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Effect of Multi-step Ageing on Corrosion Resistance and Thermal Stability of Al-Li Binary System

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Effect of multi-step ageing of Al-Li system influences the decomposition of the solid solution at the first step on morphology of precipitates at the following steps. It results in a decrease of size and increase in the volume fraction of strengthening phases. The uniform distribution of precipitates also eliminates formation of non-desirable phases or provides a discreteness to stable phase precipitates along the grain boundary and a decrease of the width of precipitates free zone along the boundary. Usage of multi-step ageing regimes allows to increases fracture toughness, corrosion resistance and thermal stability in comparison with the one-step ageing for Al-Li system.

Keywords: Multi-step ageing, Al-Li system, Volume fraction, Corrosion resistance, Thermal stability.

INTRODUCTION

Aluminum-lithium alloys are attractive especially for aerospace applications, because they offer interesting combinations of high specific strength and high specific modulus. The addition of lithium, however, generally results in decreased ductility. Attempts to improve the ductility of this alloy have been made by alloying, processing and thermo-mechanical treatments [1-3]. The precipitation process in Al-Li binary alloys has already been studied by many investigators [4-7]. Silcock concluded that the precipitation in Al-Li alloys was a two stage process; solid solution → δ′ (Al_3Li) → δ (AlLi). The δ′ phase is a quasi-equilibrium phase which is of $L1_2$ structure and perfectly coherent with the matrix [4]. The δ phase is a coarse equilibrium phase with a B32-type (NaTl) structure. It is believed that, on plastic deformation, coherent δ′-particles are sheared by moving dislocations leading to a planar dislocation distribution, which may be responsible for low ductility of this material. Transmission electron microscopy actually has revealed sheared δ′-particles in the deformed alloy [8,9].

Thus it appears that the strength and ductility of Al-Li alloys are closely related to the characteristic behaviour of dislocations. However, only a few papers have been published that describe details of the mode of interactions between dislocations and precipitates. Hosson *et al*. [10] analyzed the dislocation motion in an Al-8.0 at. % Li alloy and showed that the δ′-particles formed by aging at 488 K for 3.6×10^3 s are shearable but those formed by aging at 518 K for 4.14×10^5 s are unshearable. Their analysis, however, was only concerned with these two states of aging.

Multi-component commercial Al-Li alloys are characterized by a high degree of alloying (9-14 at. %), a considerable heterogeneity and complexity of phase transformations both under the manufacture of semi-products and their final heat treatment, that influences considerably on the alloy properties, especially on characteristic of crack resistance, fracture toughness and corrosion resistance.

Multi-step ageing is the most effective method to influence the structure and properties of multi-phase ageing alloys [11-14]. For the first time the step ageing has been developed for Al-Zn-Mg-Cu alloys. A considerable influence of preliminary zone ageing on the following phase ageing was established for them. In the present work the efficiency of multi-step ageing used for Al-Li alloys has been determined.

EXPERIMENTAL

Alloys were prepared by arc melting in an Ar atmosphere. Studies of the morphology of oversaturated solid solution (OSS) decomposition of Al-Li-Cu (1451, 1461), Al-Li-Mg-Cu (1430) and Al-Li-Mg (1424) alloys has been carried out under ageing in a wide time-temperature region on 2.5 mm sheets thickness by transmission electron microscopy (TEM), differential scanning calorimetry (DSC, METTLER TOLEDO

DSC-3), X-ray structural analysis (XRD, JDX-35HS JEOL) and resistometry. For the TEM observations, slices were taken from the deformed specimens by a rotating wheel fine cutter, and thin foils for TEM were prepared using a Twin-Jet electro-polishing apparatus. The composition of the electrolyte was 20 % $HClO_4$ and 80 % C_2H_5OH. Microstructures and dislocation configurations were examined by dark field TEM using a JEM-3010 (JEOL) high voltage electron microscope.

During heat treatment of Al-Li alloys well-known phases θ″, θ′, θ (Al_2Cu) and S″, S′, S (Al_2CuMg) for Al-Cu and Al-Cu-Mg alloys, a series of the new δ′ (Al_3Li), T_1 (Al_2CuLi), T_2 (Al_6CuLi_3), S_1 (Al_2MgLi), Al_3Zr, Al_3Sc arises at the same time. Multi-stage decomposition of oversaturated solid solution of Al-Li alloys takes place with the formation of different phases in the different time- temperature regions (Table-1).

RESULTS AND DISCUSSION

Two mechanisms of formation of hardening phases take place: (1) a primary precipitate is a nucleus of each new type precipitate and (2) an independent nucleation of a new type precipitate in the matrix, in particular, as a consequence of total or partial decomposition of the primary precipitates. There are time-temperature boundaries for a phase region.

T_{min}, below which GP-zones do not reach a critical size for transformation in the new type of precipitate at higher ageing temperature and T_{GP}, above which, GP-zones either become the nuclei of the metastable phases or dissolve and the new type precipitates form from solid solution.

The general hardening metastable δ′(Al_3Li)-phase is isomorphic to the matrix and ordered $L1_2$ type, it nucleates homogeneously. Moreover for multi-phase alloys this phase nucleates on inter-phase boundary of matrix and other phase, forming composite particles. Composite particles of two types with a shell of the metastable δ′-phase are founded (Fig. 1).

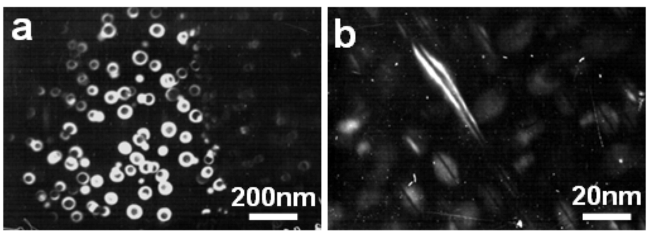

Fig. 1. Composite particles of $Al_3(Zr,Sc)$/δ′ (a) and θ′/δ′ (b) dark field image, (110) δ′ reflection

The first type particles are observed in Al-Li alloys, alloyed with Zr and Sc, and consist of two isomorphic phases ordered by type $L1_2$. The kernel is Al_3X-phase, where X can be Zr, Sc or Zr + Sc. The second type particles are formed in Al-Cu-Li alloys. The kernels of these composite particles are the non-isomorphic tetragonal θ″ and θ′-phases.

At the development of multi-step ageing a choice of time-temperature parameters is realized with an usage of diagram of phase transformation under ageing (DPTA), which have been plotted in consequence of structural investigation and definition the time-temperature boundaries of existence of different phases (Fig. 2).

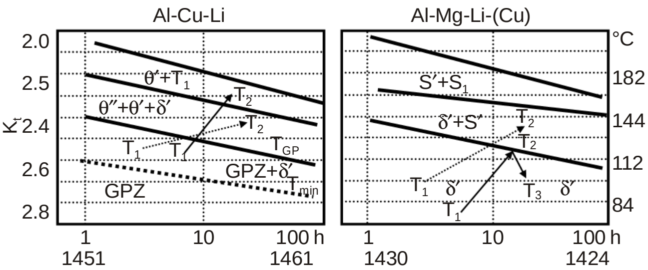

Fig. 2. Al-Li alloys DPTA and step ageing schemes

A temperature of the first step ageing for Al-Cu-Li alloys is chosen between T_{min} and T_{GP}. At the first step of ageing (120-135 °C) the maximum quantity of composite particles of θ″/δ′ phase is formed, capable to a growth at higher temperatures (145-165 °C) [15].

The density of heterogeneously nucleated θ′ and T_1 precipitates is relatively little. The high density of θ″/δ′ particles slows down both a growth of θ′-phase and further T_1 precipitation at the second ageing step.

The 2-step ageing, in comparison with 1-step ageing, ensures an intensive precipitation of finer θ″/δ′ and T_1-phases in the grain body and decrease the quantity, size of T_1 precipitates and PFZ width (Table-2, Fig. 3).

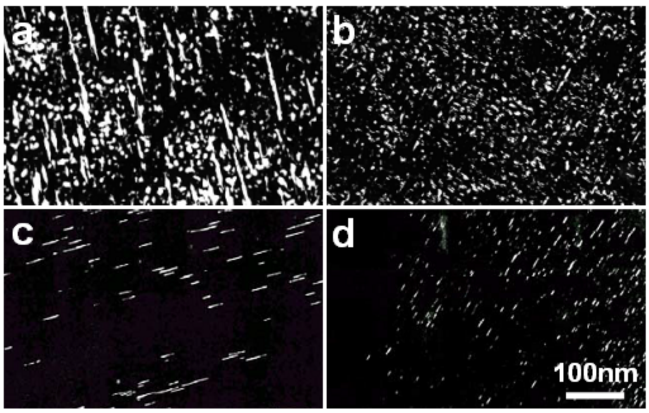

Fig. 3. Precipitates of δ′ , θ″ , θ′ (a, b) and T_1 (c, d) in 1461 alloy. (a, c); 1-step ageing, dark field image, (001) δ′ reflection. (b, d); 2-step ageing, dark field image, T_1 reflection

Multi-step ageing is effective also for 1430 Al-Li-Mg-Cu and 1424 Al-Li-Mg alloys, which have practically a single strengthening δ′-phase. At the ageing temperature of 140 °C and higher, for 1430 alloy S′ (Al_2CuMg) strengthening phase

TABLE-1
SCHEME OF Al-Li ALLOYS OVERSATURATED SOLID SOLUTION DECOMPOSITION

Alloying	Al-Mg-Li	Al-Cu-Li	Al-Mg-Li-Cu
Decomposition α-solid solution	α → δ′ → S_1	α → δ′ → GP → θ″ → θ′ → T_1	α → δ′ → S_1 → GPB → S″ → S′

TABLE-2
INFLUENCE OF AGEING REGIME ON Al-Li ALLOYS STRUCTURE CHARACTERISTICS

Alloy	Characteristics			Ageing	
				1-step	2-step
1451 Al-Cu-Li	Size of precipitate (nm)		θ″/δ′	30	16
			θ′	85	50
			T_1	70	Absent
1461 Al-Cu-Li	Size of precipitate (nm)		θ″/δ′	10 (δ′)	5 (δ′)
			θ′	60-145	20-40
			T_1	60-166	20-80
		PFZ width (nm)		70-145	40-80
1430 Al-Mg-Li-Cu	Size of precipitate (nm)	Grain body	δ′	10	4.7
		Grain boundary	S_1	6-200	Absent
			S′	4-40	1-4
		PFZ width (nm)		60-80	Absent

is formed on the dislocations, introduced by straightening and on the grain boundaries. Moreover, S_1 (Al_2MgLi)-phase precipitates on the grain boundaries at ageing temperature higher, than 150 °C. The growth of these particles results in the forming the PFZ, that results in a decrease of fracture toughness and corrosion resistance. At the ageing temperature of 100 °C a sufficiently high volume fraction (5-6 %) of fine δ′-phase precipitates is achieved during the first 3-5 h [16]. At the second step of ageing at 140 °C this volume fraction increases up to 8.5-9.0 % mainly due to the growth of precipitate size. This 2-step ageing removes the continuous S_1-phase precipitates on the grain boundaries and forming the PFZ (Fig. 4, Table-2).

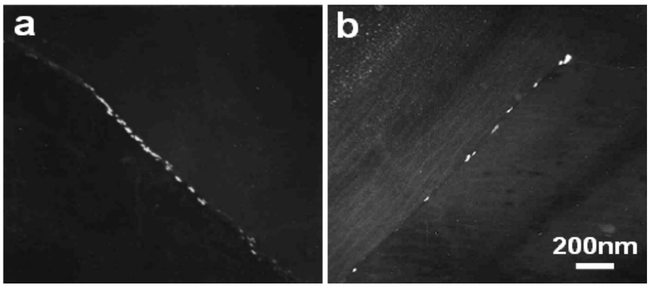

Fig. 4. Precipitates of S_1 in 1430 alloy after 1-step (a) and 2-step ageing (b)

The usage of 2-step ageing for 1451, 1461 and 1430 alloys results in an increase of fracture toughness, crack and corrosion resistance, saving the level of strength characteristics as compared with 1-step ageing (Table-3).

Low temperature ageing of 1424 Al-Li-Mg alloy at 115-125 °C ensures the best combination of mechanical and corrosion properties [17,18]. However, in this case the supersaturation of solid solution by Li is retained. By this reason an additional precipitation of fine δ′-phase can take place during long-term low temperature exposure, leading to additional strengthening and decrease of fracture toughness by 20-25 % [19-21].

The use of 3-step ageing in the temperature range of 85-120 °C allows the following: owing to the low temperature (T_1) a fine dispersion of δ′-phase precipitate in the first step, the second step ($T_2 > T_1$) provides sufficient volume of it and the third step ($T_3 < T_2$) leads to additional precipitation of δ′-phase that decreases the supersaturation of solid solution. Air quenching and 3-step ageing ensures a thermal stability of 1424 alloy properties after exposure for up to 3000 h [20,22-24].

Conclusion

• Effect of multi-step ageing of Al-Li alloys is in that it influences decomposition of the solid solution decomposition at the first step on morphology of precipitates at the following steps. It results in a decrease of size and increase in the volume fraction of strengthening phases. The uniform distribution of precipitates also eliminates formation of non-desirable phases or provides a discreteness to stable phase precipitates along the grain boundary and a decrease of the width of precipitates free zone along the boundary (PFZ).

TABLE-3
INFLUENCE OF AGEING REGIME ON Al-Li ALLOYS PROPERTIES

Alloy	Characteristics	Ageing	
		1-step	2-step
1451 Al-Cu-Li	UTS (MPa)	530	525
	YS (MPa)	780	470
	El (%)	8	10
	K_{c0} (MPa √m (B = 140 mm))	60	74
	FCPR (da/dn), mm/kcycle, ΔK = 31 MPa √m)	4	2.5
	LCF (kcycle, σ_{max} = 157 MPa, K_t = 2.6	130	170
	Exfoliation corrosion	EB	EA
1430 Al-Mg-Li-Cu	UTS (MPa)	470	465
	YS (MPa)	375	375
	El (%)	11	14
	K_{c0} (MPa √m (B = 140 mm))	60	75
	FCPR (da/dn), mm/kcycle, ΔK = 31 MPa √m)	2.2	1.2
	Exfoliation corrosion	EB	EA

• Usage of multi-step ageing regimes allows to increases fracture toughness, corrosion resistance and thermal stability in comparison with the one-step ageing for Al-Li alloys.

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