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# Effect of pre-strain and two-step aging on microstructure and stress corrosion cracking of 7050 alloy

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#### ABSTRACT

A novel heat treatment procedure that combines the pre-strain with a two-step aging was proposed to improve both the strength and stress corrosion cracking (SCC) resistance of the high strength 7050 aluminum alloy. The heat treatment included a post-quenching pre-strain of 5%, a high-temperature aging at 200 °C for several minutes, and a subsequent low-temperature aging at 120 °C for 24 h. The yield strength of the samples aged at 200 °C for 0.5 and 1 min was higher than that of the T6 sample. The SCC resistance of these samples was improved compared to the T6 sample, due to the enlargement in the size and the inter-particle distance of the grain boundaries precipitates and the decreases in the number of the GP zone. Especially, the sample aged at 200 °C for 5 min exhibited a similar SCC resistance and much higher elongation compared the T76 sample.

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#### 1. Introduction

7000 series aluminum alloys are widely used in the aircraft structures due to the high strength and low density [1]. The high strength of the 7000 series alloys is due to the fine and uniformly distributed precipitates in the matrix which precipitate during the artificial aging. The usual precipitation sequence of the 7000 series Al alloys can be summarized as: solid solution–GP zone–metastable  $\eta'$ –stable  $\eta$  (MgZn<sub>2</sub>) [2]. For the peak aged (T6) 7000 series aluminum alloys, the  $\eta'$  phase is the main precipitate; however, for the over aged temper (T7×) the  $\eta$  phase is the main precipitate [3,4].

The stress corrosion cracking (SCC) resistance is of practical importance for the industrial applications of the 7000 series aluminum alloys. Many efforts have been made to correlate the heat treatment and the resultant microstructure with the SCC behavior of the alloys. The major microstructural features that affect the SCC resistance are the grain boundary precipitates (GBP). The SCC resistance could be improved by increasing both the size and the inter-particle distance of the GBP [5–7].

Heat treatments are widely used to improve the SCC susceptibility of aluminum alloys. In the industrial practice, over aging, namely the T7× temper, is a general method to apply. Although the overaged 7000 series alloys have excellent SCC resistance, their strength is reduced by 10–15% compared to that of the T6-tempered alloys [8,9].

Therefore, extensive research efforts have been made to enhance both the strength and the SCC resistance of the 7000 series alloys. Cina [10] firstly reported the retrogression and re-aging (RRA) treatment of 7075 alloy in the 1970s. The RRA involved in annealing of T6 sample at 180–240 °C for 5–2400 s (known as retrogression), followed by cold water quenching and final T6-aging (known as reaging). After the RRA heat treatment, the precipitates on the grain boundaries of the alloys became coarse, whereas those within the grains were still fine. Thus, the SCC susceptibility of the alloys was reduced with the strength being kept to be similar to that obtained in conventional T6 sample.

Ou et al. [4,11] proposed a step-quench and aging (SQA) treatment for 7000 series alloys, in which the sample was quenched to a temperature of 200–220 °C from the solution treatment temperature and kept for 10–30 s, then quenched to room temperature, finally the quenched sample was aged to the T6 temper. This treatment improved the SCC resistance substantially due to the increased size and inter-particle distance of the GBP, while the strength of the sample was similar to that achieved in the T6 sample. However, this treatment was not suitable for the high quench sensitive 7000 series alloys.

A pre-strain is usually applied to the 7000 series alloys in the as-quenched state to relieve quench-induced internal stress [12]. It is widely recognized that the presence of the dislocations which are generated in the pre-strain process greatly influences the subsequent precipitation process, thereby affecting the mechanical

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Table 1			
Chemical compositions	of 7050 Al	allov (wt %	ሄ)

Zn	Mg	Cu	Zr	Si	Fe	Al	
6.18	2.20	2.21	0.13	0.11	0.10	Bal.	

properties of age-hardened aluminum alloys. For the 7000 series alloys, the pre-strain usually resulted in the generation of equilibrium phase  $\eta$  (MgZn<sub>2</sub>) on the dislocation network, therefore reduced the amount of the solutes for the formation of the main hardening phase  $\eta'$  in the T6 sample, which decreased the mechanical strength of the alloys [13,14]. However, it was also reported that the strain-hardening in the process of the pre-strain led to the increase in the strength of the 7000 series alloys [15].

In this paper, a novel heat treatment procedure that combines the pre-strain with a two-step aging treatment was proposed. The aims are (a) to examine the effect of the pre-strain on the strength of the high strength 7050 aluminum alloy and (b) to evaluate the microstructural evolution and SCC behavior of the samples treated with this novel treatment procedure.

#### 2. Experimental

7050 aluminum casting was used as the raw material and the chemical composition of the alloy is shown in Table 1. 20 mm thick plates were cut from the casting and were homogenized at 470 °C for 48 h, and then quenched to room temperature by using water. The homogenized plates were hot-rolled to 6 mm at 430 °C. The rolled samples were solution treated at 470 °C for 1 h in a salt bath furnace and then quenched into water at room temperature. Different treatment procedures as listed in Table 2 were used after quenching. In addition to the traditional T6, T651, and T76 treatments, a novel heat treatment procedure that combines the pre-strain and two-step aging was proposed. HTA0.5-30 samples referred to the samples aged at 200 °C for 0.5, 1, 5, 10 and 30 min, respectively.

Dog-bone shaped tensile specimens with a gauge length of 23 mm and a gauge width of 4 mm and a thickness of 2 mm were machined with their axes parallel to rolling direction. The tensile

#### Table 2

Heat treatment procedures used for 7050 Al alloy in this study

Temper	Heat treatment
T6 T651 T76	470 °C/1 h + water quench + 120 °C/24 h 470 °C/1 h + water quench + 5% pre-strain + 120 °C/24 h 470 °C/1 h + water quench + 120 °C/6 h + 165 °C/16 h
HTA	470 °C/1 h + water quench + 5% pre-strain + 200 °C/several min + 120 °C/24 h

HTA0.5-30 referred to the samples aged at 200  $^\circ C$  for 0.5, 1, 5, 10, and 30 min, respectively.

Table 3

Transverse mechanical properties of differently-treated 7050 samples at a strain rate of  $2\times 10^{-6}~\rm s^{-1}$ 

Temper	UTS (MPa)		Elongation (%)		
	In air	In 3.5% NaCl	In air	In 3.5% NaCl	r <sub>sol/air</sub> (%)
Г6	588	591	8.6	5.8	67.4
Г76	489	484	9.1	8.6	94.5
HTA0.5	589	590	8.6	5.9	68.6
HTA1	575	575	8.8	6.8	77.2
HTA5	509	497	9.6	9.1	94.8

 $r_{\text{sol/air}}$  = elongation tested in 3.5% NaCl/elongation tested in air.

properties were tested at a strain rate of  $1\times 10^{-3}\,s^{-1}$  by using an SHIMADZU AG-100KNG testing machine.

SCC resistance of the 7050 samples was evaluated using the slow strain rate test (SSRT) in the air and in a 3.5% NaCl aqueous solution with the gauge length of the specimens being completely immersed during the test. Dog-bone shaped specimens with a gauge length of 10 mm, a width of 3 mm and a thickness of 1 mm were machined from the heat-treated plates with the tensile axes perpendicular to the rolling direction. Tensile tests were conducted at room temperature with a strain rate of  $2 \times 10^{-6}$  s<sup>-1</sup> by using an Instron 5848 testing machine.

The fracture surfaces of the specimens were observed by the scanning electron microscopy (SEM). The precipitates in the samples were analyzed by transmission electron microscopy (TEM). Thin foils for TEM were prepared by twin jet-polishing in 30% HNO<sub>3</sub>, 70% ethanol solution cooled to -35 °C with liquid nitrogen at 19 V. Differential scanning calorimetry (DSC) experiments were performed on TAQ 1000. The heating rate was 10 K min<sup>-1</sup>.

#### 3. Results and discussion

Fig. 1 illustrates the tensile properties of the 7050 samples with various heat treatments. Both the yield strength (YS) and ultimate tensile strength (UTS) of the T6 sample were higher than those of the T76 one. Similar result has been reported before [8]. The T651 sample with a pre-strain of 5% prior to the aging exhibited higher YS and UTS compared to the T6 sample, and the YS increased by 9%, especially. The strength of the HTA samples decreased as the aging time increased. The YS of the HTA0.5 and HTA1 samples was slightly lower than that of the T651 sample, but still higher than that of the T6 and T76 samples. The strength of the HTA5 sample was equal to or greater than 10 min, the strength of the HTA samples was lower than that of the T76 one.

Table 3 shows the SSRT results of the differently treated samples in the air and 3.5% NaCl solution at a strain rate of  $2 \times 10^{-6}$  s<sup>-1</sup>. The UTS values of the samples tested in the air were slightly lower than



Fig. 1. Tensile properties of 7050 samples with various heat treatments.



Fig. 2. SSRT curves of differently-treated 7050 samples in 3.5% NaCl solution.

those shown in Fig. 1, since these samples were tested perpendicular to the rolling direction at a lower strain rate. The UTS values of the samples tested in the 3.5% NaCl solution were similar to those tested in the air. However, the elongations in the 3.5% NaCl solution were lower than those in the air. The T6 sample had the lowest elongation in the 3.5% NaCl solution, indicating that the T6 sample had the worst SCC resistance among these samples. Similar results were also reported in other 7000 aluminum alloys [16]. However, the elongation of all the HTA samples was larger than that of the T6 sample, indicating that the HTA procedure improved the SCC resistance of the 7050 samples.

Fig. 2 shows the SSRT curves of the various 7050 samples. In general, the longer displacement in the aqueous chloride solutions

indicates the better SCC resistance of the samples [4,16]. Similar to the results in Table 3, the T6 sample had the worst SCC resistance among these samples. Nevertheless, because of the improvement in the elongation of the HTA5 sample, the displacement of the HTA5 sample was larger than that of the T76 sample. For the HTA1 sample, the displacement was similar to that of the T76 sample, which was due to the higher YS of the HTA1 sample compared to the T76 sample. This indicates that both the displacement and the time to failure used previously to evaluate the SCC resistance [4,17] are not appropriate to the HTA samples.

In this study, we used an index  $r_{sol/air}$  to evaluate the SCCresistance, which is the ratio of the elongations of the samples tested in the 3.5% NaCl solution and in the air (shown in Table 3). The greater the  $r_{sol/air}$  value is, the better the SCC resistance is. The  $r_{sol/air}$  value of 1 implies that the material exhibits no SCC susceptibility. The lowest  $r_{sol/air}$  value observed in the T6 sample indicates that the T6 sample had the worst SCC resistance among these samples. The  $r_{sol/air}$  of the HTA samples was larger than that of the T6 sample, and increased as the aging time increased. The  $r_{sol/air}$  of the HTA5 sample was similar to that of the T76 sample, implying that they had a similar SCC resistance.

Fig. 3 presents the typical tensile fracture surfaces of the T6, T76 and HAT5 samples tested in the aqueous chloride solutions. The fracture surface of the T6 sample was dominated by the intergranular fracture with severe corrosion, showing the typical brittle fracture and SCC attack (Fig. 3(a)). In contrast, intergranular microvoid coalescences were found in the T76 sample (Fig. 3(b)). This is attributed to the preferential deformation of the precipitate free zone around the grain boundary area [18]. Furthermore, some large dimples and cleavage facets were also observed on



Fig. 3. SEM micrographs showing SSRT fracture surfaces of differently treated 7050 samples in 3.5% NaCl solution: (a) T6, (b) T76, and (c) HTA5.



Fig. 4. TEM micrographs showing precipitates within grains of 7050 samples: (a) T6, (b) T651.

the fracture surface of this sample. In the HTA5 sample, the dimples nearly disappeared and the fracture surface was characterized by the intergranular microvoid coalescences and cleavage facets (Fig. 3(c)). The predominance of the intergranular fracture suggested that the grain boundaries were the preferential paths for the SCC.

Fig. 4 shows the TEM images within the grains of the T6 and the T651 samples, respectively. For the peak aged (T6) 7050 alloy, the  $\eta'$  phase is the main precipitate and some GP zones remain in the matrix. In the present T6 sample, it was observed that the precipitates were homogeneously distributed (Fig. 4(a)). Similar result has been previously reported by Sha and Cerezo [2]. For the T651 sample with a pre-strained of 5% prior to the aging, many dislocations were generated in the pre-strain process and remained in the matrix after the aging (Fig. 4(b)). The precipitates far away from the dislocations were also homogeneously distributed. The dislocations existed in the matrix led to the increase in the strength of the sample. Therefore, the strength of the T651 sample was higher than that of the T6 sample.

Fig. 5 presents the TEM images within the grains of the HTA0.5 and HTA5 samples. It is well known that the  $\eta$  phase can be nucleated within the grains and the GP zone can be reversed at 200 °C. The reversion of the GP zone at 200 °C increased the difficulty of the nucleation of the  $\eta'$  phase that was prone to nucleate in the GP zone when the samples were aged at 120 °C, thereby producing some coarse  $\eta'$  phase particles in the matrix. For the HTA0.5 sample

(aged at 200 °C for 0.5 min), the coarse precipitates were few in the matrix (Fig. 5(a)). When the aging time was longer at 200 °C, more coarse precipitates appeared in the matrix. In the HTA5 sample the precipitates were coarser than those in the HTA0.5 and T6 samples (Fig. 5(b)). Therefore, the strength of the HTA samples decreased as the aging time increased at 200 °C. Furthermore, the dislocations would be annihilated when the samples were aged at 200 °C. Therefore, the dislocation was not found in the HTA5 sample, which might be another reason for the decrease in the strength of these samples.

The selected area electron diffraction (SAED) patterns of the T6 sample in the Al matrix (100), (112), and (111) projections are illustrated in Fig. 6. The main strong diffraction spots came from the Al matrix. The weak diffraction spots came from the precipitates [19-21]. According to the studies by Stiller et al. and Berg et al. [20,21], the diffraction spots in the  $\{1, (2n+1)/4, 0\}$  in the  $(100)_{Al}$  were from the GPI zone (Fig. 6(a)) and the diffraction spots near  $\{311\}/2$  associated with the GPII zone were observed in the  $(112)_{Al}$  projection. The diffraction features of the  $\eta'$  phase were also observed, such as diffraction spots in 1/3 and 2/3 of  $\{220\}$  in the  $(100)_{Al}$  projection and weak streaks along  $\{111\}$  direction in 1/3 and 2/3 of  $\{220\}$  in the  $\langle 112 \rangle_{Al}$  projection. This indicates that both the GP zone and the  $\eta'$  phase existed in the T6 sample. Some weak sharp diffraction spots in the three projection pattern, such as  $\{100\}, \{110\}$  in the  $\langle 100 \rangle_{Al}$  projection were from cubic structure Al<sub>3</sub>Zr.



Fig. 5. TEM micrographs showing precipitates within grains of 7050 samples: (a) HTA0.5, (b) HTA5.



Fig. 6. Selected area electron diffraction patterns of T6 sample: (a)  $\langle 100 \rangle$ , (b)  $\langle 112 \rangle$ , and (c)  $\langle 111 \rangle$  projection of Al matrix.

The SAED patterns of the HTA5 sample in the Al matrix  $\langle 1\,0\,0\rangle$ ,  $\langle 1\,1\,2\rangle$ , and  $\langle 1\,1\,1\rangle$  projections are shown in Fig. 7. The diffraction spots in 1/3 and 2/3 of  $\{2\,2\,0\}$  in the  $\langle 1\,0\,0\rangle_{Al}$  projection became stronger and the shape became complicated compared to the T6 sample. Some of the spots came from the  $\eta$  phase. The diffraction spots of the GPI zone were also observed in the  $\{1, (2n+1)/4, 0\}$  in the  $\langle 1\,0\,0\rangle_{Al}$  projection. However, the diffraction spots of the GPI zone almost disappeared in the HTA5 sample. The weak sharp diffraction spots of the Al<sub>3</sub>Zr dispersoids also existed in the three projection patterns.

Fig. 8 illustrated the DSC curves for the samples with a prestrain of 0% and 5% after quenching but without aging (hereafter referred to as 0% and 5% pre-strain sample). Recent studies [22,23] indicated that peak I in the two curves corresponded to the dissolution of the GP zone, peak II was caused by the formation of the  $\eta'$  phase, peak III was due to the transformation of the  $\eta'$  phase to the *n* phase, and peak IV was associated with the growth of the fine  $\eta$  phase. Although the 0% and 5% pre-strain samples showed the same peaks in the DSC curves, all the peaks of the 5% pre-strain samples moved to the lower temperatures, and peak I in the 5% pre-strain sample had higher intensity compared to that in the 0% pre-strain sample. It is known that the GPII zone, produced by the quenching from the high supersaturation solution above 450°C, had been assumed to be the vacancy rich clusters [21]. The quenching vacancies were easily annihilated in the dislocations produced in the pre-strain process, which accelerated the dissolution rate of the GP zone in the 5% pre-strain sample. Consequently, the intensity of peak I increased in this sample. The dislocations could also provide efficient sites for the heterogeneous nucleation of the precipitates and they were also fast diffusion paths for the precipitation elements in the aging process. In this case, the precipitates would precipitate at a lower temperature, leading to the movement of peaks II and III to lower temperatures. Therefore, for the T651 sample, the dislocations made more GP zone dissolve and the  $\eta'$  phase nucleate directly in the dislocations, which increased the number of the  $\eta'$  phase. This would be one of the reasons for the strength increment of the T651 sample.

In the T651 sample, the dislocations provided efficient sites for heterogeneous nucleation of the  $\eta'$  phase at 120 °C and accelerated the elements diffusion in the early aging process and made the  $\eta'$ phase coarsen in this process. The residual dislocations also made the strength increase compared to the T6 sample. However, for the HTA samples that were aged at 200 °C for several minutes after the pre-strain, the dislocations accelerated the heterogeneous nucleation of the  $\eta$  phase and the reversion of the GP zone, while the dislocations were also annihilated in this process. During the aging at 120 °C for 24 h, the GPI zone and the  $\eta'$  phase appeared within the grains, and the  $\eta$  phase nucleated at 200 °C grew in size. Furthermore, the GPII zone almost disappeared in the HTA5 sample since they were annihilated by the dislocations and reversed at 200 °C.

Fig. 9 shows the grain boundary precipitates in the 7050 samples with various heat treatments. The precipitates on the grain boundaries of the T6 sample were small and distributed continuously (Fig. 9(a)), but in the T76 sample the grain boundaries were decorated with the coarse and discontinuously distributed particles (Fig. 9(b)), which is similar to the previous reports [5,11]. The grain boundary precipitates in the HTA1 sample (Fig. 9(c)) were slightly larger than those in the T6 sample (Fig. 9(a)) and the interparticle distance was also larger than that in the T6 sample. With increasing the aging time at 200  $^{\circ}$ C, the size of the grain boundary precipitates and the precipitates and the largest grain boundary precipitates and the interparticle distance, but the



Fig. 7. Selected area electron diffraction patterns of HTA5 sample: (a) (100), (b) (112), and (c) (111) projection of Al matrix.



Fig. 8. DSC curves of 0% and 5% pre-strain samples prior to aging.

size was still smaller than that in the T76 sample. The small and discontinuously distributed grain boundary particles are believed to be beneficial to the toughness of the samples [24]. A slight increase in the elongation of the HTA5 sample compared to the T76 sample was observed (Table 3).

For the HTA samples aged at 200 °C, the  $\eta$  phase pre-nucleated on the grain boundaries. At the same time, some dislocations produced by the pre-strain treatment would be aggregated around the grain boundaries. Similar to the situation in the grains, the dislocations in the grain boundaries also provided heterogeneous nucleation sites for the  $\eta$  phase particles. Both the modes contributed to the nucleation of the  $\eta$  phase on the grain boundaries and the  $\eta$  phase grew up in the subsequent aging at 120 °C, which made the grain boundary precipitates enlarge and distribute discontinuously.

For the 7000 series aluminum alloys, the stress corrosion cracking resistance was related to the grain boundary precipitates. The mechanism of the SCC in the Al–Zn–Mg–Cu alloys was thought to be both anodic dissolution and hydrogen embrittlement [17]. The precipitates in the grain boundaries are Mg-rich phases in these alloys, which have the electrode potential different from the Al matrix. This would result in the anodic dissolution and form critical defects in the first stage of the SCC process in the aqueous chloride solutions. Furthermore, the hydrogen produced in the crack tip also leads to the hydrogen embrittlement in the grain boundaries.

However, in fact, the lattice diffusion coefficients of hydrogen in the FCC materials such as the aluminum alloys are very low, which means that there should be some more rapid transport modes for hydrogen. It is well known that the grain boundary is one of the rapid diffusion paths in a material which would help the hydrogen transport quickly. Moreover, Albrecht et al. [25] examined another possible mode and suggested that the mobile dislocations generated in the tip of the crack would improve the hydrogen diffusion in the matrix. Neguyen et al. [26] proposed that the increment of the precipitate size and the associated change from the GP zone to the semicoherent  $\eta'$  and incoherent  $\eta$  precipitates will result in a more homogeneous slip mode and the reduction of slip planarity, which could effectively decrease the mobile dislocation and further reduce hydrogen transported to the grain boundaries. In the



Fig. 9. TEM micrographs showing grain boundary precipitates of 7050 samples: (a) T6, (b) T76, (c) HTA1, (d) HTA5.

HTA samples, the number of the GP zone was reduced due to the reversion in the aging at  $200 \,^{\circ}$ C and the annihilation by the dislocations. Therefore, more homogeneous slip mode would appear in the SCC process in order to inhibit the hydrogen diffusion. This was a possible reason that the HTA samples had better SCC resistance compared to the T6 sample.

In addition, Song et al. [27,28] reported that the element Mg in the grain boundaries had a larger electronegativity difference between Mg and H atoms than that between Al and H atoms. Therefore, the element Mg in the grain boundaries could increase the amount of hydrogen absorbed and consequently accelerate its diffusion and enhance the solution degree of hydrogen in the grain boundaries. This resulted in the embrittlement of the grain boundaries and accelerated the growth of the stress corrosion cracks. The large size and inter-particle spacing of the grain boundary particles could decrease the anodic dissolution speed and act as the trapping sites for atomic hydrogen and create molecular hydrogen bubbles to reduce the concentration of the atomic hydrogen in the grain boundaries. Both the size and the inter-particle distance of the grain boundary precipitates in the HTA samples and the T76 sample were larger than that in the T6 sample, therefore, the SCC resistance of these samples were better than that of the T6 sample. For the HTA samples, as the aging time increased, the size and, especially, the inter-particle distance of the grain boundary precipitates enlarged, which improved the SCC resistance. The SCC resistance of the HTA5 sample was almost equal to that of the T76 sample.

#### 4. Conclusions

- 1. The strength of the T651 sample was enhanced compared to the T6 sample, since the pre-strain prior to the aging produced the dislocations in the sample and more  $\eta'$  precipitates precipitated within the grains after an aging at 120 °C for 24 h.
- 2. Although the strength of the HTA0.5 and HTA1 samples was slightly lower than that of the T651 sample, it was greatly higher than that of the T6 sample. Furthermore, the strength of the HTA5 sample was slightly higher than that of the T76 one and its elongation increased by 17%.
- 3. The size and the inter-particle distance of the grain boundary precipitates became large in the HTA samples due to the heterogeneous nucleation of the  $\eta$  phase on the grain boundaries, which was attributed to the high-temperature aging at 200 °C and the dislocation generated by the pre-strain.

4. The enlargement in the size and the inter-particle distance of the grain boundary precipitates and the decrease in the number of the GP zone all together improved the SCC resistance of the HTA samples, which had better SCC resistance than the T6 sample. Especially, the SCC resistance of the HTA5 sample was equivalent to that of the T76 sample.

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