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DIAGRAMS OF PHASE TRANSFORMATIONS IN MATRIX AND ON GRAIN BOUNDARIES ON AGEING IN Al-Li-Cu AND Al-Li-Cu-Mg ALLOYS

V. N. Ananiev, E. A. Lukina
All-Russia Institute of Light Metals, Moscow, Russia

Introduction

Artificially aged Al-Li alloy semiproducts feature grain boundary (GB) failure. One of the main factors contributing to the GB failure of Al alloys is the GB precipitates formation during supersaturated solid solution decomposition [1]. A number of stable phases formed during ageing at grain boundaries in Al-Li-Cu and Al-Li-Cu-Mg alloys was mentioned in literature [2,3]. Among such phases are the following: $\delta(\text{AlLi})$, $T_1(\text{Al}_2\text{LiCu})$, $T_2(\text{Al}_6\text{CuLi}_3)$, $S(\text{Al}_2\text{CuMg})$, $S_1(\text{Al}_2\text{LiMg})$. Types of the forming phases, their sizes and amount depend on both chemical composition of alloys and heat treatment conditions.

This paper covers study of solid solution decomposition at grain boundaries in three alloys of Al-Li-Cu-Mg and Al-Li-Cu systems to determine temperature and time ranges of the formation of the GB precipitates of various types during artificial ageing.

Experimental procedure

Experiments were conducted on 1430, 1440 and 1451 alloy sheets. Table I shows chemical compositions of the alloys. 1 mm thick sheets were produced by cold rolling with intermediate quenching to form recrystallized structure in these alloys. The sheets were solution treated and artificially aged in the air furnace. Ageing temperature and time were varied in the ranges of 120-250°C and 4-24 hrs respectively. The thin specimens for TEM were prepared by the double jet electrochemical polishing in a solution of 15% nitric acid - methanol cooled to -25°. Foils were examined using JEM-100CX II TEMSCAN electron microscope operating at an accelerating voltage 100 kV.

Table I. Chemical compositions

Alloy	Al	Li	Cu	Mg	Zr
1451	bal.	1.53	2.7	0.05	0.12
1430	bal.	1.6	1.6	2.7	0.09
1440	bal.	2.14	1.2	0.89	0.11

Results and discussion

Initial state

Grain boundaries in all three alloys in as-quenched and naturally aged condition were free from any precipitates. The very fine metastable δ' (Al₃Li) phase particles of 4 nm (max) in size precipitated homogeneously in the matrix of 1430 and 1440 alloys. Relative intensity of superstructure reflections was higher in 1440 alloy than that in 1430 alloy due to higher volume fraction of the precipitated δ' -phase. Solid solution decomposition with the formation of the δ' -phase at room temperature was not observed because of too lower lithium content of 1451 alloy sheet.

In all three alloys the Zr additions from 0.09 to 0.12 wt.% led to the formation of coherent spherical β' Al₃(Zr,Li) particles. Their nucleation and growth occur at high temperatures of 450-550°C, so during artificial ageing β' particles did not change but provided heterogeneous nucleation sites for δ' and induce the formation of "composite" δ'/β' particles [2].

Alloy 1430

With an increase in temperature and/or ageing time, the matrix δ' -particles coarsened. At the temperature of 150°C all matrix dislocations were decorated with rod-shaped, partially coherent particles of the S'(Al₂CuMg) phase [2]. Besides, the rod-shaped particles of the S(Al₂CuMg) phase formed at the high-angle grain boundaries. Their diameters were higher by an order of magnitude than those of the matrix S'-phase particles and were equal to 20-40 nm. According to TEM examinations mean length of grain boundary per one S particle was about 0.24-0.25 μm after ageing 150°C, 24 hrs.

After 16 hrs ageing at temperature of 175°C the δ' precipitate free zones (PFZ) at grain boundaries appeared. The PFZ formation was caused by precipitation of the stable GB S₁(Al₂LiMg)-phase (Fig. 1) [2]. S₁ particles had irregular shape and their maximum size was 150-200 nm. At temperatures above 200°C S₁-phase predominated not only at GB but also in the matrix.

The influence of the ageing time and temperature on GB and matrix phase composition in 1430 alloy is shown in Fig. 2 on the diagrams of phase transformations on ageing. It is seen that at temperatures of 150°C and below there are no Li-containing particles on the grain boundaries in 1430 alloy.

Alloy 1440

We didn't find any indications of the GB phase nucleation in 1440 alloy on artificial ageing at temperatures of 150°C and below. As in 1430 alloy the matrix δ' -particles coarsened on ageing. At the temperature of 150°C matrix dislocations and dislocation loops were decorated by the rod-shaped particles of phase S'(Al₂CuMg).

As the temperature increased up to 175°C, the S(Al₂CuMg) (Fig. 3a) and still very rare T₂(Al₆CuLi₃) precipitates appeared on the high-angle grain boundaries. The narrow δ' PFZ of 0.15-0.20 μm in width formed near T₂-particles. At the same time δ' PFZ was absent near the S-particles. Area density of the S-phase varied in wide limits and depended on the

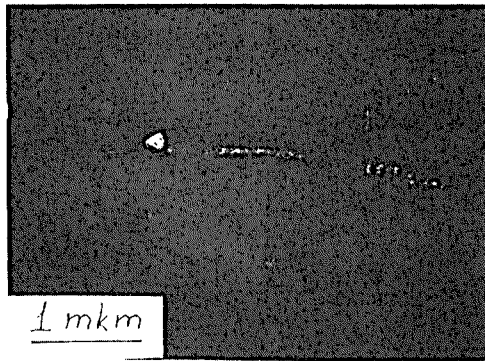


Fig. 1. GB phases S(Al_2CuMg) and $S_1(\text{Al}_2\text{LiMg})$ in 1430 alloy. (170°C, 24 h.)

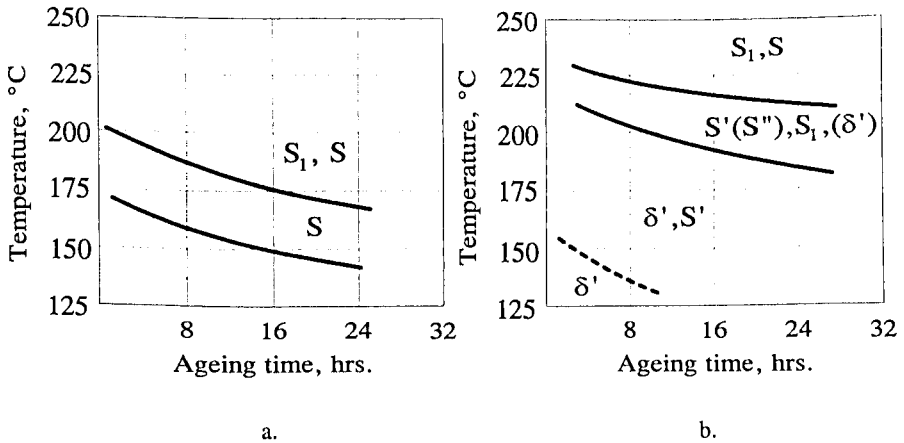
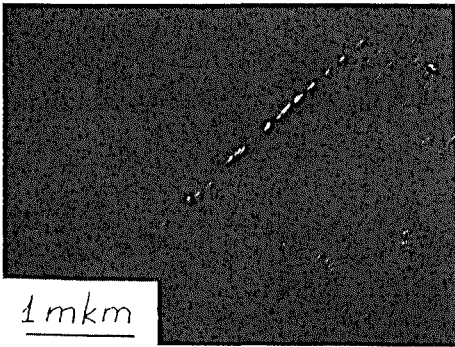
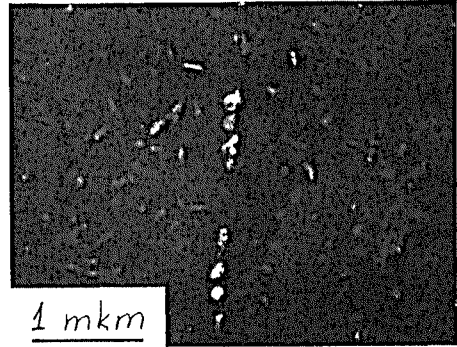


Fig. 2. 1430 alloy: phase compositions at GB (a) and in matrix (b) as a function of ageing time and temperature.



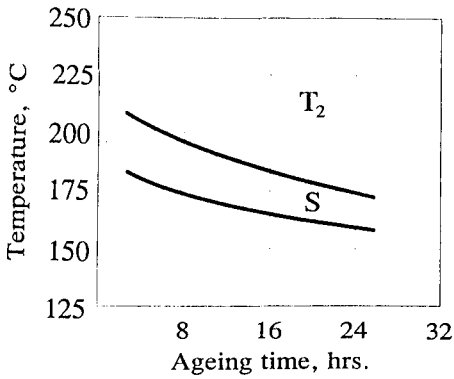
a.



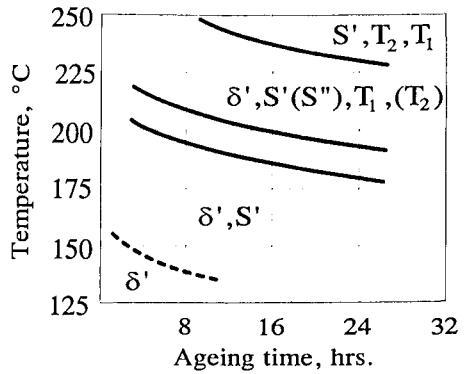
b.

Fig. 3. GB phases in 1440 alloy.

a) $S(Al_2CuMg)$, 175° , 24 h., b) $T_2(Al_6CuLi_3)$, $150^\circ C$, 24 h.



a.



b.

Fig. 4. 1440 alloy: phase composition at GB (a) and in matrix (b) as a function of ageing time and temperature.

type and local orientation of the particular grain boundary. The mean length of grain boundary per one S particle was equal to 0.26 mkm with minimal value being 0.14 mkm.

At elevated temperatures 200°C and above matrix δ' -particles rapidly coarsened. In addition to the heterogeneously nucleated S'-phase we found homogeneously nucleated in matrix needle-like S'_{hom} (Al_2CuMg) precipitates. Their growth direction was also $\{100\}_\alpha$ [2]. The T_2 -particles were surrounded by δ' PFZ formed both in the matrix and on the grain boundaries. At the temperatures of 225°C and above only T_2 -phase was observed at the GB (Fig. 3b). After ageing 225°C, 24 hrs T_2 particles size was about 0.2-0.3 mkm and GB PFZ width was about 0.4-0.5 mkm.

The results of TEM phase analysis were plotted on the diagrams of phase transformations at GB on ageing (Fig. 4). It may be noted that like in the 1430 alloy Li-containing GB phases and δ' PFZ did not form in the 1440 alloy below the certain ageing temperature.

Alloy 1451

Phase composition of the grain boundary precipitates was studied in the ageing temperature range of 120-250°C. The T_1 -phase was found to be the low temperature GB phase in 1451 alloy (Fig. 5a). At temperature of 120°C the GB T_1 -particles were detected after 16 hours ageing. The size and quantity of these precipitates were very small (mean diameter of T_1 -plates was about 20 nm after ageing 120°C, 24 hrs.).

With an increase in temperature and/or time of ageing, amount and size of the GB T_1 phase precipitates enlarged. When the local GB orientation was close to the matrix planes $\{111\}$, the T_1 particles covered almost all GB surface (like scales). Change in the type of GB phase took place in the temperature range of 200-225°C, namely: the T_1 phase was replaced by the T_2 phase. After ageing 250°C, 24 hrs the sizes of the T_2 precipitates elongated along GB were equal to 3 mkm (length) and 0.2 mkm (thickness) (Fig. 5b). GB and matrix phase compositions of 1451 alloy are plotted in Fig. 6 as a function of the ageing time and temperature.

It is well known that precipitation of GB phases in Al-Li alloys results in the formation of PFZs at grain boundaries. These zones are free from the strengthening precipitates due to alloying component depletion [4-6]. During artificial ageing of 1451 alloy two matrix phases, δ' (Al_3Li) and θ' (Al_2Cu), nucleated. Thin plates of the semicoherent θ' -phase formed and were covered by the coherent δ' -phase envelope.

It was found that the formation of the both Li and Cu containing T_1 GB phase was accompanied by appearance and growth of two zones: δ' PFZ and θ' PFZ. Fig. 7 shows the size of these zones depending on the ageing temperature. Certain facts can be noted.

1. In all cases the width of the θ' PFZ was larger than the size of the δ' PFZ.
2. Temperature dependences of both PFZ were very similar. This correlation resulted from GB T_1 -phase precipitation accompanied by not only lithium, but also copper depletion (in equiatomic amounts) of matrix near boundaries.
3. At low ageing temperatures (below 130°C), the θ' PFZs were observed even at the absence of the δ' PFZ. Judging by the fact that the size of the θ' PFZs did not tend to zero with decreasing of the ageing temperature, one could come to the conclusion that the size of the θ' PFZs was controlled not only by the solute depletion due to the GB T_1 phase formation, but also by the vacancy depletion mechanism which defined the "initial" PFZ size.

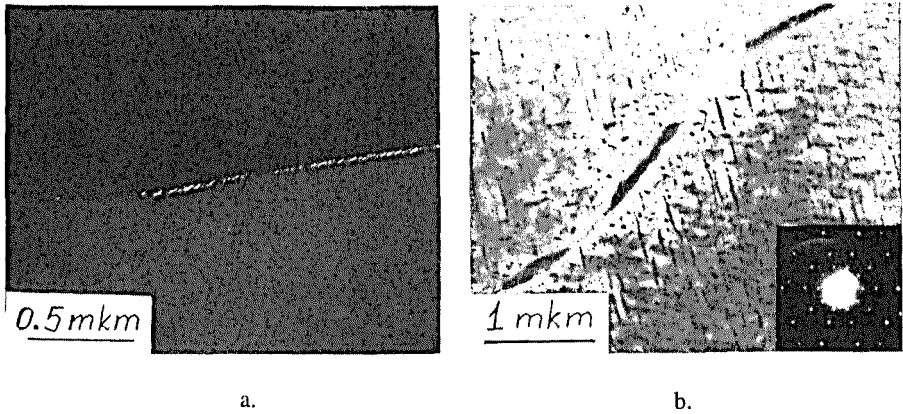


Fig. 5. GB phases in 1451 alloy.

a) $T_1(Al_2LiCu)$, 150°C, 24 h., b) $T_2(Al_6CuLi_3)$, 250°C, 24 h.

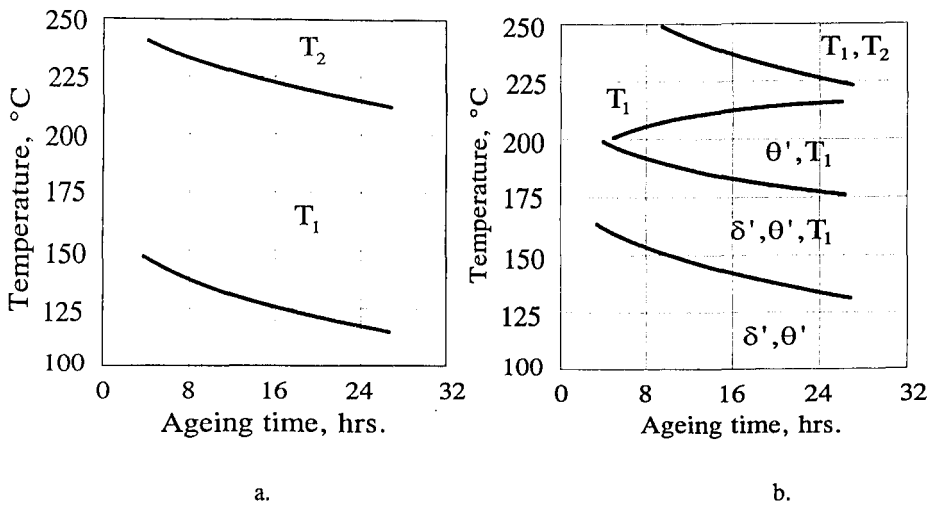


Fig. 6. 1451 alloy: phase composition at GB (a) and in matrix (b) as a function of ageing time and temperature.

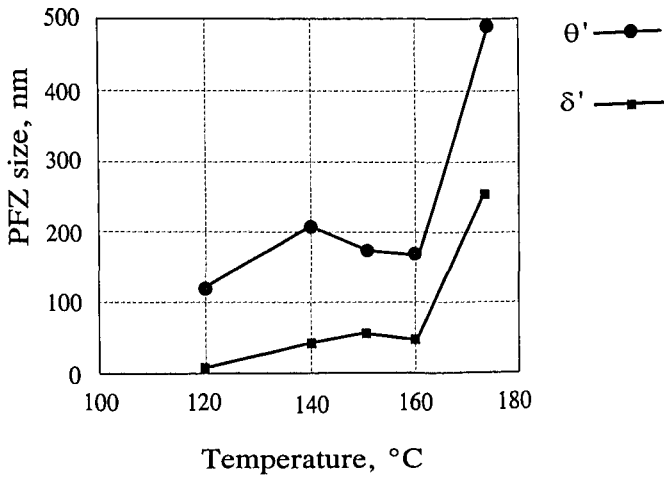


Fig. 7. δ' and θ' PFZ size as a function of ageing temperature.

4. A noticeable increase in the slope of both curves in the ageing temperature range of 160-170°C was apparently related with the intensification of solid solution decomposition at the GB with an increase in temperature.

In our opinion, when analysing the deformation localization mechanisms in Al-Li-Cu alloys it should be taken into account that apart from more or less thin layers of softened α -solid solution, rather wide layers of the $\alpha+\delta'$ phase composition exist along grain boundaries within the really used ageing temperature range. These $\alpha+\delta'$ layers are susceptible to the slip localization to the same extent as binary Al-Li alloys. Moreover, at low temperatures these quasi-binary layers are observed even at the absence of the δ' PFZ.

Conclusions

1. Solid solution decomposition at the high-angle grain boundaries was investigated in recrystallized sheets of 1430 (Al-1.6Li-1.6Cu-2.7Mg-0.09Zr), 1440 (Al-2.14Li-1.2Cu-0.9Mg-0.11Zr) and 1451 (Al-1.53Li-2.7Cu-0.05Mg-0.12Zr) alloys, artificially aged in the temperature range of 120-250°C and time range of 4-24 hrs.

2. Types of the GB phases formed in these alloys were determined. The $S(Al_2CuMg)$ and $S_1(Al_2LiMg)$ phases precipitated in 1430 alloy, while the S and $T_2 (Al_6CuLi_3)$ phases precipitated in 1440 alloy and the $T_1 (Al_2LiCu)$ and T_2 phases precipitated in 1451 alloy. GB phase compositions of all three alloys were plotted as a function of the ageing time and temperature on the diagrams of phase transformations at grain boundaries. In comparison with the decomposition occurred in 1451 (Al-Li-Cu) alloy, GB decomposition in 1430 and 1440 alloys (both of Al-Li-Cu-Mg system) featured by the absence of Li-containing low-temperature GB phases.

3. In all three alloys intensive precipitation of the GB Li-containing phases resulted in the formation of δ' PFZ along grain boundaries. 1451 alloy featured the formation of one more zone - the θ' PFZ - because of precipitation of both Li- and Cu-containing phase T_1 . θ' PFZ can exist even at the absence of δ' PFZ.

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