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Small angle X-ray scattering study of the strengthen mechanism of Al–Mg–Li alloy during retrogression and reaging treatment

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ABSTRACT

The δ′ precipitation behavior during retrogression and reaging (RRA) heat treatment in 1420 alloy has been investigated by small angle X-ray scattering (SAXS) method. SAXS results indicate that the transition interfacial layer exists between δ′ phase and matrix in earlier aging and reaging stages. The diffuse phase boundary gradually disappears with the increase of aging and reaging time. The specific inner surface (S_p) rapidly decreases during aging to retrogression. The sample of retrogression 5 min has a sharply defined phase boundary between δ′ phase and matrix. The gyration radius of the particle decreases. Some larger and smaller δ′ particles coexist in the sample. RRA treatment results in an increase in the volume fraction of δ′ phase particles.

1. Introduction

Aluminum–magnesium–lithium 1420 alloy, which was developed in the former USSR in the 1960s for aerospace application, is lightweight, weldable, corrosion resistant and high strength. The 1420 alloy is a typical precipitation hardening alloy. The precipitation of the δ′ phase in 1420 alloys has been the subject of many investigations because of its interesting phase transformations. Early studies have shown that the δ′ phase is an ordered phase with the $L1_2$ structure and that the misfit strain associated with its formation is very small [1]. These studies have also shown that the precipitation particles are coherent with the matrix and spherical in shape. Furthermore, a precursor structure formed before the precipitation has recently been interpreted as congruent ordering and spinadal decomposition [2]. In addition, we have reported the interfacial characteristic of δ′ phase [3,4]. The results also showed that the precipitation and coarsening of δ′ could interpret by spinadal decomposition.

Retrogression and reaging (RRA) treatment, as a new heat treatment, was devised for 7000 system alloys by Cina [5]. It has been employed with 7075 aluminum to obtain stress corrosion cracking (SCC) resistance equivalent to the T73 temper together with T6 strength levels. This treatment was applied to material in the T6 condition and involves a short time heat treatment in the range 200–280 °C, followed by reaging using the conditions similar to those used for the original T6 age. To the best of our knowledge, no detailed study on microstructure change during RRA treatment has been used in Al–Li system alloy by means of SAXS. On the basis of previous work, this article investigates the precipitation process of δ′ phase in 1420 Al–Mg–Li alloy during RRA treatment, which could further help us to understanding the strengthen mechanism of 1420 alloy and mastery the application of RRA treatment in Al–Li alloy.

2. Experimental

A 1420 Al–Li alloy was employed for this study with the composition shown in Table 1. After cold rolling to 1.2 mm in thickness, the samples were cut to the size of 40 mm × 10 mm × 1.2 mm and made solution in a salt bath for 40 min at 743 K, followed by ice water quenching and aged at 433 K for 10, 20 and 32 h in silicon oil, respectively. The RRA treatment consists of two stages:

Firstly, retrogression treated the samples after aging 32 h in salt bath at 593 K for 5 min and subsequently quenched in ice water.

Secondly, reaging treated at 433 K for 1, 5, 10, 20, 32 and 48 h in silicon oil, respectively.

The samples were mechanically polished to the shape of thin wafers of approximately 0.07 mm in thickness suitable for SAXS experiments. SAXS experiments were performed on D/max-rA diffractometer and a 12 kW rotating anode X-ray source. The samples were scanned with a step size of 0.02° and dwelling time of 10 s from 0.1° to 1.5°. The background intensity was subtracted.

3. Results and discussion

For an ideal two-phase structure having sharply defined phase boundaries, the asymptotic behavior of the intensity curve was

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Table 1
Chemical composition of the 1420 alloy (wt.%).

Li	Mg	Zr	Fe	Si	Al
2.16	5.47	0.13	0.14	0.006	Balance

found to obey Porod's law [6]:

$$\lim_{s\to s_{max}} [s^3 J(s)] = K'_p, \quad (1)$$

where $s = 2\sin\theta/\lambda$, 2θ is scattering angle, λ is the wavelength of the X-rays and $J(s)$ is the smeared SAXS intensity obtained experimentally using a long-slit collimation system. K'_p is Porod's constant. One can use Eq. (1) to plot $s^3J(s)$ *vs.* s profile and it will show that $s^3J(s)$ approaches a constant at large values of s. It is obtained that Porod's law is not satisfied in the samples aged at 433 K for 10 and 20 h, respectively, which is satisfied in the later stage (80 h) of aging [4]. For this case, Ruland and Koberstein [7,8] thought that the existence of a diffuse phase boundary or a transition zone between scattering particles and matrix would result in a negative slope of the plots of $s^3J(s)$ *vs.* s. This was because the electron density did not change abruptly, but changes gradually over a certain range between two phases. This structural model is called a non-ideal two-phase structure. When Porod's law is satisfied in the later stage of aging, this shows that the diffuse phase boundary or a transition interfacial layer between δ′ phase and matrix gradually disappear with the increase of aging time, and a sharply defined phase boundary arises in the later stage of aging. Ruland showed that for the non-ideal two-phase structure Eq. (2) should replace Eq. (1):

$$J(s) = \frac{K'_p}{s^3}\left(1 - \frac{2\pi^2E^2s^2}{3}\right), \quad (2)$$

where E is the width of the transition layer between the two phases. By plotting $sJ(s)$ *vs.* s^{-2}, K'_p and E can be obtained. In Fig. 1, the plots of $sJ(s)$ *vs.* s^{-2} for the aged 10 and 20 h are shown, respectively. The transition interfacial layers between δ′ phase and matrix are 3.01 and 3.47 nm. The sizes of δ′ phase are 5.99 and 7.67 nm for the aged 10 and 20 h, respectively [4,6]. This shows that the diffuse phase boundary or a transition interfacial layer between δ′ phase and matrix gradually disappears with the increase of aging time, and a sharply defined phase boundary arises in the later stage of aging 80 h. Fig. 2 shows the plots of $s^3J(s)$ *vs.* s for retrogression 5 min (a) and aging 80 h (b). The Porod's constants are 0.0019 and 0.0042 nm^{-3} for retrogression 5 min and aging 80 h, respectively. The constant K'_p is related to specific inner surface, S_p, by Eq. (3) [9]:

$$S_p = \frac{8\pi K'_p}{I_e(\Delta\rho)^2 V}, \quad (3)$$

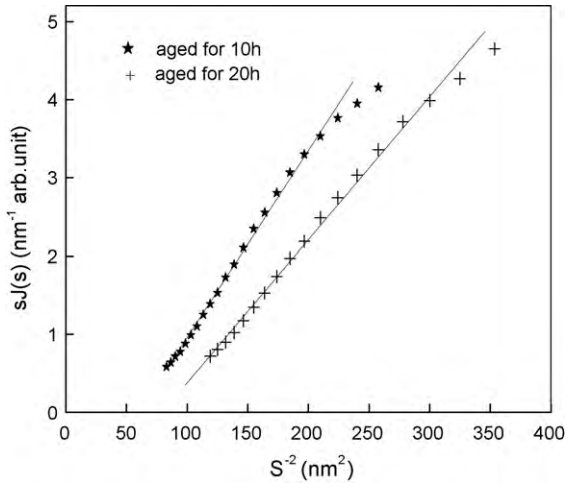

Fig. 1. The plots of $sJ(s)$ *vs.* s^{-2}.

where I_e is the scattering intensity of an electron. $\Delta\rho$ is the electron density difference between the particle and the matrix. V is the volume of sample irradiated by X-rays. I_e, $\Delta\rho$ and V are constant under unchanged experimental conditions. According to K'_ps values, we can obtain

$$S_{pA80\,h} : S_{pR5\ min} = K'_{pA80\,h} : K'_{pR5\ min} = 0.0042 : 0.0019 = 1 : 0.45, \quad (4)$$

where $S_{pA80\,h}$ and $S_{pR5\,min}$ are the specific inner surface of these samples aging 80 h and retrogression 5 min after aging 32 h, respectively. Clearly, the specific inner surface (S_p) rapidly decreases during aging to retrogression.

According to Guinier low [9]:

$$J(h) = I_eNn^2\ \exp\left(-\frac{h^2R_G^2}{3}\right), \quad (5)$$

where h is equal to $4\pi\sin\theta/\lambda$, N is the number of particles, n is the total number of electrons in the particle, R_G is gyration radius of the particle. We can calculate the gyration radius of δ′ phase from the slope of $lnJ(h) \sim h^2$ plot. The value of gyration radius should be considered as the electronic gyration radius of both the δ′ particles and the interfacial layer about their center of electron mass. Table 2 shows that the R_G values correspond to the various thermal treatment conditions. From Fig. 3, one can see that the Guinier curve of the sample of retrogression 5 min after aging 32 h satisfies a straight line within the low-angle and high-angle range, respectively. It is shown that some larger and smaller δ′ particles coexist in the sample. The largest gyration radius of δ′ particles is 9.38 nm and the smallest is 3.77 nm.

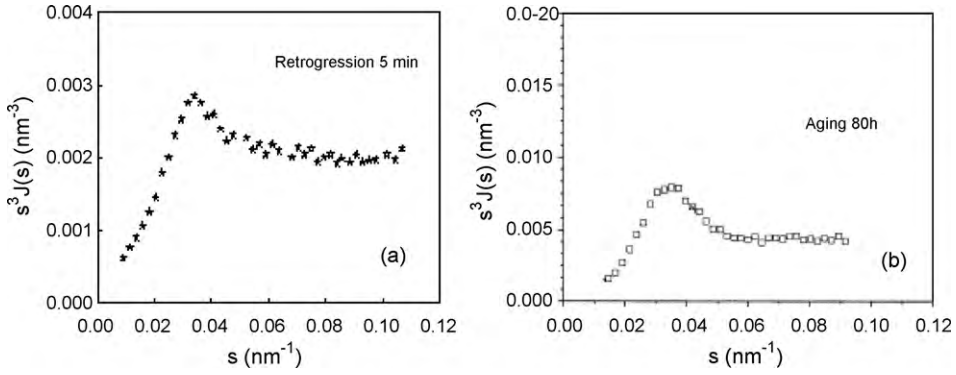

Fig. 2. $s^3J(s)$ *vs.* s plots: (a) retrogression 5 min after aging 32 h; (b) aging 80 h.

Table 2
The R_G values of different thermal treatment conditions.

Treatment condition	Aging 32 h	Reaging 1 h	Reaging 5 h	Reaging 10 h	Reaging 20 h	Reaging 32 h	Reaging 48 h
R_G (nm)	6.76	2.90	3.82	3.99	4.32	4.65	4.92

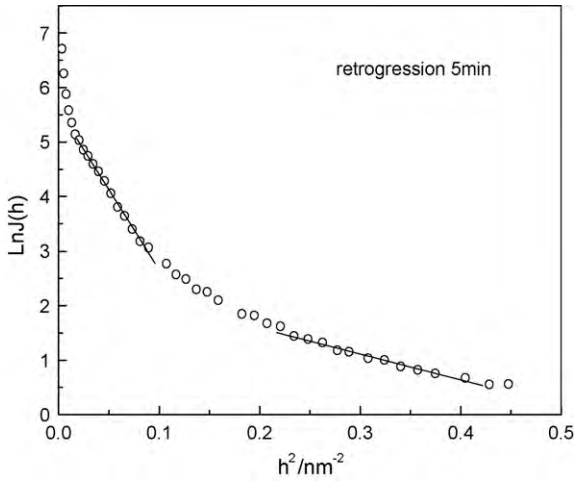

Fig. 3. The plots of $LnJ(h)$ *vs.* h^2.

Previous investigations have reported the interfacial characteristic of δ′ phase exists in binary Al–Li alloy, 8090 alloy and 1420 alloy for aging process [3,4]. These results showed that the precipitation and coarsening of δ′ could be interpreted by spinadal decomposition. In this paper we also find the similar results for 1420 alloy during RRA treatment. In the earlier stage of reaging, the δ′ phase particle quickly precipitates, and the transition interfacial layer between the δ′ phase and matrix is formed simultaneously. Because of the existence of a transition interfacial layer, we could not give an exact determination of the radius of the δ′ phase particle by SAXS or even by TEM techniques, the Table 2 shows that the initial coarsening rate of δ′ particles is quicker. In the later stages of reaging, because the transition interfacial layer gradually disappears, the experimental data on the radius of δ′ particles give the true values.

The transition interfacial layer exists between δ′ phase and matrix in the aging and reaging stages. The transition interfacial layer gradually disappears with the R_G values increase. The specific inner surface (S_p) rapidly decreases during aging to retrogression. The R_G value decreases during retrogression. Since the retrogression temperature (593 K) is higher than aging temperature (433 K), the transition interfacial layer and the smaller δ′ phase would dissolve in the first stage of RRA. However, the δ′ phase does not disappear completely because the retrogression temperature (593 K) is lower than solution temperature (743 K) and the retrogression time is short. This is why the sample of retrogression 5 min has a sharply defined phase boundary between δ′ phase and matrix, the gyration radius of the particle decreases and some larger and smaller δ′ particles coexist in the sample. In the second stage of RRA corresponding to the reaging process, the new δ′ particles surrounded the transitional interfacial layer precipitate. New δ′ phase and remaining δ′ phase coexist in the sample at the same time, so the sum total of δ′ phase particles is much more than that formed in the aging stage and the size of δ′ particles is smaller. The gyration radius of δ′ phase slowly increases in the reaging stage. The formation of new particles is the origin of larger volume fraction of the particle after reaging than after aging. It is shown that the volume fraction of δ′ phase particles is more after RRA treatment. Because of the atom concentration of lithium is definite and in the last stage the volume fraction of precipitated particles shall be more or less constant, the R_G values are smaller than the values of aging stage. RRA treatment results in an increase of the number of fine δ′ phase particles. The beneficial effect of RRA treatment on strengthen alloy and SCC resistance is believed to be due, at least partially, to the increased the volume fraction of fine δ′ phase particles.

4. Conclusion

As shown in this study, the following conclusions are drawn from the experiment results of SAXS. The existence of a transition interfacial layer, which formed in earlier aging treatment, shows that the δ′ phase precipitates by spinodal decomposition. The transition interfacial layers between δ′ phase and matrix are 3.01 and 3.47 nm, and the sizes of δ′ phase are 5.99 and 7.67 nm for the aged 10 and 20 h, respectively. This shows that the diffuse phase boundary or a transition interfacial layer between δ′ phase and matrix gradually disappears with the increase of aging time. The specific inner surface (S_p) rapidly decreases during aging to retrogression. The transition interfacial layer and the smaller δ′ particles would disappear in the retrogression stage. The sample of retrogression 5 min has a sharply defined phase boundary between δ′ phase and matrix, the gyration radius of the particle decreases and some larger and smaller δ′ particles coexist in the sample. In the stage of aging, the coarsening rate of δ′ particles is quicker than the δ′ particles in the reaging stage. RRA treatment results in an increase in the volume fraction of fine δ′ phase particles.

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