Advanced Materials Research Vols. 146-147 (2011) pp 1702-1707
Online available since 2010/Oct/27 at www.scientific.net

doi:10.4028/www.scientific.net/AMR.146-147.1702

Microstructure and Creep property of as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloys

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Keywords: magnesium alloy; Nd; creep; grain boundaries sliding; dislocation motion

Abstract. The microstructure and creep property of as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloys were studied in this paper. Results showed that the needle-like $Al_{11}Nd_3$ phase and polygonal Al_2Nd phase are the main precipitates in these alloys containing Nd, and the weight fraction of the former is much more than that of the latter. Their secondary creep rates under the applied stress of 70MPa at 175℃ decreased with the content of Nd increasing and reached the minimum value of $5.0\times10^{-8}s^{-1}$ in the Mg-6Al-6Nd alloy, which is one ninth of that of the Mg-6Al alloy. The improvement in creep property of these alloys containing Nd was mainly attributed to the needle-like $Al_{11}Nd_3$ phase, which hindered the grain boundaries sliding and dislocation motion effectively in the creep.

Introduction

Magnesium alloys are regarded as promising structural materials applied to the transport field, which has been the concern of the researchers in recent years. It has been reported that the rare earth element of Nd is an effective strengthening element to improve the properties of common magnesium alloys such as AZ and AM series. For example, Zhang etal studied the effects of Nd on the microstructure and tensile property of AM60 alloy, which indicated that small needle-like $Al_{11}Nd_3$ formed in AM60 alloy and improved its yielding and tensile strength [1]. Wang etal studied the tensile property of AM50 alloy containing Nd, which indicated the addition of Nd improved the tensile property of AM50 at room and elevated temperatures [2]. Mao etal studied the effects of 0.4-1.2%Nd on the tensile properties of Mg-6Al alloy and found that the tensile strength and elongation both arrived at the highest value when the added amount of Nd reached 1.2% [3]. All studies as above indicate that the strengthening effects of Nd on Mg-Al alloy are obvious, but those studies on the creep property of Mg-Al alloys containing high content of Nd and the contributions of precipitating phases containing Nd to the creep property have seldom been reported. In the present paper, the Mg-6Al alloys containing 2,4,6%Nd are prepared, respectively. The microstructure and creep property of as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloys were studied and the main contributions of main precipitating phases to creep property are analyzed.

Experimental

The nominal compositions of two studied alloys are Mg-6Al and Mg-6Al-6Nd. Commercially pure Mg, Al (>99.99%) were first melted in a crucible electric resistance furnace under a protection of flux cover, then the rare earth Nd was added to the melt in the form of Mg-20%Nd master alloy at 780 °C. The melt was held at 780 °C for 30 mins and poured at 730 °C into a steel mold that was kept at room temperature. The measured compositions of all studied alloys detected by inductively-coupled plasma (ICP) spectroscopy are Mg-6.1Al, Mg-5.8Al-1.7Nd, Mg-5.7Al-3.8Nd and Mg-5.8Al-5.7Nd, respectively, which are denoted as AN60、AN62、AN64 and AN66, respectively. The creep samples were 25mm in gauge length and 5.0mm in diameter. The microstructure of Mg-6Al-*x*Nd(*x*=2,4,6) alloys were analyzed by X-ray diffraction(XRD) , scanning electron microscopy (SEM) and energy dispersive spectrometer (EDS) .The creep tests were performed by using CSS-3902 creep testing machine under the applied stresses of 70MPa at 175℃. In order to clarify the possible contributions of main precipitating phases in the creep, the microstructure of AN66 alloy after creep was studied by optical microcopy (OM) and transmission electron microscopy (TEM).

Results and discussion

Microstructure of as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloys. Fig. 1 shows the XRD patterns of as-cast Mg-6Al-*x*Nd(*x*=0,2,4,6) alloys. Fig. 1(a) indicates that the diffraction peaks of α-Mg and $Mg_{17}Al_{12}$ emerge in the Mg-6Al alloy. It is generally noted that Mg-6Al alloy is mainly composed of α-Mg and $Mg_{17}Al_{12}$ phase according to the Mg-Al phase diagram. However, in this experiment, the diffraction peaks of $Mg_{17}Al_{12}$ were very low. It implies that the precipitating amount of $Mg_{17}Al_{12}$ phase is very little, which is related to the high cooling rate in the solidification process. Fig. 1(b,c,d) indicate that the additional diffraction peaks of Al_2Nd and $Al_{11}Nd_3$ emerge in the Mg-6Al- *x*Nd(*x*=2,4,6) alloys.

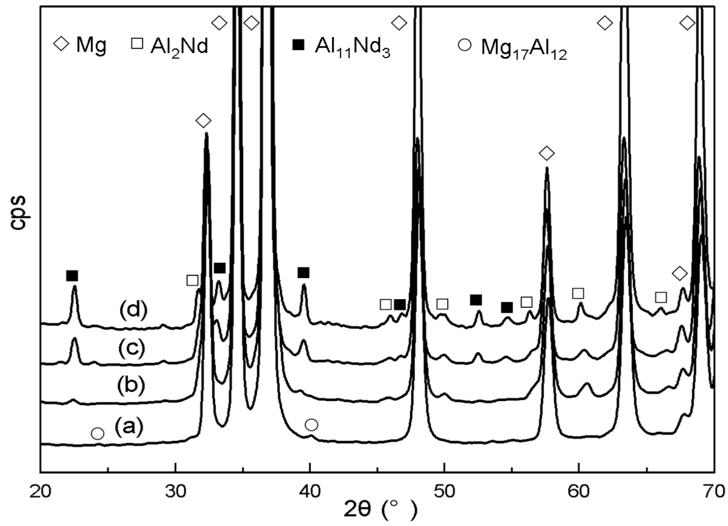

Fig.1 X-ray diffraction patterns of as-cast Mg-6Al-*x*Nd(*x*=0,2,4,6) alloys
(a) AN60; (b) AN62; (c) AN64; (d) AN66

Fig. 2 shows the microstructure of as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloys. Their SEM images in Fig. 2(a,b,c) exhibit that one irregular polygon-like phase and the other fiber-like phase are formed in the Mg-6Al-*x*Nd(*x*=2,4,6) alloys, and the amount of the latter is much more than that of the former. The EDS analysis is conducted to identify the composition of precipitating phases (denoted as A and B, respectively). Results show that the irregular polygon-like and fiber-like phases consist of Al and Nd elements and their atomic ratios are 2.2 and 3.4, respectively, which are well in agreement with the stoichiometric ratio 2:1 and 11:3. Based on the results of XRD analysis, the chemical formula of Al-Nd precipitating phases can be conformed to be Al_2Nd and $Al_{11}Nd_3$.

In order to find out the weight fractions of Al_2Nd and $Al_{11}Nd_3$ in the investigated alloys, one kind of calculation method as below was applied. The contents of Al and Nd in these alloys were obtained by ICP. The content of Al reacted with Nd can be calculated after the content of Al dissolved alloys matrix is identified. According to the contents of Al and Nd element of the two precipitating phases, the weight fractions of $Al_{11}Nd_3$ and Al_2Nd can be obtained. It is noted in reference [4] that the content of Al solid saluted in Mg matrix can be evaluated based on the precise measurement of the lattice constant of Mg-Al alloy. The lattice parameter a_0 as a function of Al content *C*(weight percent) in Mg-Al solid solution can be obtained by the following equation.

$$a_0 = 0.32094 - 3.9922 \times 10^{-4} C_{Al} \quad (1)$$

According to Eq.1, the content of Al solid saluted in Mg-Al matrix of Mg-6Al-*x*Nd(*x*=2,4,6) alloys can be calculated. The weight fractions of precipitating phase, $Al_{11}Nd_3$ and Al_2Nd are calculated and listed in Table 1 It can be seen from Table 1, when the content of Nd increased from 2wt.% to 6wt.%, the content of $Al_{11}Nd_3$ increased from 2.16wt% to 8.44 wt% while the content of Al_2Nd only increased from 0.57 wt% to 0.96wt.%. In the three alloys, the contents of $Al_{11}Nd_3$ are higher than those of Al_2Nd and the increasing range of $Al_{11}Nd_3$ with the content of Nd is much higher than that of Al_2Nd, implying there is a sharp difference between the effects of Nd on the contents of the two Al-Nd phases.

Table 1 Weight fractions of $Al_{11}Nd_3$ and Al_2Nd in Mg-6Al-*x*Nd(*x*=2,4,6) alloys

alloys	a_0 /nm	Al solid saluted in Mg matrix / wt.%	$Al_{11}Nd_3$ /wt.%	Al_2Nd /wt.%
AN62	0.319039	4.76	2.16	0.57
AN64	0.319624	3.29	5.53	0.72
AN66	0.320102	2.10	8.44	0.96

In order to clarify the distribution of two phases in these investigated alloys containing Nd, taking the AN64 alloys as an example, its optical image (OM) is shown in Fig. 2(d). It can be seen that $Al_{11}Nd_3$ are mainly located along the grain boundaries and dendritic boundaries while Al_2Nd with a larger morphology (denoted as C and D) are mainly situated in grains interiors. It is known that the diffusion rate of a solute element in solid solution is extremely slow. For example, the diffusion coefficients of Nd in Mg are about $1.0\times10^{-17}m^2s^{-1}$ at 500℃[5].Therefore, it is difficult to form Al_2Nd by diffusion of Al and Nd after α-Mg has precipitated. It can be inferred that Al_2Nd precipitates prior to α-Mg. Because the migration rate of precipitating phases in melt is comparatively high, it is easy for the small particulates of Al_2Nd to cluster and grow, which is also in agreement with the larger morphology of Al_2Nd. With α-Mg precipitating, Al and Nd would be enriched in the surplus liquid according to the Mg-rich corner of Al-Mg and Mg-Nd phase diagram [6]. Once the activity of Al and Nd in the surplus liquid arrives at the thermodynamic conditions of precipitation of $Al_{11}Nd_3$, $Al_{11}Nd_3$ would precipitate instead of Al_2Nd. Therefore, that is to say the distribution and fraction of the two phases are related to the actual solidification process.

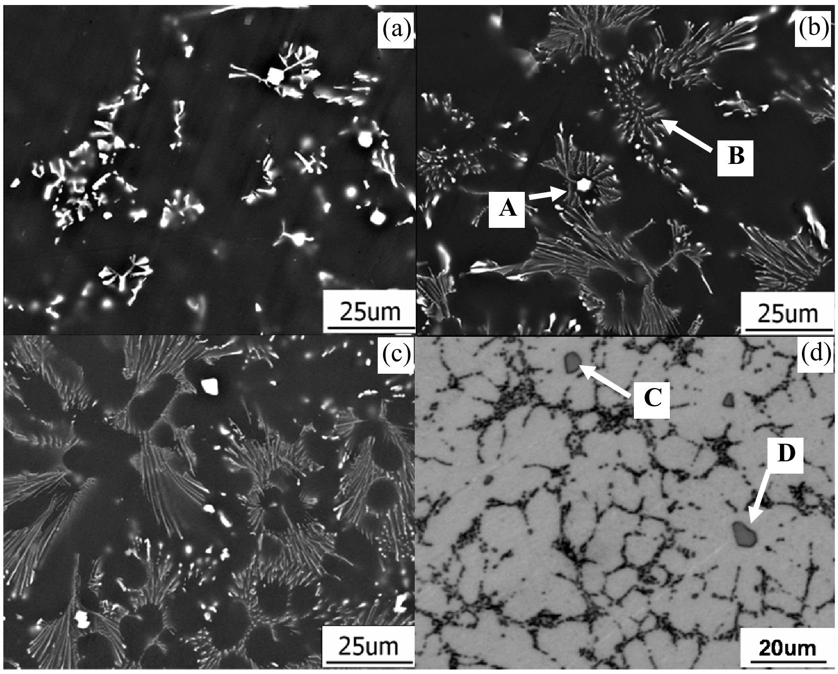

Fig.2 Microstructure of as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloy
(a) SEM image of AN62 alloy; (b) SEM image of AN64 alloy;
(c) SEM image of AN66 alloy;(d) OM image of AN64 alloy

Creep property of as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloys. The creep curves and secondary creep rates of as-cast Mg-6Al-*x*Nd(*x*=0,2,4,6) alloys are showed in Fig.3. Fig.3(a) indicates that the creep life for AN60 is only about 60h and the total creep amount (δ) of Mg-6Al-*x*Nd(*x*=2,4,6) alloys for 100h decreases with the increase in the addition of Nd. For example, δ_{AN66} decreases to one half of that of δ_{AN62}. Fig.3(b) indicates that the secondary creep rates of as-cast Mg-6Al-*x*Nd(*x*=0,2,4,6) alloys also decrease with the increase in the addition of Nd. The secondary creep rate of AN66 ($5.0\times10^{-8}s^{-1}$) reached one ninth of that of AN60 ($4.5\times10^{-7}s^{-1}$).

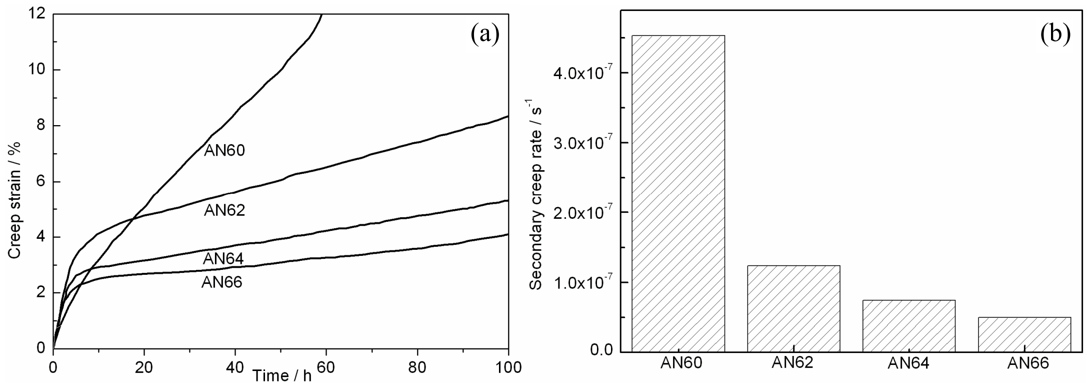

Fig. 3 Creep property of as-cast Mg-6Al-xNd(x=0,2,4,6) alloys under the applied stress of 70MPa at 175℃

Taking AN66 alloy as an example, its microstructure after creep for 100h under the applied stress of 70MPa at 175℃ was observed by using OM and TEM and the results are listed in Fig.4 and Fig.5, respectively. Fig.4(a) indicates that some $Al_{11}Nd_3$ crystallize across the grain boundaries such as A and provides a bridging effect between two adjacent grains [7]. Its schematic diagram can be described by Fig.4(b), which indicates that these $Al_{11}Nd_3$ are able to hinder the grains sliding each other effectively. For the common Mg-Al series alloys, their creep mechanism is greatly affected by the instability of the microstructure of grains boundaries. For example, the failure of creep for AZ91 alloy is related to the precipitation of $Mg_{17}Al_{12}$ with a low melting point of 438℃ in the creep [8]. It is easy for $Mg_{17}Al_{12}$ to be softened at the temperatures for creep and is difficult to hinder the grain boundaries sliding while $Al_{11}Nd_3$ with the melting point of 1235℃ is more thermally stable and hinder the grain boundaries sliding more effectively [9]. Fig.5(a) shows the high density dislocations emerge in the vicinity of needle-like precipitating phase. Fig.5(b) shows this phase is $Al_{11}Nd_3$

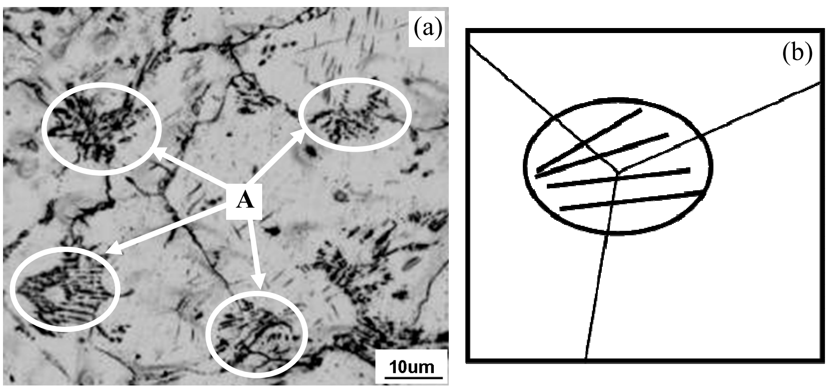

Fig.4 $Al_{11}Nd_3$ phase located in grain boundaries of AN66 alloy
(a) $Al_{11}Nd_3$ phase located in grain boundaries;
(b) Schematic diagram of $Al_{11}Nd_3$ phase cross grains

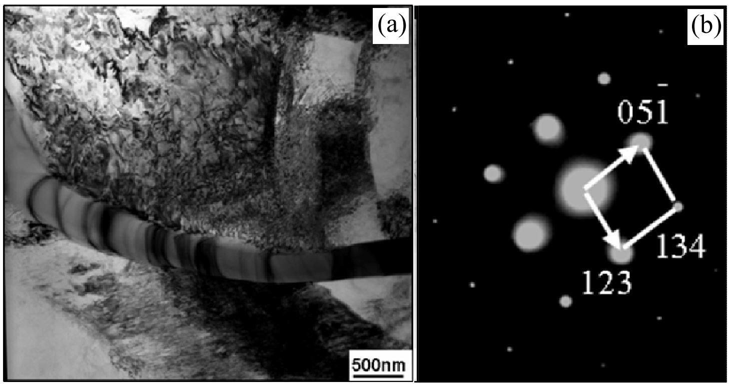

Fig.5 Microstructure changes of as-cast AN66 alloy after creep at 175℃ under the stress of 70MPa for 100h
(a) High density dislocations in the vicinity of needle-like precipitating phase;
(b) SADP of precipitating phase

(a=0.4359nm, b=1.2924nm, c=1.0017nm) [9]. Both indicates that $Al_{11}Nd_3$ phase also leads to the dislocation pile-ups and hinder the dislocations motion. In all, the contributions of Nd to the improvement of creep property of Mg-6Al alloy was mainly contributed to the needle-like $Al_{11}Nd_3$ phase ,which hindered the grain boundaries sliding and dislocations motion effectively in the creep of Mg-6Al alloy.

Conclusions

(1) The needle-like $Al_{11}Nd_3$ phase and polygonal Al_2Nd phase are the main precipitating phases in as-cast Mg-6Al-*x*Nd(*x*=2,4,6) alloys, and the weight fraction of the former is much more than that of the latter. The needle-like $Al_{11}Nd_3$ phase is the main precipitating phase in the Mg-6Al alloys containing Nd.
(2) The secondary creep rates of as-cast Mg-6Al-*x*Nd(*x*=0,2,4,6) decreased with the content of Nd increasing and reached the minimum value of $5.0\times10^{-8}s^{-1}$ at the content of 6.0.%Nd, which is one ninth of that of Mg-6Al alloy. The improvement in creep property of Mg-6Al-*x*Nd(*x*=2,4,6) alloys was mainly attributed to the needle-like $Al_{11}Nd_3$ phase, which hindered the grain boundaries sliding and dislocation motion effectively in the creep of Mg-6Al alloy.

Acknowledgements

This work is financially supported by the Major state Basic Research Development Program of China (No. 2007CB613706), Project of Key Disciplines Development: “Resources, Environment and Recycling Economy” Interdisciplinary under the Jurisdiction of Beijing Municipality (No. 0330005412901) and Project of Team Construction of Science and Technology Innovation for Students, Beijing University of Technology (CTD-2009-17).

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Advances in Superalloys
doi:10.4028/www.scientific.net/AMR.146-147

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