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EFFECT OF HEATING RATE ON PRECIPITATION OF PEAK-AGED Al-Li (8091) ALLOY – A QUANTITATIVE APPROACH

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Abstract

Samples of Al-Li (8091) alloy were solution treated at 540ºC for 30 min, water-quenched and continuously heated in oil at 0.05 and 0.1°C/min rates, from room temperature to 190ºC when the peak hardness was achieved. Having reached 190ºC at a pre-determined heating rate samples were water-quenched followed by cooling in liquid nitrogen. Precipitate identification performed by transmission electron microscopy (TEM) revealed the presence of strengthening phases formed during heating to 190ºC: δ' (Al_3Li), S (Al_2CuMg), and β' (Al_3Zr). Applying the method of quantitative metallographic analysis (light microscopy and TEM) size, shape, density, distribution and other characteristics of precipitates were determined.
Key words: continuous heating, precipitation, morphology, strengthening phases

Introduction

Reduction in weight of aerospace components is an important necessity to enhance the fuel, structural as well as pay load efficiency of aerospace vehicles. Addition of lithium to aluminum is known to result in a decrease of density by about 3%, associated with an increase in modulus of elasticity by about 6%. Therefore, aluminum–lithium (Al–Li) alloys [1,2] are finding extensive applications mainly as materials for aerospace structures due to their superior specific strength, modulus and good cryogenic toughness properties, compared with other conventional Al alloys and composites. Therefore, Al–Li alloys are employed as materials for various aerospace and other transportation components.

Precipitation strengthening has long occupied an important position among available strengthening mechanisms and has been extensively exploited in Al-Li alloys.

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By optimal choice of precipitation process fine precipitates could be created inside the grains, at grain and subgrain boundaries providing favorable properties of the investigated alloy.

Several metastable and stable phases, including GP zones, θ'', θ', δ', β', S', S, T_1, T_2, δ are known to form in Al–Li alloys depending on the particular alloy chemistry and treatment condition [3]. It is widely reported that the most dominant precipitation hardening mechanism in these alloys is through the formation of ordered δ' (Al_3Li) phase. δ' phase precipitates as spherical nuclei and is coherent with the Al-rich matrix. Optimization of the treatment conditions to avoid over-aging is essential to obtain maximum hardness and tensile strength. Addition of minor alloying elements like Cu and Mg, aids in the formation of semi-coherent lath-shaped S' (Al_2CuMg) phase, as a precursor for S phase of the same composition, nucleating homogeneously in the matrix. S and S' phases do not undergo shearing [4,5], but dislocations loops around these precipitate serve as obstacles to moving dislocations enhancing the tendency for homogeneous deformation of the alloy, and, in turn, increasing its ductility. An optimum microstructural mixture of δ' which is responsible for effective precipitation hardening and S' and S phases which prevent co-planar slip, is vital for engineering applications of the alloy. It is obvious that the microstructural characteristics impose a strong influence on the mechanical behavior of the Al–Li alloys. Further, tailoring of the microstructure depending on the specific end application is vital to ensure optimum performance of the components. In view of the above, identification precipitates of Al–Li alloys subjected to different heat treatments and their quantitative characterization can assist in assessing the optimal properties of the alloy.

The objective of this paper was to study the precipitation mechanism of Al-Li alloy (8091) at strengthening maximum (peak hardness) applying quantitative microstructural analysis. Also, the identification of precipitates was one of the aims of this paper.

Experimental procedure

Material and heat treatment

Al-Li alloy (8091) with chemical composition 2.40 Cu, 2.0 Mg, 0.70 Zr, 1.15 Li (mass %) was solution treated at 540ºC for 30 min, water-quenched and continuously heated in oil at different heating rates (0.05 and 0.1°C/min), from room temperature to 190ºC. According to previous experiments [6] it was well documented that by aging of the supersaturated Al-Li (8091) solid solution the maximum value of hardness (peak hardness) was achieved at this temperature. Reaching 190°C at pre-determined continuous heating, samples were water-quenched then cooled in liquid nitrogen in order in to prevent natural aging. Holding time at 190ºC was 700 and 50min for samples heated at rates of 0.05 and 0.1°C/min, respectively.

Methods

Particles precipitated during heating to 190°C at rates of 0.05 and 0.1°C/min were identified and their quantitative parameters (size, shape, density, distribution etc.) defined. Transmission electron microscopy (TEM) and light microscopy in bright and

polarized light were used for examination of microstructure and qualitative and quantitative analysis of samples subjected to above described heat treatment.

Samples with dimensions 10x10x1mm were thinned by emmery paper until the foil with thicknes between 0.1 and 0.08mm was obtained. The disks with diameter of 3mm punched from this foil were electrochemically polished. The polishing was performed at the room temperature in the „Jet-pol" aparatus using the mixture of 10% $HClO_4$ and 90% ethanol, under the following condition: voltage – 14V, current – 20mA, time of polishing – approximatelly 60s. TEM „JEM 100C–JEOL" used for microstructural examination was operated at 100kV.

Vickers hardness with load of 10 kgf was applied to measure hardness of heat treated samples.

Quantitative microstructural analysis was carried out on semi-automatic device for picture analysis with automatic data processing, using microphotographs produced by light and TEM. Structural parameters which best define processes and features were measured. They are primarily the parameters which characterize precipitated particles, i.e. the particle size, the volume fraction and the numerical density, as well as the distance between the particles.

At the same time, through the shape factor, the shape of particle was defined on the basis of the following expression:

$$F_L = \frac{4\pi A}{L} \qquad (1)$$

where: A – surface of particle section, L – interparticle distance in chain of particles , F_L –shape factor of particles [6].

For measurement of microstructural parameters, linear and method of surface measurement, or even both, were simultaneously used depending on the shape and size of particles. The priority is given to the method of surface measurement due to the shape of particles and a great number of obtained data [7]. Empirical equation for calculation of ultimate free path (distance between particles) is given by the equation:

$$\lambda = \frac{1 - Vv}{P_L} \qquad (2)$$

where: λ – average free path between the precipitated particles (nm), obtained by linear method of measurement, V_V – volume fraction of the precipitation particles in %, linear and surface methods of measurement, P_L – number of cut particles per length unit of the particle, linear and surface methods of measurement.

Using the method of surface measurement, the following parameters are defined:

A – surface section of maximum diameter of precipitate, μm^2

l_1 – l ength of the lath-shaped particle, nm

l_2 – width of the lath-shaped particle, nm

d_1 – longer diameter of spherical particle, nm

d_2 – shorter diameter of spherical particle, nm.

F_L – particle factor perimeter which for circle and ellipse equals 1.

On the basis of quantitative measurement the following characteristics are calculated:

N_A- numerical density (particle number per surface unit)

V_V- volume fraction of particles, %.

Histograms of particles distribution were used for determination of their quantitative parameters.

Results and discussion

TEM micrograph illustrating strengthening phases such as δ' and β' (metastable Al_3Zr phase) precipitates in the Al-rich matrix is shown in Fig. 1 (a, b).

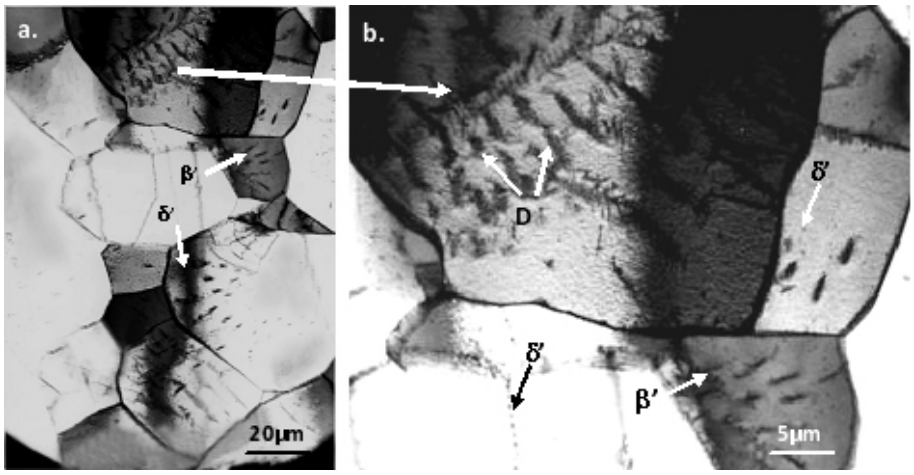

Fig. 1. TEM micrograph. Microstructure during continuous heating rate of 0.05°C/min to 190°C.

Very fine particles of δ' phase can be more clearly seen in Fig. 1b which is an enlarged detail of Fig. 1a. Arrays of dislocations (D) represent preferential sites for formation of precipitates of S phase. TEM micrographs of samples continuously heated at rates of 0.05 and 0.1°C/min is shown in Fig. 2 (a, b).

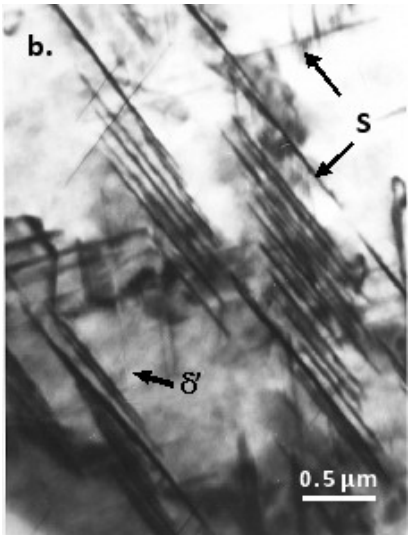

Fig. 2. TEM micrographs of samples heated with different rates to 190°C; a. 0.05°C/min and b. 0.1°C/min

Fine δ' particles together with lath-shaped S particles may be seen in the matrix of both samples. Also, dislocation loops of various diameters are present in Al-matrix (Fig. 2a). Quantitative morphological characteristics of δ' particles as a function of heating rate are given in Table 1.

Table 1. Quantitative morphological parameters of δ'particles as a function of heating rates

Heating rate, °C/min	d_1 (nm)			d_2 (nm)			λ (nm)			F_L		
	min	max	av	min	max	av	min	max	av	min	max	av
0.05	30	62	46	31	44	38	92	408	250	0.9	1.0	0.95
0.1	38	111	62	32	86	52	108	534	321	0.85	1.0	0.93

Where: d_1 – longer diameter of spherical particle, nm; d_2 – shorter diameter of spherical particle, nm and F_L – factor of particle perimeter which for circle and ellipse equals 1.

The results from Table 1 suggest that the shape of δ' particles is more elliptical than spherical with the size approximately 26% larger during the faster cooling rate.

The results of quantitative measurement of dimensions (length and width) of S laths and the distance between these particles are shown in Table 2.

Table 2. Quantitative parameters of S precipitate, heating rate – 0.05°C/min

l_1 (nm)			l_2 (nm)			λ (nm)		
min.	max.	av.	min.	max.	av.	min.	max.	av.
167	1359	712	19	97	47	100	1568	549

Where: l_1 – length of the lath-shaped particle, nm; l_2 – width of the lath-shaped particle, nm; λ – average free path between the precipitated particles (nm).

From the results of Table 3 it is clear that one dimension (length) of S precipitates is much larger than other dimension (width).

Parameters defining structural parameters of δ' precipitates and diameters of dislocation loops are listed in Table 3.

Table 3. Structural parameters of δ' precipitates and diameters (L) of dislocation loops

δ' precipitates		S precipitates		
V_V (%)	N_A (nm^{-2})	L_1 (nm)	L_2 (nm)	av. (nm)
		min.	max.	
2.4	9	143	292	235

Where: V_V – volume fraction of particles, %; N_A – numerical density (particle number per surface unit).

With the volume fraction of 2.4% δ' particles are homogeneously distributed in the matrix during slower heating (0.05°C/min) [8]. During heating precipitation of S particles is enhanced by vacancies which are formed at δ' particles. Namely, these newly developed vacancies may form dislocations loops which represent preferable sites for nucleation of S particles [9]. Formation of vacancies and dislocation loops have a direct influence on the density and uniform distribution of S particles.

Comparing the values of average distance, λ, between δ' and S particles (250 and 549, respectively, see Tables 2 and 3, heating rate 0.05°C/min) it is obvious that the smaller and more densely distibuted δ' particles play a decisive role in strengthening of Al-matrix.

The values of hardness of previously solution treated samples and heated at different rates to 190°C are shown in Table 4.

Table 4. The maximum values of hardness as a function of continuous heating rate

Heating rate, °C/min	Time (min)	Hardness (HV10)
0.05°C/min heating from room temperature to 190°C	2200	154
0.1°C/min heating from room temperature to 190°C	940	119.5

With the lower heating rate higher hardness was obtained than in the case when the higher heating rate was applied. Higher values of hardness may be ascribed to the smaller size of δ' particles and their homogeneous and dense distribution in the Al-matrix (see Table 2).

Conclusion

Precipitation in Al-Li (8091) alloy was studied during heating (0.05 and 0.1°C/min) from room temperature to peak hardness (190°C). Size, shape, density, distribution and other characteristics of precipitates were determined applying the method of quantitative metallographic analysis.

Precipitate identification performed by transmission electron microscopy (TEM) revealed the presence of strengthening phases formed during heating to 190ºC: δ' (Al_3Li), S (Al_2CuMg), and β' (Al_3Zr).

Very fine δ' particles rather elliptic than spherical in shape together with lath-shaped S phase may be seen in samples heated with both heating rates. Also, dislocation loops of various diameters are present in Al-matrix.

The size of δ' particles is approximately 26% larger during the faster cooling rate.

With the lower heating rate higher hardness was obtained than in the case when the higher heating rate was applied. Higher values of hardness may be ascribed to the smaller size of δ' particles and their homogeneous and dense distribution in the Al-matrix

References

[1] E.A. Starke Jr., F.S. Lin, Met. Trans. A 13A (1982) 2259–2269.
[2] P.J. Gregson, H.M. Flower, Acta Met. 33 (1985) 527–537.
[3] N. E. Prasad, A.A. Gokhale, P. R. Rao, Sadhana 28 (2003) 209–246.
[4] S. Ceresara, A. Giarda, A. Sanchez, Phil. Mag. 35 (1977) 97–110.
[5] P.J. Gregson, H.M. Flower, C.N.J. Tite, A.K. Mukhopadhya, Mat. Sci. Techn. 2 (1986) 349–353.
[6] O. Novitović, B. Jordović, M. Rogulic Supplement to the study of hardness change in alloy Al-Li-Cu-Mg-Zn, Aluminium, 67 (1991) 11-15.
[7] S.A. Saltykov, Stereometričeskaja metalografija, Metallurgia, Moskva (1970).
[8] O.Novitovic, Precipitation process in the alloy system Al-Li-Cu-Mg-Zn, Doctoral dissertation, TMF University of Belgrade (1994).
[9] R. Grimes, A. Cornish, W. Miller, M.A. Reynolds Met. and Mat. June (1985) 357-363