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Mechanical properties, corrosion behaviors and microstructures of 7075 aluminium alloy with various aging treatments

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Abstract: The influence of two novel aging treatments, T6I6 (130 ℃, 80 min + 65 ℃, 240 h+130 ℃, 18 h) and high-temperature pre-precipitation(HTPP) aging (445 ℃, 30 min+120 ℃, 24 h) on the tensile properties, intergranular corrosion, exfoliation corrosion behaviors and microstructures of 7075 Al alloy was studied, which were compared with the T6, T73 and RRA treatments. Fine η' precipitate with high density was obtained in the alloy with the T6 and RRA treatments. The η' precipitate density in the HTPP aged alloy is decreased due to the formation of coarse particles during the pre-precipitation process at high temperature of 445 ℃. The 7075-T6I6 alloy possesses higher precipitate density and whole precipitate volume fraction within the grain than the 7075-T73 alloy, and its whole precipitate volume fraction is even greater than that of the 7075-T6 alloy. Compared with T6 treatment, the RRA, T73, T6I6 and HTPP aging treatments cause the discontinuous distribution of the η precipitates at the grain boundary, which decreases the intergranular corrosion and exfoliation corrosion susceptibility of the alloy. Meanwhile, the T6I6 and RRA treatments can keep the high strength of the 7075 Al alloy, but the studied HTPP aging and T73 treatments lower its strength.

Key words: 7075 Al alloy; aging treatment; tensile property; corrosion behavior; microstructure

1 Introduction

7××× series Al alloys have been widely used as structural materials in aeronautical industries due to their attractive comprehensive properties, such as low density, high strength, ductility, toughness and resistance to fatigue[1−3]. However, 7××× series Al alloys are sensitive to localized corrosion, such as intergranular corrosion, exfoliation corrosion and stress corrosion cracking(SCC). Therefore, further applications of the 7××× series Al alloys require enhanced corrosion resistance.

The corrosion resistance of the 7××× series Al alloys can be modified by heat-treatment. It is known that although the 7××× series Al alloys with T6 treatment possess high strength, their localized corrosion resistance is poor. To increase their corrosion resistance, over-aging treatments such as T73, T76 and T74 have been developed. However, the strength of the 7××× series Al alloys with these over-aging treatments is decreased. The 7××× series Al alloys with retrogression and re-aging (RRA) treatment possess high strength and good SCC resistance[4−5]. However, the RRA treatment can not be used for large-section Al alloys due to its very short retrogression time[6].

To keep the high strength of the 7××× series Al alloys and improve their corrosion resistance simultaneously, novel heat treatments have been developed. CHEN et al[7−8] and HUANG et al[9−10] advanced a novel aging treatment, called high-temperature pre-precipitation(HTPP) aging treatment. It was found that the HTPP aging treatment not only kept the high strength of 7A52 and 7055 Al alloys, but also enhanced their resistance to intergranular corrosion, exfoliation corrosion and SCC[7−10]. Recently, another aging treatment, secondary aging, has been developed. This process involves interrupting a normal T6 temper with a period when the alloys are held at a lower temperature. It was found that T6I6, one of the secondary

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aging tempers, typically displayed simultaneous improvements in the strength, hardness and fracture toughness of many Al alloys[11−13].

7075 Al alloy has a long history, and it is still widely used in aeronautical industries. However, few reports about the effects of the secondary aging and HTPP aging treatments on its mechanical properties and corrosion behaviors have been investigated. In the present work, the comparative study of the effects of the various heat treatments, especially the secondary aging and HTPP aging on its tensile properties, corrosion behaviors and microstructures was carried out.

2 Experimental

The alloy used for this study was an as-received commercial 7075 Al alloy plate with a thickness of 2 mm. The alloy plate has undergone various aging treatments of T6, T73, RRA, T6I6 and HTPP aging after solution treatment and quenching. The various heat treatment processes are listed in Table 1.

Table 1 Various heat treatment processes applied to 7075 Al alloy

Aging state	Process
T6	Solution-treatment at 470 ℃ for 1 h, quenching in cold water, and aging at 120 ℃ for 24 h
T73	Solution-treatment at 470 ℃ for 1 h, quenching in cold water, aging at 120 ℃ for 24 h, and aging at 160 ℃ for 30 h
RRA	Solution-treatment at 470 ℃ for 1 h, quenching in cold water, aging at 120 ℃ for 24 h, retrogression at 203 ℃ for 10 min, and re-aging at 120 ℃ for 24 h
T6I6	Solution-treatment at 470 ℃ for 1 h, quenching in cold water, pre-aging at 130 ℃ for 80 min, interrupted aging at 65 ℃ for 240 h, and re-aging at 130 ℃ for 18 h
HTPP aging	Solution-treatment at 470 ℃ for 1 h, cooling to 445 ℃ in resistance oven and maintaining for 30 min for pre-precipitation, quenching in cold water, and aging at 120 ℃ for 24 h

The tensile specimens with a gauge of 30 mm in length and 8 mm in width were cut from the aged 7075 Al alloy plate. The tensile tests were carried out using an INSTRON 4507 testing machine at a tensile speed of 2 mm/min.

Specimens for intergranular corrosion and exfoliation corrosion were cut from the aged 7075 Al alloy plate. The surface for corrosion was ground with abrasive papers, polished with Cr_2O_3 powder, rinsed with acetone, degreased with distilled water and then dried in air. The other surfaces of the specimens were sealed with paraffin. The intergranular corrosion test was performed according to the standard of GB7998−97[14]. The corrosion medium was the solution of 57 g/L NaCl + 10 mL/L H_2O_2, and its temperature was maintained at (35±2) ℃ using a thermostatic bath. After 6 h of immersion in the corrosion medium, the sectional surface of the corroded samples were ground and polished, and the average intergranular corrosion depth was measured with a metallographic microscope with scale.

The accelerated exfoliation corrosion test was performed at room temperature according to EXCO test of ASTM G34−79[15]. The EXCO solution of 4.0 mL/L NaCl+0.5 mL/L KNO_3+0.1 mL/L HNO_3 (pH0.4) was used. After 48 h of immersion in the EXCO solution, the corrosion morphologies of the 7075 Al alloy with various aging treatments were recorded.

The microstructures were observed with a Tecnai G^2 20 Transmission Electron Microscope(TEM). The TEM samples of the 7075 Al alloy with various aging treatments were prepared by conventional electrolytic etching method using a solution of 25% HNO_3+75% CH_3OH. Then the precipitate distribution within the grain and of the area around the grain boundary was observed with the Tecnai G^2 20 TEM.

To explain the different intergranular corrosion sensitivity, in addition to the η($MgZn_2$) precipitate distribution at the grain boundary, the corrosion mechanism associated with the precipitate of η should also be known. Here, it was investigated with the Tecnai G^2 20 TEM. Two TEM samples of the 7075 Al alloy aged at 175 ℃ for 52 h were prepared. The objective of the aging treatment of high temperature was to coarsen the η precipitates. Then, one of the TEM samples was observed with the Tecnai G^2 TEM to obtain its original microstructure. Another TEM sample was immersed in a 3.5% NaCl solution for 2 h, and then observed with the Tecnai G^2 TEM.

3 Results and discussion

Table 2 lists the tensile properties of the 7075 Al alloy with the various aging treatments. It is found that the 7075 Al alloy with the T6, RRA and T6I6 treatments

Table 2 Strength of 7075 Al alloy with various treatments

Aging state	Tensile strength/MPa	Yield strength/MPa
T6	559.8	504.4
T73	527.9	464.6
RRA	568.2	511.8
T6I6	563.0	507.3
HTPP	538.4	468.5

possesses high strength. The T73 and HTPP aging treatments lower its strength. Meanwhile, it is also found that the 7075-T73 alloy possesses the lowest strength.

The intergranular corrosion susceptibility of the 7075 Al alloy is greatly dependent on the heat treatment. The representative intergranular corrosion morphologies and the average corrosion depth of the 7075 Al alloy with various heat treatments are presented in Fig.1 and Table 3, respectively. Compared with the T6 treatment, the RRA and T6I6 treatments not only keep the high strength of the 7075 Al alloy, but also increase its intergranular corrosion resistance. The T73 and HTPP aging treatments can increase its intergranular corrosion resistance, but lower its strength.

Table 3 Average intergranular corrosion depth of 7075 Al alloy with various treatments

Aging state	Average intergranular corrosion depth/mm
T6	0.063 5
T73	0.043 9
RRA	0.036 1
T6I6	0.051 7
HTPP	0.049 8

Fig.2 shows the exfoliation corrosion morphologies of the 7075 Al alloy with various heat treatments. Small lifted surface layers are observed on the alloy. It is found that most of the lifted surface layers on the alloy with the T6 and T6I6 treatments are peeled off and raised to the upside of the solution with gas blisters caused by electrochemical reaction during the corrosion process. The alloy with the T6 and T6I6 treatments is more susceptible to exfoliation corrosion than that with the RRA, T73 and HTPP aging treatments. Meanwhile, it is also found that the 7075-T6 alloy is a little more susceptible to exfoliation corrosion than the 7075-T6I6 alloy, as seen in Figs.2(a) and (d).

Fig.3 shows the TEM bright field micrographs within the grain of the 7075 Al alloy with the T6, T73 and RRA treatments. The precipitates of η' with fine size and high density are observed within the grain of the alloy with the T6 and RRA treatments (see Figs.3(a) and (b)). This micro-structural feature is consistent with their

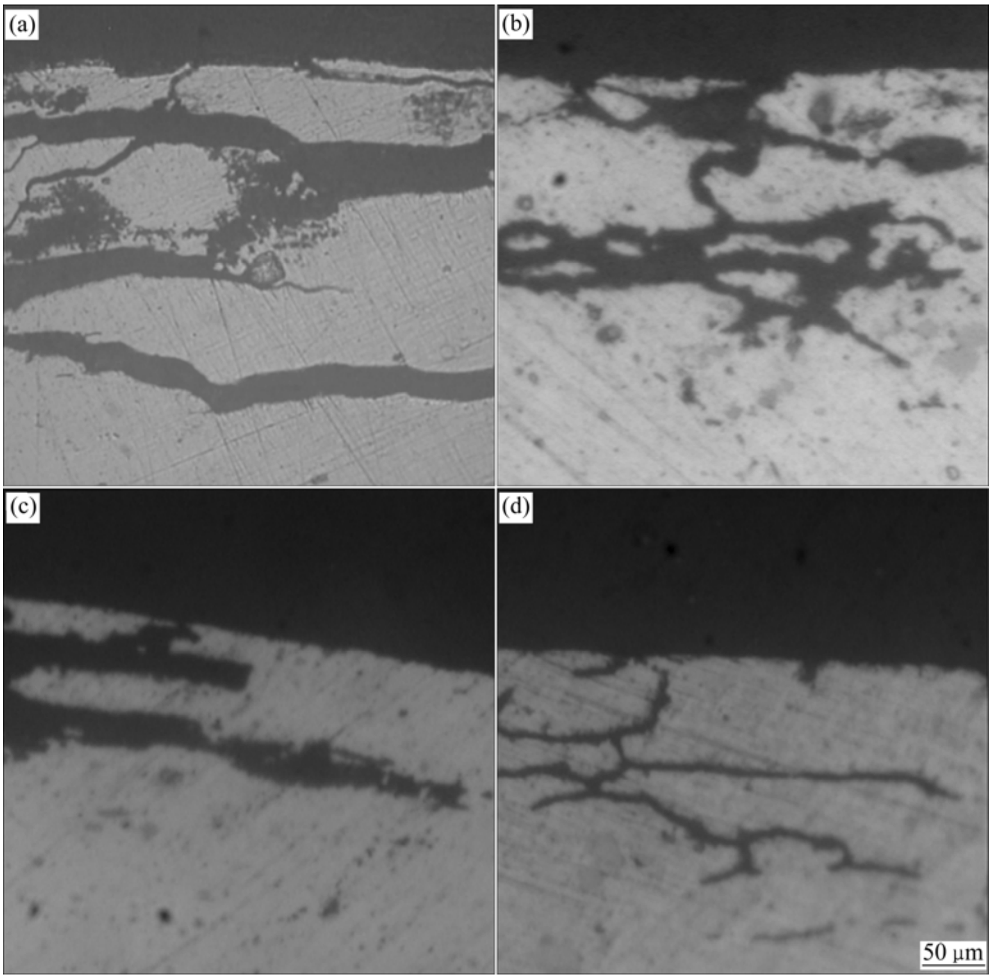

Fig.1 Representative intergranular corrosion morphologies of 7075 Al alloy with T6 (a), T73 (b), T6I6 (c) and HTPP (d) aging treatments

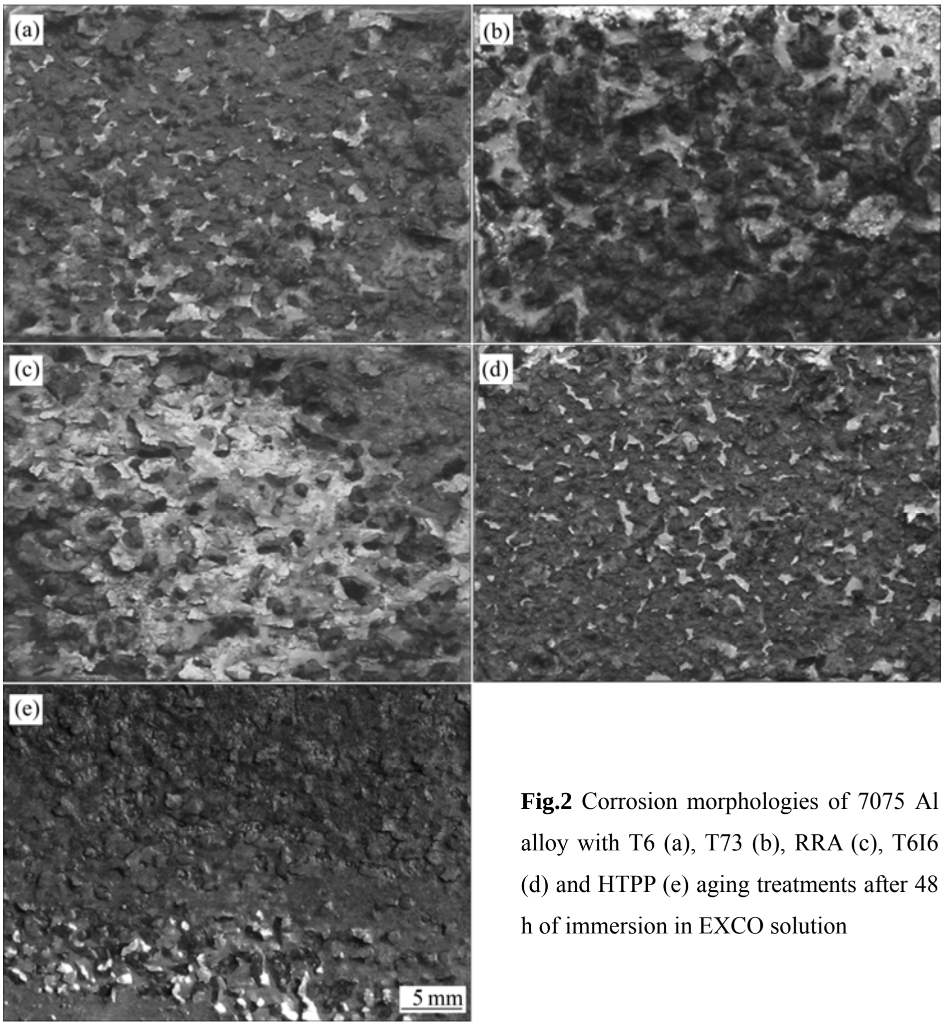

Fig.2 Corrosion morphologies of 7075 Al alloy with T6 (a), T73 (b), RRA (c), T6I6 (d) and HTPP (e) aging treatments after 48 h of immersion in EXCO solution

high strength. The T73 treatment is a two-step aging treatment. During the first aging process at 120 ℃ for 24 h (T6), the phases of η' are precipitated within the grain. Then, during the second aging at a higher temperature of 160 ℃ for a longer time of 30 h, the η' precipitates coarsen and their density is decreased, as seen in Fig.3(c). Sequentially, compared with the T6 temper, the T73 treatment lowers the strength of the 7075 Al alloy.

Fine η' precipitate is observed in the 7075 Al alloy with the HTPP aging treatment, just as in the 7075-T6 alloy. However, its density is clearly lowered (see Fig.4(a)), which is consistent with the lower strength of the HTPP aged alloy. Meanwhile, some coarse particles are also observed (see Fig.4(b)). The EDAX analysis shows that these coarse particles contain the alloying element of Zn, Mg and Cu, as seen in Fig.4(c). The lower density of the η' precipitate and the formation of the coarse particles can be explained by the pre-precipitation process. During the pre-precipitation process at a high temperature of 445 ℃, some equilibrium phases can form and easily coarsen, resulting in a decrease in solute fraction. Therefore, during the aging process at a lower temperature of 120 ℃, the super-saturation degree of the solute is decreased, leading to a lower density of the η' precipitates in the HTPP aged 7075 Al alloy. HUANG et al[9−10] reported that the HTPP aging did not deteriorate the strength of the 7A52 Al alloy. However, in this case, the HTPP aging causes the formation of some coarse particles within the grain of the 7075 Al alloy and lowers its strength. This phenomenon might be associated with an unsuitable pre-precipitation process, but it is still unclear and further study is needed.

Fig.5 shows the TEM bright field micrographs within the grain of the 7075-T6I6 Al alloy. Coarse precipitates of η' are observed. However, compared with the 7075-T73 alloy, its density and whole precipitate volume fraction are obviously higher, which may be one cause of its high strength. The T6I6 treatment is a three-step aging treatment. During the pre-aging at 130 ℃ for 80 min, the η' phases nucleate within the grain. During the interrupted aging at a lower temperature of 65 ℃ for a much longer time of 240 h, greater super-saturation degree causes a greater number

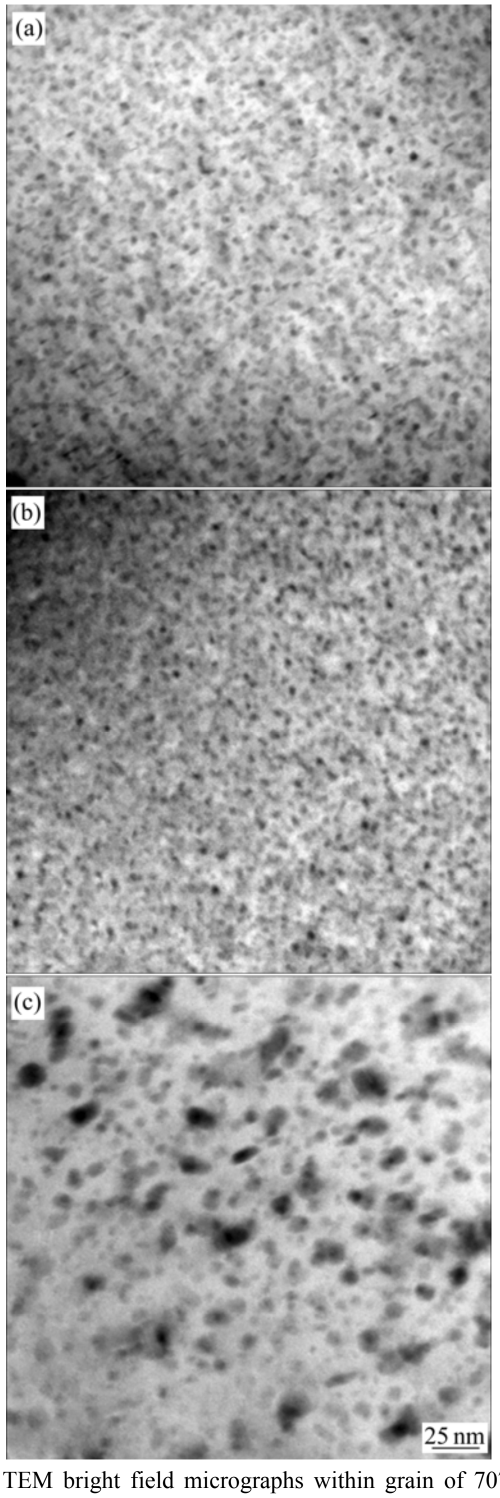

Fig.3 TEM bright field micrographs within grain of 7075 Al alloy with T6(a), RRA(b) and T73(c) treatments

of finer and more densely dispersed precipitates[16], as shown in Fig.5(b). Although some η' precipitates grow and coarsen during the re-aging process at 130 ℃ for 18 h, there is greater whole precipitate volume fraction in the final microstructure of the 7075-T6I6 alloy, as compared with the 7075-T6 alloy.

Fig.6 shows the TEM bright field micrographs of the area around the grain boundary of the 7075 Al alloy

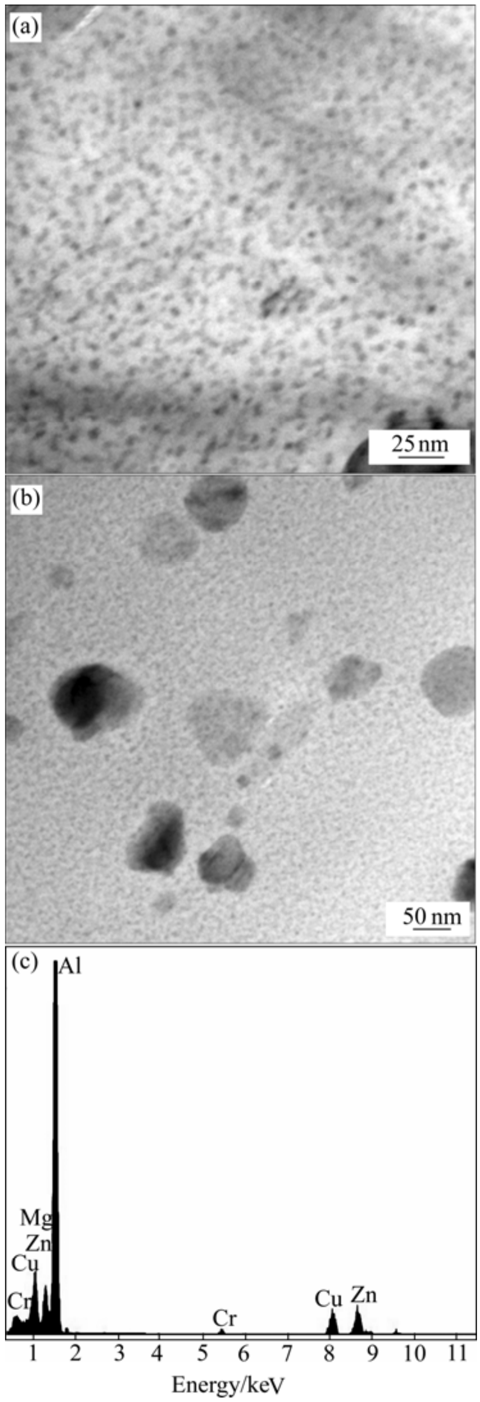

Fig.4 TEM bright field micrographs(a, b) and EDAX spectrum (c) of coarse particles within grain of HTPP aged 7075 Al alloy

with the T6, RRA, T73 and HTPP aging treatments. The precipitate of η is distributed continuously at the grain boundary of the 7075-T6 Al alloy, as seen in Fig.6(a). However, it is distributed discontinuously at the grain boundary of the 7075 Al alloy with the RRA, T73 and HTPP aging treatments, as seen in Figs.6(b), (c) and (d).

A lot of research work has been reported on the grain boundary microstructure formation of the 7××× series Al alloys with the RRA and T73 treatments

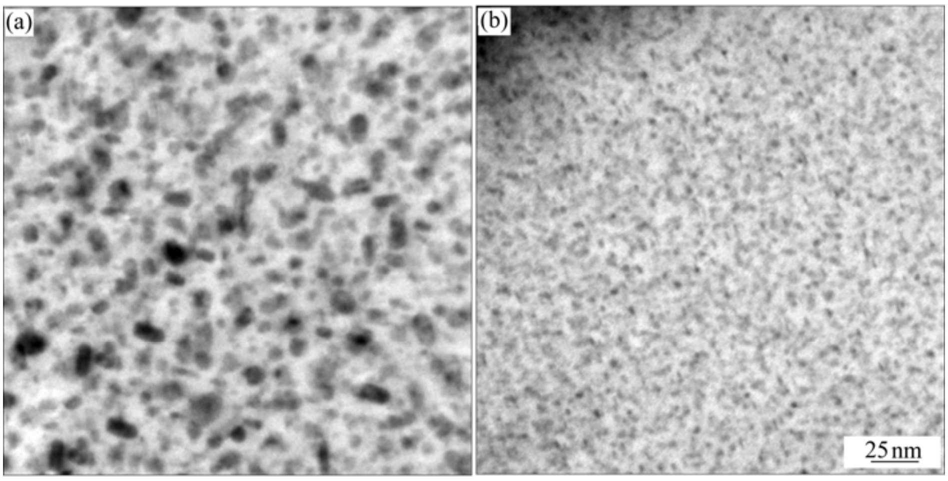

Fig.5 TEM bright field micrographs within grain of 7075 Al alloy: (a) T6I6; (b) 130 ℃, 80 min+65 ℃, 240 h

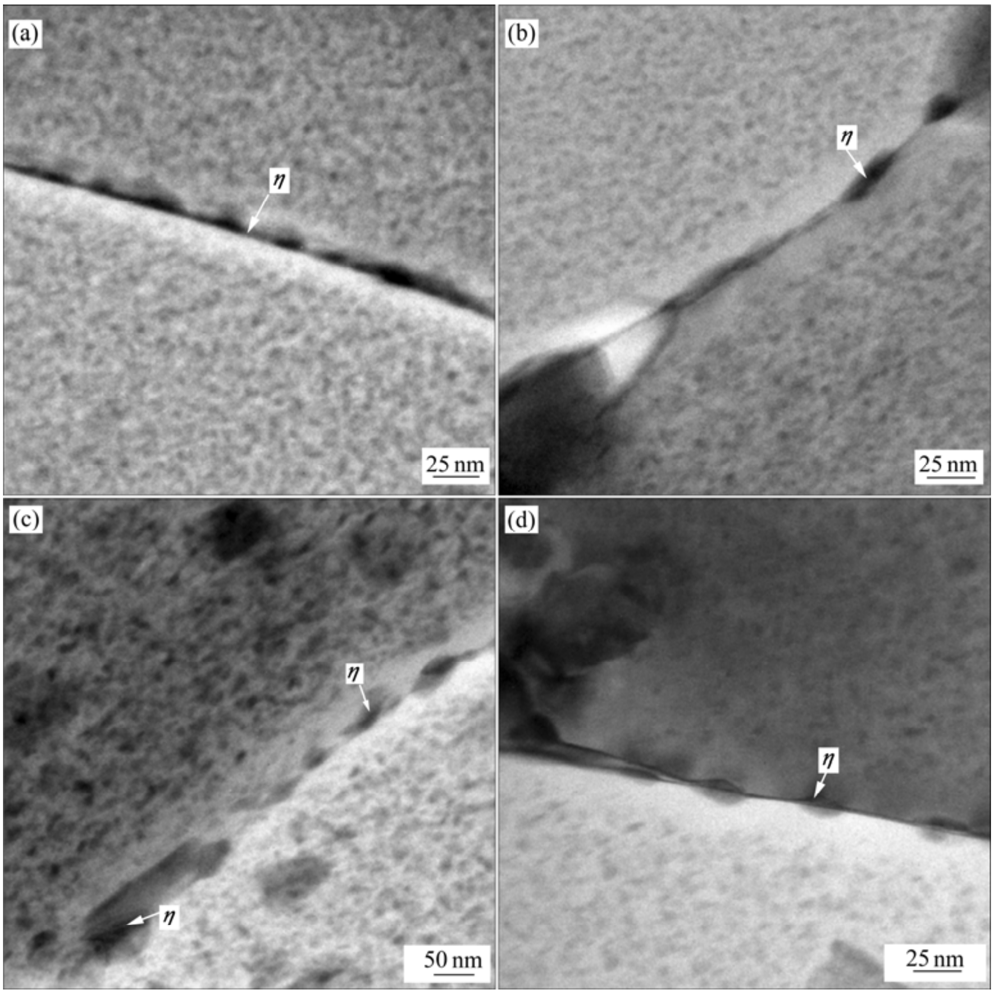

Fig.6 TEM bright field micrographs of area around grain boundary of 7075 Al alloys with T6 (a), RRA(b), T73(c) and HTPP(d) aging treatments

[5, 6, 17−18]. The discontinuous distribution of the η precipitates at the grain boundary in the HTPP aged 7075 Al alloy is correlated with the pre-precipitation process at the temperature of 445 ℃. At this pre-precipitation temperature, some equilibrium phases precipitate at the grain boundary. After the alloy is quenched and then aged at 120 ℃, the η precipitates grow on these phases that formed during the pre-precipitation process[10]. Because of the low super-saturation degree at the pre-precipitation temperature close to the solution temperature, these pre-formed phases possess a much low density, which leads to the discontinuous distribution of the η precipitates at the grain boundary of the HTPP aged 7075 Al alloy.

The precipitate of η is also distributed discontinuously at the grain boundary of the 7075-T6I6 Al alloy, as seen in Fig.7(a). During the pre-aging process at 130 ℃ for 80 min, the η precipitates should nucleate continuously at the grain boundary. Then, during the interrupted aging process at 65 ℃ for a much longer time of 240 h, the η precipitates accumulate to form discontinuous strip-like morphology, as seen in Fig.7(b). During the re-aging process at 130 ℃ for 18 h, the η precipitates are coarsened.

From the TEM observation of the area around the grain boundary, it is also found that there is large spacing between the η precipitates of the 7075-RRA Al alloy. Although the strip-like η precipitate of the 7075-T6I6 alloy is distributed discontinuously at the grain boundary, its whole volume fraction is greater than that of the alloy with the T73, RRA and HTPP aging treatments.

To explain the different corrosion susceptibility of the 7075 Al alloy, besides the precipitate distribution at the grain boundary, its electrochemical behavior also should be known. From our previous research[19], it was known that the corrosion potential of the η precipitate was negative with respect to that of the alloy base at its adjacent periphery, and its corrosion current density was much greater, indicating that the η precipitate was more sensitive to corrosion than the alloy base. The TEM bright field micrographs of the area around grain boundary of the 7075 Al alloy aged at 175 ℃ for 52 h are shown in Fig.8. The grain boundary shows coarse equilibrium η precipitates. It is clearly seen that the color of the η precipitates is darker than that of the alloy base at their adjacent periphery, as shown in Fig.8(a). After 2 h of immersion of the TEM sample in the 3.5% NaCl solution, it is found that the color of the η precipitates turns white, being opposite to their original color, as

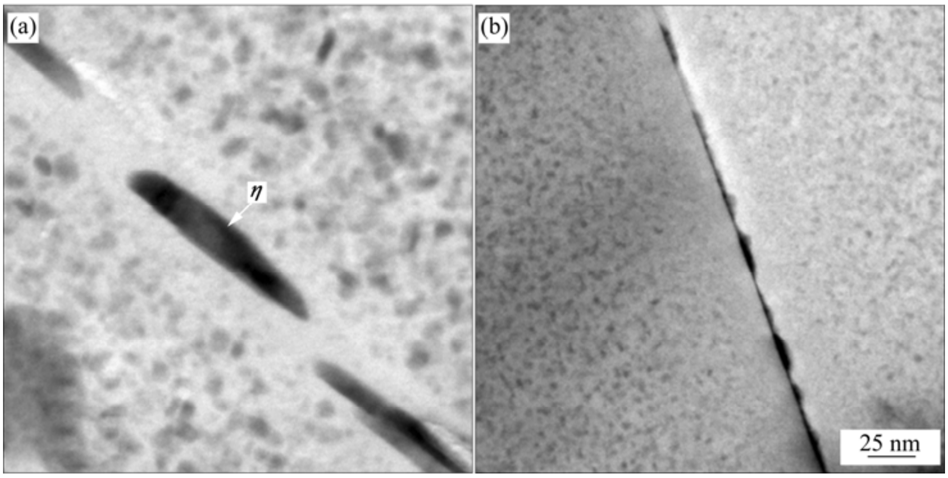

Fig.7 TEM bright field micrographs of area around grain boundary of 7075 Al alloy: (a) T6I6; (b) 130 ℃, 80 min+65 ℃, 240 h

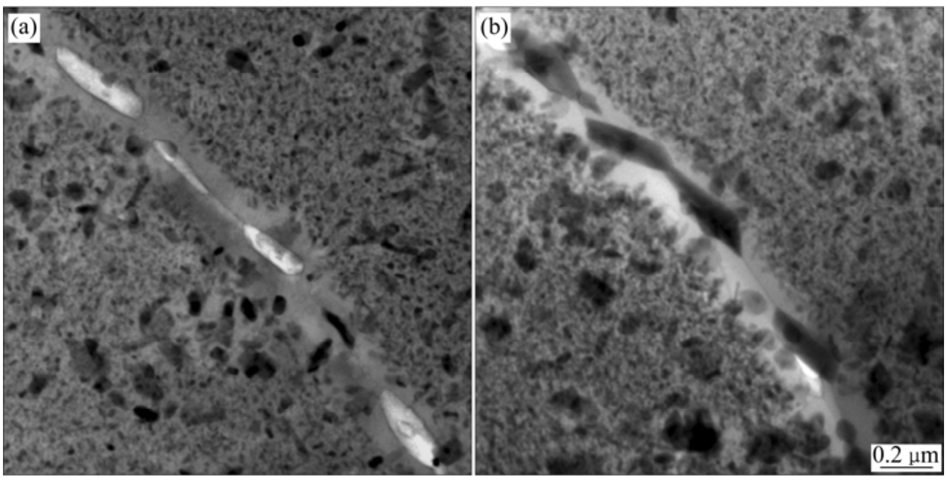

Fig.8 TEM bright field micrographs of area around grain boundary of 7075 Al alloy (a) and after 2 h (b) of immersion in 3.5% NaCl solution

seen in Fig.8(b). The color variation indicates that the precipitates of η in the 7075 alloy are preferentially attacked. The preferential corrosion of the η precipitates at the grain boundary is in agreement with the electrochemical behaviors of the precipitate of η and the alloy base. Therefore, as the 7075 Al alloy is immersed in the 3.5% NaCl solution, the η precipitates will act as anodic zones and be preferentially attacked. However, the alloy base at their adjacent periphery is protected as a cathodic zone.

The precipitates of η are distributed continuously at the grain boundary of the 7075-T6 Al alloy (see Fig.6(a)). Therefore, there exists an active corrosion path resulting from the galvanic reaction between the anodic precipitates of η at the grain boundary and the alloy base at their adjacent periphery, which leads to its greatest susceptibility to intergranular corrosion. The precipitates of η at the grain boundary are distributed discontinuously in the 7075 Al alloys with the RRA, T73, T6I6 and HTPP aging treatments, no continuous corrosion path exists in these alloys, and their susceptibility to intergranular corrosion is decreased. The spacing between the η precipitates at the grain boundary of the 7075-RRA Al alloy is the largest, and its susceptibility to intergranular corrosion is the lowest.

Exfoliation corrosion is developed from intergranular corrosion. During intergranular corrosion process, the corrosion product accumulates at the grain boundary, resulting in a wedging force and finally lifting the alloy surface. The more sensitive the alloy is to intergranular corrosion, the more susceptible it is to exfoliation corrosion. This can explain why the RRA, T73, T6I6 and HTPP aging treatments decrease the exfoliation corrosion susceptibility of the 7075 Al alloy.

4 Conclusions

1) The used HTPP aging treatment lowers the η' precipitate density of the 7075 Al alloy due to the precipitation of some coarse particles during the pre-precipitation process at the temperature of 445 ℃. The 7075-T6I6 alloy possesses higher precipitate density and whole precipitate volume fraction within the grain than the 7075-T73 alloy, and its whole precipitate volume fraction is even greater than that of the 7075-T6 alloy, due to the interrupted aging process at a low temperature of 65 ℃ for a much long time of 240 h.

2) The precipitate of η is distributed continuously at the grain boundary of the 7075-T6 Al alloy. However, the RRA, T73, T6I6 and HTPP aging treatments cause the discontinuous distribution of the η precipitates at the grain boundary.

3) Compared with the T6 treatment, the RRA and T6I6 treatments not only keep the high strength of the 7075 Al alloy, but also increase its resistance to intergranular corrosion and exfoliation corrosion. The T73 and the HTPP aging treatments increase its corrosion resistance, but lower its strength.

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