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# Heterogeneous Precipitation in 1424 Al–Mg–Li Alloy

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## Abstract

Heterogeneous precipitation on grain and subgrain boundaries of 1424 alloy sheets has been studied within the present work. The presence of different phase structural precipitations is typical for the grain and subgrain boundaries in addition to equilibrium $S_1$ ($Al_2LiMg$) phase. In particular, ranges, containing superfine grains are revealed. Formation of these structures is determined by the electron-structural state of lithium atom in Al-Li alloys. There are three of those states. These are substitutional atoms, having a covalent bond with close aluminium atom – covalent lithium; substitutional atoms as $Li^+$ ion – metallic lithium and interstitial atoms as $Li^+$ ion – interstitial lithium. The phase composition and structure of heterogeneous precipitations depends on the deformation and heat treatment regimes. Taking into a consideration all the three states of lithium atoms mechanism of those structures formation has been considered.

## 1. Introduction

As a rule, a banded subgrain (grain) structure is formed in sheets under a deformation and heat treatment of Al–Mg–Li alloys. The anisotropic nature of this structure influences considerably the mechanical properties of the material especially on the anisotropy of strength characteristics and elongation. One of the reasons, why the equiaxial recrystallized structure is not formed, is the fact, that grain and subgrain boundaries are immobilised with precipitates.

The tasks put forward in the present work are to discover the types of grain and subgrain precipitations formed in 1424 alloy sheets and to study a regularity of heterogeneous nucleation and growth of phases on grain and subgrain boundaries.

## 2. Material and Methods

Investigation has been carried out on the sheets of the most perspective 1424 Al-Mg-Li alloy having a high crack and corrosion resistance, a good weldability and fracture toughness [1].

The sheets of 4,5 mm thickness are manufactured by following scheme: hot deformation + cold deformation with intermediate annealing + air quenching + ageing. The air quenching is carried out after solid solution heat treatment at the temperature 530$^o$C (TG). One-step ageing in the temperature interval from 100$^o$C to 200$^o$C and three-step ageing ensuring a thermal stability of 1424 alloy under a long exposure at the temperature 85$^o$C are studied. TEM is fulfilled using JEM200CX. Thin films for TEM are prepared by jet electropolishing with the electrolyte at –38$^o$C using "TENUPOL-5". Microstructure of specimens is revealed by the chemical etching and observed using LEICA DMIRM.

## 3. Results and Discussion

The typical structure of 1424 alloy sheets after final heat treatment has fibres, "subfibres" and recrystallized grains (Figure 1).

Figure 1: Microstructure of 1424TG1 Alloy (125$^o$C, 32 h)

The layers appearing as a result of geometrical change of ingot grains have been called as fibres. Under a cold deformation with intermediate annealing thinner fibres formed inside these fibres. They can be called as "subfibres". Boundaries of fibres and "subfibres" are decorated with particles (Figure 1). These particles are thermodynamically stable at the SSHT temperature (530°C). We'll call them stable. The phase composition analysis of ithem requires a subsequent study. It is possible, that during solid solution heat treatment a recrystallization process is taking place. The rate of the grain boundary movement under recrystallization along the decorated boundaries of fibres and "subfibres" is much higher, than across them (Figure 1). Steps are formed on the grain boundaries during recrystallization (Figure 2). A size and geometry of steps is defined by the character of particles disposition, decorating the boundary. Stable particles form a bonded structure in the recrystallized grains, being a continuation of "subfibres" boundaries.


Figure 2: Recrystallized grain boundary, stable particles are noted with arrows, bright field (125°, 32 h).

Each "subfibre" is extended along a deformation direction. It is a chain of fine subgrains. A fine structure of two "subfibres" A and B and a boundary between them are presented in Figure 3.

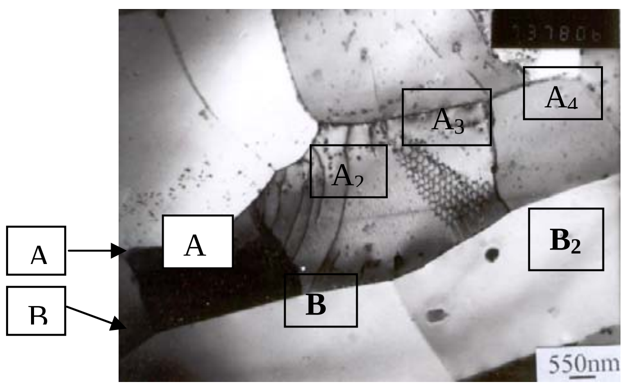
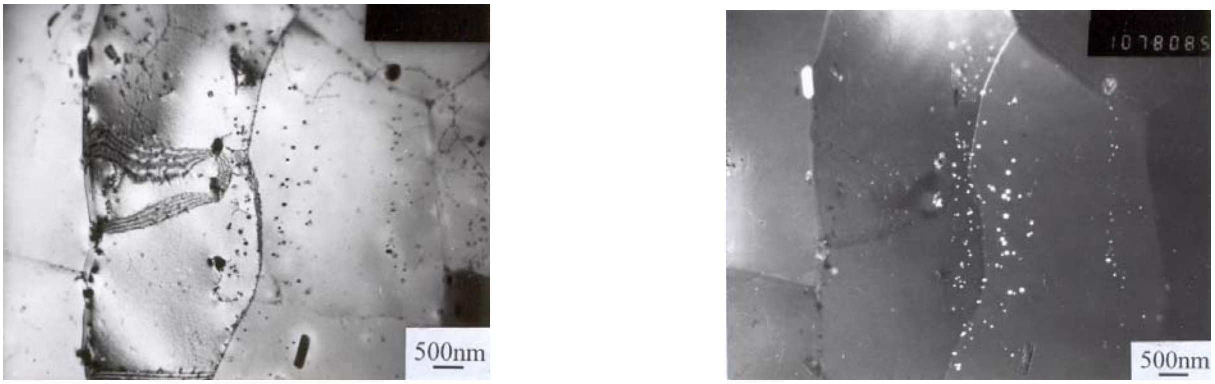



Figure 3: Bright field electron micrograph showing the structure of "subfibres".

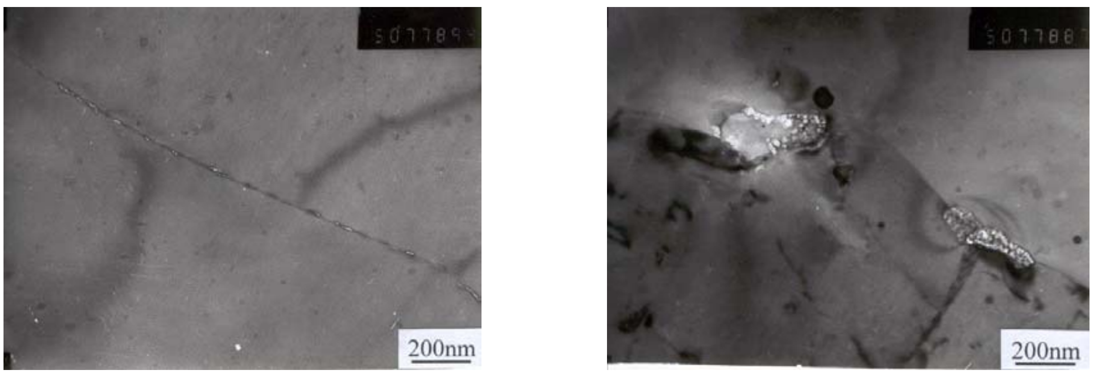
"Subfibres" consist of subgrains $A_1$, $A_2$, $A_3$ and $B_1$, $B_2$ correspondingly. Boundaries between "subfibres" A and B have misorientation angles (6-7)°. But for boundaries between subgrains in one "subfibre" (between $A_2$ and $A_3$, $B_1$ and $B_2$ and etc.) a misorientation constitutes approximately (0.2-1)°. Boundaries similar to the boundaries of "subfibres" are observed under an intensive plastic deformation and are called as knife boundaries [2], or as low-energetic dislocation structures [3].

Study of heterogeneous nucleation and growth of phases shows, that subgrain boundaries, having low misorientation angles (approximately 0.2-1°), are not decorated with precipitates as a rule. "Subfibres" boundaries are decorated with $\delta_{non}$-phase particles (Figure 4), found and studied by us earlier [4,5].




a b

Figure 4: "Subfibre" boundary: a) bright field (125°C, 32 h), b) dark field using $\delta'$ -phase diffracted spot.




a b

Figure 5: Superfine grain structures: a) uniformly located along a boundary (TX); b) generated as a result of accumulative recrystallization (TX+ 85°C, 2000 h)

Ranges, containing superfine grains are observed after three-step aging (TX) on high-angle boundaries of grains (Figure 5a). After a long low-temperature exposure more coarse grains are formed as a result of accumulative recrystallization in these ranges (Figure 5b).

## 4. Discussion

It is known [6], that lithium atoms in a solid solution of aluminum alloys may be present in three electron-structural states, determined by the type of their chemical bond (covalent or metallic) and their position in f.c.c.-lattice (substitutional atom or interstitial atom). A lot of metallic lithium is non-equilibrium in the quenched condition. Particles of $\delta_{non}$-phase contain metallic lithium [4]. While aging in temperature ranges higher than 85°C (exposure time 4000 h) and 175°C (1 h) a dislocation structure is formed near $\delta_{non}$- precipitations. It means, that every particle of $\delta_{non}$-phase becomes a center of strong elastic distortions in the matrix. Around particles substantial internal stresses appear, a relaxation of which results in formation of dislocation structure and it is observed by a weak beam method (Figure 6).

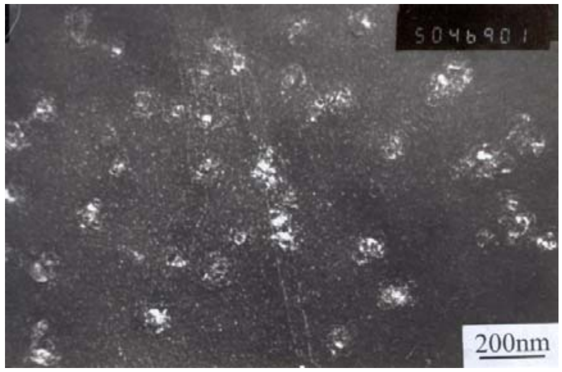



Figure 6: Weak beam (g; 3g ) revealing dislocation structure around $\delta_{non}$- precipitates (150°C, 32 h).

This process can be explained by the transformation of non-equilibrium metallic lithium in equilibrium, covalent lithium. The density of dislocations around $\delta_{non}$- precipitations increases under an increase of temperature (at annealing and aging) and exposure at aging. During heat treatment ranges with superfine grains 20-40 nm are formed near the particles, in the place of dislocation. Then inside these ranges more coarse grains grow according to a mechanism of accumulative recrystallition (Figure 5b). A constitution of these ranges was studied by local microdiffraction method [5].

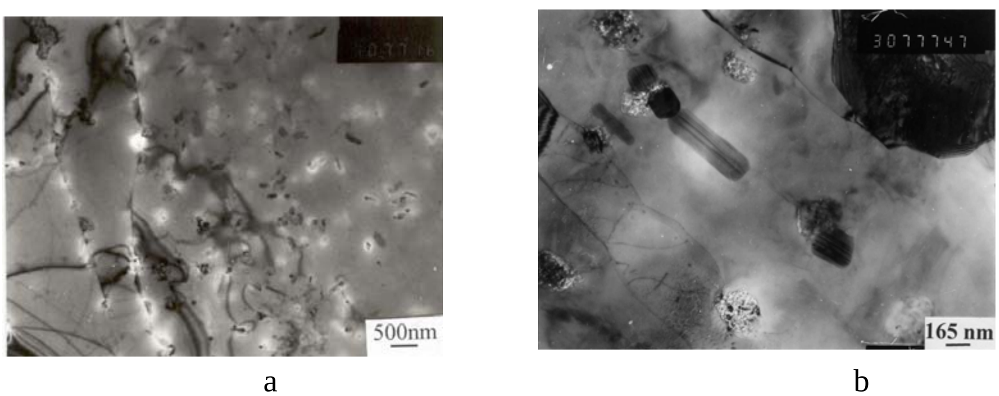



Figure 7: $S_1$- phase precipitates, light field: a) (200, 2 h); (390°C, 1 h).

At temperatures higher, than 150°C a typical heterogeneous decomposition takes place with a formation of $S_1$- phase ($Al_2MgLi$) (Figure 7). Under aging at 150°C and higher complex structural-phase formations are observed. They are the ranges, in which phases $S_1$, $\delta_{non}$ and superfine grain structure coexist simultaneously. $S_1$ may be inside superfine grain structure, and $\delta_{non}$-phase - inside $S_1$-phase [4].

Besides that, after air quenching from 530°C and age-hardening $\delta_{non}$-phase precipitates line up along high-angle and "subfibres" boundaries forming stripes (Figure 8).

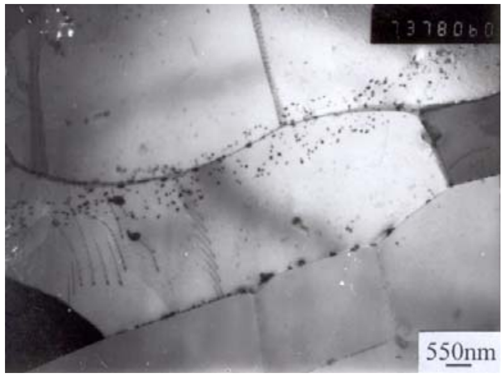



Figure 8: Stripe of $\delta_{non}$-phase particles accumulation along a "subfibre" boundary, bright field (125°C, 32 h).

The same stripes are generated along bands of stable particles inside recrystallized grains (Figure 2). It should be noted, that $\delta_{non}$-phase particles may be formed both during cooling after SSHT and aging. By diffraction method it is established, that orientation relation of $\delta_{non}$-phase lattice with a matrix both of deformed grain and recrystallized grain is the same and coincides with the one established in the previous work [4]. It means, that $\delta_{non}$-phase particles are formed after recrystallization. This may be a consequence of inhomogeneity of metallic lithium concentration, which is preserved at heating of SST and is not eliminated under forming of recrystallized grain. A formation of a certain number of non-equilibrium interstitial lithium atoms, having ion structure, under air cooling after annealing may be a reason of concentration inhomogeneity. These interstitial atoms diffuse rapidly to high-angle boundaries and in the first place on "subfibres" boundaries. During a subsequent cold deformation a great number of non-equilibrium deformation vacancies are formed in the alloy. These vacancies, while flowing to grain boundaries meet with interstitial lithium atoms and a substitutial solid solution is formed. The following operations of cold rolling, annealing and air cooling result in a growth of this effect. Under cooling after SSHT and ageing accumulations of $\delta_{non}$-phase particles enriched with metallic lithium are formed in the ranges.

## 5. Conclusions

1. The studied sheets of 1424 have a fibrous structure, elongated along the direction of deformation. The structure of the alloy after a final heat treatment is mainly non-recrystallized, partially recrystallized. Recrystallized grains are elongated along the direction of deformation.
2. In the alloy, that has been deformed, boundaries of fibres and "subfibres" are fixed with particles, which are thermodynamically stable at the temperature of SSHT up to 530°C.
3. Under ageing the ranges with superfine grain structure are generated on high-angle boundaries.
4. Under cooling after SSHT and following ageing at the temperatures lower, than 150°C, the stripes of $\delta_{non}$-phase particles accumulation are generated in the alloy.
5. At the ageing temperatures higher, than 150°C complex structural-phase formations, containing $S_1$- phase ($Al_2MgLi$) particles, $\delta_{non}$-phase and superfine grain structure are generated in the alloy.

## References

[1] J.N.Fridlyander, L.B. Khoklatova, N.I.Kolobnev, L.B. Ber, E.I.Shvechkov, K.-N.Rendigs, G.Tempus. Proceedings of Int. Cong. Advan. Mat. and Proc. Munich, Germany, 2001.
[2] M.A.Shtremel. "Alloys strength". MISiS, II, 29, 1997
[3] I.A.Mazurina, O.Sh.Sitdikov, R.O.Kaybyshev, Physics of metals and physical metallurgy, v. 94, №4, 104-112, 2002
[4] A.A.Alekseev, E.A.Lukina, M.I.Ermolova, L.B.Khokhlatova. Proceed. of ICAA8, Cambridge, UK, 2002.
[5] L.B.Khokhlatova, L.B. Ber, A.A.Alekseev, N.I.Kolobnev, O.G.Ukolova, E.A.Lukina. Proceed. of ICAA8, Cambridge, UK, 2002
[6] J.N. Fridlyander, K.V.Chuistov, A.L.Beresina, N.I.Kolobnev, "Aluminium-Lithium Alloys. Structure and Properties", Kiev: Naukova Dumka, 1992