanisms were also analyzed.

2 EXPERIMENTAL

2.1 Experimental materials

Rare earth additions were prepared in form of cerium rich misch metal. Chemical compositions of AZ91 alloy and misch metal are listed in Table 1.

2.2 Experimental procedure

AZ91 magnesium alloy was melted in crucial electric resistance furnace. Rare earths were added when the temperature of the melt was about 730 ℃ and then the melt was held at 730 ℃ for 30 min before being cast to make sure that rare earths were completely dissolved. AZ91 alloy and AZ91-xRE alloys with rare earth addition amounts of 1%, 2% and 3% were studied.

The alloys were cast into resin-coated sand spiral specimen moulds to investigate the influence of rare earths on fluidity, which was determined by the change of length (mm) of spiral specimens. Matrix microhardness of the alloys were tested in MHT-1 microhardness meter with a load of 0.1 N ($HV_{0.01}$). By casting test bars in permanent mould, the effects of rare earths on tensile properties were studied. The pouring and mould temperature were (730 ± 3) ℃ and (200 ± 10) ℃, respectively. Tensile tests at ambient and elevated temperatures were conducted on a Shimadzu AG-100KNA material test machine.

① **Received date:** Mar. 2, 1999; **accepted date:** May 24, 1999

Table 1　Chemical compositions of AZ91 alloy and misch metal　(%)

AZ91	Al	Zn	Mn	Be	Si	Fe	Cu	Ni	Mg
	8.12	0.923	0.142	0.00067	0.0607	0.0148	< 0.004	0.0042	Balance
Misch metal	Ce	La	Nd	Pr	Fe	Si	Mg	Mn	Ca	P
	50.2	26.67	15.28	5.37	0.65	0.01	0.38	0.11	0.01	0.003

Fracture surfaces were examined in a Philips SEM515 microscopy.

3 RESULTS AND DISUSSION

3.1 Microstructure

Fig.1 shows the influence of rare earths on the microstructure of AZ91 magnesium alloy. The microstructure of AZ91 alloy is composed of α(Mg) matrix and $Mg_{17}Al_{12}$ phase, which precipitates along grain boundaries (Fig.1 (a)). After rare earths are added, a rod-like intermetallic phase —$Al_{11}RE_3$ phase is observed (Fig.1 (b), (c)), which was called Al_4RE by some researchers[10,13]. Because of the high chemical stability of $Al_{11}RE_3$, rare earths are combined with Al and form $Al_{11}RE_3$ until all the available rare earths are used without any formation of pseudobinary Mg-RE phase or pseudoternary Mg-Al-RE phase, shown as Fig.1(b) and (c). The melting points of $Al_{11}RE_3$ phase are higher than 1 200 ℃ and it is the main enhancing phase at elevated temperatures.

The formation of $Al_{11}RE_3$ consumes some aluminum atoms, which greatly reduces the amount and size of $Mg_{17}Al_{12}$ phase (Fig.1(b), (c)) after the additions of rare earths. With increasing the amount of addition, the $Mg_{17}Al_{12}$ phase is gradually fined and no coarse and continuous $Mg_{17}Al_{12}$ can be found any more. On the contrary, the amount of the rod-like $Al_{11}RE_3$ phase increases and its size coarsens (Fig.1 (c)).

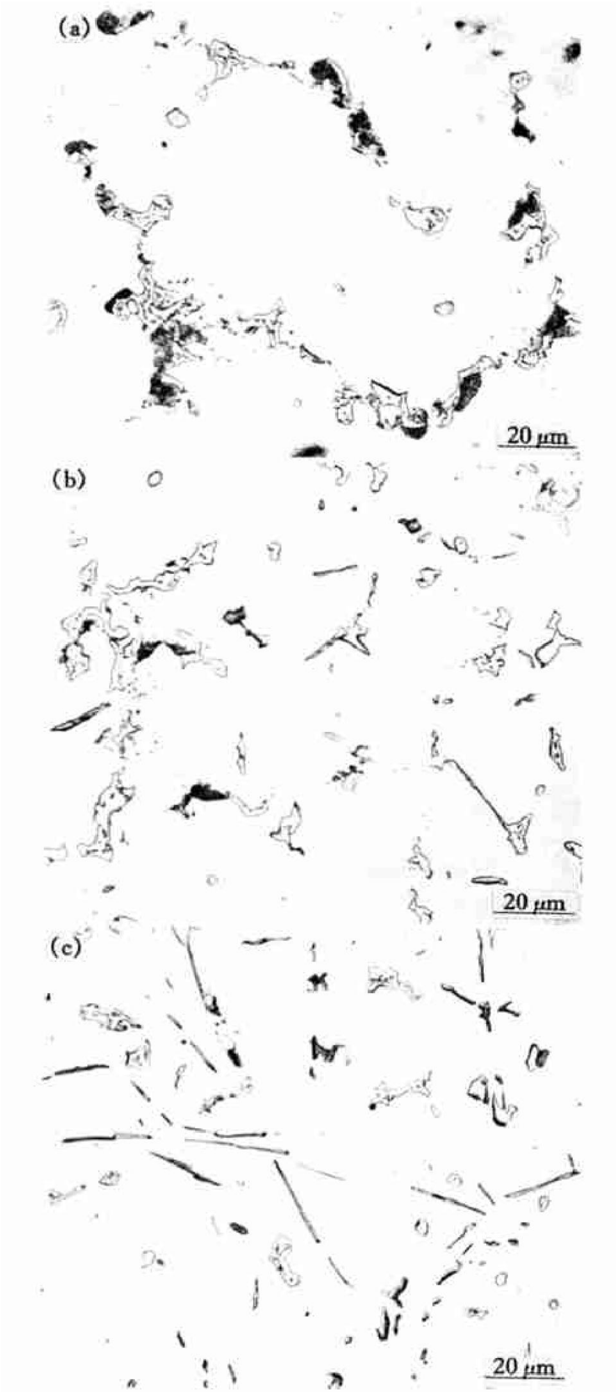

Fig.1　Microstructures of alloys with different RE additions
(a) —AZ91; (b) —AZ91-1RE; (c) —AZ91-2RE

3.2 Effects of rare earths on properties

3.2.1　Fluidity

Fig.2 shows the fluidity of the alloys with different rare earth additions. With increasing rare earths addition amount, fluidity decreases at the beginning and then increases. With rare earths addition amount of 3 %, the fluidity length is 731 mm, longer than that (681 mm) of AZ91 alloy, and tends to go on increasing with further rare earths additions (Fig.2).

The influences of rare earths on fluidity of magnesium alloys can be concluded as follows:

1) Rare earths have a high susceptibility to oxidation and reaction with fluxes[6,14,15], so the inclusions in melt are increased and fluidity resistance increases, which is disadvantageous to fluidity.

2) Rare earths reduce the freezing range of magnesium alloys, which is beneficial to fluidity.

3) $Al_{11}RE_3$ has high latent heat (approximately 55 kJ/ mol)[11]. Following the formation of $Al_{11}RE_3$, much heat gives out, which slows down temperature decreasing and prolongs fluidizing time.

With relatively less rare earth additions, the first fact played the leading role, which results in the decrease of fluidity. With further rare earths addi-

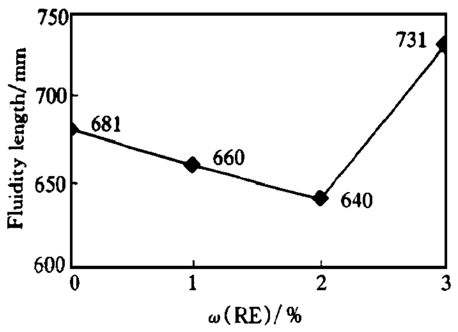

Fig.2 Fluidity of alloys with different RE additions

tions, more $Al_{11}RE_3$ phase is formed and the improvement effects become more important and then fluidity is increased (Fig.2).

3.2.2 Microhardness

Table 2 shows the effects of the addition amount of RE on microhardness of the matrix of alloy. As shown in Table 3, with increasing rare earth addition amount, microhardness of the matrix increases due to solid solution strengthening. However the microhardnesses of AZ91-2RE alloy and AZ91-3RE alloy have very little difference. This can be explained by the poor solid solubility of rare earths in magnesium[17], which is further reduced for the presence of Al[2]. When rare earths has increased to a certain content in magnesium alloys, solid solution of rare earths in matrix may saturate and further additions would only cause coarsening and increasing $Al_{11}RE_3$ phase without any further solid solution strengthening.

Table 2 Effects of RE on microhardness of the matrix

Alloy	$HV_{0.001}$
AZ91	60
AZ91-1RE	64.5
AZ91-2RE	71.5
AZ91-3RE	72

3.2.3 Tensile properties at ambient temperature

Table 3 shows the effects of rare earths on tensile strength and elongation tested at ambient temperature. Compared to AZ91 alloy, ultimate tensile strength (σ_b) of the alloys with increasing RE addition amount has little difference, yield strength ($\sigma_{0.2}$) somewhat increases and the elongation (δ) gradually decreases with increasing the amount of rare earth addition. According to Nair and co-workers, cerium rich misch metal had little effects on the ambient temperature tensile properties of magnesium alloys in as-cast condition[1], as shown in Table 3.

From above discussion on microhardness, after solid solution saturation of rare earths in matrix, $Al_{11}RE_3$ phase begin to coarsen, causing the decreasing of elongation. The precipitation of $Al_{11}RE_3$ phase impedes the formation and incipient development of the cracks, which improves the yield strength.

Table 3 Effects of RE addition on tensile properties at ambient temperature

Alloy	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%
AZ91	165	94	3.01
AZ91-1RE	164	107	2.29
AZ91-2RE	166	101	1.93
AZ91-3RE	167	103	1.76

3.2.4 High temperature tensile properties at 150 ℃

Table 4 gives the high temperature tensile results of the alloys with or without rare earths additions at 150 ℃ (short-time test). As can be seen in Table 4, rare earths greatly improves the high temperature tensile strength and elongation.

Table 4 Effects of RE addition on tensile properties at 150 ℃

Alloy	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%
AZ91	124	56	3.81
AZ91-1RE	165	91	7.20
AZ91-2RE	177	82	10.08

In AZ91 alloy, the main enhancing phase is $Mg_{17}Al_{12}$, which has low melting point of approximately 462 ℃ and poor thermal stability. $Mg_{17}Al_{12}$ phase can readily coarsen and soften at the temperatures exceeding 120 ℃[5,6]. In addition, $Mg_{17}Al_{12}$ precipitation has a cubic crystal structure incoherent with the HCP magnesium matrix, which leads to the fragility of Mg/$Mg_{17}Al_{12}$ interface. All of these lead to the poor high temperature tensile properties of AZ91 alloy at 150 ℃ (Table 4).

Adding rare earths to AZ91 forms $Al_{11}RE_3$ precipitation in the alloy. $Al_{11}RE_3$ along with the low diffusion speed of rare earths elements in magnesium at elevated temperatures, has much high melting points[10], which makes $Al_{11}RE_3$ have high thermal stability. $Al_{11}RE_3$ phase could retain stability in heat treatments at 500 ℃[8]. So sliding of grain boundaries and development of cracks are effectively prevented at elevated temperatures and high temperature properties are improved. Melting points of typical precipitations in the studied alloys are refered to Ref.[10].

AZ91-2RE alloy has higher ultimate tensile strength (σ_b) and elongation (δ) than AZ91-1RE alloy but relatively lower yield strength ($\sigma_{0.2}$) (Table 5), which indicates that the increase of $Al_{11}RE_3$ phase has little effect on improving the elastic strain ability. Improving effects of $Al_{11}RE_3$ on high temperature properties are mainly to prevent the later stage development of the cracks.

3.3 Fracture analyses

Fig.3 shows SEM images of the tensile fracture surface of AZ91 alloy and AZ91-xRE alloys tested at ambient and elevated temperatures.

Fig.3(a) is the image of AZ91 alloy at ambient temperature, which has a long secondary crack in the middle. Fig.3(b) is a local magnified image of Fig.3(a), which also has a deep secondary crack in the middle. Fig.3(b) is composed of small cleavage planes and steps. On some planes, cleavage rivers can be seen. The tensile fracture surface of AZ91 alloy tested at ambient temperature shows the characteristic of typical cleavage fracture. Fig.3(c) and (d) show the morphologies of ambient temperature tensile fracture surface of AZ91-1RE alloy. Fig.3(d) is local magnified image of Fig.3(c).

Compared with Fig.3(a) and (b), Fig.3(d) has less regular cleavage planes and steps. This can be explained by Fig.4. In cleavage fracture, two parallel cracks with relatively high differential altitude can form steps through the pattern of secondary cleavage or tearing. During the development of cracks, the two patterns are interchangeable. Local areas of Fig.3(d) assume the characteristic of quasi-cleavage and cleavage rivers can still be found on some small planes. Generally, the ambient temperature tensile fracture of AZ91-1RE is mainly cleavage.

Fig.3(e) and (f) show the morphologies of high temperature tensile fracture surface of AZ91-2RE alloy tested at 150 ℃. Fig.3(f) is a local magnified image of Fig.3(e). Compared with ambient temperature tensile fracture surface of alloys with rare earth additions (Fig.3(c) and (d)), the quasi-cleavage of Fig.3(e) and (f) is more apparant. Cracks appeared in local areas develop and finally form morphologies of pits. The fracture procedure is shown in Fig.5 schematically[18]. It is also a kind of brittle fracture but different with cleavage. It is a rather complicated fracture pattern. The planes on the fracture surface

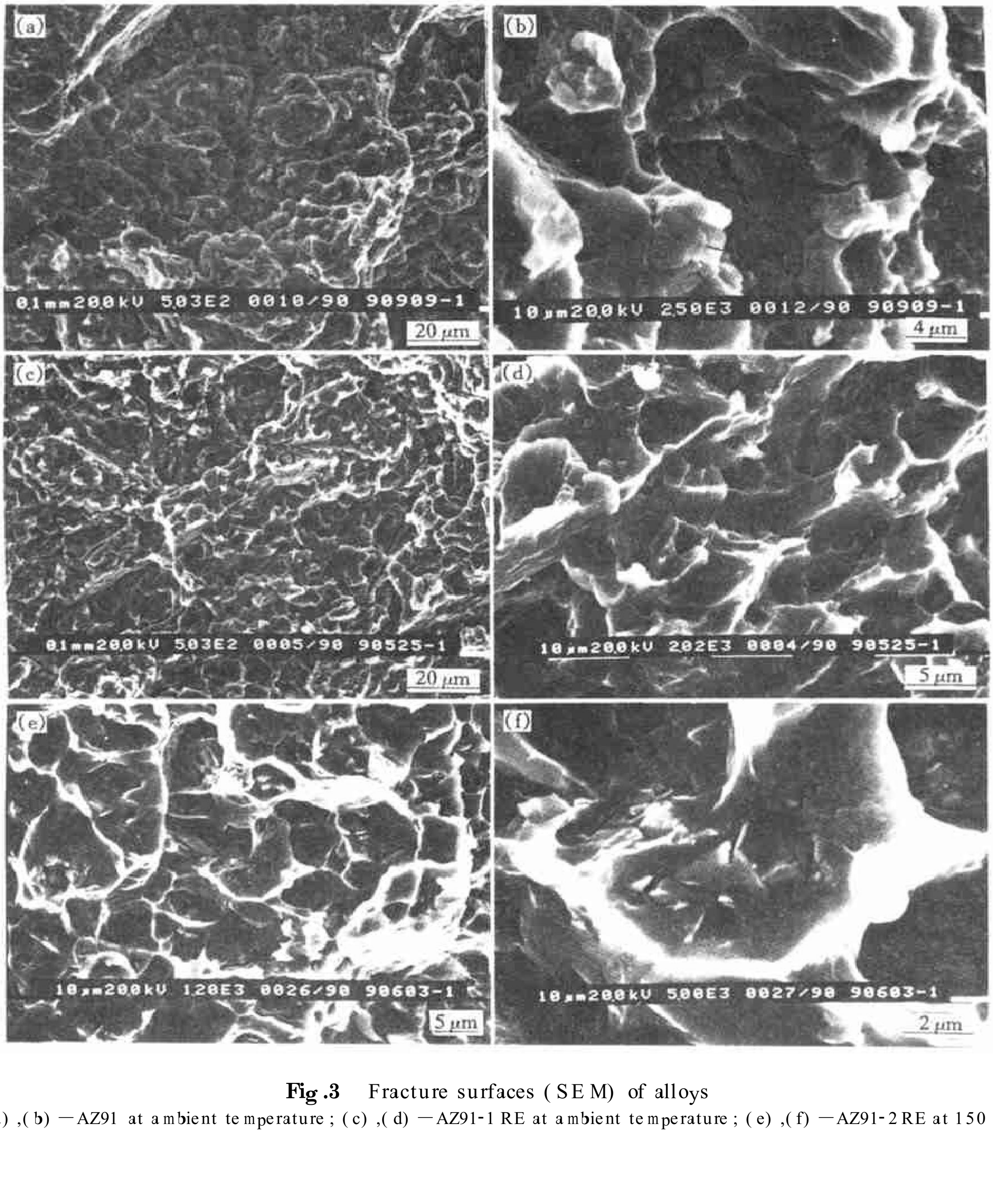

Fig.3 Fracture surfaces (SEM) of alloys

(a),(b) —AZ91 at ambient temperature; (c),(d) —AZ91-1RE at ambient temperature; (e),(f) —AZ91-2RE at 150 ℃

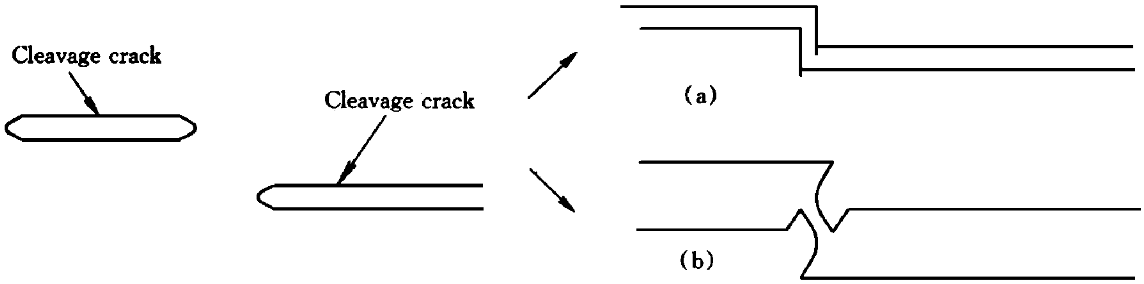

Fig.4 Formation of steps through secondary cleavage (a) and tearing (b)

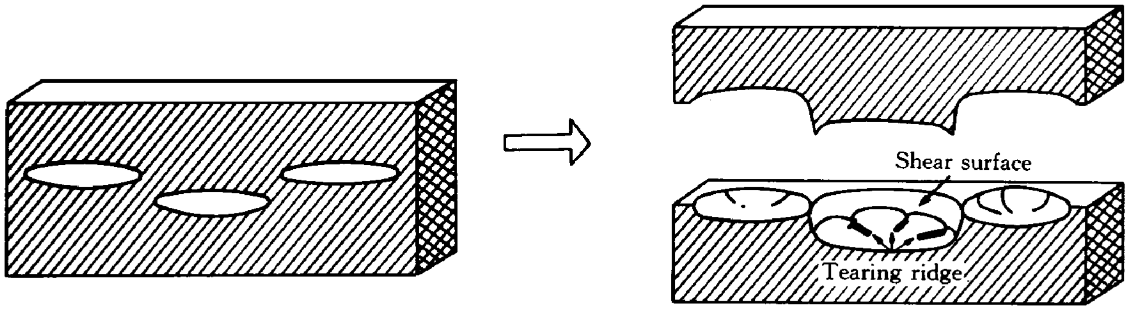

Fig.5 Schematic of quasi-cleavage fracture

are not coherent with certain crystal orientation but formed through combination of local formed micro-cracks. During the combination, tearing ridges are formed (Fig.3(e)). The bottoms of the pits are not strict cleavage planes but consist of several somewhat sunken planes with developed secondary cracks (Fig. 3(f)). The high temperature tensile fracture of AZ91-2RE alloy tested at 150 ℃ is mainly quasi-cleavage fracture.

REFERENCES

[1] Nair K S and Mittal M C. Rare earths in magnesium alloys [J]. Materials Science Forum, 1988, 30: 89.

[2] Pettersen G, Westengen H, Hoier R, et al. Microstructure of a pressure die cast magnesium 4% aluminum alloy [J]. Materials Science and Engineering, 1996, A207 (1): 115.

[3] SUN Yang-shan, WEN Kun-zhong and YUAN Guang-yin. Effects of Sn addition on microstructure and mechanical properties of magnesium alloys [J]. The Chinese Journal of Nonferrous Metals, (in Chinese), 1999, 9 (1): 55.

[4] Sanschagrin A, Tremblay R and Angers R. Mechanical properties and microstructure of new magnesium-lithium base alloys [J]. Materials Science and Engineering, 1996, A220(1~2): 69.

[5] Polmear I J. Magnesium alloys and applications [J]. Materials Science and Technology, 1994, 10(1): 1.

[6] Polmear I J. Recent development in light metals [J]. Materials Transaction JIM, 1996, 37(1): 12.

[7] LI Y and Jones H. Structure and mechanical properties of rapidly solidified magnesium based Mg-Zn-RE alloys consolidated by extrusion [J]. Materials Science and Technology, 1996, 12(12): 81.

[8] WEI L Y, Dunlop G L and Westengen H. Development of microstructure in cast Mg-Al-Rare earth alloys [J]. Materials Science and Technology, 1996, 12(9): 741.

[9] Ferro R, Saccone A and Borzone G. Rare earths metals in light alloys [J]. Journal of the Chinese Rare Earth Society, (in Chinese), 1997, 15(3): 262.

[10] LI Y and Jones H. Effect of rare earth and silicon additions on structure and properties of melt spun Mg-9Al-1Zn alloy [J]. Materials Science and Technology, 1996, 12(8): 651.

[11] WEI L Y and Dunlop G L. The solidification behavior of Mg-Al-Rare earth alloys [J]. Journal of Alloys and Compounds, 1996, 232: 264.

[12] Lee Sunghak, Lee Seung and Kim D H. Effect of Y, Sr and Nd additions on the microstructure and microfracture mechanism of squeeze-cast AZ91-X magnesium alloys [J]. Metallurgical and Materials Transactions A, 1998, 29A(4): 1221.

[13] WEI L Y, Dunlop G L and Westengen H. Age hardening and precipitation in a cast magnesium-rare-earth alloy [J]. Journal of Materials Science, 1996, 31(2): 387.

[14] LUO Zhi-ping, ZHANG Shao-qing, TANG Ya-li, et al. Thermal dynamic analysis of rare earth in the solution of magnesium alloys [J]. Journal of the Chinese Rare Earth Society, (in Chinese), 1995, 13(2): 119.

[15] Luo A and Pekgüleryüz M O. Cast magnesium alloys for elevated applications [J]. Journal of Materials Science, 1994, 29(20): 5259.

[16] WANG Qu-dong, LU Yi-zhen, ZENG Xiao-qin, et al. Application of rare earth in cast magnesium alloys [J]. Special Casting & Nonferrous Alloys, (in Chinese), 1999, (1): 43.

[17] Das S K and Dvis L A. High performance aerospace alloys via rapid solidification processing [J]. Materials Science and Engineering, 1988, 98: 1.

[18] Engel L and Klinger H. An Atlas of Metal Damage. Translated by MENG Xi-ming, (in Chinese) [M]. Beijing: Mechanical Industry Press, 1990: 30~31.

(Edited by HUANG Jin-song)