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Influence of Retrogression and Reaging Heat Treatment on Calorimetric Behavior of EN AW-7049A Alloy

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Aluminum alloy EN AW-7049A samples in artificial (T6 temper) and after multi-stage aging (T77 temper, i.e. retrogression and reaging treatment - RRA), were investigated by means of differential scanning calorimetry (DSC). DSC was employed to distinguish the dissolution behavior of different metastable and Cu-enriched precipitate populations in EN AW-7049A alloy in the T6 and T77 tempers. DSC thermographs exhibited two consecutive endothermic peaks in the T6 temper, which correspond to the sequential dissolution of metastable semi-coherent phases. First peak corresponds to the main strengthening η' phase, the second peak is associated with the dissolution of thermally more stable Cu-enriched η′/incipient η precipitates. These Cu-rich precipitates enhance yield strength and hardness by increasing dislocation resistance and significantly improve thermal stability by retarding precipitate coarsening and overaging. In the T77 temper, only a single endothermic peak was observed, indicating a reduced concentration of metastable η′ precipitates and the predominance of coarser η′ or incoherent equilibrium η precipitates formed during the retrogression and reaging treatment. The dissolution and transformation of metastable semi-coherent precipitates during RRA promote the nucleation and growth of the stable incoherent η phase, particularly at grain boundaries, leading to strength reduction. Consequently, the increased fraction and coarsening of η precipitates account for the lower strength of the EN AW-7049A alloy in the T77 temper compared with the initial T6 temper.

Key words: **aluminum alloy EN AW-7049A, T6 and T77 temper, DSC.**

Introduction

ALUMINUM alloys of the 7xxx series belong to the group of alloys in which high strength, toughness and corrosion resistance are achieved by age hardening. The precipitation process in 7xxx series alloys generally takes place according to the following scheme [1-3]:

SSS (supersaturated solid solution) → stable coherent GP zones → semi-coherent intermediate η' (MgZn) phase → stable incoherent η (MgZn2) and some of Cu rich phases (T-(Cu, Zn, Al)49Mg32, M-(Cu, Zn, Al)2Mg, S-Al2CuMg).

Due to its high mechanical properties and high strength to density ratio, 7xxx series aluminum alloys are widely used in the automotive, aviation and military industry. In order to improve toughness and corrosion resistance, many studies [2, 4-7] had been done with the aim to determine optimal parameters of the heat treatment of retrogression and reaging (RRA, temper T77) that provide better corrosion resistance, without or with minimal loss of strength compared to the initial T6 temper.

The concept of retrogression and reversion (reaging), i.e. RRA, was first developed by China and its collaborators in 1974 within the Israeli aircraft industry (Israel Aircraft Industries) [5, 8] and it consisted of two stages:

1. retrogression of aluminum alloy series 7xxx in the T6 temper at a temperature located in the temperature interval within the two-phase (α + η) region of the Al–Zn–Mg phase diagram, where η ($MgZn_2$) is the equilibrium strengthening phase [9], [10], followed by quenching in water,
2. reversion – reaging of the alloy according to the parameters of artificial aging of the initial T6 temper.

The main mechanism controlling strength and ductility in AlZnMg(-Cu/-Si) alloys is precipitation strengthening. During retrogression and reaging of Al–Zn–Mg–Cu alloys, the relevant precipitation phases include GP zones, metastable η′ and Cu-enriched η′ precipitates that partially dissolve during retrogression, and the stable incoherent η ($MgZn_2$) phase that nucleates and grows during reaging, particularly at grain boundaries [1-3, 5, 8, 12]. The aim of this work was to investigate the influence of different heat treatments on the dissolution of metastable strengthening precipitates (GP zones, η′ and Cu-enriched η′ precipitates) and the precipitation of stable phases (η) using differential scanning calorimetry (DSC) [2, 9, 13]. DSC measures heat flow associated with solid-state reactions, such as precipitate dissolution and transformation, providing information on their characteristic temperatures and enthalpy changes, rather than phase chemical composition.

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Materials and methods

The material used in this study was aluminum alloy EN AW 7049A, received in the form of extruded rods, Ø90 mm, produced by "Impol" Slovenska Bistrica, Slovenia. To obtain optimum combination of the mechanical properties, as received alloy was solution heat treated, quenched and artificially aged to the T6 temper in accordance to the following heat treatment parameters:

solution heat treatment at 470 °C/2 h + Water Quench (WQ) + artificial aging at 120 °C/24 h.

Artificially aged EN AW-7049A alloy samples in the T6 temper were further heat treated by using a retrogression and reaging (RRA) process, according to the following scheme:

retrogression at 180 ºC/7 min + Water Quench (WQ) + reaging at 120 ºC/24 h.

The samples were mechanically tested by means of hardness measurements and tensile testing. Hardness measurements were determined by using the Brinell hardness method (HB2,5/62,5/30''), according to EN ISO 6506-1, using the "Wolpert Diatestor 2RC" hardness tester. The hardness was determined as a mean value of the results of five measurements. The tensile test was performed on a round specimen, taken from the longitudinal direction of the extruded rod, by using "Shimadzu Servopulser" testing machine with extensometer, at the strain rate of 0.125 mm/s.

The results of the mechanical testing of EN AW-7049 alloy in T6 and T77 temper are presented in the previous work [12].

For the DSC testing, the heat treated samples were cut into 4.5×3.5×1 mm specimens and cleaned in an ultrasonic bath with alcohol. The analysis of phase transformations was performed on a differential scanning calorimetry device type "DSC Q20 - TA Instruments". The instrument was calibrated with indium (In) at a heating rate of 2 °C/min in a nitrogen atmosphere with a gas flow rate of 50 ml/min. The temperature range of the tests was from 0 °C to 390 °C, while the tests were performed at heating rates of 10 °C/min, 20 °C/min and 40 °C/min.

Results and discussion

The chemical composition of the alloy used is given in Table 1, while the mechanical properties of the alloy in T6 and T77 temper are listed in Table 2.

Table 1 Chemical composition, wt.%.

Zn	Mg	Cu	Mn	Si	Fe	Cr	Zr	Ti	Al
7.6	2.88	1.51	0.25	0.06	0.11	0.15	0.1	0.07	ball

Table 2 Mechanical properties of the EN AW-7049 alloy for T6 and T77 tempers.

Temper	HB	$R_{p0.2}$, MPa	Rm, MPa	A, %
T6	182	710	746	8
T77	186	644	676	5

The DSC heating curves for aluminium alloy EN AW-7049A in T6 temper and T77 temper (RRA regime) are shown in Figure 1 and 2 respectively.

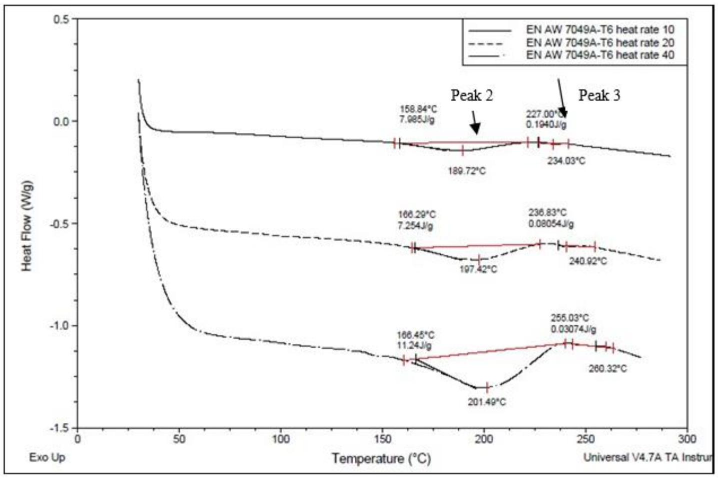

Figure 1 DSC thermograph for alloy EN AW-7049A T6, at the heating rates of 10 °C/min, 20 °C/min and 40 °C/min.

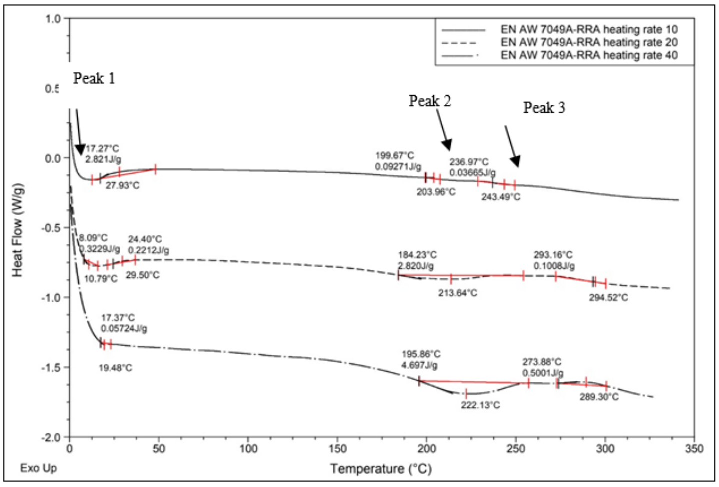

Figure 2 DSC thermograph for alloy EN AW-7049A RRA, at the heating rates of 10 °C/min, 20 °C/min and 40 °C/min.

Heat flow is plotted as a function of specimen temperature so that upward peaks correspond with the exothermic reaction, such as the reaction of precipitation. Downward peaks correspond with the endothermic reaction, such as the dissolution of precipitates. Also, as it can be seen on the given DSC thermograms, the applied heating rate during the DSC process has a great influence on the detection of individual peaks. Table 3 shows the temperature at which endothermic and exothermic peaks occur for the alloy EN AW-7049A for different tempers.

Table 3 Temperatures of the endo- and exo-thermic peaks

temper	Heating rate, °C/min	peak 1, °C	peak 2, °C	peak 3, °C
T6	10.0	-	189.72 (endo)	234.03 (endo)
	20.0	-	197.42 (endo)	240.92 (endo)
	40.0	-	201.49 (endo)	260.32 (endo)
RRA	10.0	27.93 (exo)	203.96 (endo)	243.49 (exo)
	20.0	29.50 (exo)	213.64 (endo)	294.52 (exo)
	40.0	-	222.13 (endo)	289.30 (exo)

Analyzing the thermogram of the alloy in the T6 temper, a broad endothermic peak is first observed followed by another smaller endothermic one (Figure 1). With an increase of the heating rate, the peaks shift to higher temperatures. Thus, the temperature of the appearance of peak 2 shifted from 190 °C at the heating rate of 10 °C/min to 202 °C at the heating rate of 40 °C/min. This behavior was also observed by other authors [14-16]. The appearance of endothermic peaks points to competing processes of dissolution of η´ precipitates and precipitates containing Cu (T-(Cu, Zn, Al)49Mg32, M-(Cu, Zn, Al)2Mg or S-Al2CuMg). Based on previous studies [14–16], within the temperature range corresponding to peaks 2 and 3, peak 2 is attributed to the dissolution of semi-coherent η′ precipitates, whereas peak 3 corresponds to the dissolution of Cu-rich second-phase particles. In addition, the second endothermic peak (peak 3) is absent at a heating rate of 40 °C/min. The difficulty in resolving the two endothermic peaks in the DSC curves is most likely associated with an excessively high heating rate, which leads to peak overlap. The use of a high heating rate may suppress or obscure the DSC peaks corresponding to precipitate-related phase transformations and changes in precipitate volume fraction. At high heating rates, diffusion-controlled processes such as precipitate dissolution and phase transformations of precipitates may not be fully captured in the DSC signal, leading to overlapping or suppressed thermal peaks. Consequently, the thermal effects associated with different precipitate types or precipitate-related phase transformations may not be distinguishable as separate DSC peaks [14, 17, 18].

In the present case, the volume fraction of one of the Cu-rich precipitates is likely too low to generate a measurable endothermic signal. Consequently, its dissolution may either overlap with the dissolution of other precipitates leading to a single combined endothermic peak or remain undetectable altogether. This would explain the absence of a distinct endothermic peak associated with the dissolution of ternary Cu-rich phases in EN AW-7049A.

For the EN AW-7049A alloy in the T6 temper, no exothermic peaks were observed during DSC heating. This behavior is consistent with literature for peak-aged 7xxx series alloys [13, 14, 16-18]. In the T6 temper, the precipitation sequence (supersaturated solid solution → GP zones → η′) has already largely occurred during artificial aging. According to several authors [14-16], the exothermic reaction associated with the formation of the equilibrium η ($MgZn_2$) phase typically occurs at temperatures above approximately 280 °C. The absence of such an exothermic peak in the present study therefore indicates that the selected DSC temperature range was too low and/or that the heating rate was sufficiently high to shift the precipitation reaction to higher temperatures**,** beyond the measurement window. This explanation is in agreement with reported DSC studies on 7xxx alloys, where increasing heating rates shift precipitation reactions to higher temperatures and reduce peak intensity [11].

The DSC curves of the EN AW 7049A alloy samples treated by the RRA (T77 temper) exhibit slightly altered peak trend. Namely, a small exothermic peak first appears at very low temperatures, and it refers to the initial formation of GP zones [2, 14-16]. This exothermic peak is followed by the formation of a broad endothermic peak which in that temperature interval corresponds predominantly to the process of dissolution of the main strengthening η′ precipitate [6]. The exothermic peak 3 that occurs immediately after the endothermic peak 2, correspond to competing reaction of precipitation of the equilibrium η phase [6, 14-16]. The complete absence of the second endothermic peak, which was observed in the T6 temper as peak 3, is most likely a consequence of the partial dissolution of Cu-enriched precipitates and the dominance of fine η′ precipitates formed during artificial aging [8, 11–14]. Differential scanning calorimetry (DSC) studies of Al–Zn–Mg–Cu alloys show distinct endothermic and exothermic peaks associated with dissolution and precipitation reactions, respectively, where the first endothermic peak typically corresponds to the dissolution of metastable η′ precipitates and subsequent peaks can be related to additional dissolution or transformation events in the microstructure [11, 20]. In the T77 temper, extended aging promotes the transformation of metastable η′ into the equilibrium η phase and coarsening of precipitates, leading to a microstructure dominated by larger, incoherent η precipitates. The nucleation and growth of incoherent η precipitates in T77 is a major factor contributing to the observed strength decrease compared to T6, as overaging reduces the number density of fine strengthening η′ precipitates and increases the volume fraction of coarser, stable η precipitates that are less effective at impeding dislocation motion, resulting in lower tensile strength, Table 2. Although the T77 temper exhibits a slightly higher hardness (186 HB) compared to the T6 temper (182 HB), Table 2, this does not necessarily imply higher tensile strength. Hardness reflects the local resistance to plastic deformation, whereas tensile properties are governed by the overall microstructural homogeneity and the effectiveness of load transfer across the material. During the RRA treatment, the coarsening and partial loss of coherency of η′ precipitates, as well as the increased fraction of incoherent η phase at grain boundaries, may locally increase resistance to indentation while simultaneously reducing the uniform strengthening effect under tensile loading. Consequently, the observed increase in hardness in the T77 temper can coexist with a reduction in tensile strength. This behavior has been documented in high-strength Al–Zn–Mg–Cu alloys, where DSC thermograms reveal that fine η′ precipitates disappear and stable η phases grow with prolonged aging, correlating with reduced mechanical properties relative to the peak-aged T6 condition [9-11, 14].

Conclusion

DSC analysis was performed on samples of the EN AW-7049A alloy in T6 and T77 tempers and the results of the analysis confirmed that:

- T77 temper provides a more complete dissolution of remaining η′ precipitates from T6 temper followed by nucleation and growth of η precipitates which affects the loss of strength of the EN AW-7049A alloy compared to the initial T6 temper,
- Higher strength of the EN AW-7049A alloy in T6 temper compared to the T77 temper, is due to strengthening effect of coherent and semi-coherent phases (η′ and some of Cu-enriched phases (T-(Cu, Zn, Al)49Mg32, M-(Cu, Zn, Al)2Mg or S-Al2CuMg)).

The results provide thermodynamic insight into precipitate evolution during T77 temper (RRA regime) and clarify the mechanisms responsible for strength differences between the T6 and T77 tempers. These findings are relevant for optimizing heat treatment parameters of high-strength 7xxx aluminum alloys in applications requiring controlled strength and microstructural stability.

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Uticaj termičkog tretmana retrogresije i reverzije na kalorimetrijske karakteristike legure EN AW-7049A

Uzorci aluminijumske legure EN AW-7049A u veštački starenom (T6) i nakon primene višestepenog starenja (T77, tj. tretman retrogresije i ponovnog starenja – RRA) ispitivani su metodom diferencijalne skenirajuće kalorimetrije (DSC). Rezultati DSC-a pokazali su postojanje dva uzastopna endotermna pika u T6 stanju, koja odgovaraju sekvenci taloženja neravnotežnih polu-koherentnih faza. Prvi pik odgovara glavnom nosiocu čvrstoće, neravnotežnoj η′ fazi, dok je drugi pik povezan sa taloženjem termički stabilnijih faza bogatih bakrom. Ove faze bogate bakrom povećavaju granicu tečenja i tvrdoću legure tako što pružaju veću otpornost kretanju dislokacija, a značajno poboljšavaju i termičku stabilnost taloga u prestarenom stanju. U T77 stanju je primećen samo jedan endotermni pik, što ukazuje na smanjenu koncentraciju neravnotežnih η′ taloga i dominaciju krupnijih η′ ili nekohernih, ravnotežnih η taloga formiranih tokom tretmana retrogresije i ponovnog starenja. Rastvaranje i transformacija neravnotežnih polu-koherentnih taloga tokom RRA tretmana podstiče nukleaciju i rast stabilne, nekohentne η faze, posebno na granicama zrna, što dovodi do smanjenja čvrstoće. Shodno tome, povećan udeo i ogrubljavanje η taloga objašnjavaju nižu čvrstoću legure EN AW-7049A u T77 u poređenju sa početnim T6 stanjem.

Ključne reči: **aluminijumska legura EN AW-7049A, T6 i T77 stanje, DSC.**