Enhancing Stress Corrosion Cracking Resistance in Al-Zn-Mg-Cu Alloy through Inhibiting Recrystallization

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Abstract. In this work, the effect of recrystallization inhibition in Al-Zn-Mg-Cu (7010) alloy towards the stress corrosion cracking (SCC) behavior was studied. For this purpose, Sc addition (0.25 wt. %) was made to Al-Zn-Mg-Cu alloy and the SCC behavior of the base and Sc-bearing alloys in peak-aged condition was examined using slow strain rate testing (SSRT). The base 7010 Al alloy showed 10 % elongation, 9.9 % reduction in area and 561 MPa ultimate tensile strength (UTS), when tested in air. The ductility of the base alloy dropped to 3 % and 3.3 % in terms of elongation and reduction in area, respectively, when tested in 3.5 % NaCl solution, showing its high susceptibility to SCC. On the other hand, the 0.25 wt. % Sc containing alloy showed a significant improvement in ductility not only in air but also in 3.5 % NaCl solution, without any loss in the UTS. Thus, the 0.25 wt. % Sc containing alloy exhibited 13.4 % elongation, 15.8 % reduction in area and 560 MPa UTS in air and 12.5 % elongation, 16.4 % reduction in area and 560 MPa UTS in 3.5 % NaCl solution. The study shows that inhibiting recrystallization in 7010 alloy through Sc addition, improves the SCC resistance substantially even in the peak aged condition.

Introduction

There is a high demand for Al alloys possessing high strength to weight ratio for applications in aircraft and aerospace industries, and hence research is constantly directed towards imparting higher strength to Al alloys. However, it is noted that high strength Al alloys are prone to stress corrosion cracking (SCC), particularly at near peak strength condition [1, 2]. Their resistance to SCC can be increased by over ageing, but with a loss of strength [3, 4]. Recently, we studied the SCC behavior of 7010 Al alloy subjected to multi-step aging i.e. two-step peak aged and three-step over aged [5]. The peak aged alloy showed high susceptibility to SCC, that is the reduction in area dropped from 9.9 % (in air) to 3.3 % (in 3.5 % NaCl) and elongation dropped from 10 % (in air) to 3 % (in 3.5 % NaCl). However, over aged alloy showed a significant improvement in the SCC resistance. For example, the reduction in area dropped from 28.1 % (in air) to just 24.4 % (in 3.5 % NaCl) and there was no drop in the elongation (~10 %) when tested at a strain rate of 10^{-6}/s. It should be noted that this reduction in SCC susceptibility was made possible at the expense of 10-13 % loss in UTS due to over ageing. Thus, it showed a UTS of 490 MPa in over aged condition when subjected to 10^{-6}/s strain rate in 3.5 % NaCl solution. The study showed the coarsening of the grain boundary precipitates (GBPs) in the over aged alloy as opposed to fine and continuous distribution of GBPs in the peak aged alloy has improved the SCC resistance in the former. Further, the study showed that recrystallized grains are more prone to SCC [6].

Literature shows that Sc additions improve the strength of various Al alloys. In 1971, Willey [6] patented that small addition of Sc results in enhancing specific strengthening of Al alloys. It was later found that Sc is beneficial even in improving the strength of high strength Al alloys such as Al-Zn-Mg-Cu-Zr alloy [8]. Combined additions of Sc and Zr were found to be even more effective in Al-Zn-Mg alloys [9]. The strengthening effect is due to a combination of (a) the presence of fine $Al_3Sc_xZ_{1-x}$ precipitates that formed during homogenization and (b) the substantial grain refinement in the as-cast structure [9, 10]. More importantly, $Al_3Sc_xZ_{1-x}$ precipitates are reported to be effective in inhibiting recrystallization [8, 10]. Recent studies have shown that 0.25 wt. % Sc addition to an Al-Zn-Mg-Cu-Zr base 7010 alloy improves the peak aged strength properties without any compromise in the ductility of the alloy [11]. Thus, the tensile properties of the base alloy in the peak aged condition are: 0.2 % proof stress = 528 MPa, UTS = 624 MPa, and elongation = 12.5 % (for 50mm gauge length) and that of 0.25 wt. % Sc containing alloy in peak aged condition : 0.2 % proof stress = 560MPa, UTS = 645 MPa, and elongation = 12.5% (for 50mm gauge length).

Only a limited study has been made on the effect of Sc on the SCC behavior of high strength Al alloys. Wang et al. [11] reported that Sc additions have no measurable effect on the SCC susceptibility of Al-5Mg alloys. Wu et al. [8] stated that Sc improves the SCC resistance of an Al-Zn-Mg-Cu-Zr alloy having composition similar to that of 7055 Al alloy. They suggested that the high SCC resistance is due to the finer grain size and homogeneous distribution of $Al_3Sc_xZr_{1-x}$ precipitates. Elagin et al. [13] have also reported an increase in SCC resistance of Al-Zn-Mg alloy due to Sc addition. However, no work has been reported on the SCC behavior of Sc containing Al-Zn-Mg-Cu-Zr (7010) system, where, Zr and Sc could simultaneously influence their property. Hence, in this work the SCC behavior of 0.25 wt. % Sc containing 7010 alloy at peak aged condition was studied and is compared with the base alloy in the similarly heat treated condition.

Experimental Procedure

The weight percent composition of the base 7010 Al alloy examined in the present work is Zn(6.30)-Mg(2.30)-Cu(1.55)-Zr(0.14)-Fe(0.09)-Si(0.06)-Al(Bal). 5 mm thick sheet material was produced from ingots of 7010 alloy and then heat-treated to two-step peak ageing (solution treated at 465°C, followed by water quenching at room temperature and aged at 100°C/8 h and 120°C/8 h) condition. Further, 7010 was alloyed with 0.25 wt. % Sc and then peak ageing treatment was provided. For optical microscopy, samples were polished to 1000 grade SiC paper and final polishing was carried out with 1μm diamond paste. They were then cleaned with methanol, etched with Keller's reagent (5ml HNO_3, 3ml HCl, 2ml HF and 190ml distilled water) and examined through the optical microscope (Nikon FX-35A). Transmission electron microscope (TEM) Philips CM200 was used to study the microstructure of the alloy. Sample preparation was done by first slicing 5mm thick sheets to 0.2 mm foil, using an abrasive cutter and then punching the foil to obtain 3mm diameter discs. The discs were further thinned by a twin-jet electro-polishing unit. A solution of 30 % nitric acid and 70 % methanol, maintained at - 18 °C, was used for electro polishing at 12 V. Slow strain rate testing (SSRT) as per ASTM G129-00 standard was carried out using a tensile testing machine. This method involves testing specimens in air and 3.5 % NaCl solution at 10^{-6}/s strain rate. Flat specimens were prepared from sheet materials (5mm thick) as per ASTM 557M-94 standard (50mm gauge length, 12.5mm gauge width and 5mm thick). Tensile properties were evaluated in the long transverse direction. U-bend testing was carried out as per ASTM G30-94. Specimens were polished to 1000 grade SiC paper and degreased by acetone before testing. Representative fracture surfaces from SSRT and U-bend specimens were examined using scanning electron microscope (SEM) to identify the mode of fracture.

Results and Discussion

Figs. 1a and b represent optical micrographs showing grain structure developed in the base alloy and the Sc containing alloy, respectively. In both the cases, the original grains are elongated along the rolling direction. Further, small recrystallized grains could be observed in the base alloy. On the other hand, the grain structure of the Sc containing alloy is without any recrystallization. This is consistent with the previously reported observations that the movement of the grain boundary required for recrystallization is difficult in Al-Zn-Mg-Cu alloys that contain both Zr and Sc [11]. Figs. 2(a) and (b) represent transmission electron micrographs showing development of microstructure in the peak aged condition in the base alloy and the Sc-containing alloy, respectively. The TEM bright field images shown in Figs. 2a and b were taken near $<110>_{Al}$ zone. Our TEM studies, in agreement with those reported recently on the similarly treated base alloy [11], showed that the intragranular precipitates are based on the strengthening η' phase, and that the coarse GBPs are based on the equilibrium η ($MgZn_2$) phase. Similar precipitates are also present at similar locations in the peak aged alloy containing Sc. However, one major difference being the observation that the grain boundary particles in the Sc-bearing alloy are not interconnected as compared to the base alloy.

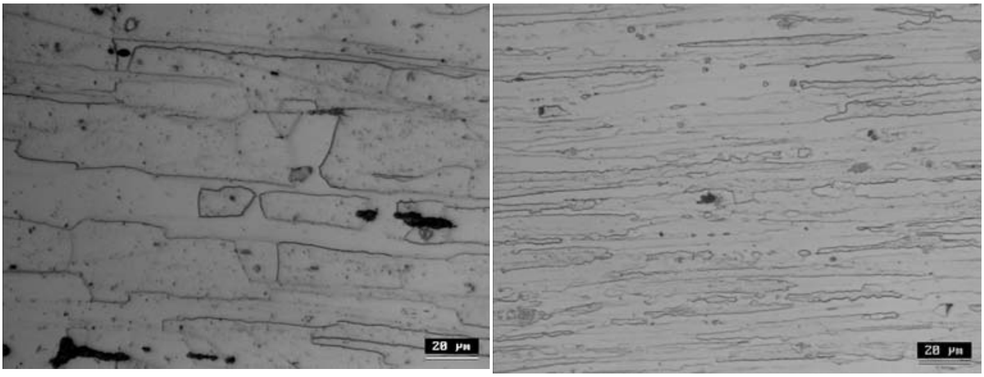


Fig. 1 Optical micrographs of (a) base 7010 alloy and; (b) 0.25 wt % Sc containing 7010 alloy (long transverse direction, etchant – Keller's reagent)

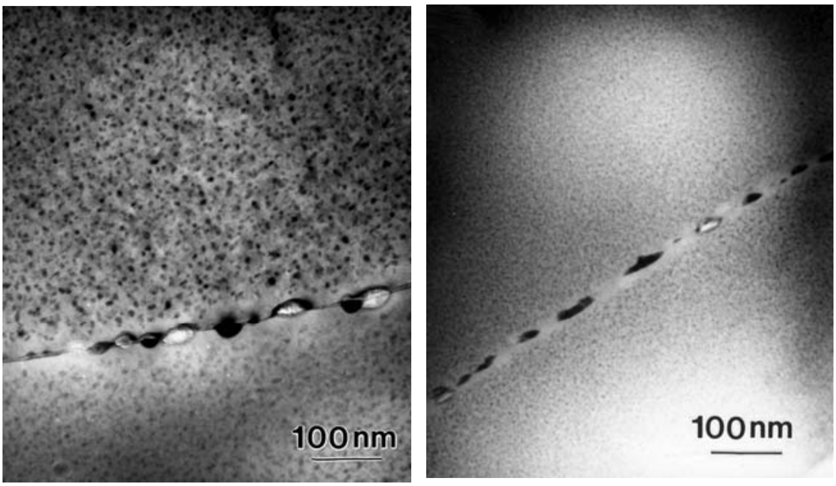


Fig. 2 TEM micrographs of (a) base 7010 alloy and (b) 0.25 wt % Sc containing alloy.

In order to obtain an idea about changes, if any, in the chemistry of the GBPs due to the presence of Sc in the alloy, the GBPs were analyzed using Energy Dispersive X-ray analysis (EDX). In Table 1, the EDX data showing the concentrations of only the major alloying elements i.e. Zn, Mg and Cu are presented. No significant difference in the chemistry between the GBPs of the base alloy and those of the alloy containing Sc could be observed.

Table 1 EDX data of grain boundary precipitates of base and 0.25 wt % Sc containing 7010 alloy.

Element (in wt.%)	Base alloy	0.25 wt.% Sc containing alloy
Zinc	10.4	8.6
Magnesium	6.3	5.2
Copper	3.9	2.7

Table 2 shows the SSRT results of base and 0.25 wt % Sc containing alloys in the peak aged condition, tested in air and 3.5 % NaCl solution at 10^{-6}/s strain rate. The base alloy shows good ductility in air i.e. 10 % elongation and 9.9 % reduction in area, and a UTS of 561 MPa. However, in 3.5 % NaCl solution, the alloy shows a reduction in its mechanical properties. For example, the elongation, reduction in area and UTS decreased to 3 %, 3.3 % and 515 MPa respectively. This shows that the peak aged alloy is highly susceptible to SCC. SCC susceptibility indices calculated by taking the ratio of a particular mechanical property, obtained from SSRT tests in environment to its corresponding value in air show very poor values. The SCC indices of both % elongation and reduction in area were found to be 0.3.

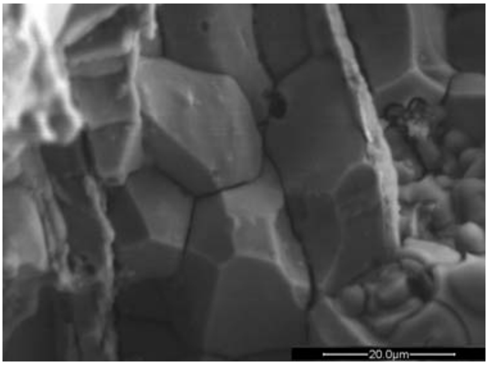
Table 2 SSRT results of base and 0.25 wt % Sc containing 7010 alloy in peak aged condition, tested in air and 3.5 % NaCl solution at 10^{-6}/s strain rate.

Mechanical Properties	Base alloy		0.25 wt.% Sc containing alloy	
	Air	3.5% NaCl	Air	3.5% NaCl
Elongation %	10.0	3.0	13.4	12.5
Reduction in area %	9.9	3.3	15.8	16.4
Ultimate tensile strength (MPa)	561	515	560	560

The alloy with 0.25 wt. % Sc addition exhibits an improvement in ductility, when tested in air that is the elongation and reduction in area have increased to 13.4% and 15.8% respectively. Interestingly, the alloy did not show any significant loss in ductility (12.5 % elongation and 16.4 % reduction in area) when tested in 3.5 % NaCl solution, indicating that it exhibits very high resistance to SCC. It should be noticed that there was no loss in the UTS value when compared to the test in air (560 MPa). Comparing the SSRT results of base alloy to that of 0.25 wt. % Sc containing alloy both obtained in 3.5 % NaCl solution, it becomes clear that the latter exhibits about 4 fold increase in elongation and 5 fold increase in reduction in area in comparison with the former, in spite of the latter exhibiting a 10 % higher UTS than the former. The improvement in the ductility clearly shows the high EAC resistance of the Sc containing alloy even with its high UTS values.

Wu et al. [8] report that Sc addition improved the SCC resistance of an Al-Zn-Mg-Cu-Zr alloy (the composition being similar to that of alloy 7055) subjected to retrogression and reaging treatment (RRA) and to a little extent in the peak aged condition of the alloy. However, in our study, high SCC resistance and high strength were achieved in the peak aged condition of 0.25 wt. % Sc containing 7010 alloy. It is worth while to compare the UTS of 0.25 wt. % Sc containing peak aged alloy in 3.5 % NaCl with that of base over aged alloy which exhibits high resistance to SCC, as reported earlier [5,6]. The former shows 14 % increase in the UTS compared to the latter, which is to be noted.

The fracture surfaces of both the alloys tested in 3.5 % NaCl were observed under SEM to identify the mode of failure. The base 7010 alloy fractograph revealed that the grains were attacked along the grain boundaries (Fig. 3 a). Close examination of these fractographs revealed that many of these grains have sizes ranging from 5 to 25 μm. The implication is that these grains represent the recrystallized grains and not the primary pan-cake shaped grains, and therefore, it is these recrystallized grains that were preferentially attacked along the grain boundaries. In the case of Sc-bearing alloy, mixed mode failure (quasi-cleavage) of the fracture sample was seen (Fig. 3b). The grains were elongated and scattered dimples were observed indicating clear deformation of the grains.



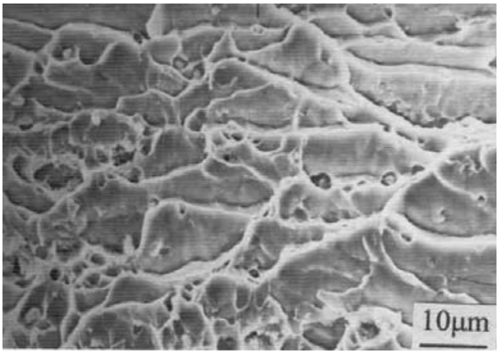


Fig. 3 SEM fractographs of : (a) base 7010 alloy reveals intergranular attack (b) 0.25 wt % Sc containing 7010 alloy shows mix mode of failure .

Wu et al. [8] suggested that the high SCC resistance observed in the Sc containing Al-Zn-Mg-Cu-Zr alloy (composition similar to 7055) could be due to refined and unrecrystallized grain structure. The present study confirmed such propositions showing that the recrystallized grains were indeed more prone to SCC exhibiting typical intergranular failure of these grains. Such an attack was, however, only prominent in the base alloy 7010 since they contained recrystallized grains. On the other hand, the Sc containing alloy did not show any such attack due to the fact that Sc addition inhibited the recrystallization. We further point out that yet another major reason for the superior SCC resistance of the Sc-bearing alloy is the widely spaced grain boundary particles as opposed to the interconnected nature of the grain boundary particles in the base alloy. The non-interconnected nature of the grain boundary particles in the Sc-bearing alloy will have delayed the SCC crack propagation.

Conclusion

The SSRT results and fractograph analysis suggest that addition of 0.25 wt. % Sc to Al-Zn-Mg-Cu alloy is beneficial against SCC even in the peak aged condition. Noticeably, no loss in the UTS was observed in the alloy due to the corrosive environment (3.5 % NaCl). The improvement in the SCC resistance of the Sc-bearing alloy is attributed to a combination of the unrecrystallized grain structure together with the non-interconnected nature of the grain boundary particles.

References

[1] M.O. Speidel: Metall. Trans. A Vol. 6A (1975), p.631.
[2] T.D. Burleigh: Corrosion, Vol. 47 (1991), p.89.
[3] I.J. Polmear: *Light Alloys*, Edward Arnold, Metallurgy and Material Science Series, London, U.K, (1989), p.89.
[4] J.E. Hatch: *Aluminium Properties and Physical Metallurgy,* Materials Park, OH, ASM International, (1984), p.270.
[5] M. Bobby Kannan, V.S. Raja, R. Raman, and A.K. Mukhopadhyay: Corrosion Vol. 59 (2003), p.881.
[6] M. Bobby Kannan, *Role of multistep aging and scandium addition on the environmentally assisted cracking and exfoliation behaviour of 7010 Al alloy*, Ph.D. Thesis, Indian Institute of Technology Bombay, India, (2005)
[7] L.A. Willey, US Patent 3619181, 1971.
[8] Yi-Lei Wu, F.H. (Sam) Froes, Chenggong Li, and Alex Alvarez: Metall. and Mater. Trans. A Vol. 30A (1999), p.1017.
[9] Yin Zhi-min, Yang Lei, Pang Qing-lin, and Jiang Feng: Trans. of the Nonferrous Metals Soc. of China Vol. 11 (2001), p.822.
[10] Y.W. Riddle, T.H. Sanders Jr.: Mater. Sci. Forum Vol.331-337 (2000), p.799.
[11] A.K. Mukhopadhyay, G.M. Reddy, K.S. Prasad, S.V. Kamat, A. Dutta, and C.Mondal : *J.T.Staley Honorary Symposium on Al Alloys,* (ed. M. Tiryakioglu) Advances in the Metallurgy of Al Alloys, ASM International, (2001), Indianapolis, USA, pp.63-71.
[12] W. P. Wang, K.S. Kumar, and C.L. Briant: Chemistry and Electrochemistry of Corrosion and Stress Corrosion Cracking: A Symposium Honoring the Contributions of R.W. Staehle, Proceedings of Symposium, New Orleans, USA, (2001), p.573.
[13] V.I. Elagin, V.V. Zakharov, A.A. Petrova, and E.V. Vyshegorodtseva: Russ. Metall Vol. 4 (1983), p.143.