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Characterization of the Interface between δ′ Phase and Matrix in 1420 Alloy by SAXS

C. Zhi-Gang

Beijing Aeronautical Technology Research Center, Beijing 100076, People's Republic of China

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Abstract

The interface between δ′ phase and matrix in 1420 alloy, which was processed by aging treatments and retrogression and reaging (RRA) treatments, has been characterized by small angle X-ray scattering (SAXS) technique. The results indicate that there exists a transition zone between δ′ phase and matrix for precipitates produced by the two heat treatments. The existence of a transition zone indicates that the δ′ phase forms by spinodal decomposition. These results also indicate that the RRA treatments can make the transition zone disappear quickly.

1. Introduction

The Al-Li alloys have received considerable attention because of their potential applications for aircraft structure. The precipitation of the δ′ phase in binary Al-Li alloys has become a subject of many investigations for its interesting phase transformations. It has been shown in the early studies [1] that the δ′ phase is an ordered phase with a $L1_2$ 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 recently interpreted as congruent ordering and spinodal decomposition [2]. Recently, we have reported the interfacial characteristic of δ′ phase in binary Al-Li alloy [3], 8090 alloy [4] and 1420 alloy [5]. The results also showed that the precipitation and coarsening of δ′ could be interpreted by spinodal decomposition.

Aluminum-magnesium-lithium 1420 alloy, which was developed in the former USSR in the 1960s for aerospace application such as the fully welded MIG-29 wing box, has excellent lightweight, weldable, corrosion resistant and strength. The 1420 alloy is a typical precipitation-hardening alloy. Its precipitation aging sequence is

$$\text{Supersaturated solid solution} \rightarrow \delta'(Al_3Li) \rightarrow \text{T-phase } (Al_2MgLi).$$

As in binary Al-Li alloys, 1420 Al-Li alloy is primarily strengthened by the homogeneous precipitation of metastable δ′(Al_3Li). The equilibrium T-phase(Al_2MgLi) has been found to occur on grain boundaries.

As the main hardening and strengthening phase, the precipitation of δ′(Al_3Li) in 1420 Al-Li alloy has been the subject of many investigations [5, 6].

In the present investigation we have characterized the interface between δ' phase and matrix in 1420 alloy, which was processed by (1) aging, and (2) retrogression and reaging (RRA) treatments, by the Small-Angle X-Ray Scattering (SAXS) technique.

2. Experimental

Aluminum-magnesium-lithium alloy 1420 was employed in this study. The composition of this alloy is shown in Table 1.

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

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

After cold rolling to 1.2 mm in thickness, the samples were cut to the size of 40×10×1.2 mm and solutionized in salt bath for 20 min at 700K and subsequently quenched into ice water. Isothermal aging treatments were performed at 433K for 5h, 10h and 100h in silicon oil. The RRA treatment consists of three stages, including (1) isothermal aging treatment performed at 433K for 32 h in silicon oil, (2) retrogression treatment in salt bath at 593K for 5 min and subsequently quenched in ice water, and (3) reaging treatment at 433K for 5h, 48h and 80h in silicon oil. The samples were mechanical polishing 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 equipped with a four-slit collimation system and a 12kW rotating anode X-ray source. The samples were scanned with a step size of 0.02° and dwell time of 10 seconds 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 SAXS intensity curve was found to obey Porod's law [7, 8]:

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

where $s = 2\sin\theta/\lambda$, 2θ is the scattering angle, λ is the wavelength of X-rays, $J(s)$ is the smeared SAXS intensity obtained experimentally using a long-slit collimation system, and K'_p is Porod's constant. One can use equation (1) to plot $s^3J(s)$ versus s profile and this equation shows that $s^3J(s)$ approaches constant at large values of s.

The particle size distribution or the particle shape does not affect the asymptotic behavior of the scattering curve. It depends only on the total surface area of the matter in the sample.

Figure 1 shows plot of $s^3J(s)$ *versus* s for the 1420 (Al-Mg-Li) alloy aged at 433K for 5, 10h and 100h. Clearly, Figure 1 shows that Porod's law is not satisfied in the early stage of aging, while Porod's law is satisfied for samples aged 100h.

For the negative slope, Ruland [9] and Koberstein et al. [10] think that the existence of a diffuse phase boundary or a transition zone between scattering particle and matrix causes a depletion of high-angle scattering and result in a negative slope of the plots of $s^3J(s)$ *versus* s, and this is because the electron density does not change abruptly, but changes

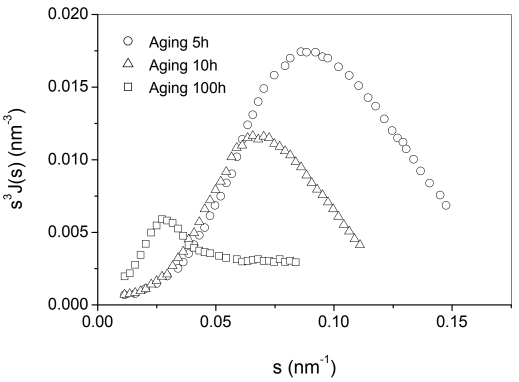

Figure 1: $s^3J(s)$ *versus* s plots. 1420 alloy aged at 433K for 5, 10h and 100h.

gradually over a certain range between the two phases. This structural model is called a non-ideal two phase structure.

For non-ideal two phase structures, Vonk [11] proposed a modified form of the Porod's law, with a linear electron-density gradient across the interface, and this is given by

$$\lim \; J(s) = \frac{k_p^{'}}{s^3}\left(1 - \frac{2\pi^2 E^2 s^2}{3}\right) \qquad (2)$$

where *E* is the thickness of the transition zone. One can use Equation (2) to determine the thickness *E* from the slope and the intercept of *sJ(s) versus* s^{-2} curve. Figure 2 shows a plot of *sJ(s) versus* s^{-2} for samples aged at 433K for 5 and 10h. The thickness values obtained are 2.18 nm and 3.07 nm, respectively. Moreover, in order to compare the thickness of the interfacial layer with the dimensions of the δ' particles, the values of Guinier radius were evaluated from the Guinier plots [8].

The values are equal to 4.36 and 6.19 nm for samples aged at 5 and 10h, respectively. Because the dispersive interfacial layer affects the scattering intensity, the Guinier radius should be considered as the electronic gyration radius of both the δ' particle and the interfacial layer about their center of electron mass.

The existence of a transition zone, which gradually disappears during aging, suggests that the δ' phase may develop and coarsen via spinodal decomposition. According to Cahn's spinodal decomposition theory [12], we think that the δ' phase forms by a homogeneous spinodal reaction and not by a nucleation reaction.

Figure 3 shows plot of *$s^3J(s)$ versus s* for samples reaged at 433K for 5, 48h and 80h. Clearly, Figure. 3 shows that Porod's law is not agreed in the early stage of aging, while Porod's law is satisfied for the samples aged 48h. One can also use Equation (2) to determine the thickness E in samples reaged for 5 h.

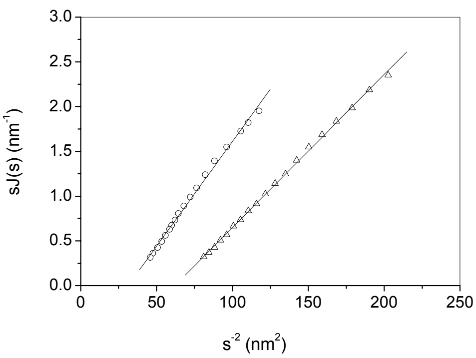

Figure 2: sJ(s) *versus* s^{-2} plots. Symbols as in Figure 1. The data in Figure 2 correspond to Porod's zone in Figure 1.

Comparing with the Figure1 and the Figure3, we notice that the transition zone between δ′ phase and matrix disappears more quickly (48 h) in RRA treatments than in aging treatments (100 h). This is because the retrogression temperature (593K) was higher than aging temperature (433K), the transitional interfacial zone and δ′ phase would disappear in the retrogression treatment. However, the δ′ phase did not disappear completely because the retrogression temperature (593K) lower than solution temperature (700K). In the reaging treatment, new particles of the δ′ phase precipitated. The new and the remaining δ′ phase particles coexist in the sample at the same time, so the number of the δ′ phase particles precipitated during RRA treatments was more than that in samples with a single aging treatment.

Because the lithium concentration in the sample is constant, we expect that the transition interfacial zone disappears more quickly during the coarsening of δ′ particles.

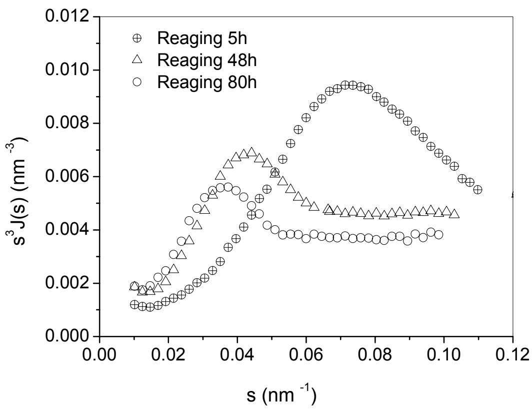

Figure3: $s^3J(s)$ *versus* s plots. 1420 alloy reaged at 433K for 5, 48 and 80h.

4. Conclusion

1) A transition zone exists between δ′ phase and matrix in aging and RRA treatments. The existence of a transition zone shows that the δ′ particles of in 1420 alloy precipitate by spinodal decomposition, but not by a nucleation reaction, in RRA treatment process.
2) Retrogression and reaging (RRA) treatment can makes the transition zone disappear quickly.

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