THE HEAT TREATMENT EFFECT ON THERMAL STABILITY OF AL-LI ALLOYS AT LOW TEMPERATURES.

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ABSTRACT Variations of structure and properties of Al-Li-Mg-Sc-Zr alloy (type 1421) and Al-Li-Cu-Sc-Zr alloy (type 1460) during long low temperature exposure (LLTE) and influence to these changes of special stabilizing heat treatment were examined.

After standard ageing regimes in aluminium alloys with lithium equilibrium or nonequilibrium supersaturation of lithium in a solid solution is retained. This leads to enlargement of δ′ phase volume fraction during LLTE. Slow cooling from ageing temperature and two-stage ageing with first high temperature and second low temperature stages decreases residual supersaturation of alloys for lithium and prevents δ′ phase fine-dispersed particles formation that is typical for of supersaturated Li solid solution decomposition at exposure temperatures. Mechanical properties of sheets are growing more stable after such treatment.

Keywords: *Al-Li alloys, δ′phase, long low temperature exposure (LLTE), slow cooling, mechanical properties, stability.*

1. INTRODUCTION

On a way of the Al-Li alloys wide application in aircraft industry it is necessary to solve a number of problems. One of major — the stability of the semiproduct properties during all the airplane life cycle. The resource of perspective designs makes 30000 h, i.e. almost 3.5 years in air, thus general term of operation is estimated by decades. During this term the constructional materials are subjected to various influences, including, thermal. In this connection the properties stability of new alloys under long low temperature exposure (LLTE) is of interest.

The evaluation of alloy properties thermal stability is carried out on the basis of comparison of properties before and after exposure, accompanied by calculation of relative changes in percentage. Thermal exposures carried out at temperatures from room temperature up to 100–120°C, up to 10^4 h are usually used [1-9]. The data published show lower stability of Al-Li alloy properties under LLTE conditions in comparison with 2000, 6000 and 7000 alloys [4,6,9].

It was found [2,10] that the alloys with a lower content of alkali metal impurities (Na, K) and H were less sensitive to the effect of LLTE on fracture toughness and yield strength. This approach requires design of special casters for vacuum refining of the melt and increases the primary cost of products. The mechanism of the effect of Na, K and H on thermal stability is not clear. In opinion of the majority of the authors the main driving force of processes governing changes in properties of aged Al-Li alloys during LLTE is high residual supersaturation of solid solution with Li. To increase the stability in Al-Li-Cu alloys it was offered to lower the Li content down to 1.1 – 1.5 wt. % to avoid δ′ phase formation [7,11]. The hardening was provided with a stable and more high-temperature phase T_1 (Al_2LiCu) formation. Shortages resulted from a lower Li content, such as an increase in density and deterioration of specific properties are obvious as well. In [12] on Al-6.5 Li, Al-9.5 Li, Al-11.5 Li (in at. %) alloys was shown that the character of the solid solution decomposition during LLTE and changes of strength and fracture energy greatly depended on the Li contents and on the exposure temperature. At temperature of 70°C, the properties during LLTE didn't vary, though fine dispersed δ′ precipitates or the ordered solid solution domains by a diameter about 2 nm were observed. At higher exposure temperatures the increase of strength and fracture

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energy reduction were observed. These changes were accompanied by increase of δ′ phase volume fraction at the expense of pre-existing δ′ particles growth and nucleation of new fine δ′ precipitates from a solid solution. As it was indicated in [12], in more complex Al-Li-Cu-(Mg) alloys the specific laws can largely change. The ways how to improve the Al-Li alloys stability under LLTE were not considered in [12]. It was shown [9], that changes in properties of 8090 alloy sheets in T81 temper during exposure 70°C, 4 weeks could be eliminated due to reversion ageing (190°C, 5 min). Realization of the reversion ageing under industrial conditions is rather difficult, especially when the material was already used in airplane.

Thus, the effective methods for thermal stabilization of the Al-Li alloys under LLTE conditions have not been offered yet. The goal of the present work is to investigate the structure and properties evolution of aged Al-Li-Mg-Sc-Zr and Al-Li-Cu-Sc-Zr alloys sheets under LLTE conditions and to develop heat treatment schedules which allow to improve their thermal stability.

2. EXPERIMENTAL

The study was carried out on the industrial 1.5 mm sheets Al-Li-Mg-Sc-Zr (type 1421) and Al-Li-Cu-Sc-Zr (type 1460) alloys. Two LLTE regimes were used: 85°C, 1000 h and 95°C, 300 h. After solution treatment alloys were cold water quenched and aged 120°C, 10 h for Al-Li-Mg-Sc-Zr alloy and 120°C, 20 h + 170°C, 12 h for Al-Li-Cu-Sc-Zr alloy. The changes in sheets structure were studied by TEM, DSC and X-ray analysis techniques. δ′ phase X-ray line $(110)_{\delta'}$ integral intensity and effective width were measured on DRON-3 diffractometer using monochromatic CuK_{α} radiation. $(110)_{\delta'}$ X-ray line integral intensity was normalized to integral intensity of the correspondent matrix $(220)_{\alpha}$ line. This value is proportional to the volume fraction of the δ′ phase (f) [13]. $(110)_{\delta'}$ width was determined by the moment method after background subtraction. This value is inversely proportional to an averaged size of the δ′ phase particles ($\bar{r}$) [14]. In a binary Al-2.2 wt % Li alloy for several aging regimes (f) and ($\bar{r}$) have been determined by TEM. Using these samples as the standards for X-ray method, (f) and ($\bar{r}$) for Al-Li-Mg-Sc-Zr and Al-Li-Cu-Sc-Zr alloys were determined by X-ray measurements.

3. RESULTS AND DISCUSSION

Mechanical properties in three directions, fracture toughness and fatigue crack propagation rate (FCPR) of aged sheets before and after LLTE are presented in Table 1. According to the data [1-9] LLTE gives some growth of the strength properties and the essential reduction of relative elongation and fracture toughness. The changes of Al-Li-Cu-Sc-Zr alloy mechanical properties that occur during LLTE at 85°C are shown in Fig. 1. The strength properties appreciably grow up to 500 h, and elongation strongly decreases up to 200 h.

Table 1. Properties of aged sheets before and after LLTE 85°C, 1000 h.

Properties		Al-Li-Mg-Sc-Zr, 120°C, 10 h			Al-Li-Cu-Sc-Zr, 120°C, 20 h +170°C, 12 h.		
		Before LLTE	After LLTE	\, %	Before LLTE	After LLTE	\, %
UTS, MPa	L	476	499	5	537	592	10
	T	477	499	5	519	585	13
	45°	434	434	0	467	525	12
YS, MPa	L	353	363	3	482	534	11
	T	346	361	4	471	534	13
	45°	297	300	1	406	458	13
El, %	L	8.6	7.1	-17	10.4	6.5	-38
	T	14.3	10.5	-27	12.8	12.3	-4
	45°	20.0	19.2	-4	14.4	14.8	3
Kc_O, MPa·$m^{1/2}$	(b = 100 mm)	94.6	80.2	-15	75.3	67.0	-11
FCPR, mm/Cycle at \K=31.2 MPa·$m^{1/2}$ (b = 160 mm)		2.5	3.1	24	2.4	2.1	-13

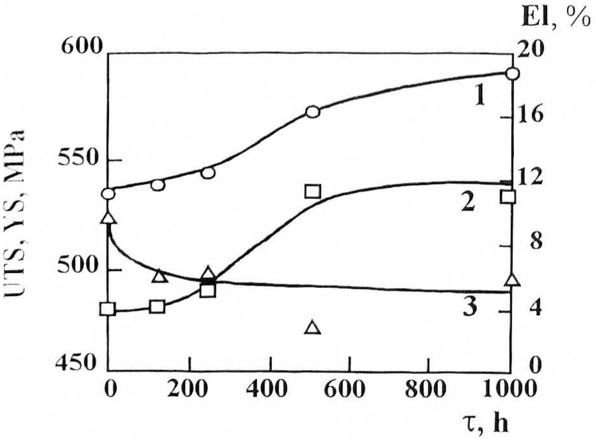

Fig. 1. **Al-Li-Cu-Sc-Zr alloy, 120°C, 20 h + 170°C, 12 h. UTS (1), YS (2), El (3) changes at 85°C.**

The TEM investigation of both alloys indicates that LLTE doesn't change phase composition within grains: δ′ precipitates were observed in Al-Li-Mg-Sc-Zr alloy; δ′, θ′ and T_1 precipitates — in Al-Li-Cu-Sc-Zr alloy. The essential distinction in character of precipitates is the appearance of fine δ′ precipitates that could be detected in the matrix of Al-Li-Cu-Sc-Zr alloy along with large precipitates formed at 170°C ageing (Fig. 2). It was difficult to notice the new fine δ′ precipitates appearance in Al-Li-Mg-Sc-Zr alloy because of low temperature of final aging (120°C). The increase of δ′ phase volume fraction (f) during LLTE was observed by X-ray method for both alloys (Fig.3). The averaged δ′ phase precipitates size ($\bar{\ell}$) in Al-Li-Cu-Sc-Zr is about twice as large than that for Al-Li-Mg-Sc-Zr alloy. Some increase in $\bar{\ell}$ under LLTE for both alloys was observed. Since after LLTE fine δ′ phase precipitates are observed by TEM (Fig. 2), it means that the major part of δ′ phase slight increase in $\bar{\ell}$ was due to the growth pre-existing large δ′ precipitates.

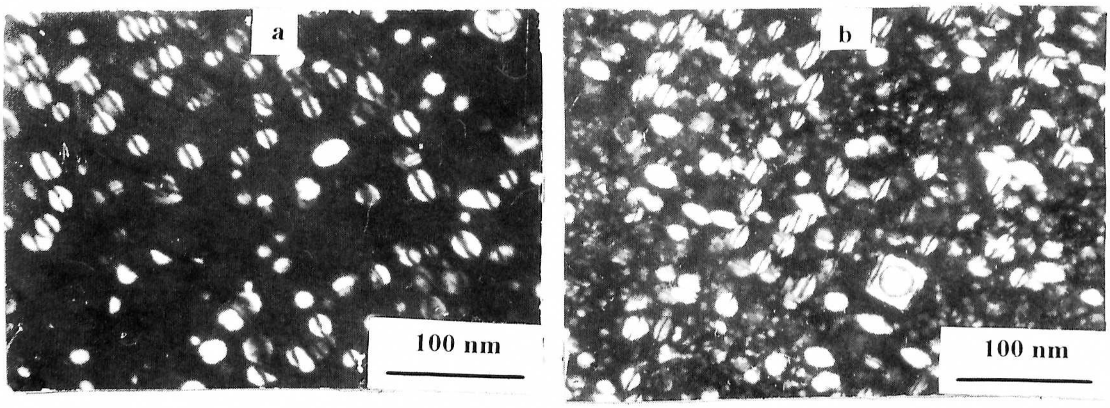

Fig. 2. **Al-Li-Cu-Sc-Zr alloy. Ageing 120°C, 20 h + 170°C, 12 h. Dark field images of the additional fine dispersed δ′ phase matrix precipitates before (a) and after (b) exposure 85°C, 1000 h**

The increase of the δ′ phase volume fraction in Al-Li-Mg-Sc-Zr alloy under LLTE was confirmed by the enlargement of δ′ phase DSC dissolution peak in 130–230°C temperature interval (Fig. 4).

The changes in a δ′ phase volume fraction during LLTE can be caused by two reasons.

1. According to δ′ phase solvus in binary Al-Li alloys [15] the quasiequilibrium difference in Li content between aging temperature (170°C) for Al-Li-Cu-Sc-Zr alloy and exposure temperature (85°C) is close to 1 at.. %. It causes the essential increase of δ′ phase volume fraction during LLTE (~ approximately 4 vol. %), that should result in growth of strength. Diffusion processes at exposure temperature may lead to quasiequilibrium condition with the appropriate volume fraction increase of the fine δ′ precipitates nucleated from a supersaturated solid solution. The changes in equilibrium

solubility with difference of aging temperature and LLTE temperature caused the changes in morphology of δ′ precipitates, that is schematically illustrated in a Fig. 5 (upper scheme). This figure explains experimentally observable changes (Fig. 2).

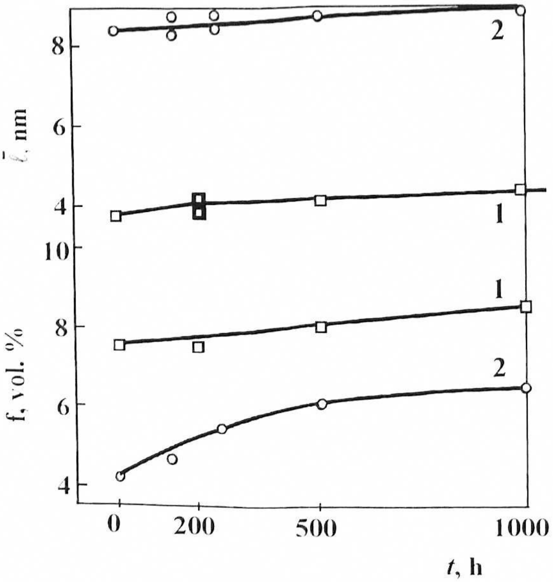

Fig. 3. δ′ phase volume fraction (f) and averaged precipitates size (ℓ) changes at 85°C. (1) — Al-Li-Mg-Sc-Zr alloy, 120°C, 10 h; (2) — Al-Li-Cu-Sc-Zr alloy, 120°C, 20 h + 170°C, 12 h.

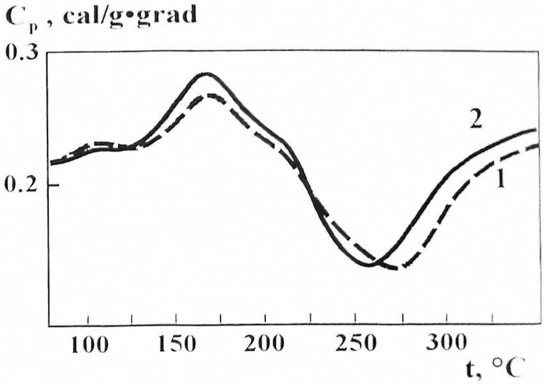

Fig. 4. The influence of LLTE (85°C, 1000 h) on DSC curves of Al-Li-Mg-Sc-Zr alloy, after ageing 120°C, 10 h. (1) — before LLTE, (2) — after LLTE.

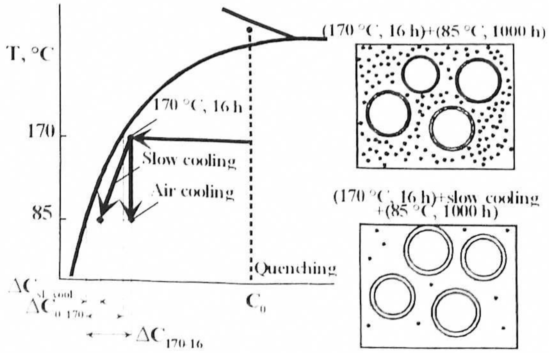

Fig. 5. The effect of the slow cooling after artificial ageing and subsequent LLTE on the δ′ precipitates morphology. The Li supersaturation is controlled by the difference in quasiequilibrium solubility between the ageing and exposure temperatures.

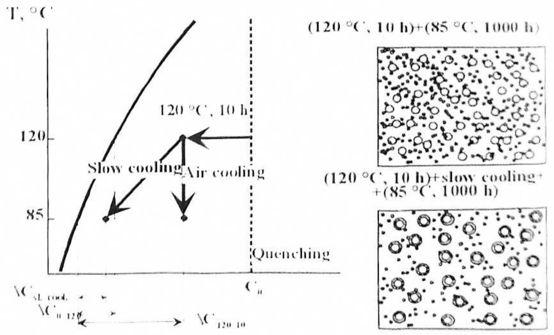

Fig. 6. The effect of the slow cooling after artificial ageing and subsequent LLTE on the δ′ precipitates morphology. The Li supersaturation is controlled by nonequilibrium solubility after artificial ageing.

2 The second reason of the δ′ phase volume fraction increase during LLTE, which can be effective for Al-Li-Mg-Sc-Zr alloy, aged at 120°C, 10 h (the condition far from complete δ′ phase precipitation), when the significant part of Li atoms remains in the solid solution, is the δ′

precipitates formation from the nonequilibrium solid solution which has stayed supersaturated after the ending of ageing (see a schematic Fig. 6).

Measured after exposure increase in strength is obviously caused by the growth of the δ′ phase volume fraction. The decrease in plasticity and fracture toughness under LLTE can be explained by occurrence in a matrix fine dispersed δ′ phase precipitates, which reduce stress relaxation at growing crack front. According to the stated above assumptions to reduce the possibility of formation in process LLTE fine δ′ precipitates the schemes of thermal processing allowing to decrease the residual Li solid solution supersaturation and to receive favorable morphology δ′ precipitates were offered. The best results have given two schemes: (1) slow cooling from ageing temperature and (2) introduction after a high-temperature step of ageing a low-temperature step.

The TEM researches have shown (Fig. 7, 8), that after heat treatment under the specified heat treatment schemes in Al-Li-Mg-Sc-Zr alloy the δ′ particles of two sizes are observed — larger precipitates, obviously, were generated with high-temperature step of ageing (170°C, 16 h), and minor precipitates were generated with the slow cooling, or on the second low-temperature step of aging (120°C, 10 h). After LLTE the particle morphology appreciably not changed, more fine δ′ particles are not formed, Li supersaturation has decreased, and the additional δ′ phase precipitation during LLTE apparently occurs on pre-existing particles.

The mechanical properties of Al-Li-Mg-Sc-Zr alloy sheets before and after LLTE, heat treated with and without application of stabilizing heat treatment are given in Table 2. The offered schemes of thermal processing greatly raise the Al-Li alloys stability during LLTE.

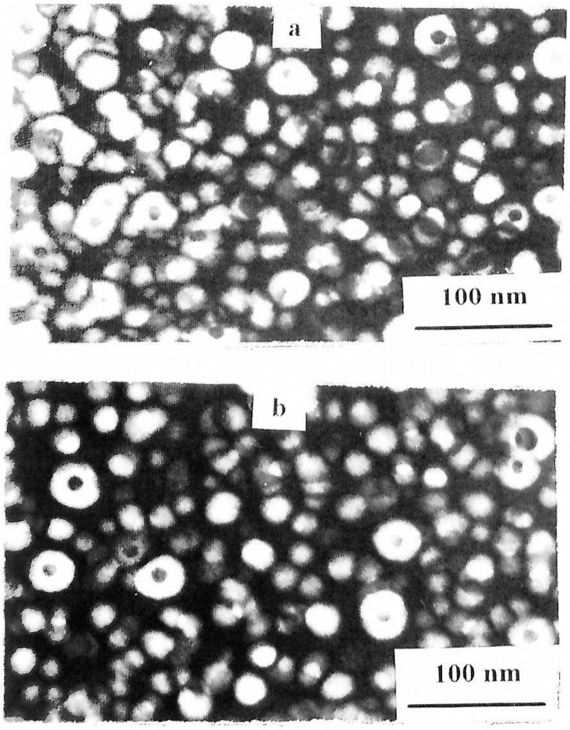

Fig. 7. δ′ precipitates in Al-Li-Mg-Sc-Zr alloy before and after LLTE 95°C, 300 h. Dark field images after (a) 170°C, 16 h + slow cooling; (b) the same + LLTE.

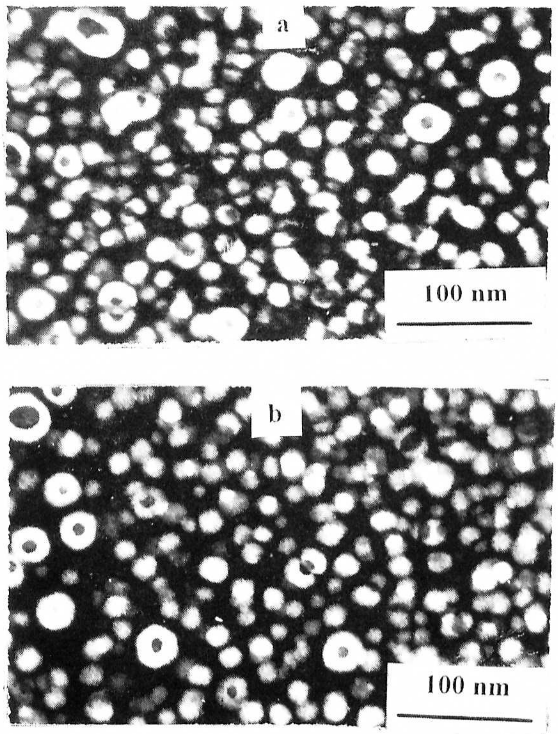

Fig. 8. δ′ precipitates in Al-Li-Mg-Sc-Zr alloy before and after LLTE 95°C, 300 h. Dark field images after (a) 170°C, 16 h + 120°C, 10 h; (b) the same + LLTE.

Table 2. Effect LLTE 95°C, 300 h on mechanical properties of Al-Li-Mg-Sc-Zr alloy sheets aged by standard and stabilizing regimes.

Ageing regime		Properties		
		UTS, MPa	YS, MPa	δ, %
120 °C, 10 h	Before LLTE	476	339	12.9
	After LLTE	509	370	9.9
	Δ, %	6.9	9.1	− 23.3
170 °C, 16 h	Before LLTE	489	332	10.6
	After LLTE	502	375	7.7
	Δ, %	2.7	13.0	− 27.4
170 °C, 16 h + slow cooling	Before LLTE	512	382	8.3
	After LLTE	527	387	8.4
	Δ, %	2.9	1.3	1.2
170 °C, 10 h + 120°C, 10 h	Before LLTE	502	371	8.9
	After LLTE	527	394	9.2
	Δ, %	5.0	6.2	3.4

4. CONCLUSIONS

Aged Al-Li-Mg-Sc-Zr (type 1421) and Al-Li-Cu-Sc-Zr (type 1460) alloys during long low temperature exposure (LLTE) (85°C, 1000 h and 95°C, 300 h) undergo decomposition with increase of δ′ phase volume fraction. The cause of this are the changes in quasiequilibrium solubility with difference of aging temperature and LLTE temperature and the fine dispersed δ′ precipitates formation from the nonequilibrium solid solution which has stayed supersaturated after the ending of ageing. Slow cooling from ageing temperature and two-stage ageing with first high temperature and second low temperature stages prevent formation of δ′ phase fine-dispersed particles at LLTE temperatures.

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