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ScienceDirect
Trans. Nonferrous Met. Soc. China 20(2010) s453−s457
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Effect of precipitation on internal friction of AZ91 magnesium alloy

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Received 23 September 2009; accepted 30 January 2010

Abstract: The effect of precipitation on the internal friction (IF) of AZ91 magnesium alloy was investigated by using X-ray diffraction (XRD) analysis, scanning electron microscope (SEM) observation, and dynamic mechanical analysis (DMA). Six different states of alloy were prepared by applying different heat treatment processes: as-cast, in-complete solid solution, complete solid solution, micro-precipitation, continuous precipitation and continuous−discontinuous precipitation. It was found that the internal friction of in-completely solid-solutionized, completely solid-solutionized and micro-precipitated specimens showed a similar characteristic, and the grain boundary relaxation is completed depressed due to the Al atoms supersaturated in the α-Mg solution. However, a thermal relaxation internal friction peak was observed for continuously precipitated and continuously−discontinuously precipitated specimens at around 438 K and frequency of about 1 Hz, which was attributed to the grain boundaries relaxation. Furthermore, it was found that the relaxation of the β-$Mg_{17}Al_{12}$/α-Mg phase interfaces should give its contribution to the background internal friction in the as-cast, continuously precipitated and continuously−discontinuously precipitated specimens.
Key words: precipitation; solutionization; isothermal treatment; aging; internal friction

1 Introduction

Magnesium alloys are suitable for aerospace and other transport applications due to their low density, high specific strength and high damping capacity[1]. The major limiting factors in using magnesium include its rapid loss of strength with an increase in service temperature and poor creep resistance[2]. With alloying by elements Al and Zn, AZ91 alloy appears which exhibits excellent mechanical properties over pure magnesium. It was found that AZ91 alloy was strengthened by β-$Mg_{17}Al_{12}$ precipitation, and the morphology of β phase can be easily changed and controlled by implementing different heat treatments[3−5]. The internal friction (IF) of AZ91 alloy has just been studied in the recent years. The effect of heat-treatment on damping properties was studied[6], and several IF peaks associated with the precipitation are found in AZ91 alloy[7−8]. However, the effect of β precipitation on the IF properties of AZ91 alloy is not clear.

Accordingly, the AZ91 alloy was investigated in order to give a clear physical view of the effect of precipitation process on internal fiction. It is also expected that the results are useful for the optimization of heat processing technologies of the AZ91 alloy.

2 Experimental

The AZ91 alloy with the nominal composition of Mg-9.0%Al-1.0%Zn (mass fraction) was prepared in vacuum induction furnace under the protection of Ar gas. The plate-shaped specimens with the dimensions of 60.0 mm×10.0 mm×0.8 mm used for the IF measurements were machined from the ingot using an electric sparking machine and subsequently heat-treated in accordance with the conditions summarized in Table 1. The heat treatment parameters were selected based on Ref.[5] where TANG investigated the effect of heat-treatment on the morphology of AZ80 alloy which has the same precipitation process as AZ91 alloy.

The measurement of IF was carried out on a TA dynamic mechanical analyzer (DMA) with dual cantilever mode. The internal friction was determined by Q^{-1}=tan δ, where δ was the lag angle between the applied strain and the response stress. For the measurement of temperature dependent IF, the test conditions were as follows: the strain amplitude (ε) was 0.01%, the vibration frequency (f) was 1 Hz, the temperature (T) range was

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Table1 Heat-treatment conditions for damping specimen

State	Heat treatment process
C	As-cast
S_1	Solution treatment for 1 h at 688 K, water quenching
S_2	Solution treatment for 3 h at 688 K, water quenching
I	Isothermal treatment for 3 h at 688 K, air cooling
A_1	Solution treatment for 3 h at 688K, water quenching; aging for 2 h at 583K, air cooling
A_2	Solution treatment for 3 h at 688 K, water quenching; aging for 2 h at 523K, air cooling

from 303 to 623 K and the heating rate ($\dot{T}$) was 3 K/min.

X-ray diffraction (XRD) analyses as well as scanning electron microscope (SEM) observation were also conducted to characterize the evolution of the microstructures. The specimens for SEM were prepared by standard techniques at room temperature and the used etching solution was 4% concentrated HNO_3 and 96% ethanol (volume fraction).

3 Results and discussion

3.1 Characterization of microstructures

The microstructures of the AZ91 alloy at different states were clarified by XRD and SEM examination. As indicated in Fig.1, there are α-Mg and β-$Mg_{17}Al_{12}$ phase co-existing in the as-cast, S_1, A_1 and A_2 specimens, while there is only α-Mg phase existing in the S_2 and I specimens. It should be found that after different heat treatments, the grain size changes little (shown in Fig.2).

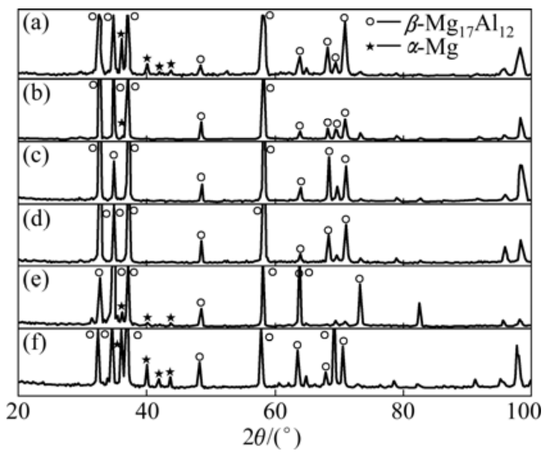

Fig.1 XRD patterns of specimens in different states: (a) As-cast; (b) S_1; (c) S_2; (d) I; (e) A_1; (f) A_2

From Fig.2(a) it can be seen that the as-cast AZ91 alloy contains a net-shaped microstructure distributing along the α-Mg grains, which is divorced eutectic compound (α-Mg+β-$Mg_{17}Al_{12}$). For S_1 specimen, most of the β precipitates dissolve in the matrix and only a little β phases exist around the grain boundaries as shown in Fig.2(b). While after S_2 treatment, the β precipitates completely dissolve in the matrix, forming supersaturated α-Mg solution and no precipitates are found at the grain boundaries (Fig.2(c)). For I treatment, it could be found from Fig.2(d) that there is a paucity of granular β phase distributing at the grain boundaries which are formed during air cooling. TANG[5] found that when aging at higher temperature after solution treatment, the β-$Mg_{17}Al_{12}$ grain were generated in a continuous precipitation, while at lower temperature, both continuous and discontinuous precipitation would happen. From Fig.2(e) it can be seen that after A_1 process, there is a number of graininess β phase distributing along the grain boundaries and abundant rhombus-plate β phase symmetrically distributing in the grains, both of which are the products of continuous precipitation. Whereas, after A_2 process, there is substantive lamellar β phase distributing in some grains but no graininess or rhombus-plate β phase can be seen (Fig.2(f)). The lamellar β phase is the product of discontinuous precipitation. The SEM observation is corresponded with relevant XRD analysis except I treated specimen in which no precipitates are found in XRD analysis due to the fact that the content of β phase is too small to be detected.

Therefore, we have six states of alloy with different β phase morphologies by solution and isothermal or aging heat treatment, and they are: C, as-cast; S_1, in-complete solid solution; S_2, complete solid solution; I, microprecipitation; A_1, continuous precipitation and A_2, continuous−discontinuous precipitation.

3.2 Examination of IF behavior

Fig.3 shows the comparison of IF—temperature curves for the specimens in different states. It can be found that the IF of different specimens increases with increasing temperature. Comparing the IF values among the as-cast, solutionized, isothermally treated and aged specimens, it can be seen that no significant differences are found after different solution treatment and isothermal treatment, and the IF value for as-cast alloy is higher than that of solutionized and isothermally treated alloys over the test temperature range. In addition, no IF peak is found in as-cast, solutionized and isothermally treated specimens. However, there is an obvious IF peak at around 433 K in the aged specimens, and the background IF increases with decreasing aging temperature. Correlating the results in Fig.3 with those in Figs.1 and 2, it is clear that the IF peak in the aged specimens is related to β precipitates.

To clarify the origins of this IF peak in the aged specimens, the dependence of the IF measurement under different frequencies (1, 10 and 100 Hz) was taken on A_1

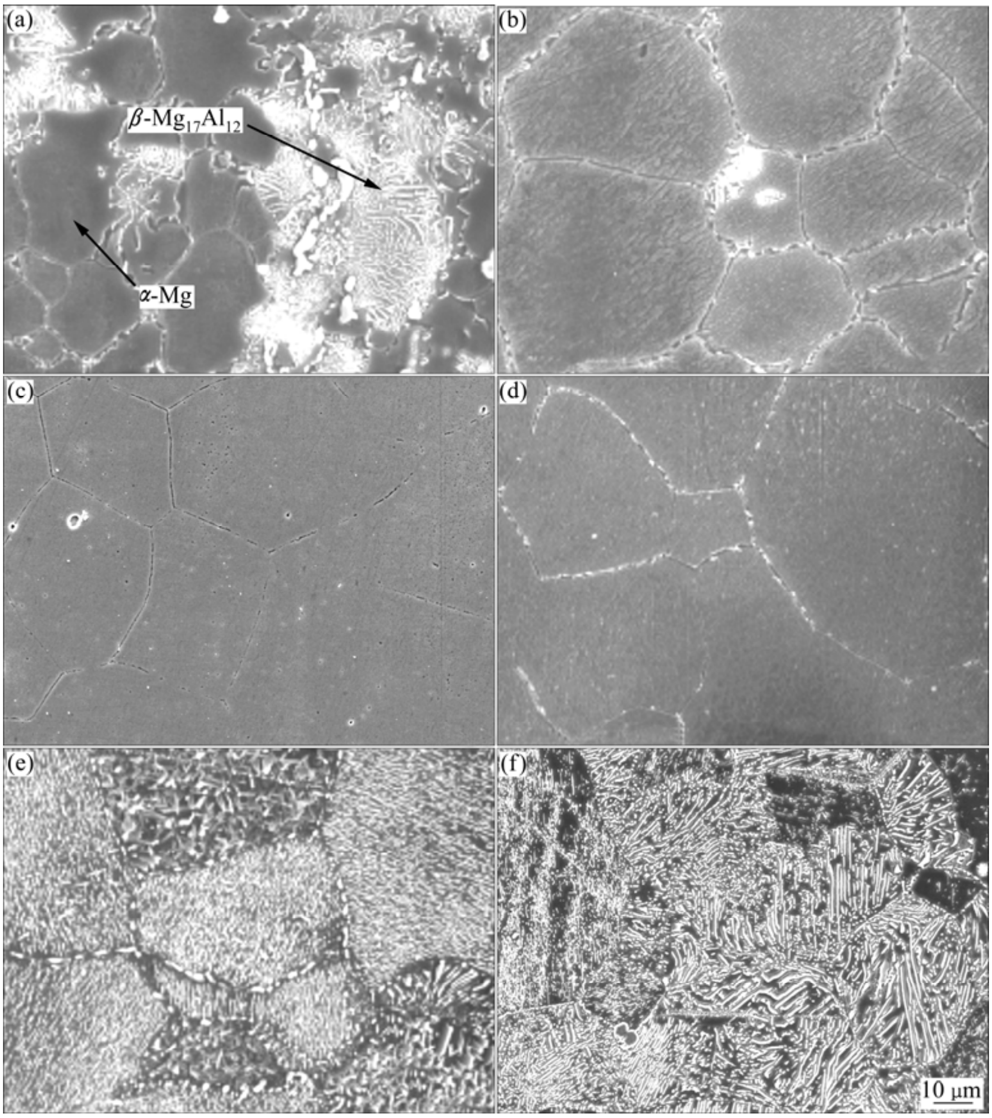

Fig.2 SEM images of specimens in different states: (a) As-cast; (b) S_1; (c) S_2; (d) I; (e) A_1; (f) A_2

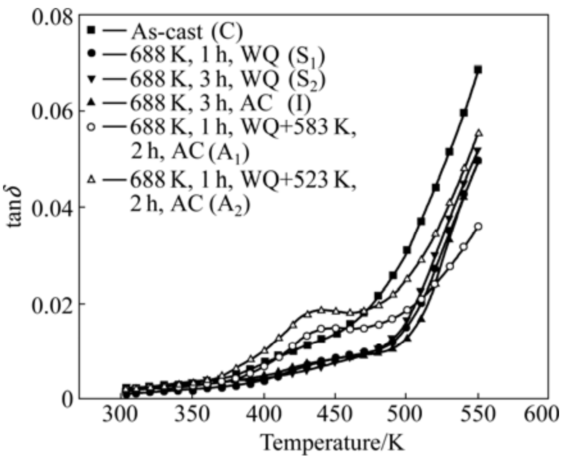

Fig.3 IF—temperature curves of specimens in different states

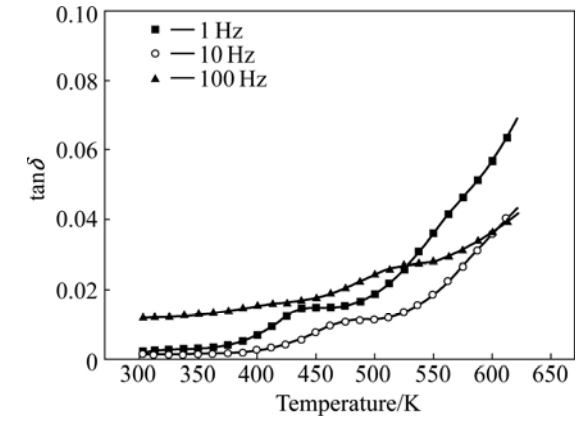

Fig.4 Dependence of IF peak on measuring frequency for A_1 treated specimens at strain amplitude of 0.01% and heating rate of 3 K/min

treated specimens at the strain amplitude of 0.01% and the result was shown in Fig.4. It can be seen that this IF peak is frequency-dependent. The peak position shifts towards higher temperature (from 438.2 to 511.5 K) as the frequency increases, showing typical relaxation nature.

For a thermally activated relaxation process, the relaxation time should obey the Arrhenius law[9−10]:

$$\tau=\tau_0\exp[H/(kT)] \quad (1)$$

where τ_0 is the pre-exponential factor and H is the activation energy of the relaxation process. At the peak position, $\omega\tau_p=1$ should be satisfied, where $\omega=2\pi f$ is the angular frequency and τ_p is the relaxation time at the peak temperature. With the τ_p values corresponding to different frequencies and according to the data in Fig.4, the Arrhenius plot of $\ln(\omega/s^{-1})$ against $(10^3/T_p)(K^{-1})$ can be given, as shown in Fig.5. From the slope of the Arrhenius plot, H=1.26 eV is obtained. The activation energy is comparable to grain boundary relaxation activation energy of 1.31 eV in AZ91 alloy[7], suggesting that the IF peak is probably related to grain boundary relaxation.

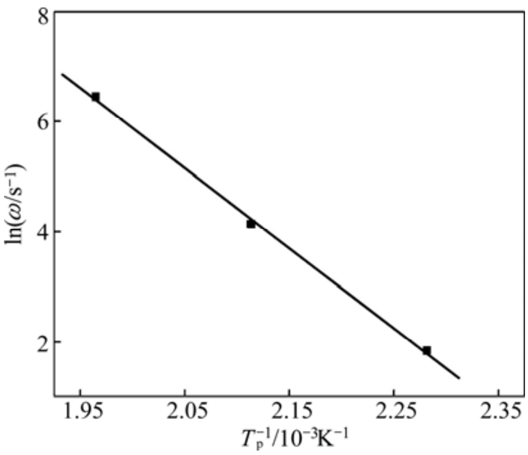

Fig.5 Logarithm of circular frequency against reciprocal of net peak temperature based on data of Fig.4

It is known that the precipitation in the boundaries in certain conditions can induce an IF peak named precipitation grain boundary peak. Compared with the clean grain boundary peak, the precipitation grain boundary peak usually locates at a lower temperature [11]. In the present study, the IF peak of aged specimens belongs to precipitation grain boundary peak due to the much lower peak temperature than the clean grain boundary peak of Mg which is around 503 K at 1 Hz [11−12]. Based on the KE's theory[13], the controlling factor of grain boundary relaxation is the diffusion process in the grain boundary. As a consequence, the distance between the precipitates at the grain boundaries should be a crucial factor in affecting the parameters of IF peak, such as, the peak intensity. After the solution treatment or isothermal treatment (S_1, S_2 and I), although there is a paucity of β phase in the grain boundaries, α-Mg solution is still in the supersaturated state and thereby there will be a number of solute atoms preferentially distributing along the grain boundaries that play a pinning role in preventing the diffusion of Mg atoms along the grain boundaries, which makes the motion of grain boundaries more difficult and thus no IF peak is found. However, substantive β-$Mg_{17}Al_{12}$ phases with different patterns precipitate and symmetrically distribute in the grains or along the grain boundaries (Figs.2(e)−(f)) after aging process, which means that most of the solute Al atoms precipitate in the β phase and decrease the concentration of solute Al atoms in the α-Mg solution not only in the grains but also in the grain boundaries. Thus, the diffusion of Mg atoms along grain boundaries becomes easy and consequently an IF peak is generated. Although there are abundant β precipitates in the as-cast alloy, there is still no IF peak in this study. The appearance of considerable big β phase distributing along grain boundaries (Fig.2(a)) may give its contribution to the vanishing of IF peak, because the dimension of big β phase is too large to transmit Mg atoms compared with the distance of diffusion in the relaxation process which is in an atomic scale.

Besides the grain boundaries relaxation in the aged specimen, another relaxation mechanism should be considered. It is found that background IF after aging process increases, as shown in Fig.3. Based on microstructure observation (shown in Fig.2), it is suggested that the interface between β-$Mg_{17}Al_{12}$/α-Mg should gives its contribution to the background IF. It has been known that $Mg_{17}Al_{12}$ precipitates have a cubic structure with $a=b=c$=10.54 Å, while the matrix has a hexagonal structure with $a=b$=3.209 Å and c=5.211 Å [14]. Because of the large difference between the lattice parameter of these two phases, an incoherent interface is generated. SCHOECK[15] predicted that the IF could be generated in incoherent interface, the relaxation was proportional to the volume of the precipitate, and several IF peaks were found in different alloys[16−18]. In this study, only solutionized or isothermally treated specimens have no or a paucity of β precipitates in accompany with the lowest background IF, which is opposite to the results of the as-cast and aged specimen (Fig.2). Consequently, the differences of background IF can be attributed to the effect of interface relaxation of the β-$Mg_{17}Al_{12}$/α-Mg interface. However, no evidence is given to the contribution of β-$Mg_{17}Al_{12}$/α-Mg interface to the grain boundary relaxation in this study.

4 Conclusions

1) The internal friction of incomplete solid solution, complete solid solution and micro-precipitation specimens show a similar characteristic, and the grain boundary relaxation is completely depressed due to the α-Mg in supersaturated state.

2) A thermal relaxation internal friction peak is observed on continuous precipitation and continuous—

discontinuous precipitation specimens at around 438 K at a frequency of about 1 Hz, and the mechanism of this peak is proved to attribute to the grain boundaries relaxation.

3) The relaxation of the β-$Mg_{17}Al_{12}$/α-Mg phase interfaces should give its contribution to the background IF in the as-cast, continuous precipitation and continuous-discontinuous precipitation specimens.

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(Edited by FANG Jing-hua)