Effects of High Temperature Retrogression and Re-aging Treatments on Microstructures and Properties of 7150 Alloy

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In this paper, the influence of high temperature retrogression and re-aging treatment (HTRRA) on properties and microstructures of 7150 aluminum alloy has been studied and properties comparisons of the alloy in various heat treated states, as T6, T77 and HTRRA, have been made. The material in its as-received T6 temper state was retrogressed at 455℃, 425℃, 400℃ for different durations between 10 seconds and 60 minutes. Subsequent re-aging was performed at the T6 aging condition (120℃ for 24 hours). The alloy after HTRRA treatment (retrogressed at 425℃ for 30 minutes) exhibits hardness up to 178HV with retention of an electric conductivity of 35.5 %IACS, which is close to that of the level of 7150-T77 temper. The decrease in hardness and increase in electric conductivity, comparing with the alloy in T6 temper, are associated with the formation of S (Al_2CuMg) grain interior phase and θ (Al_2Cu) phase in grain boundary. Alloys undergone the HTRRA treatment show less sensitivity to retrogression time and might be applied to thick section components.

Key words: Al-Zn-Mg-Cu alloy; high temperature retrogression; RRA;

1 Introduction

7000 series aluminum alloys (Al-Zn–Mg-Cu) have been widely used in the aircraft structures, due to their attractive comprehensive properties. The series of alloys in the peak hardening condition (T6 temper) is very sensitive to stress corrosion cracking (SCC). Compared with the T6 temper, over-aged condition (T7 temper) can overcome the poor resistance to SCC, reducing its strength 10–15 % [1]. While the retrogression and re-aging (RRA) treatment invented by CINA [2] in 1974 can enhance the resistance of aluminum alloy to stress corrosion up to or close to the level of T73 temper whilst maintain the tensile strength of T6 temper.

The evolution of microstructure and properties of RRA have been extensively studied[3-6], their researches indicate that the drop in hardness for the short retrogression time is due to the partial dissolution of G.P. zone and small η`precipitates, whilst the growth and the transformation to η′precipitates of existing η`precipitates cause a rapid increase in hardness. As the retrogression time is longer, the coarsening of precipitates attributes to the drop in hardness after the second maximum of hardness [3-5]. While the increase in hardness during reaging is due to the occurrence of new precipitates and the increase of volume fraction of the precipitates[5]. On the other hand, the retrogression treatment produces rapid coarsening of the grain boundary precipitates[4], similar to the T7 temper, improving the SCC resistance owing to the coarsen grain boundary precipitates as permanent capture of hydrogen atoms[6].

However, RRA treatment was suitable for thin section plate, due to the short retrogression time at a relative high retrogression temperature of 200~280℃[3,4]. Many works focused on extending retrogression time at lower retrogression temperatures with an optimum combination of mechanical properties and the resistance to corrosion [7-9], making it possible to apply to thicker component. The detention of the reaction kinetics occurring during low temperatures retrogression caused a

longer retrogression time[7]. MURAT BAYDOGAN, et al.[8] indicated that as the retrogression temperature was lowered to 170℃, the retrogression time for 7075 aluminum alloy could be extended to 60 minutes, obtaining an optimum combination of hardness and SCC resistance. Li, et al.[9] found that A A7150 alloy retrogressed at 175℃ for 3 h can possesses good exfoliation corrosion resistance without strength loss.

Although there are many works focusing on the microstructure and the properties of RRA treatment at retrogression temperatures (170~280℃), few published data concerned the effect of high retrogression temperatures above 400 ℃. The purpose of the present study is to investigate the influence of high retrogression temperatures above 400 ℃ on the microstructure and properties of 7150 aluminum alloy.

2 Experimental Procedures

The material used in this study was commercially available 7150-T77 aluminum alloy sheets and was delivered in the form of a 2mm×15mm×15mm plate, whose chemical composition range is given in Table 1. The specimens were solution treated by heating to 475℃ for 60min; water quenching then were artificially pre-aged at 120℃ for 24 h. Then, this was followed by retrogression for various times at 455℃, 425℃ and 400℃, respectively (and subsequent cold water quench). All specimens were re-aged at 120℃ for 24 h.

The thermodynamic calculation for the equilibrium phase diagram of 7150 aluminum alloy was carried out by means of Thermo-Calc software. The Vickers hardness test was performed on HVA-10A low load Vickers hardness test machine. The load of each test was 98 N for 30s. Each point represented the mean value of measurements on 6 points. The variation of electric conductivity, an indication for the alloy's susceptibility to stress corrosion cracking[10], can be identified by 7501 eddy-current device.

Optical microscopy examinations were carried out with an Olympus Neophot2 optical microscope. The morphology and chemical composition of precipitates within grain and grain boundary were examined by using scanning electron microscopy(Apollo 300 with field emission gun(FEG) and Energy dispersive X-ray spectroscopy(EDS) operated at 20KV).

Table 1 Chemical Compositions of 7150Aluminum Alloy (mass fraction, %)

Zn	Mg	Cu	Zr	Fe	Si	Al
5.9~6.9	2.0~2.7	1.9~2.5	0.08~0.15	≤0.15	≤0.12	Bal.

3 Results

3.1 Hardness property

Fig.1a shows the hardness curves of the retrogressed 7150 aluminum alloy as a function of retrogression time at retrogression temperatures of 455℃, 425℃ and 400℃，respectively. For all three temperatures, a similar retrogression behavior is observed. Following retrogression times, the hardness is rapidly decreased, followed by a slight drop to a minimum hardness level. And the curves show that the minimum hardness values varied depending on retrogression temperature. At retrogression temperature 455℃, the hardness level reaches to 84HV, while the hardness level declines to 75HV at 425℃ and 71HV at 400℃. The variations of hardness during retrogression at 455℃, 425℃ and 400℃ for different times and re-aging treatments are shown in Fig.1b. The hardness is initially dropped to valley value in hardness, and then increased slightly to a maximum plateau after which there are no further changes in hardness. Furthermore, the curves show that the higher the retrogression temperature, the higher hardness will exhibit. The plateau hardness of 7150

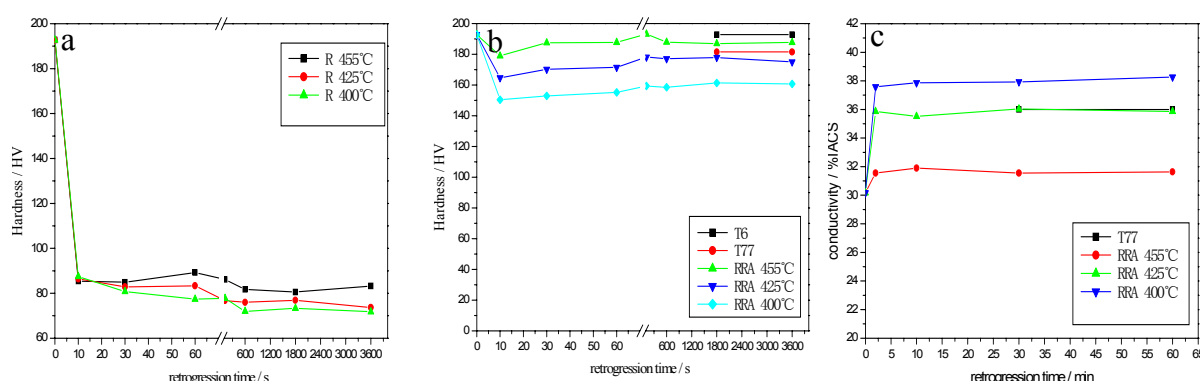aluminum alloy retrogressed at 400℃ reaches to about 160 HV. As the retrogression temperature is

Fig.1 Properties curves of 7150 as a function of retrogression time at retrogression temperatures of 455℃, 425℃
and 400℃.(a-hardness of retrogression; b-hardness of retrogression and re-aging; c- electric conductivity of
retrogression and re-aging)

elevated to 425℃, the corresponding hardness is improved to about 178HV, close to the hardness of 7150-T77 temper. And the alloy retrogressed at 455℃ possesses high hardness in the vicinity of that of 7150-T6 temper.

3.2 Electric conductivity

Fig.1c shows the electric conductivity of the alloys, when retrogressed at 455℃, 425℃ and 400 ℃ for different times and consequent re-aging treatment, sharply increase to maximum values. Moreover, the curves show that the electric conductivity of RRA increases with the retrogression temperature declining. And the conductivity of the alloy retrogressed at 425℃ is dramatically increased to 35.5 %IACS, close to the level of T77 temper.

3.3 Microstructure characterization

The optical photographs of 7150 alloy with different retrogression temperatures for 30min and re-aging are showed in Fig.2. Precipitates are not observed at both grain interior and grain boundary in 7150-T6 condition. Compared to 7150-T6, the coarse grain boundary precipitates dispersed in the HTRRA treated 7150 alloy retrogressed at 455℃ for 30min. Both dispersed grain interior precipitates and grain boundary precipitates are observed in the alloy retrogressed at temperature of 425℃. However, the alloy retrogressed at 400℃ exhibits much fine precipitates dispersed on grain interior and gain boundaries as well in comparison with that retrogressed at 425℃. Fig.3 shows FEG photograph of precipitates in grain boundary and grain interior of 7150 alloy retrogressed at 425℃ for 30min. It is evident that grain boundary precipitates are coarse and dispersed. And Fig.3b indicates that large precipitates are formed in grain interior with a low number density.

The chemical compositions of the precipitates on grain boundary and grain interior formed during the high temperature retrogression treatment are semi-quantitatively analyzed by EDS and the result is shown in Table 2. It can be seen that the chemical composition of the precipitates on grain boundary is close to the composition of stoichiometric phase θ (Al_2Cu). The formation of θ phase in grain boundary might be a consequence of the diffusion of Cu, Mg and Zn elements. When the alloy is retrogressed at 425℃, the element of Cu, Mg and Zn dissolved into matrix, However, the diffusion of Cu is slower than Mg, Zn, the retainment of Cu element results in the formation of θ phase in grain boundary. While the chemical compositions of the grain interior precipitates contained too much Al, as is shown in Table 2, is owing to the fact that the size of grain interior precipitates is relatively small, about 300 nm, and the Al composition of alloy matrix are tested by EDS. Considering above factors, the chemical compositions of the grain interior precipitates could be close to the stoichiometric S(Al_2CuMg) phase or perhaps T($Al_2Mg_3Zn_3$) phase. On the other

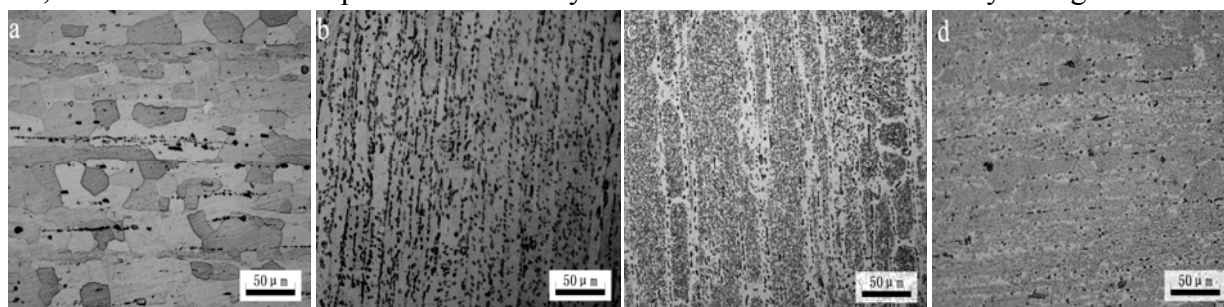hand, it is more stable for S phase on thermodynamics for 7150 aluminum alloy retrogressed at

Fig.2 Optical photographs of 7150 alloy with different retrogression temperatures

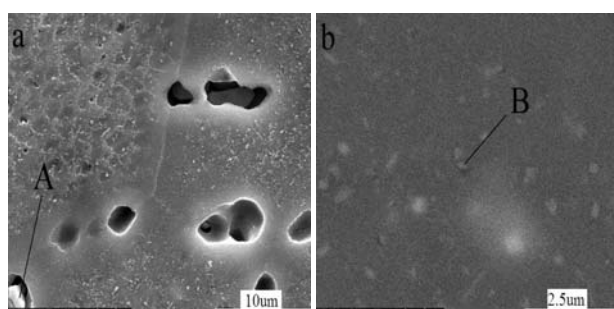a-T6; b-retrogressed at 455℃/30min c- retrogressed at 425℃/30min; d- retrogressed at 400℃/30min

Fig.3 images retrogression at 425℃/30min
(a) FEG-SEM; (b) FEG-BSE grain interior;

Table 2 Chemical compositions (at.%) of phases of 7150 alloys retrogressed at 425℃

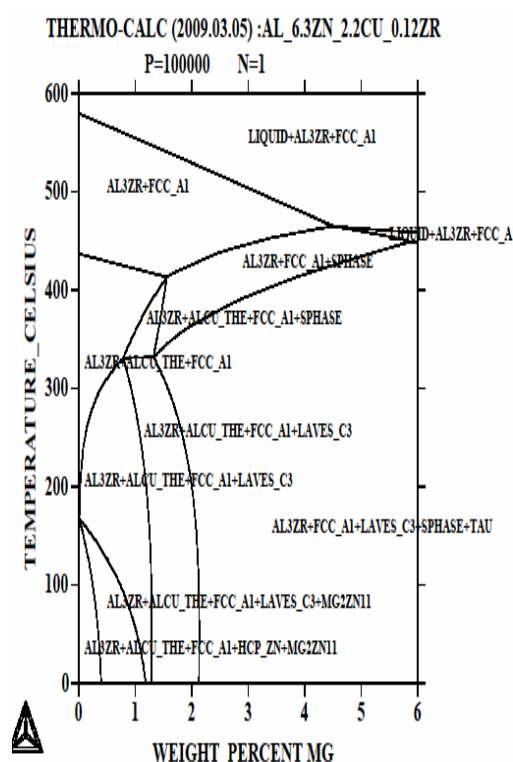|	Mg	Cu	Al	Zn
A(grain boundary precipitates)	2.86	29.45	66.71	0.98
B(grain interior precipitates)	2.64	3.26	91.53	2.58

Fig.4 the phase program of Al6.3Zn2.2Cu0.12ZrMgx

425℃, according to the phase program of Al6.3Zn2.2Cu0.12ZrMgx (Fig.4), where the Mg composition of 7150 is about 2.6 wt%. And S phase can nucleate in the primary $MgZn_2$ phase, concomitant with growth and coarsening of S phase during homogenization treatment in commercial Al–Zn–Mg–Cu alloys[11-13]. Furthermore, the metastable solubility of zinc may range up to 30 wt.% for S phase[14]. Therefore, it is reasonable to assume that S phase precipitates in grain interior during retrogression at 425℃.

4 Discussion

The GP zones, predominantly populating the grain interior under the 7150-T6 temper condition[15], are thermodynamic instable when the alloy are retrogressed above 400℃. And most of GP zones quickly dissolve into matrix, causing a sharply decrease in hardness and a dramatically increase in matrix solubility because of the high diffusion rate of Cu, Mg and Zn elements at the high

retrogression temperature. The dissolution of few large precipitates results in increasing gradually to the saturation of matrix solubility during the initial retrogression stage, where the hardness of the alloy depends on the solution strength of matrix in relation to matrix solubility. The alloy retrogressed at 455℃ is in the (α+Al₃Zr) phase region (see in Fig.4) and possesses microstructure near to solution (see in Fig.2b), which is stable to obtain hardness independent of retrogression times. While S phase grain interior and θ phase in grain boundary are formed in the alloy retrogressed at 425℃. The coarsening rate of S phase is very slow due to the high thermostability[16], as a consequence of which the hardness does not decline with retrogression times. On the other hand, the formation of S phase and θ phase results in a decrease of solubility and hardness of matrix. Compared to retrogressed at 425℃, the alloy retrogressed at temperature 400℃ obtains more S phase and less solubility of matrix. However, it is faint for coarse and low densely distributed S phase in contribution to hardness of alloys, and then solution strengthening plays a critical role in enhancing hardness of the alloy retrogressed at 400℃-455℃. Thus, the higher matrix solubility obtained after higher temperature retrogression[17], the higher hardness the alloys will possess.

The precipitation leads to a dramatically increase in hardness when the alloy is reaged at 120℃ for 24h after retrogression at high temperature. And the effect of reaging hardening makes a major contribution to the hardness of the alloy, which is related to the matrix solubility. The hardness of the alloy by HTRRA treatment gradually increases to a maximum hardness due to the gradual increase in matrix solubility during retrogression. Moreover, a higher retrogression temperature, increasing the dissolution degree, promotes matrix solubility and hardness after reaging treatment. The 7150 HTRRA treatment (retrogression at 455℃) obtains the highest hardness in comparison with 425℃ and 400℃. On the other hand, an increase in matrix solubility is beneficial to the formation of more stable precipitates [17]. The grain boundary precipitates are formed in the alloy retrogressed at 455℃, reducing the solubility of matrix and the driving force of re-ageing, with the result that the hardness lowers to 7150-T6 temper. However, the reduction in the matrix solubility leads to a rapid increase in electric conductivity[18]. The formation of S phase grain interior and θ phase in grain boundary increase electric conductivity for the 7150 RRA treatment. Compared to that retrogressed at 425℃, the electric conductivity of the alloy retrogressed at 400℃ obtains higher electric conductivity, due to the formation of much S phase. Furthermore, the alloy retrogressed at temperature above 400℃ is not susceptible to the retrogression time in comparison to retrogression at regular temperature, and the alloy at retrogression temperature 425℃ possesses an optimum combination of hardness and electric conductivity, close to the level of T77 temper. Therefore, it might be a new way, in a sense, for high temperature retrogression and re-aging to apply to thick section components.

5 Conclusion

This work has elucidated the response of 7150 aluminum alloy to high temperature retrogression and re-aging treatment in terms of hardness, electric conductivity and microstructure. For the retrogression temperature of 455℃, 425℃ and 400℃, the hardness dramatically decreased to a minimum hardness level during retrogression and then rapidly increased by re-aging treatment. The formation of grain interior S phase and θ phase in grain boundary resulted in a decrease in hardness while an increase in conductivity. The alloy after certain HTRRA treatment (see retrogressed at 425℃ for 30 minutes) obtains hardness up to 178HV with retention of an electric conductivity of 35.5 %IACS, close to the level of 7150-T77 temper. Moreover, the microstructures of alloys retrogressed at temperatures above 400℃ are less sensitivity to the retrogression time, thus, the HTRRA treatment might be a substitute process for obtaining satisfied combination properties of

Al-Zn-Mg-Cu alloys and applied to thick section components

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