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A COMPREHENSIVE STUDY ON THE EFFECT OF RETROGRESSION AND RE-AGING ON THE PROPERTIES OF ALUMINIUM ALLOY CONFORMING TO AA 7049 SPECIFICATION

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Abstract

The Al-Zn-Mg-Cu alloys have been widely used as aircraft structure materials because of their high strength to density ratio. These alloys in the T6 temper are known to be highly susceptible to stress corrosion cracking (SCC). In the present investigation, a comprehensive study on the effect of RRA treatment on the strength and corrosion properties of AA 7049 aluminium alloy has been made. The retrogression heat treatment is varied from 2 min to 60 min at temperatures between 180^{0}C and 240^{0}C; the re-aging treatment was done for 24 hours at 120^{0}C. Within the scope of this investigation, the results indicate that, the susceptibility for corrosion can be decreased by subjecting the alloy for RRA treatment. It is found that the grain boundary precipitate coarsening and enrichment of copper in the grain boundary precipitate are the factors responsible for improvement in reducing susceptibility to SCC.

1.0 Introduction

Amongst the aluminium alloys, high strength accomplishment has been promising with Al-Zn-Mg-Cu alloys. Over the years, age-hardneable aluminium alloys belonging to the Al-Zn-Mg-Cu family (7xxx series) have proved useful as structural materials primarily because of their unique combination of low density, high strength and good corrosion resistance. But 7xxx series of aluminium alloys in the T6 temper are known to be highly susceptible to Stress Corrosion Cracking (SCC). B.Cina [1] first discovered a heat treatment phenomenon known as Retrogression and Re-aging (RRA) where-in resistance to SCC is enhanced without losing yield or tensile strength. RRA process essentially consists of three steps: firstly, pre- age the material at low temperature (120^{o}C) after solution treatment; then, reheat or retrogress the alloy for a adequate period of time at high temperature (180^{o}C–240^{o}C); finally, re-age the alloy at a low temperature (120^{o}C). A high strength due to T6 and the good SCC resistance for T73 condition can be achieved simultaneously by the optimum RRA process. [2]

Various studies have been conducted to characterize the microstructural evolution during heat treatment conditions, in order to gain insight into which microstructural parameters control the strength & stress corrosion cracking resistance of high strength aluminium alloys of 7xxx series. It is observed that the microstructure of AA7075-T73 shows grain boundary precipitates that are much coarser than those in the T6 temper. Furthermore, it has been shown that longer retrogression time during the RRA process corresponds to grain boundary precipitate coarsening, which resemble the T73 temper and matrix precipitate resemble the T6 temper. This combination results in good performance with respect to both resistance to stress corrosion cracking and mechanical strength. [3-6]

The paper presents the findings on the effect of heat treatment on the properties and corrosion behaviour of high strength aluminium alloy conforming to AA 7049 specification. The published information on the effect of various temper conditions such as T6, T73 and RRA for this alloy is very limited. In the present work, an attempt has been made to examine the properties and correlate the phase transformations that occur during RRA using Transmission Electron Microscopy (TEM) with electrical conductivity measurements.

2.0 Experimental

The raw material of AA7049 alloy required for this study was in the extruded form of 60 mm diameter rods. The chemical composition of the alloy is given in Table 1. The extrusions were checked for soundness using ultrasonic flaw detector (Kraut Kramer make) with 2mm flat bottom hole (FBH) standard reference block. The acceptance standard used was Class A as per AMS 2630B. The extruded rods were ascertained for absence of defects like cracks, porosity or cavity. The mechanical properties of the extruded raw materials in short transverse (S.T) were evaluated on six nos. of samples and the results are given in Table 2.

Table.1. Chemical Composition of extruded aluminium alloy conforming toAA7049.

Element	Specification, wt %	Actual, wt %
Zn	7.2 – 8.4	7.82
Mg	2.1 - 3.1	2.23
Cu	1.2 -1.9	1.44
Fe	0.5 max	0.22
Si	0.4 max	0.25
Mn	0.5 max	0.29
Ti &Zr	0.5 max	0.16
Al	Rem.	Rem.

Table 2: Properties (in short transverse direction) of AA7049 extruded extrusions before heat treatment

Hardness BHN	Conductivity % IACS	0.2 % P.S MPa	UTS MPa	% El(4d)
83-87	37.8-38.4	179-182	272-287	7.42-7.82

In the present work, heat treatment was carried out to three different tempers viz., T6, T73 and T77 (RRA). All the samples were solution treated at 470^{0}C with a soaking duration of 120 minutes and then subsequently water quenched. These samples were aged to different tempers. The maximum time delay between quenching and aging was 15 minutes. For attaining T6 temper, the solution treated samples were aged at 120^{0}C for 24 hours. Similarly for attaining T73 temper the solution treated samples were left for natural ageing for a period of 48 hours. These samples were then subjected to two steps of artificial ageing of 120 ^{0}C for 24 hours, air cooled to room temperature followed by aging at 160 ^{0}C for 14 hours and air cooling to room temperature. For RRA treatment all specimens were initially heat treated to T6 temper and then subjected to RRA heat treatment which consisted of retrogression treatment at 180^{0}C to 240^{0}C, for a time duration varying from 2 minutes to 60 minutes. All the specimens were water quenched after retrogression and subsequently re-aged at 120^{0}C for 24 hours. After aging treatment the samples were air-cooled to room temperature.

All the heat treated samples were evaluated for mechanical properties using TIRA-make Universal Testing machine at room temperature in accordance with ASTM E8 standard. Max electronics-make Conductivity meter, having range 10% to 120% IACS (International Annealed Copper Standard) where 1%IACS = 0.58MSm−1, was used to perform electrical conductivity measurements of the heat treated samples. Exfoliation corrosion tests were performed in accordance with ASTM G34 standard and the intergranular tests were done as per the standard ASTM-G-110-92. The microstructure was characterized using a Philips-make CM20 TEM operating at 200kV.

3.0 Results and discussion

3.1. Role of RRA on tensile properties and conductivity

The variations in Ultimate Tensile Strength (UTS), 0.2%Proof Stress (PS) & Ductility (%El) with different heat treatment tempers i.e T6, T73 & RRA in transverse direction are plotted in Figs. 1- 3 respectively. The UTS for T6 temper was 583MPa and for T73 temper was 497MPa. Similarly conductivity for T6 temper was 29.4 %IACS and for T73 temper 35 %IACS as shown in Fig 4.

From Fig. 1 & 2, it is evident that with lower retrogression temperature of 180^{0}C, the UTS value achieved is marginally higher than or in line with T6 temper for the retrogression time durations beyond 10 min. However, 0.2%PS is higher when compared to the strength at T6 temper for all the retrogression time employed in this investigation. At higher temperature range of 200^{0}C to 240^{0}C, it seen that UTS is maintained at T6 temper level up to 05-10 minutes of retrogression time and there is appreciable drop in UTS at higher retrogression time durations thereafter.

The 0.2%PS is superior to T6 temper for all retrogression treatments at temperature of 180^{0}C and time durations up to 20 minutes in the remaining temperatures range of 200^{0}C to 240^{0}C. This is further corroborated by the fact that at lower retrogression temperature of 180^{0}C – 200^{0}C there is appreciable change in conductivity when compared with T6 temper and is in the range of 30-32%. Whereas at higher retrogression temperature of 200^{0}C and time duration beyond 30 minutes; retrogression temperature of 220^{0}C -240^{0}C and time duration beyond 20 min, there is steep increase in electrical conductivity & a corresponding drop in strength. From fig 1-2 & 4, it is clear that the electrical conductivity has an exactly reverse trend as that of strength.. The ductility in transverse direction is low and is due to the inherently directional nature of working of the product. The ductility is about ~1% along the short transverse direction for T6 temper. It is about ~5% at T73 temper. The ductility remains unaffected up to 30 min at RRA 180^{0}C and upto 5 min -10 min at RRA 200^{0}C -RRA 240 ^{0}C condition. Beyond these retrogression time durations there is an improvement in ductility in short transverse direction and again drops after longer retrogression times. This confirms the fact that there exists an optimum RRA regime for AA 7049 aluminium alloy extrusions.

TEM micrographs of the alloy in peak aged T6 temper condition revealed the presence of fine precipitates structure of GP zones and metastable phase η' which are attributable to the strengthening of alloy, as can be seen in fig. 5a. The Selected area diffraction (SAD) analysis of this study indicated that majority of the precipitates are of η' type and more stable GP-II zones are present in smaller amount. These findings are reported by the authors of this paper elsewhere[9]. TEM micrograph of the alloy in T73 temper condition as shown in fig. 5b reveals coarser precipitates with uniform distribution inside the grains. The SAD patterns from the T73 temper reveal several spots from the equilibrium precipitate η and less from η'. These precipitates are essentially η with η' precipitates being present in a smaller extent. The microstructures resulting from RRA temper(fig.5c & fig.5d.) are similar to that of T6 temper in the sense that there is a fine distribution of η' precipitates in the aluminum matrix grains and the coarser η precipitates which are distributed in the matrix are similar but bigger in size compared to that in T73 temper(Fig.5c.) The observations are in close agreement with those in the literature [5-8].

The higher strength achieved at RRA temper as compared to that of T6 temper suggests that, at T6 temper the material may be in the under-aged condition. Similar results are reported for the alloy AA 7010 [8] which suggest that the dissolution of unstable phases during retrogression and subsequent precipitation occurred after RRA condition might have increased the overall concentration of η' particles than that of T6 condition. In the present study it was observed that there is an increase in the presence of concentration of η' particles which is a strengthening phase, in RRA condition. The volume fraction of η' particles at T6 condition are less when compared to RRA condition. This reason is attributed for the increase of strength due to RRA treatment. At higher retrogression temperature and time durations there will be coarsening of strengthening precipitates. In the study on AA 7010 alloy [8] it is suggested that the strength would fall as coarsening of precipitates dominates. In the present study, the TEM micrographs at higher RRA temperature and time durations shows the coarsened precipitate morphology and corresponding low strength values. Therefore, higher retrogression temperature and longer retrogression duration results in reduction in strength while increase in ductility.

The TEM micrographs of AA 7049 aluminium alloy at T6 temper as shown in fig. 5a, reveals closely spaced precipitates along the grain boundaries. On the other hand, the TEM micrograph at RRA 200^{0}C for a duration of 45 minutes, as shown in fig 5c, reveals a coarser and widely spaced precipitates at the grain boundaries. Due to the presence of elongated grains in extrusions, crack propagation is much easier in the short transverse orientation, affecting in the process the tensile properties, particularly ductility. In addition to this, the formation precipitates as a continuous network along the grain boundary will adversely affect the ductility. This is the reason for obtaining lower ductility during initial retrogression cycle and also in T6 condition.[8]

It is observed that, the retrogression for longer duration followed by re-aging, breaks the continuous type precipitate network at the grain boundaries by coarsening of the precipitates. This reason is attributed for restoring of ductility due to RRA treatment. However, at higher retrogression temperature range of 220^0C–240^0C, for longer retrogression duration, the matrix precipitates also gets coarsened along with the grain boundary precipitates. Due to this a drop in ductility was observed at these conditions.

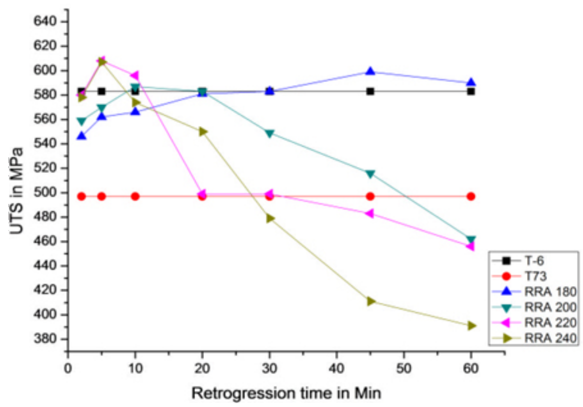


Fig. 1 Variation of Ultimate Tensile Strength with Retrogression Time as compared with T6 & T73 tempers given by straight lines parallel to time axis.

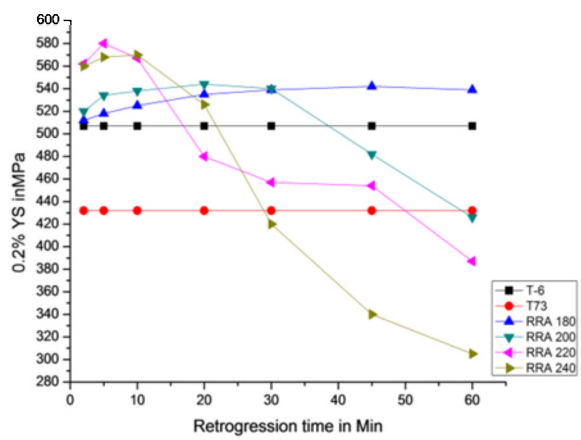


Fig. 2 Variation of 0.2%Proof Strength with Retrogression Time as compared with T6 & T73 tempers given by straight lines parallel to time axis.

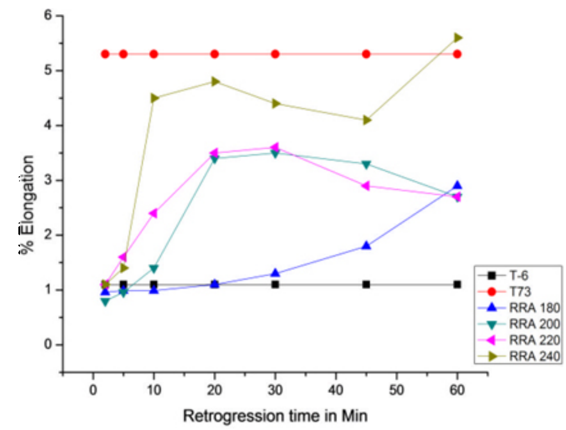


Fig. 3 Variation of Elongation % (in transverse direction) with Retrogression Time as compared with T6 & T73 tempers given by straight lines parallel to time axis.

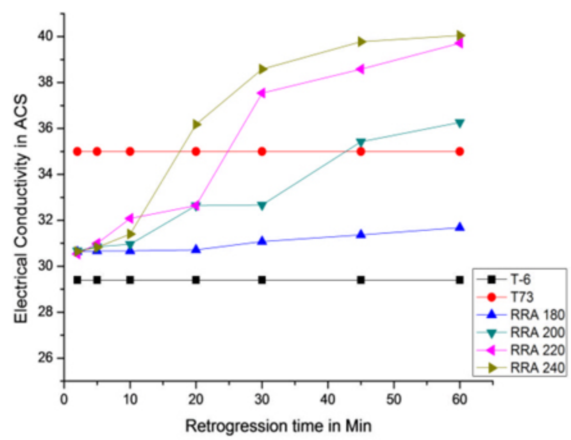
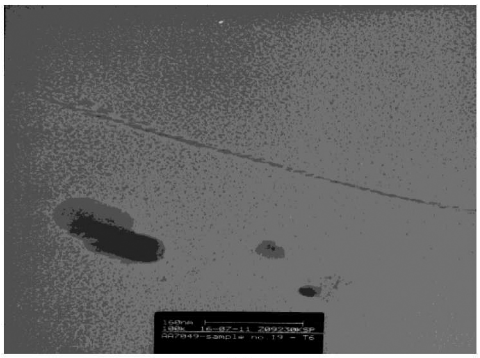


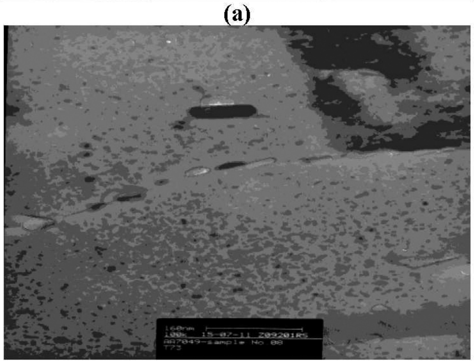
Fig. 4 Variation of Electrical Conductivity with Retrogression Time as compared with T6 & T73 tempers given by straight lines parallel to time axis.

3.1. Effect of RRA on corrosion resistance

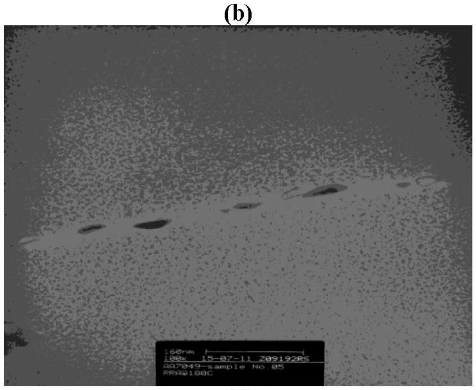
Inter-granular corrosion (IGC) is the selective attack of the grain boundary region with no appreciable attack within the grain. It has been found from the results of intergranular corrosion (IGC) studies that, the test specimens subjected to T73 temper heat treatment has better IGC resistance when compared to the IGC resistance of test specimens subjected to T6 temper heat treatment. Also, from the results of IGC test, it has been found that, the susceptibility of extruded AA 7049 aluminium alloy for intergranular corrosion can be decreased by subjecting the alloy for retrogression and re-aging treatment, particularly in the range of 200^0C to 240^0C.



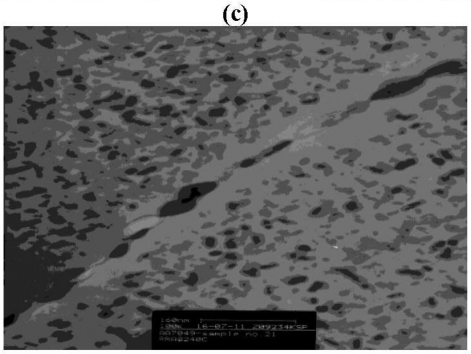
(a)



(b)

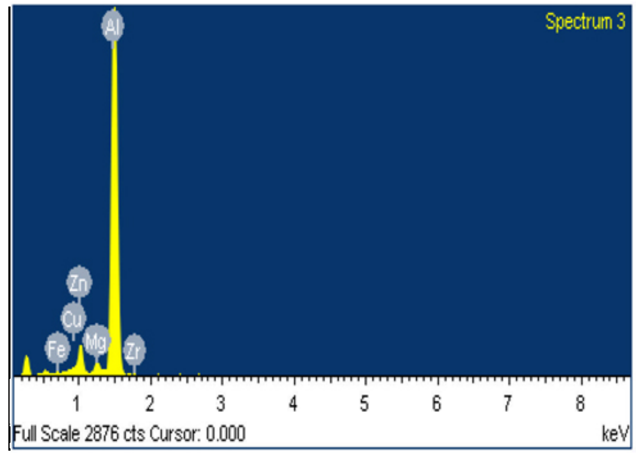


(c)



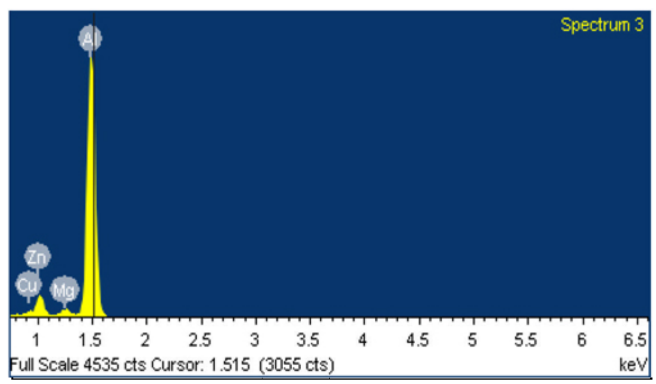
(d)

Fig. 5. (a). Representative bright field TEM micrographs pertaining to T6 temper ;
(b). Representative bright field TEM micrographs pertaining to over-aged T73 temper;
(c). Representative bright field TEM micrographs pertaining to RRA 200^{0}C-45min;
(d). Representative bright field TEM micrographs pertaining to 240^{0}C-60min



Element	Weight %	Atomic %
Mg K	1.77	2.11
Al K	86.61	92.73
Fe L	0.00	0.00
Cu L	1.98	0.90
Zn L	9.64	4.26
Zr L	0.00	0.00
Totals	100.00	100.00

Figure 6 (a): Chemical composition of the precipitate at the grain boundary of extruded AA 7049 aluminium alloy subjected to T6 temper heat treatment



Element	Weight %	Atomic %
Mg K	1.66	1.99
Al K	85.84	92.43
Fe L	0.00	0.00
Cu L	2.53	1.16
Zn L	9.97	4.43
Zr L	0.00	0.00
Totals	100.00	100.00

Figure 6 (b): Chemical composition of the precipitate at the grain boundary of extruded AA 7049 aluminium alloy subjected to retrogression treatment at 200^{0}C, for a time duration of 45min, followed by re-aging.

The Exfoliation corrosion test revealed that the RRA treatment has led to better resistance in corrosion when compared to T6 temper. In the retrogression temperature range of 200^0C to 240^0C, for time duration of 45 minutes, it has been observed that, the exfoliation corrosion resistance is comparable to the exfoliation corrosion resistance of test samples subjected to T73 temper heat treatment. Similar observations of improved resistance to exfoliation upon RRA are reported by Es-Said et al [13].

The literatures indicate that SCC performance is strongly influenced by alloy composition and type of aging temper [10-12]. The enhanced stress corrosion cracking resistance of 7075 aluminium alloy was attributed to the reduction of the Cu concentration within the PFZ and incorporating Cu into the grain boundary precipitates [9]. In the present work, EDX analysis has been carried out to evaluate copper concentration in the grain boundary precipitates in T6 temper and RRA condition. The average copper content (2.53%) present in the grain boundary precipitate at RRA 200-45 condition [Fig6(b)] has been higher when compared to the average copper content (1.98%)[Fig6(a)] present in the grain boundary precipitate at T6 temper condition and comparable to the average copper content (2.4%) in the grain boundary precipitate at T73 temper condition. The copper enrichment of the coarse grain boundary precipitates would play an vital role in improving SCC resistance; a similar behavior was also observed in RRA treated 7010 alloy [8]. Also, the discontinuous network of the grain boundary precipitates by coarsening during RRA as shown in Fig. 5 c would also reduce the continuous path available for stress corrosion crack propagation.

Conclusions

1. The RRA treatment has influence on electrical conductivity. The electrical conductivity increases with increase in the retrogression time duration.
2. The nature of precipitate present at the grain boundary affects the tensile ductility in short transverse direction. The ductility is restored at retrogression time duration of 20 minutes and above at the retrogression temperatures of 200^0C-240^0C.
3. The T6 temper microstructure reveals the presence of fine precipitates essentially of η'-type and small amounts of fine GP zones, distributed uniformly in the aluminium matrix and at the grain boundary the precipitates are slightly coarser and continuous in nature.
4. The T73 temper microstructure reveals the presence of coarse precipitates essentially of η-type distributed uniformly in the aluminium matrix and at the grain boundary the precipitates are being highly coarser and discontinuous in nature.
5. Retrogression process is responsible for the dissolution of the less stable precipitates (GP zones and the finer particles of η'-type precipitates) inside the grains; which is influenced by the retrogression temperature. Also the grain boundary precipitates grows and becomes discontinuously spaced.
6. Re-ageing process enables the formation of more stable η-type precipitates and its volume fraction rises during the RRA process. At the grain boundary the precipitates transforms and becomes similar to the T73 condition.
7. Therefore, due to RRA process, the η'-type and η-type precipitates are dispersed in the alloy matrix along with coarser and sparsely distributed η-type precipitates at the grain boundaries, together with precipitates free zones. This enables the AA 7049 aluminium alloy to retain strength of T6 temper while attain the thermodynamic stability of the T73 temper.
8. It is found that coarsening of grain boundary precipitates and its copper enrichment occurred during RRA, the factors responsible for improvement in corrosion resistance.

References:

1. CINA B.M, GAN. R., Reducing the susceptibility of Alloys particularly Aluminum Alloys to Stress Corrosion Cracking, Israel Aircraft Industries Ltd, U.S. Patent 3856584, 1974:12-24.
2. RAJAN K, WALLACE W, BEDDOES J., Microstructural study of the high strength stress corrosion resistant 7055 aluminium alloy, [J]. Mater. Sci.,17(1982): 2817-2824.
3. DANH H C, KRISHNA RAJAN, WALLACE W., A TEM study of microstructural changes during RRA in 7075 aluminium alloy, [J]. Metall. Trans., 14A (1983):1843-1850.
4. PARK J K, ARDELL A J., Effect of Retrogression and Re-aging treatments on the microstructure of Al-7075-T651, [J]. Metall. Trans., 15A (1984) :1531-1543.
5. VIANA F, PINTO A M P, SANTOS H M C, LOPES A B., Retrogression and re-ageing of 7075 aluminium alloy; microstructural characterization, [J]. Mater. Proc. Tech, 92-93 (1999): 54-59.
6. PARK J K, ARDELL A J., Correlation between microstructure and calorimetric behavior of aluminium alloy 7075 and Al-Zn-Mg alloys in various tempers, [J]. Material Science & Engineering A, 114 (1989):197-203.
7. GAZDA A, WARMUZEK M, WIERZCHOWSKI W., DTA investigation of the retrogression and re-ageing in some Al-Zn-Mg-Cu alloys, [J]. Thermo-chemical. Acta., 303 (1997): 197- 202.
8. ANGAPPAN M, SAMPATH V, ASHOK B, DEEPKUMAR V P., Retrogression and re-aging treatment on short transverse tensile properties of 7010 aluminium alloy extrusions, [J]. Materials and Design, 32 (2011): 4050–4053
9. RANGANATHA R, ANIL KUMAR V, VAISHAKI S NANDI, BHAT R R, MURALIDHARA B K., Multi-stage heat treatment of aluminium alloy AA 7049, Transactions of Nonferrous Metal Society of China, 23 (2013): 1570-1575
10. MUKHOPADHYAY AK, PRASAD KS, KUMAR V, REDDY GM, KAMAT SV, VARMA VK. Key microstructural features responsible for improved SCC resistance and weld-ability in 7xxx series Al alloys containing micro/trace alloying additions. Material Science Forum 2006; 519–521:315–20.
11. PUIGGALI M, ZIELINSKI A, OLIVE JM, RENAULD E, DESJARDINS D, CID M. Effect of microstructures on SCC of Al–Zn–Mg–Cu alloy. Corrosion Science. 1998; 40(4/5):805–19.
12. SARKAR B, MAREK M, STARKE JR EA. The effect of copper content and heat treatment on the stress corrosion characteristics of Al–6Zn–2Mg–X Cu alloys. Metallurgical Transactions A 1981; 12 A: 1929–43.
13. O.S. ES-SAID, W.E. FRAZIER AND E.W. LEE, The effect of retrogression and reaging on the properties of the 7249 aluminium alloy, Journal of Material Science, January 2003, P 45 – 48.