PRECIPITATION OF S′ PHASE IN AN 8090 Al–Li ALLOY①

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ABSTRACT

The precipitation of S′ phase in an 8090 Al–Li alloy under various aging conditions was investigated by transmission electron microscopy. The results show that a small quantity of S′ precipitates precipitated on the grain and subgrain boundaries for the specimens aged to peak hardness during single aging. Cold working prior to aging promoted the widespread precipitation of S′ phase because of increasing dislocation density. In natural aging the vacancies bound by Li atoms were released and formed dislocation loops. S′ precipitates preferentially nucleated and grew on the dislocation loops.

Key words: Al–Li alloys S′ phase aging precipitation

1 INTRODUCTION

The development of aerospace industry requires material scientists to supply structural materials of much lower-density and higher strength. The studies of Al–Li based alloys have been developed rapidly in recent years to meet this demand. The density of aluminum is reduced by about 3% for each weight percent addition of lithium, while the Young′s modulus is increased by about 6%. If Al–Li alloys are used as the replacement of 2024 and 7075 commercial alloys, the weights of aircraft and spacecraft components could be reduced by about 10～15%. However, the toughness and ductility of Al–Li alloys are lower than those of 2000 and 7000 series aluminum alloys. These characteristics have been attributed one or several of the following phenomena: (a) the intense coplanar slip associated with shearable precipitates; (b) localization of strain in a soft precipitate free zones adjacent to grain boundaries; (c) the presence of grain boundary precipitates; (d) grain boundary segregation of tramp elements such as sodium, potassium and hydrogen [1–2].

The present ways to improve the ductility and fracture toughness of Al–Li alloys are mainly by the optimization of heat treatment conditions, decreasing impurities contents and addition of alloying elements, such as copper and magnesium. It has been reported that in 8090 and 2091 Al–Li alloys widespread precipitation of fine laths of S′ phase can promote homogeneous deformation, therefore fracture toughness was improved [3–5]. Thus, it is important to understand the mechanism of nucleation and growth of S′ phase and effects of heat

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treatment conditions on S′ precipitation for the improvement of properties in Al–Li alloys. The purpose of this paper is to investigate the microstructural development in an 8090 Al–Li alloy under various aging conditions by transmission electron microscopy. The emphasis has been placed on determining the effects of aging time, natural aging and cold working prior to aging on the precipitation of S′ phase in the alloy studied.

2 EXPERIMENTAL

The chemical compositions of the alloy used in this investigation were Al–2.7, Li–1.2, Cu–0.9, Mg–0.14, Zr–0.15, Fe–0.15, Si–0.05 (wt–%). An ingot was scaled, hot and cold rolled to 2 mm sheet. The specimens were solution treated at 530 ℃ for 40 min and quenched into ice-water. In order to prove depletion of Li atoms during solution treatment, the specimens were sealed into heat-resisting glass tubes. After quenching, a series of specimens were aged at 190 ℃ for various periods immediately. Some specimens were cold rolled by 2 ~ 15% after quenching and then aged at 190 ℃ for 16 h. Some others were aged at room temperature for 7 d before aging at 190 ℃. The specimens for the examination using transmission electron microscopy were prepared by the twin jet electropolishing technique in a 33 vol. –%HNO_3 and 67 vol. –% CH_3OH solution at –20 ℃. TEM observation was carried out using H800 with an accelerating voltage of 200 kV.

3 RESULTS

3.1 *The Precipitation of S′ Phase in Single-Step Aging*

TEM observation showed that the δ′ precipitation was very fast. In the early stage of aging widespread and uniform precipitation of very fine δ′ particles was observed. In contrast to δ′ precipitation, the precipitation of S′ phase is very sluggish, before peak-aging S′ precipitates were not found. For the specimens aged to peak hardness at 190 ℃, a small amount of S′ phase heterogeneously nucleated on grain and subgrain boundaries was observed, Fig. 1 (a). With increasing aging time the number of S′ precipitates in the matrix increased. Fig. 1 (b) showed a larger amount of lath-shaped S′ precipitates together with coarser δ′ particles in the specimens aged at 190 ℃ for 100 h.

3.2 *Effect of Natural Aging on the Precipitation of S′ Phase*

The TEM observations showed that the

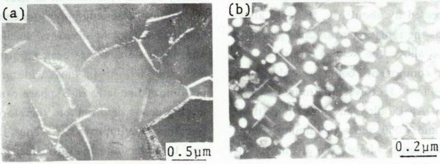

Fig. 1 Dark field images of S′ precipitates in single aging

(a)—aged for 16 h at 190 ℃; (b)—aged for 100 h at 190 ℃

precipitation of S′ phase in the specimens naturally aged and artificially aged was different from that in the specimens without natural aging. Fig. 2 is the dark field images of S′ precipitates in the specimens aged at 190 ℃ after natural aging for 7 d. As shown in Fig. 2 (a), for the specimens aged at 190 ℃ for 5 h, a larger amount of lath-shaped S′ precipitates precipitated on dislocation loops, some dislocation loops decorated by S′ laths were overlapped each other. With increasing aging time S′ precipitates grew and the traces of dislocation loops became nuclear, see Fig. 2 (b). On the contrary, for the specimens directly aged at 190 ℃ without natural aging the dislocation loops decorated by S′ phase were not found. This implies that natural aging prior to artificial aging promoted the precipitation of S′ phase because of enhancing the formation of dislocation loops.

3. 3 *Effect of Cold Working Prior to Aging on S′ Precipitation*

For the specimens which were cold rolled after solution treatment and then aged at 190 ℃, the rate of the precipitation of S′ phase became fast. As shown in Fig. 3 a, some S′ precipitates were present in the matrix for the specimens aged at 190 ℃ for 5 h after rolled by 5%. In peak aged condition, the uniform and widespread S′ precipitates were present. TEM micrographs of precipitates clearly show that the density and homogentity of S′ precipitates increased as the degree of cold rolling increased from 2% to 8%. For the specimens which were cold rolled by 15% the dispersion of S′ phase became inhomogeneous again and the density of S′ particles decreased. This shows moderate pre-deformation can promote uniform precipitation of S′ in the matrix.

4 DISCUSSION

4.1 *Effect of Vacancies on the Precipitation of S′ Phase*

It is known that in Li-free Al–Cu–Mg alloys the nucleation of S′ takes place preferentially on the high density of dislocation loops and helices produced by the condensation of vacancies on quenching after solution treatment[6–7]. Such defects are absent in the Li–containing alloys, this may be attributed to strong lithium atom to vacancy binding energy which inhibits vacancy condensation and formation of helices and loops on quenching[8]. In the alloy tested no loops or no helices after

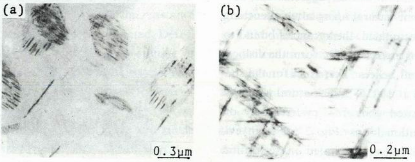

Fig. 2 Bright field images of S′ precipitates

(a)—natural aged for 7 d and aged at 190 ℃ for 5 h; (b)—natural aged for 7 d and aged at 190 ℃ for 16 h

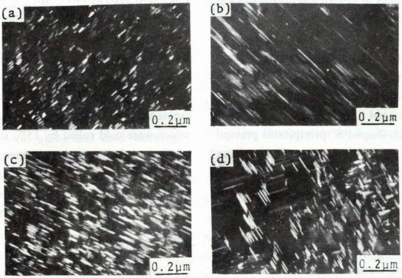

Fig. 3 S′ precipitates in the specimens pre-deformed and artificially aged at 190 ℃

(a)—5% rolling + 190 ℃ / 5 h; (b)—2% rolling + 190 ℃ / 16 h;
(c)—8% rolling + 190 ℃ / 16 h; (d)—15% rolling + 190 ℃ / 16 h

solution treatment and quenching were observed. Therefore, in the specimens aged directly to peak hardness at 190 ℃ after quenching, there was only a small amount of S′ phase heterogeneously nucleated on grain and subgrain boundaries, see Fig. 1 (a).

On the other hand, since the precipitation of δ' was rapid, even rapid cooling during quenching can not inhibit the precipitation δ' phase [9] too. So it can be suggested that during subsequent natural aging after quenching δ' will be precipitated, the vacancies bound to lithium atoms are released to form the dislocation loops and helices. Therefore, for the specimens aged at 190 ℃ after natural aging, S′ phase nucleated and grew preferentially on these dislocation loops. Fig. 2 (a) is an evidence that S′ laths precipitated on dislocation loops condensed by vacancies. In addition, vacancies may interact with Cu, Mg atoms and accelerate diffusion process of these atoms and make Cu, Mg atoms near dislocation loops. This is favourable for the precipitation of S′ phase on these sites. Fig. 4 shows S′ precipitation free zones along grain boundaries in the specimens naturally aged and then aged at 190 ℃. It is associated with the depletion of vacancies near the grain boundaries acting as an efficient sink for vacancies.

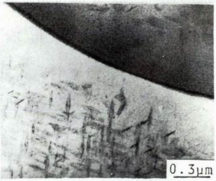

Fig. 4 S′-precipitation free zones in grain boundaries

The release of vacancies occurs during

aging at 190 ℃ after quenching immediately, but no dislocation loops were observed. The reason is perhaps that when the specimens were aged at higher temperature, vacancies can diffuse rapidly to the surfaces, grain boundaries and dislocation loops. Even if the dislocation loops were formed, they might disappear quickly on account of the action of the linear tension of dislocations. So the dislocation loops were notable to become the stable nucleation sites of S′ phase and thus dislocation loops decorated by S′ were not observed in the specimens aged at 190 ℃ directly. As reported by Alekseyev *et al*[10], the growth of S′ particles was accompanied by the absorption of vacancies. This is the reason why the transition of vacancies from loop to interphase face may be seen as a reduction in the areas of the loops when S′ particles on a loop increase in size and quantity. This must finally end in total disappearance. The results of this observation are in agreement with the analysis and observation by Alekseyev *et al.*

4.2 *Effect of Dislocation on S′ Precipitation*

It is known that the nucleation of precipitates occurs preferentially on dislocations because when dislocations are present in the matrix their elastic strain accommodates the strain energy of the precipitate and the resistance to growth of the precipitates is lowered. Deformation prior to aging introduces a number of dislocations in the matrix to produce adequate heterogeneous nucleation sites, thus the precipitation of S′ phase was promoted. Fig. 5 shows S′ precipitates nucleated and grew at a single dislocation line. From this, it can be seen that lath-shaped S′ precipitates are parallel to each other. S′ phase is orthorhombic with a = 4.00, b = 9.23 and c = 7.14 Å [11]. The value of crystallographic parameter a of S′ precipitate is about the same as that of aluminum lattice planes. However, the misfit values along b and c axes are large. There is largest tensive stress field in the matrix along b axis of the precipitate. On the other hand, a dislocation can best accommodate the lattice misfit between a precipitate and the matrix if the Burgers vector of the dislocation is parallel to the misfit vector of the precipitate [12]. Therefore, it is considered that a series of parallel S′ laths precipitated along a single dislocation line is the result of lowering the strain energy.

Fig. 5 Precipitation of S′ phase along single dislocation line

As seen from above, S′ precipitation tends to be denser and more homogeneous as the degree of cold working increases from 2% to 8%. This is attributed with the dislocation structures introduced by cold working. Fig. 6 is TEM micrographs of dislocations induced by deformation in the specimens after quenching. From this it can be seen that when the amount of cold rolling increased from 2% to 8%, the density and homogeneity of dislocations increased. However, when the amount of deformation was of more large value, such as 15%, the homogeneity of dislocation dispersion decreased and there were some dislocation cells. So S′ distribution became again

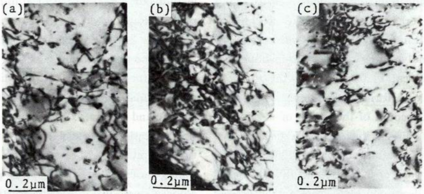

Fig. 6 Dislocation structures introduced by cold working

(a)—2% rolling; (b)—8% rolling; (c)—15% rolling

inhomogeneous. This implies that in order to develop a sufficiently dense distribution of S′ precipitates, proper predeformation is required.

5 CONCLUSIONS

(1) In single-step aging, the precipitation of S′ phase in 8090 alloy is sluggish, and a small number of S′ precipitates nucleated and grew at grain and subgrain boundaries under peak-aged condition at 190 ℃, while homogeneous nucleation of S′ phase occurs under the overaged condition.

(2) During natural aging before artificial aging at 190 ℃ vacancies bound by Li atoms were released to form dislocation loops, S′ precipitates were nucleated and grew preferentially on these dislocation loops. Growth of S′ particles is accompanied by the absorption of vacancies, thus the areas of the loop are diminished until total disappeared.

(3) Pre-deformation prior to artificial aging promoted the precipitation of S′ phase because of the increasing density of dislocations. When degree of cold working was increased up to 8%, the density and homogeneity of S′ precipitates increased. Large deformation decreased the homogeneity of S′ distribution again because the dislocations introduced by deformation tend to form tangle arrangement and cell structures.

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