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Small-angle x-ray scattering study of the formation and growth of δ' in binary Al–7.87at% Li alloy

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The formation and growth of δ' particles in an alloy of Al–7.87at% Li have been investigated using the small angle x-ray scattering technique. It has been shown that a transition zone, which exists between the δ' phase and the matrix after ageing for 16 h, disappears after ageing for 32 h. The existence of a transition zone indicates that the δ' phase is precipitated in the process of spinodal decomposition. This result also indicates that the growth of δ' particles should be divided into two stages. During the first stage, the δ' phase increases eating up the transition zone before ageing for 32 h, and in the second stage it grows by swallowing up each other after ageing for 32 h. In the latter stage, the larger and smaller δ' particles can coexist in the alloy.

Keywords: Al–Li alloy, δ' phase, transition zone
PACC: 8130M, 6110F

1. Introduction

Al–Li alloys have received considerable attention because of their potential applications for constructing aircraft structures. The precipitation of the δ' phase in binary Al–Li alloys has become a subject of many investigations for its interesting phase transformation. It has been shown in earlier studies[1] that the δ' phase is an ordered phase with an $L1_2$ structure and the misfit strain associated with its formation is very small. These studies have also shown that the precipitated particles are coherent with the matrix and spherical in shape. Furthermore, a precursor structure, formed before the precipitation, has been interpreted as a congruent ordering and spinodal decomposition.[2] Recently, we have reported on the interfacial characteristic of the δ' phase in the binary Al–Li alloy,[3] 8090 alloy[4] and 1420 alloy.[5] The results have also shown that the precipitation and coarsening of δ' could be interpreted by spinodal decomposition.

In this paper, the formation and growth of δ' particles in the Al–7.87at% Li alloy are investigated using the small angle x-ray scattering (SAXS) technique.

2.Experimental details

A high-purity Al–7.87at% Li binary alloy was used for this study. After cold rolling to 1.2 mm in thickness, the samples ($40\times10\times1.2\,\mathrm{mm}^3$) were solution-treated in a salt bath for 20 min at 800K and subsequently quenched into ice water. The as-quenched samples then underwent mechanical polishing to the shape of thin wafers of approximately 0.1 mm in thickness, suitable for SAXS experiments when Cu$K\alpha$ radiation was used. Isothermal ageing treatments were performed at 463 K for 16, 32 and 64 h in a silicon oil stove. SAXS experiments were performed on a D/max-rA diffractometer equipped with a four-slit collimation system and a 12 kW rotating anode x-ray source. The samples were scanned with a step size of 0.02° and dwell time of 10 s from 0.1° to 1.5°.

3. Results

For an ideal two-phase structure having sharply defined phase boundaries, the asymptotic behaviour of the SAXS intensity curve was found to obey Porod's law[6–8]

$$\lim_{s\to s_{\max}}[s^3J(s)] = K'_{\mathrm{P}}, \qquad (1)$$

where s=2sinθ/λ, 2θ is the 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. $K'_{\rm P}$ is Porod's constant. One can use Eq.(1) to plot the $s^3J(s)$ versus s profile, and it will show that $s^3J(s)$ approaches a constant at large values of s. The asymptotic behaviour of the scattering curve is not affected by the particle size distribution or the particle shape. It depends only on the total surface area of the constituent in the sample.

Figure 1 shows a plot of $s^3J(s)$ versus s for the Al–7.87at% Li alloy samples aged at 463 K for 16, 32 and 64 h, respectively. It is clear from Fig.1 that Porod's law is not satisfied for 16 h ageing. In this case, Ruland[9] and Koberstein *et al.*[10] considered that the existence of a diffuse phase boundary or a transition zone between scattering particles and the matrix will cause a depletion of high-angle scattering and result in a negative slope of the plots of $s^3J(s)$ versus s. This is because the electron density does not change abruptly, but changes gradually over a certain range between the two phases. This structural model is called a non-ideal two-phase structure. As in previous studies,[3−5] we can determine the thickness of the transitional zone which formed for 16 h ageing; the thickness is 4.51 nm.

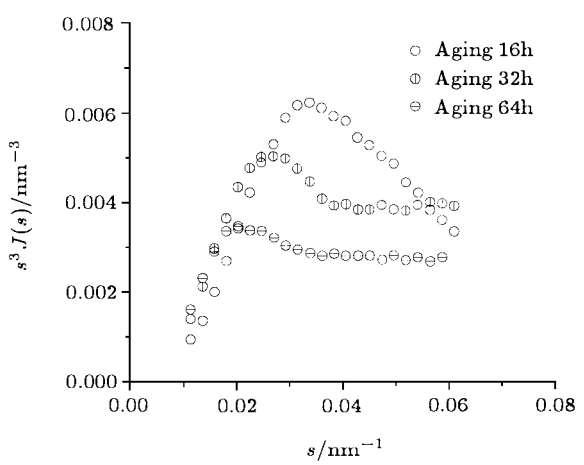


Fig.1. $s^3J(s)$ versus s plots. Al–7.87at% Li alloy aged at 463 K for 16, 32 and 64 h.

It is also seen from Fig.1 that Porod's law can be satisfied for samples aged for 32 and 64 h. It indicates that the diffuse phase boundary or a transition zone between the δ' phase and matrix disappears gradually with an increase of ageing time, while a sharply defined phase boundary forms during the later stages of 32 and 64 h of ageing. The values of Porod's constant ($K'_{\rm P}$) are equal to 0.0038 and 0.0027 nm^{-3}, respectively.

The constant $K'_{\rm P}$ is related to a specific inner surface, $S_{\rm P}$, by the following equation[11]

$$S_{\rm P} = 8\pi K'_{\rm P}/[I_{\rm e}(\Delta\rho)^2 V], \tag{2}$$

where $I_{\rm e}$ is the scattering intensity of an electron, $\Delta\rho$ is the electron density difference between the particle and the matrix, and V is the volume of the sample irradiated by x-rays. $I_{\rm e}$, $\Delta\rho$ and V are constant under unchanged experimental conditions. According to the values of $K'_{\rm P}$, we can obtain

$$S_{\rm P32h} : S_{\rm P64h} = K'_{\rm P32h} : K'_{\rm P64h}$$
$$= 0.0038 : 0.0027 = 1 : 0.71.$$

Clearly, after the sharply defined phase boundaries have formed, the specific inner surface ($S_{\rm P}$) gradually decreases with ageing time.

In addition, comparing Fig.1 with our previous results,[3−5] we notice that transition zone between the δ' phase and matrix disappears more quickly (32 h) than that of samples of Al–9.74at% Li alloy (over 64 h), 8090 alloy (95 h) and 1420 alloy (100 h).

Figure 2 shows a plot of $\ln J(h)$ versus h^2 for samples of the Al–7.87at% Li alloy aged at 463 K for 16 and 64 h. According to Guinier's law,[12] the scattering intensity of a long-slit collimation system is

$$J(h) = J_{\rm e} N n^2 \exp\left(-\frac{h^2 R_{\rm G}^2}{3}\right), \tag{3}$$

where $J_{\rm e}$ is the scattering intensity of an electron, $h{=}4\pi\sin\theta/\lambda$, $\theta = \varepsilon/2$, ε is the scattering angle, N is the number of particles, n is the total number of electrons in the particle, and $R_{\rm G}$ is the gyration radius of the particle. We can calculate the gyration radius of δ' particles from the slope of the $\ln J(h)$ versus h^2 curve. From Fig.2, one can see that the Guinier curve of the sample aged for 16 h satisfies a straight line within the low-angle range (h^2 <0.15 nm^{-2}), and the value of gyration radius is 9.16 nm. This value should be considered as the electronic gyration radius of both δ' particles and the transition zone about their centre of electron mass. However, one should also note that the Guinier curve of the sample aged for 64 h has a curvature at h^2=0.08 nm^{-2}. It is shown that some larger and smaller δ' particles coexist in the sample aged for 64 h. In this case, one can determine approximately the gyration radius of the large and small δ' particles from the different slopes of $\ln J(h)$ versus h^2 curves.[12,13] For the sample aged for 64 h, the largest gyration radius of δ' particles is 15.74 nm and the smallest is 3.75 nm.

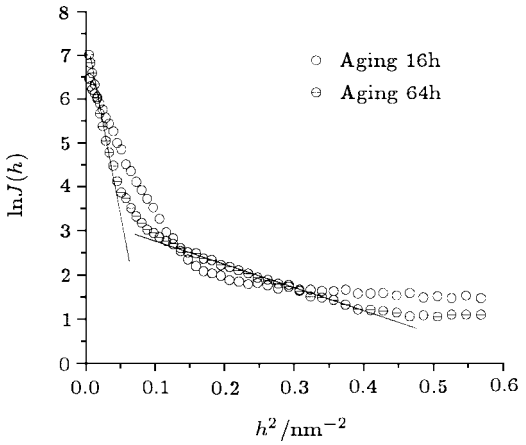


Fig.2. $\ln J(h)$ versus h^2 plots. Al–7.87at% Li alloy aged at 463 K for 16 and 64 h.

4. Discussion and conclusion

In our previous investigations we have reported on the interfacial characteristic of the δ' phase in binary Al–Li alloy, 8090 alloy and 1420 alloy in the ageing process. These results have shown that the precipitation and coarsening of δ' particles could be interpreted by a spinodal decomposition process. In this paper we have also found similar results for the Al–7.87at% Li alloy.

4.1. The formation of δ' particles in the ageing process

According to the above results, there is a transition zone between the δ' phase and matrix in the first stage of ageing (16 h). The existence of a transition zone shows that the δ' particles precipitate via spinodal decomposition, and they precipitate via a homogeneous spinodal reaction, but not via a nucleation reaction in the ageing process. The established spinodal decomposition theory[14] can help us to understand the formation of δ' phase particles. At first, the supersaturated solid solution matrix is separated into regions low in lithium and regions rich in lithium through diffusion. Then the development of the δ' particles via modulation in concentration of the constituents may be associated with an increase of lithium concentration in the region rich of lithium until they are enlarged to form discrete δ' particles. Because the lithium atom concentration of the sample is lower than that of the samples used in Refs.[3]–[5], the transition zone would disappear quickly with an increase in the ageing time.

4.2. The growth of δ' particles in the ageing process

According to the spinodal decomposition theory and the above results, the coarsening process of δ' particles is to eat up the transition zones through diffusion in the initial 32 h of ageing, and then swallow up each other after the initial 32 h. This is why, when the sharply defined phase boundaries form in δ' particles, the specific inner surface (S_P) decreases and the larger and smaller δ' particles coexist at the same time. Hence, the coarsening process of δ' particles should be divided into two stages. The first stage is the growth of δ' particles by eating up the transition zones, and the second stage is the growth of δ' particles via swallowing up each other.

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