

Effect of high-temperature pre-precipitation on microstructure and properties of 7055 aluminum alloy^①

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Abstract: The near-solvus pre-precipitation following higher temperature solution treatment was performed on 7055 aluminum alloy. The effect of the pre-precipitation on the microstructure, age hardening and stress corrosion cracking of 7055 alloy was investigated. The optical and transmission electron microscopy results show that the near-solvus pre-precipitation can be limited to grain boundary and enhance the discontinuity of grain boundary precipitates in the sequent age. The stress corrosion cracking resistance of aged 7055 alloys could be improved with non-deteriorated strength and plasticity via the pre-precipitation.

Key words: 7055 aluminum alloy; stress corrosion; precipitation; solution

CLC number: TG 249.9

Document code: A

1 INTRODUCTION

Al-Zn-Mg-Cu series high-strength aluminum alloys with high strength-to-density ratio and excellent mechanical properties have been the primary structural materials of aircraft in the space and ground transportation vehicles. The development and extensive utilization of high-strength 7xxx series are hampered by poor corrosion resistance, especially stress corrosion cracking (SCC) resistance. Over the past few years, the studies about stress corrosion cracking and ageing treatment process have been proceeding in order to obtain materials that are not susceptible to stress corrosion cracking but have optimum strength^[1, 2].

Although the mechanism on stress corrosion susceptibility is intricate and affected by all kinds of microstructure factors, the two common ideas have been formed that grain boundary precipitates by corrosive media first dissolved in the anode and atomic hydrogen enriching at grain boundaries led to hydrogen embrittlement (HE)^[3-5]. The quantity and dispersion of matrix precipitates and discontinuity of grain boundary precipitates determined the strength and resistance to corrosion of 7xxx series high-strength aluminum alloy separately^[3-7]. Hitherto, precipitates have been only usually adjusted and controlled in the ageing sequence^[1-3, 6-10]. Presently there are three ageing processes frequently used in 7xxx series aluminum alloys, namely, peak strength temper (T6), over-aged temper (T73) and RRA or T77 temper. T6 tempered materials have the highest strength but poor resistance to stress corrosion and exfoliation corrosion. Over-aged process can improve stress corrosion

cracking resistance by impelling grain boundary precipitates to grow and present discontinuous distribution, but synchronously matrix precipitates grow up and strength of alloys lose 10% - 15%. RRA or T77 temper can combine strength with resistance to corrosion favorably by precipitates in matrix presenting as T6 temper and precipitates at grain boundaries presenting as over-aged state by retrogression, it is difficult to extensively apply it in the industry because this process demands heat preservation for a little time at retrogression temperature^[1, 11, 12].

In order to obtain effects resembling retrogression in RRA and to probe into new heat treatment methods, the near-solvus pre-precipitation following higher temperature solution treatment on 7055 high-strength aluminum alloy is investigated. The purpose is to improve the synthetic properties of alloys, especially to resist stress corrosion cracking by the near-solvus pre-precipitation (a little dynamic) limited to grain boundary altering precipitation state in matrix and grain boundaries in the subsequent ageing process. 7055 high-strength aluminum alloy has been developed on the basis of 7050 and 7150 alloys by increasing the contents of Zn, Cu in alloy. It is necessary that improvements in stress corrosion cracking resistance of this alloy are in favor of its applications even if it has very high strength-to-density ratio^[1, 13].

2 EXPERIMENTAL

The composition of the 7055 alloy was Al-8.4Zn-2.2Mg-2.6Cu-0.20Zr (mass fraction, %).

① Received date: 2003 - 01 - 21; Accepted date: 2003 - 03 - 26

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99.9% high purity aluminum was adopted as raw materials. Melting of alloy proceeded in a crucible furnace. Cu and Zr were added to Al-Zr-Mg melts in intermediate metals. The temperature of alloy melt was kept at 700–740 °C. The melt was refined by adding 0.2%–0.4% C_2Cl_6 for 10–15 min, and then cast into cylindrical iron moulds with diameter of 45 mm. The as-cast 7055 alloy ingots were homogenized by holding at 455–470 °C for 50 h.

The homogenized ingots were extruded at 400–430 °C into 15 mm diameter bars with an extrusion ratio of 9 in 500 t load extrusion machine. The extrusion canister diameter was 45 mm. It was necessary that the rate of extrusion was properly controlled so as to assure the uniformity of deformation structure.

Solution (pre-precipitation) treatment of extrusion bars is as follows: firstly, samples were held at 450 °C and 470 °C separately for 1 h, then kept at 485 °C for 3 h, and finally the temperature descended to 480 °C or 475 °C for 30 min. The soluble bars were quenched in ambient temperature water, and then artificial ageing treatment proceeded.

Six samples with different solution (pre-precipitation) and ageing treatments were compared. 1[#]–3[#] samples were held at 485 °C for 3 h and then directly quenched in cold water, 4[#]–6[#] samples were held at 480 °C for 30 min after being kept at 485 °C for 3 h, subsequently quenched in cold water. The proper condition of T6 temper of this alloy was 130 °C, 24 h by measuring the rules that ageing hardening (HRB) of 7055 alloys varies as temperature and time. Solution (pre-precipitation) and ageing process of all the samples are shown in Table 1.

Stress corrosion testing was performed according to the Chinese GB12445.1–90 specification stress corrosion test method for double cantilever beam samples in high strength aluminium alloys. Testing medium was 3.5% (mass fraction) NaCl solution and its temperature was controlled at (35 ± 1) °C. For K_{Ic} corresponding to crack expansion in stress corrosion, the dimension of samples accorded

with $B \geq 2.5(K_{Ic}/\sigma_{0.2})^2$ and $(1-a) \geq 2.5(K_{Ic}/\sigma_{0.2})^2$.

All properties measurements were made at ambient temperature on the specimens machined from heat-treated extrusion bars. Electric resistance rate of bars were determined using SX1931 digital ohmmeter and four probes methods. The HBRVU-187.5 type sclerometer was used to measure microhardnesses (HRB). Tensile properties were determined on the specimens with dimensions of 8 mm diameter and 48 mm gauge length by an Instron 8032 mechanical testing machine.

Metallographic specimens of quenching after solution and pre-precipitation were prepared. Microstructure was observed by using optical microscope. The distribution of precipitates in matrix and grain boundaries after pre-precipitation and ageing process in alloys were observed in a JEOL-100 transmission electron microscope.

3 RESULTS

3.1 Soluble (pre-precipitation) structure

As shown in Figs. 1(a), (b), (d) and Figs. 2(a), (b), minute precipitated particles did not exist in alloys structure after isothermal treatment at 485 °C, but minute precipitates appeared in alloys structure by isothermal treatment at 485 °C followed by quenching at 480 °C or 475 °C. Comparably, precipitates were mainly located at grain boundary by isothermal treatment at 485 °C followed by quenching at 480 °C, and precipitates existed both in the grain and at grain boundary and were excessive by isothermal treatment at 485 °C followed by quenching at 475 °C. As shown in Figs. 1(c) and (d), different from isothermal treatment at 485 °C following quenching at 475 °C, alloys after isothermal treatment and quenching at 475 °C had no precipitation in the grain and at grain boundary, namely precipitation was independent of lower temperature treatment.

Table 1 Solution (pre-precipitation)-ageing process parameters of 7055 aluminum alloys

Series	Solution (pre-precipitation)	Ageing process
1 [#]	450 °C, 1 h+ 470 °C, 1 h+ 485 °C, 3 h	130 °C, 24 h
2 [#]	450 °C, 1 h+ 470 °C, 1 h+ 485 °C, 3 h	173 °C, 3 h+ 130 °C, 24 h
3 [#]	450 °C, 1 h+ 470 °C, 1 h+ 485 °C, 3 h	130 °C, 24 h+ 173 °C, 3 h+ 130 °C, 24 h
4 [#]	450 °C, 1 h+ 470 °C, 1 h+ 485 °C, 3 h+ 480 °C, 0.5 h	130 °C, 24 h
5 [#]	450 °C, 1 h+ 470 °C, 1 h+ 485 °C, 3 h+ 480 °C, 0.5 h	173 °C, 3 h+ 130 °C, 24 h
6 [#]	450 °C, 1 h+ 470 °C, 1 h+ 485 °C, 3 h+ 480 °C, 0.5 h	130 °C, 24 h+ 173 °C, 3 h+ 130 °C, 24 h

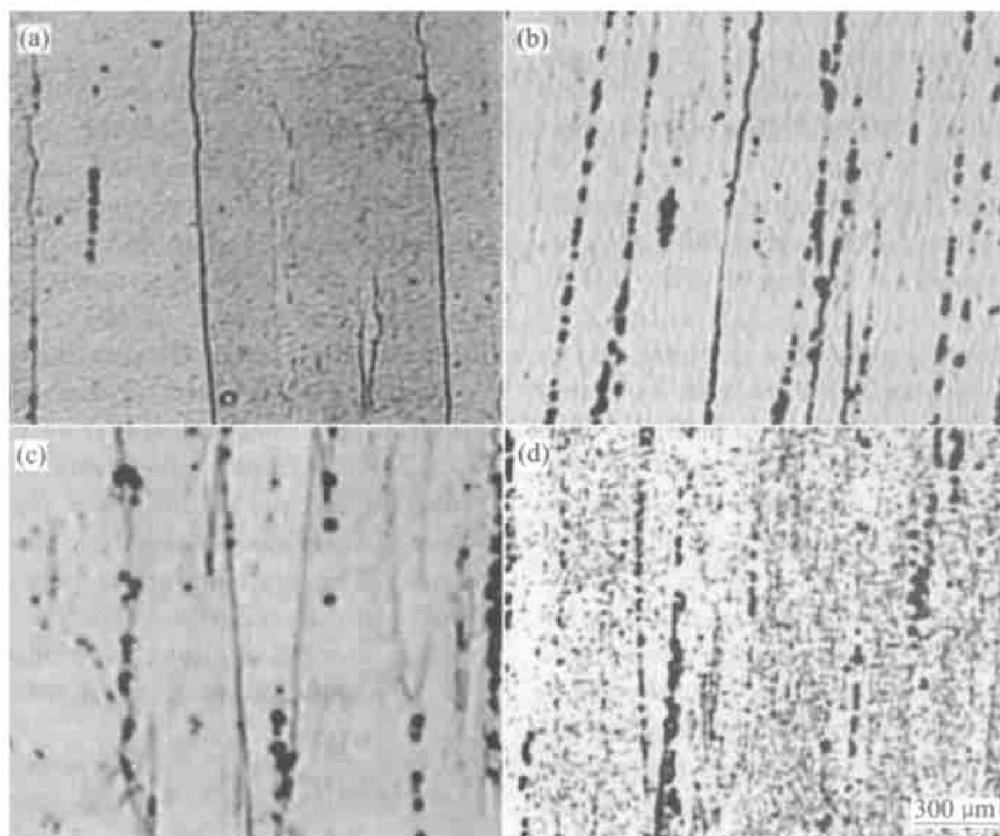


Fig. 1 Optical photographs of 7055 aluminum alloys solution-treated at different temperatures

(a) $-485\text{ }^{\circ}\text{C}$, 3 h; (b) $-485\text{ }^{\circ}\text{C}$, 3 h and $480\text{ }^{\circ}\text{C}$, 0.5 h;
(c) $-475\text{ }^{\circ}\text{C}$, 3 h; (d) $485\text{ }^{\circ}\text{C}$, 3 h and $475\text{ }^{\circ}\text{C}$, 0.5 h

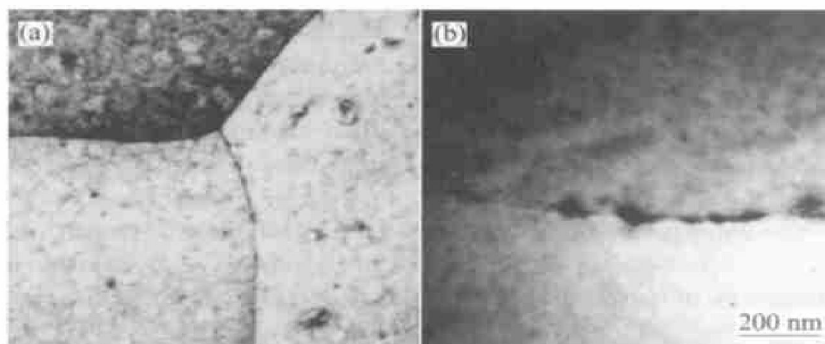


Fig. 2 TEM bright field images of quenched 7055 alloys solution-treated at different temperatures

(a) $-485\text{ }^{\circ}\text{C}$ for 3 h; (b) $-485\text{ }^{\circ}\text{C}$ for 3 h and $480\text{ }^{\circ}\text{C}$ for 0.5 h

3.2 Peak ageing condition

The time evolution of hardness (HRB) at $120\text{ }^{\circ}\text{C}$, $130\text{ }^{\circ}\text{C}$, $140\text{ }^{\circ}\text{C}$ are represented in Fig. 3. It is shown that peak ageing (T6) condition of this alloy was not at $120\text{ }^{\circ}\text{C}$ for 24 h but at $130\text{ }^{\circ}\text{C}$ for 24 h.

3.3 Hardness, electric resistance and tensile property

As shown in Table 2, the electric resistance of the samples by pre-precipitation all descended after ageing but their hardness was basically uniform, and yield strength and tensile strength were appreciably enhanced, and elongation was 10% or so. According to the relation of electric resistance with resistance to stress corrosion, stress corrosion cracking resistance

of 7055 alloy could be improved to some degree by

Table 2 Hardness, electric resistance and tensile properties of 7055 alloys

Series	HRB	$\rho/(\text{n}\Omega\cdot\text{m})$	σ_b/MPa	$\sigma_{0.2}/\text{MPa}$	$\delta/\%$	$\phi/\%$
1 [#]	98.0	62.3	718	697	10.5	8.1
2 [#]	98.5	52.9	667	656	10.1	26.4
3 [#]	97.0	53.7	691	680	12.3	15.6
4 [#]	98.0	57.5	755	738	10.0	7.6
5 [#]	97.6	52.1	682	671	9.5	18.9
6 [#]	96.5	53.0	705	699	9.4	15.6

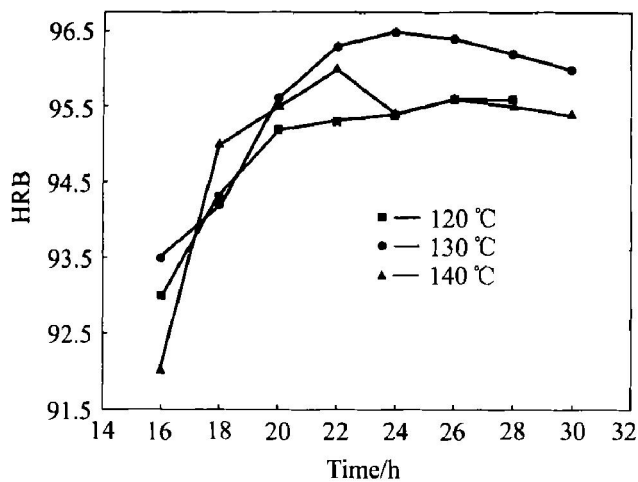


Fig. 3 Ageing hardening of 7055 alloys at different temperatures

pre-precipitation at high temperatures.

3.4 Stress corrosion susceptibility

A related diagrammatic curves of growth rate of stress corrosion cracking and stress intensity factor of double cantilever bialk samples is shown in Fig. 4. In peak ageing (1[#] and 4[#]), over ageing

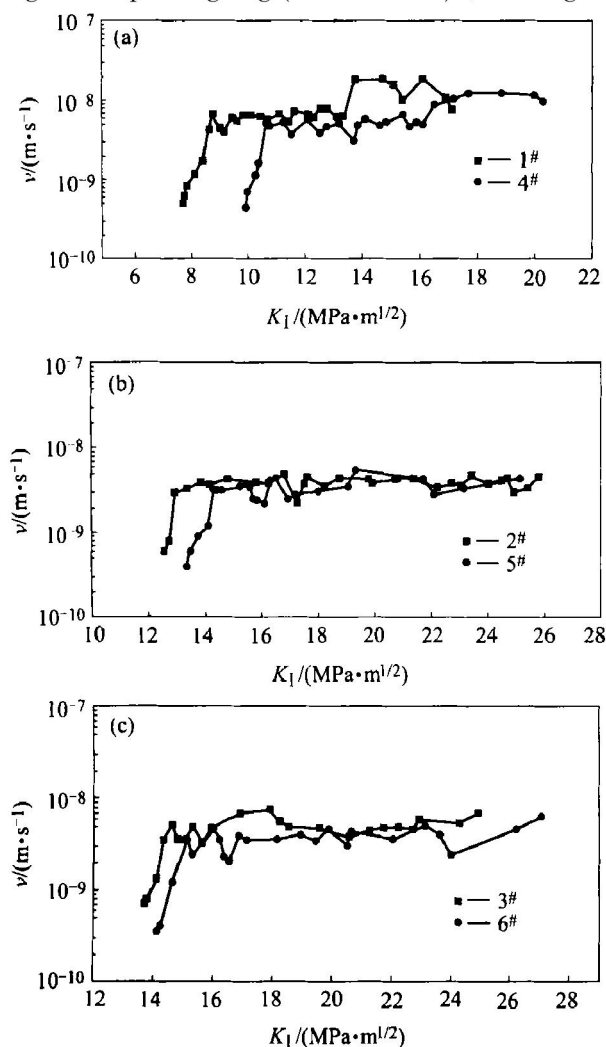


Fig. 4 Effect of pre-precipitation on stress corrosion cracking of 7055 aluminum alloys

(2[#] and 5[#]) and retrogression and re-ageing (3[#] and 6[#]), stress corrosion cracking of the samples of pre-precipitation at high temperatures was compared with quenching after solution in Figs. 4(a), (b), (c). It was discovered that compared with the samples with quenching after solution, the resistance to stress corrosion of the samples with pre-precipitation after solution was obviously enhanced below flat zone of stress corrosion rate under the above ageing conditions.

4 DISCUSSION

Precipitation strengthening is a primary mechanism in 7xxx series high strength aluminium alloy, but 7xxx series are strengthened by precipitates, at the same time the stress corrosion cracking resistance currently descended. Due to the fact that at the nucleation and growth of the precipitates in grain were different from that at grain boundaries in thermodynamics and dynamics, precipitated structure in grain and at grain boundaries are discrepant obviously. Precipitates in grain contains commonly G. P. zone, η' , η phase; yet precipitates at grain boundaries are η phase and precipitate-free zones (PFZ) exits at grain boundaries. Characteristic of these precipitated structure changes easily with parameters of heat treatment processes^[3, 6, 10]. A small quantity of equilibrium precipitates η phases and a great deal of small dimension and dispersed metastable phases (G. P. zone, η') contributes to precipitation hardening and enhancement of alloy strength, moreover, size and spacing of precipitates at grain boundaries (η phase) are larger, and stress corrosion cracking resistance is higher^[3-8, 12]. Because grain boundary precipitates by stress corrosion first dissolved as the anode, stress corrosion cracking resistance were enhanced by enlarging spacing of grain boundary precipitates. Besides, atomic hydrogen enriching at coarse grain boundary precipitates leads to transgression of molecule hydrogen and decreases the content of molecule hydrogen at grain boundary, ultimately restrains hydrogen embrittlement. Therefore, 7xxx series could be provided with high strength and favourable corrosion resistance simultaneously by utilizing heat treatment process to extend the difference of precipitates in grain and at grain boundary.

So far, there have been abundant researches about 7xxx series alloys which were optimized by altering ageing sequence. In ageing, alloy matrix was at the state of high saturation, the control of precipitated structure mostly depended on dynamics of course. In fact, when alloy matrix is located at low saturation (near-solvus) high temperature, precipitation of grain and grain boundary are controlled by thermodynamics. By adjusting temperature to confine over-saturation to some degree, grain precipitation is

limited by nucleation energy, yet grain boundary precipitation exists, and then the quantity of grain boundary precipitation is restricted and adjusted by measuring temperature, namely over-saturation. As shown in Fig. 1 and Fig. 2, near-solution temperature precipitates structure confirms that near-solvus temperature precipitation could limit the precipitation to grain boundary. As shown in Fig. 4, the testing results of stress corrosion indicated that the near-solvus pre-precipitation can enhance SCC resistance obviously. The results illuminate that the effects of near-solution pre-precipitation at high temperature on SCC resistance abide by the common relation of stress corrosion property and structure.

Table 2 shows that pre-precipitation at high temperature does not lower the tensile strength of the alloy, instead improves it slightly. Microscopical yield and the fracture of the alloy at tensile states contains intricate microcosmic plastic deformation and the process of budding and extent of cracking. High temperature pre-precipitation enhancing alloy strength is possibly concerned with alteration of grain boundary precipitation state to restrain budding and extent of cracking, its exact mechanism deserves advanced research.

5 CONCLUSIONS

1) Lower temperature precipitation process after high temperature solution can form discontinuous grain boundary precipitated phases under condition of near-solution, and increases size and spacing of grain boundary precipitation particles at ageing sequence.

2) High temperature near-solvus pre-precipitation process can not only retain alloy strength but also enhance alloy resistance to stress corrosion cracking.

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(Edited by LONG Huai-zhong)