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Research Article

STUDY OF STRESS CORROSION CRACKING OF AA7075-T6 ALUMINUM ALLOY BY CHROMATE COATINGS IN AERIAL INDUSTRY

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ABSTRACT

Stress corrosion as one of the most important industrial injuries that always cause problems operation of industrial components. Stress corrosion of parts which are used in the aerospace industry could lead to irreparable disasters. Coating as a very effective way could be used to protect the aluminum against oxidation and corrosion as well as stress corrosion. Chromate conversion coating is one of these coatings that show a good corrosion and abrasion resistance at high and low temperatures. In this study, AA7075-T6 aluminum alloy was chromate conversion coated with an industrial solution (Alodine). Accordance with the G-38 and G-47 ASTM standards, stress corrosion resistance of the coated AA7075-T6 samples were investigated in NaCl 3.5% saline solution and the results were compared with the raw samples. SEM studies of surface of the immersed C-ring samples showed that macro cracks were initiated in the chromate conversion coated samples after about 1150 hrs. Finally, the mechanism of the stress corrosion of the coated samples was suggested using results of TEM, EDS, EDX and SEM characterization methods as well as findings of TOEFL corrosion test. The microscopic studies also revealed the destructive effects of Al2CuMg in the chromate conversion coating. The rate determining step of the corrosion reaction, seems to be breaking up the outer layer of the chromate coat and initiating of defects at the interface of the film - the metal that the latter one lead to dissolution of active base metal

Keywords: *Chromate Conversion Coating, C-ring Test, Stress Corrosion, Aluminum 7075-T6 Alloy*

INTRODUCTION

In recent years, high strength aluminum alloys (7XXX) due to ratio of strength to high weight and their resistance to uniform corrosion have been widely used in aerospace industry especially aircraft. Yet, presence by various alloying elements despite rise of sedimentation stability in aluminum has subjected aluminum alloys to local corrosions. This problem derives from the fact that a wide range of intermetallic compositions which develop during thermal operations in structure of these alloys are in a wide range in sake of electrochemical potential which result in formation of galvanic couples and ultimately formation of holes and cracks. Aluminium alloy 7075 together with elements zinc, magnesium, silicon, chromium and copper with high strength which have a wide use in various industries are subjected to local corrosions especially pitting and intergranular corrosion. These corrosions can be a starting point for various corrosions such as exfoliation, stress corrosion cracking (SCC) and fatigue cracks in aluminum alloys.T6 operations refer to dissolution heat treatment and artificial aging; this process will have various applications in products followed by cold-working and subsequent heat-treatment, where modifications are applied with precipitation hardening heat treatment so as to access mechanical features and dimensional stability of aluminum alloys. The reason for high strength of aluminum alloys lies on fine and uniform distribution of (T6) precipitates of secondary phases in the network which is obtained during aging operations. Unfortunately, this group acquired in maximum hardness loses their strength to stress corrosion cracking during T6 heat operations.

T6 aging operations on Aluminium alloy 7075 result in high strength of this alloy and fine and uniform distribution of (T6)precipitates of secondary phases in the network, for which the preparations stages are as follows (Meng, 2003):
470°C/3h ► water quenching ►120°C/24 h

Aluminum alloys 7075-T6 due to high strength and low density has a wide application in aerial industry. Aluminum alloy AA7075-T6 is used in the top surface of aircraft. Further, aluminum alloy AA7075-T6 is used in most of original frames and structures attached to the landing gear to the fuselage (Ramesh *et al.*, 2010). Chromate conversion coating is a type of conversion coating used to passivate aluminum, zinc, cadmium, copper, silver, magnesium, and tin alloys. Conversion coatings are coatings for metals where the part surface is converted into the coating with a chemical or electro-chemical process. Chromate is called to the chemical and electrochemical operations on metals and metal coatings of the solutions in which chromic acid, dichromate or chromate exist. The result from such operations can be protective coating processes including trivalent and hexavalent chromium compounds on the metal surface. Chromate coating can be chemically or electrochemically developed.
On Aluminium, reaction I can be as follow:
$2Al + Cr_2O_7^{-3} + 2H^{+} + 2H_2O \rightarrow Al_2O_3 + 2Cr(OH)_3$
Where the present study by appling T6 heat operations on Aluminium alloy 7075 and chromate conversion coating via submersion method was conducted, aiming at analyzing and reporting standard studies and experiments on aluminum alloy AA7075-T6. Examination on stress corrosion cracking of aluminum alloys at halliday environments if of importance. Halliday environments containing chloride or bromide ions can break top layer on aluminum surface, resulting in loss on aluminum surface. Stress corrosion cracking implies cracking due to combined effect of corrosion with stress. One of the most important forms of stress corrosion cracking can be stress corrosion cracking deriving from chloride, which it can be assumed as a type of intergranular corrosion (Meng, 2003).
Meng and Frankel in a study entitled "Effect of Copper Content on Chromate Conversion Coating Protection of 7xxx-T6 Aluminum Alloys" stated that Chromate conversion coating (CCC) protection of untreated and acid-pretreated 7xxx Al alloys with various Cu content in aerated chloride solution as well as the polarization behavior of as-polished alloys in deaerated chloride solution were investigated using polarization techniques in conjunction with metallography, scanning electron microscopy (SEM), and x-ray photoelectron spectroscopy (XPS). Only one breakdown potential was found in all alloys, and the value was slightly higher than the second breakdown potential for bare alloys. Breakdown of CCCs occurred on the matrix region. Electrochemical impedance spectroscopy (EIS) tests were used to show that alloyed Cu improved the CCC protection of polished AA7xxx-T6 samples, but Cu enrichment on the surface degraded the CCC protection of AA7xxx-T6 samples. The relevance of polarization and EIS results to salt spray testing is also addressed (Meng, 2003).
Ramesh *et al.,* (2010) conducted a study entitled Stress corrosion cracking of Al7075 alloy processed by equal channel angular pressing, in which the aim of the present work was to evaluate the stress corrosion cracking (SCC) behavior of the annealed Al-7075 alloy before and after Equal Channel Angular Pressing (ECAP). The SCC behavior of the Al-7075 alloy before and after ECAP was evaluated using slow strain rate testing (SSRT). Tensile and SCC behavior of the UNECAPed and ECAPed samples were compared. The elongation and ultimate tensile strength (UTS) was decreases 1.25, 1.1 times respectively after SCC in the annealed Al 7075.
After ECAP, about 1.6 times decreases in elongation and 1.09 times decrease in UTS is observed. The decrease in ductility is more as compared to UTS. The fracture surface analysis (from the SSRT tests in 3.5% NaCl solution) revealed predominant ductile failure in the before ECAP and mixed (quasi cleavage) mode of failure was observed after ECAP. Though the SCC resistance decreases due to ECAP, this appears a positive sign that the SCC may be improved by modifying process parameters and the condition of the sample (Berukshtis and Klark, 1996).

MATERIALS AND METHODS

The samples were defined and prepared for testing under standards ASTM G-38 and ASTM G-47. Standard ASTM G-38 expresses the method for preparing sample for testing, and standard ASTM G-47 considers sampling method, type of sample, preparation of sample, testing environment and how to be subjected to corrosion for determination of subjecting to stress corrosion cracking. Firstly the sample alloy 7075-T6 has been analyzed to reach to a chemical composition of used alloy by means of Quantometre device, where chemical composition has been represented in table 1.

Table 1: Chemical composition of aluminum alloy AA7075-T6

Weight percent	**%Al**	**%/Mn**	**%Cu**	**%Zn**	**%Si**	**%Mg**	**%Fe**	**%Ti**	**%Cr**
Amount	Residual	0.04	1.83	5.91	0.06	2.42	0.26	0.03	0.20

Ten aluminum alloys AA7075-T6 samples regarding standard ASTM G-38 perpendicular to the rolling direction of coated sheets by length of 60mm, width of 20mm and thickness of 2mm were cut and five samples were prepared for chromate coating operations. Temperature of operations has been in range of 45-50 ° C, ended in 20 minutes. Before chromate coating operations, grease trap was applied on piece and then it must be cleansed with water. Chromate percent in coating has been in range of 4% to 5%. After chromate coating operations, the pieces were washed with distilled water in temperature of 70° C. the samples were classified to two groups: 1- simple group without coating, 2-group coated with chromate. Thereafter, all the samples regarding standard ASTM G-47 and stages in figure 1 in form of C named C-Ring were prepared. Then, according to figure 1, to develop a fixed stress, two heads of sheets were closed to each other by means of a long bolt. Now, before entering the pieces into solution, the pieces must be colored at the area bolted for avoiding corrosion at these areas.

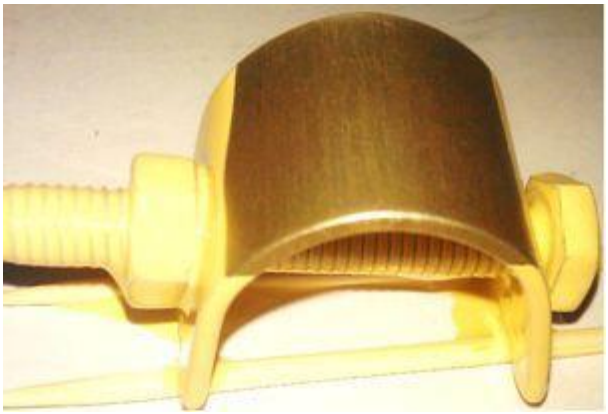

Figure 1: Structure of sample and coating the sample

To examine corrosion behavior of the samples with chromate coating operations in aqueous solution containing 3.5 wt% NaCl, Tafel extrapolation method and electrochemical impedance spectroscopy were used. Tri- polar electrode method was used for electrochemical tests. Simple and chromate aluminum alloys AA7075-T6 samples as working electrode, electrode Ag / AgCl containing 1 M KCl solution as the reference electrode, and platinum electrode as the auxiliary electrode, were used. The samples were classified into two groups: aluminum alloys 7075-T6 with coating chromate and simple aluminum alloys 7075-T6. The samples were submerged in considered electrolyte for 40 minutes, and immediately Tafel extrapolation method and electrochemical impedance spectroscopy were applied on them. Then, 3.5 wt.% NaCl solution was prepared for each piece in separate container. When the metal submerges into water or chlorides salts such as sodium chloride, the rate of corrosion due to reduction of diffusion of oxygen in the cathode region reduces. Chloride plays a major role in corrosion. Chloride causes destruction and perforation of the protective oxide layer. According to studies by Berukshtis and Klark, this ion in marine atmospheres causes the rate of corrosion in aluminum be for 22 times more than rate of corrosion in rural atmospheres (Yuhchae, 2004).

Examining 28 days from the starting day of corrosion due to penetration of chloride ions in samples of aluminum to oxide aluminum, macroscopic cracks were observed in simple and without coating samples, and after 48 days by observing macroscopic cracks in the samples coated with chromate, firstly light microscopy and scanning electron microscope (SEM) and EDX, as well as transmission electron microscopy (TEM) and EDS were used.

RESULTS AND DISCUSSION

Chemical Examination of Aluminum Alloys AA7075-T6

As seen in quantometer test, high presence of the elements such as Mg, Cu, Zn can be seen. Aging phenomenon is the most important phenomenon in alloys group 7000. According to aluminum alloys AA7075-T6 during aging, clusters Cu and Mg develop regular areas with similar interface. These areas enjoy bar-like morphology containing Mg and Cu. Rest of aging causes increasing similar strains by developing secondary phases. The secondary phase generally enjoys Al2CuMg and germinates on dislocations and dislocation loops so as to reduce germination energy (Dieter, 1986). Electrochemical deposition behavior relies on its primary phase. This deposit firstly is anodic, but over the time due to high speed at corrosion of Mg, the deposit will be empty from Mg, being cathode and causing corrosion around the deposit.

Examination of Electrochemical Tests

As the samples were classified into two groups: 1- simple group without coating (group 1 and 4), 2-group coated with chromate (group 2 and 3). Further, according to figure 2 and comparison of corrosion rate in samples 1 to 4 for NaCl 3.5% saline solution represented in tables 2 and 3, it can observe that Tafel extrapolation method confirms electrochemical impedance spectroscopy, where on samples 2 and 3 have the least rate of corrosion.

Table 2: Electrochemical impedance features and thickness of oxide layer

d_2(nm)	n_{C2}	C_2 (µF/cm^2)	R_2 (Ωcm^2)	Sample
13.4	0.89	0.66	$0.9*10^7$	1
1.53	0.93	0.28	$1.3*10^8$	2
1.43	0.91	0.31	$1.1*10^8$	3
13.8	0.91	0.64	$1*10^7$	4

Table 3: Results of Tafel extrapolation test for samples

The corrosion rate in year (M.m in year)	(i_{corr}) Corrosion current density (A/cm^2)	(E_{corr}) Corrosion voltage (V)	Sample
$2.67*10^{-3}$	$2.45*10^{-7}$	-0.51	1
$1.5*10^{-4}$	$1.38*10^{-8}$	-0.66	2
$2.46*10^{-4}$	$2.26*10^{-8}$	-0.52	3
$1.05*10^{-3}$	$9.65*10^{-8}$	-.54	4

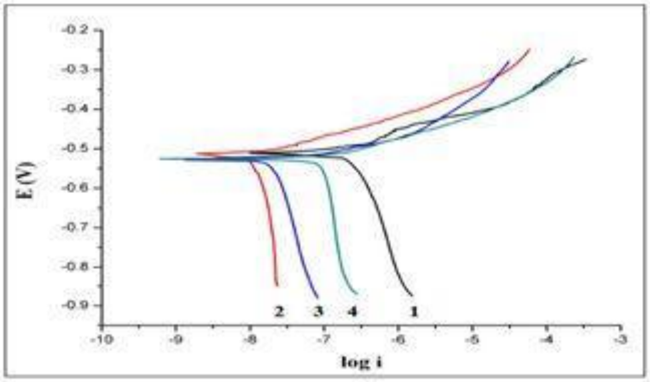

Figure 2: Diagram of Tafel extrapolation test for samples 1-4

Examination of light microscope on simple group without coating and group coated with chromate

Corrosion occurs faster and chloride penetrates easier in the simple and without coating samples regarding lack of coating, whereby Al2O3 passivation layer breaks and the preparations for crack are provided. According to light microscope examinations with magnification (50), it was observed that migration of chloride ions from the solution to the left side not only has been perpendicular to surface, but also it has been able to break grain boundaries and move in parallel to grain boundaries. Geometry of the crack tip can be observed in section b, figure 3. According to this figure, the starting area of corrosion and another area in which corrosion progresses are seen. According to section c, a part of crack in the chromate coating samples can be seen, in which changing the direction of crack in grain boundaries due to strength of coating can be observed.

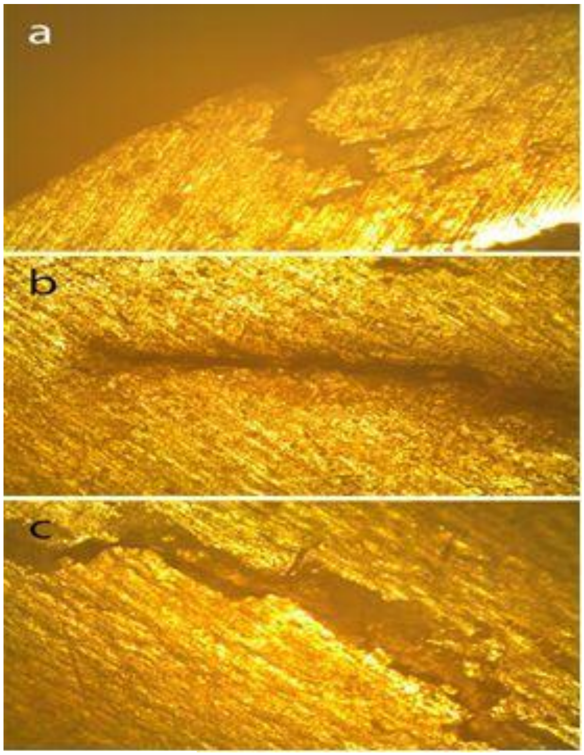

Figure 3: Light microscope images of samples with magnification (50)

a- Chloride penetration in both directions perpendicular and parallel to the surface of the alloy Al7075-T6
b- Crack in Al7075-T6
c- Crack in Al7075-T6 with chromate coating

Scanning electron microscope (SEM) and EDX on simple group without coating and group coated with chromate

To examine deposits and analyze the samples properly, scanning electron microscope (SEM) was used. Aluminum is of active-passive metals which represents less corrosion in range of PH (4.4-8.4). Extent of corrosion in aluminum practically relies on its protection layer, where oxide layer (hydrated aluminum oxide ($Al_2O_3 . X H_2O$)) has been developed in the simple samples regarding fixed and tensile stress in their surface. It should be noted that hydrated aluminum oxide ($Al_2O_3 . X H_2O$) develops easier regarding a broken and repaired cycle and penetration of chloride ions. SEM microscope images relating to simple group without coating and group coated with chromate in figure 4 as well as deposits of hydrated aluminum oxide ($Al_2O_3 . X H_2O$) and NaCl crystals can be seen. Further, the holes developed via chloride ions and water develops a poor hole for starting crack.

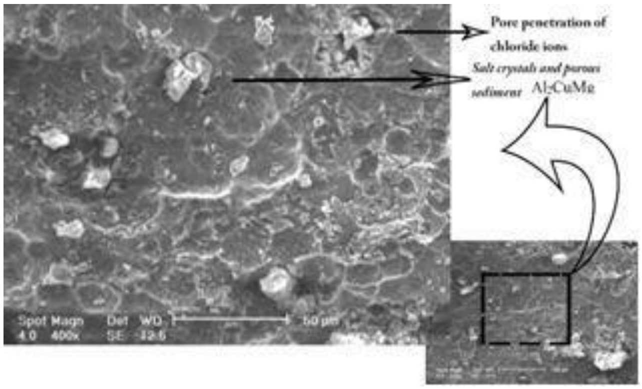

Figure 4: SEM image for simple group without coating and group coated with chromate with magnification (400)

According to image SEM for the crack arisen from stress corrosion in simple sample, it can observe breakage of oxide layer developed inside hole of crack; Refer to figure 5.

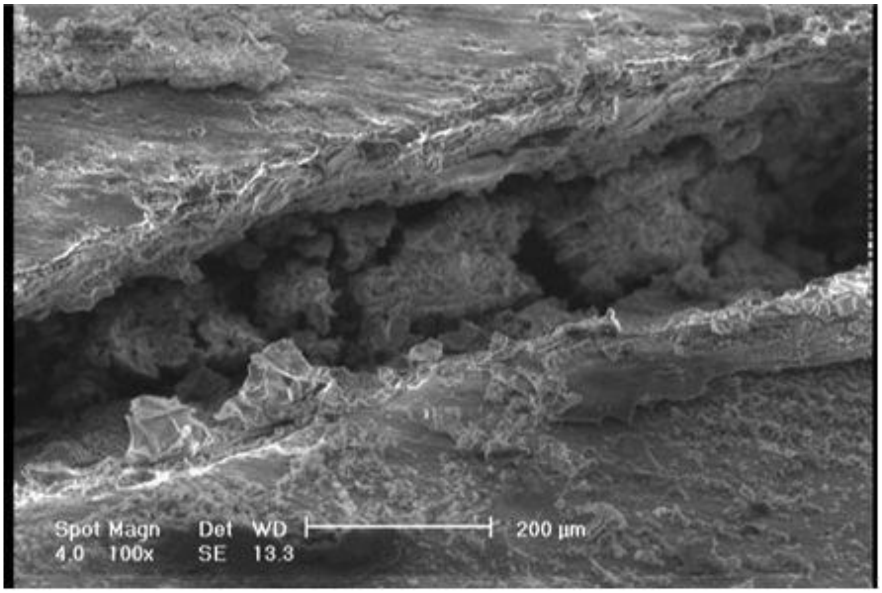

Figure 5: Image SEM for the crack arisen from stress corrosion in simple sample with magnification (100)

In the samples coated with chromate, there will be corrosion only while the stress develops microcracks in a long duration, and then microcracks pave the way for invasion of chlorine ions for corrosion and release of Al^{+3}. Yet, in these samples, it cannot neglect role of oxide Cr2o3, which helps for reducing speed of corrosion. But, with regard to fragility of oxide Cr2o3, oxide Cr2o3 during stress gradually breaks and remains as porous in opening of crack. Here, hydrolysis of metal ions affects speed at corrosion, resulting in activation of the surface at gap area in crack.

According to the reaction as follow:

$$Al^{+3} + H_2O \leftrightarrow Al(OH)^{+2} + H^{+}$$

It can know PH at crack area acidic, that hydrolysis of Al ions in form of this reaction causes rise of acidic solution in crack area.

According to examination of deposit and existing elements on surface of the sample, analysis of EDX on surface was fulfilled. This has been indicated in figure 6, the elements have been represented in table 4. High extent of Chlorine on surface is confirmed.

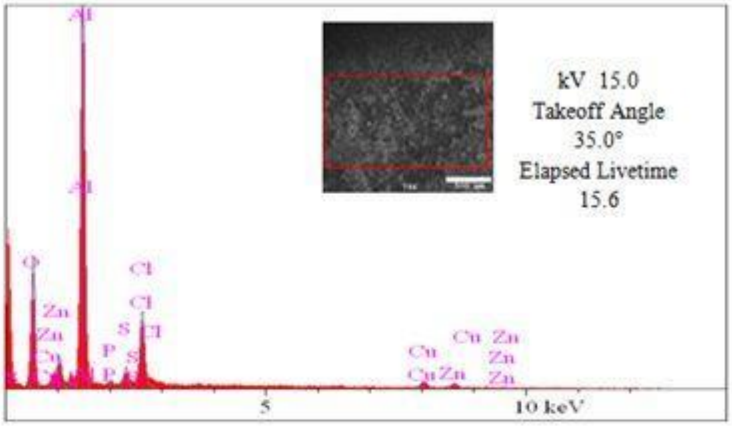

Figure 6: Analysis of EDX from surface of simple group without coating and group coated with chromate

Table 4: The results relating to analysis of EDX from surface of simple group without coating and group coated with chromate

Elt.	Line	Intensity (c/s)	Atomic %	Conc	Units	
O	Ka	395.91	41.78	26.21	wt.%	
Na	Ka	91.35	3.39	3.06	wt.%	
Mg	Ka	61.15	1.79	1.71	wt.%	
Al	Ka	1,477.97	36.44	38.54	wt.%	
P	Ka	37.23	0.95	1.15	wt.%	
S	Ka	84.29	1.92	2.41	wt.%	
Cl	Ka	313.28	6.83	9.50	wt.%	
Cu	Ka	47.93	3.41	8.48	wt.%	
Zn	Ka	36.78	3.49	8.94	wt.%	
			100.00	100.00	wt.%	Total

According to figure 7, the crack arisen from stress corrosion can be observed in the sample coated with chromate.

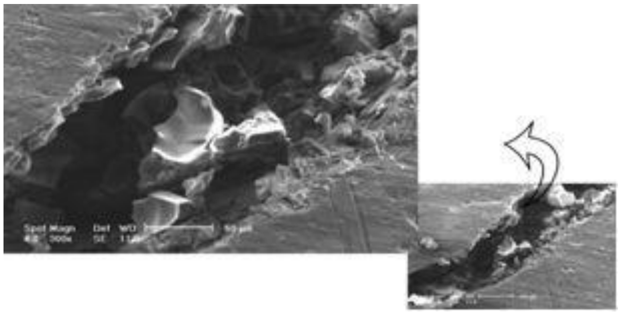

Figure 7: Image SEM for the crack arisen from stress corrosion in sample coated with chromate with magnification (300)

Deposits that can be of Oxidation reduction and binary compounds are clearly shown in Cr2O3/ CrO4 or Cr2O3/ Cr2O7. According to Chromate conversion coating, presence by fluoride ion as an activator is mainly considered for protection. Growth of film in CCCs without fluoride ion keeps going slowly. Dissolution of fluoride causes aluminum oxide (Al2O3) firstly stops on the surface, and then allows oxidation of chromium(Cr^{+3}/ Cr_6) for reaction, in order that sedimentary coating develops. The reactions are as follow (Vargel, 2004).

$$Al_2O_3 + 6HF \rightarrow 2AlF_3 \text{ (soluble)} + 3H_2O$$

$$AlOOH + 3HF \rightarrow AlF_3 \text{ (soluble)} + 2H_2O$$

$$CrOOH + 3HF \rightarrow CrF_3 \text{ (soluble)} + 2H_2O$$

According to examination on the deposit and existing elements in crack of sample, analysis of EDX was fulfilled on crack tip; this has been represented in figure 8, and the extent of elements has been represented in table 5. As said, chromium by forming Cr_2O_3 avoids from progression of chloride ions and reduces its amount rather than the state with simple sample. But, it must be noted that continuing fixed stress at this area, crack will break inside the gap and porosity will occur.

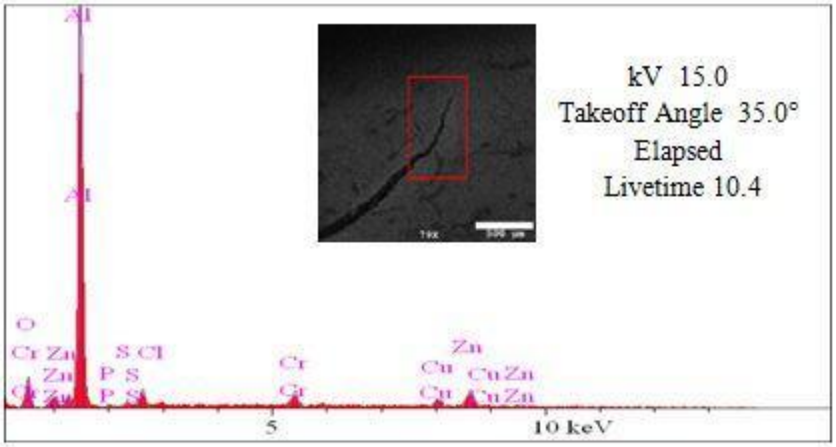

Figure 8: Analysis of EDX was on crack tip in the sample coated with chromate Al7075-T6

Table 5: Results relating to analysis of EDX from crack tip in the sample coated with chromate

Elt.	Line	Intensity (c/s)	Atomic %	Conc	Units	
O	Ka	64.84	11.88	5.56	wt.%	
Na	Ka	18.63	1.41	0.95	wt.%	
Mg	Ka	33.13	2.04	1.45	wt.%	
Al	Ka	1,150.18	57.82	45.61	wt.%	
P	Ka	10.85	0.54	0.49	wt.%	
S	Ka	15.82	0.68	0.64	wt.%	
Cl	Ka	59.40	2.38	2.46	wt.%	
Cr	Ka	67.58	3.27	4.96	wt.%	
Cu	Ka	47.42	5.77	10.71	wt.%	
Zn	Ka	86.99	14.21	27.17	wt.%	
			100.00	100.00	wt.%	Total

Examination of Transmission electron microscopy (TEM) in the sample coated with chromate

According to figure 9, as seen, image of transmission electron microscopy (TEM) in which thickness of coated layer is about ~150-300 nm sah been represented.

Figure 9 image of transmission electron microscopy (TEM) with magnification (60000) of Chromate conversion coating on aluminum alloys (7075-T6)

Mg is an active metal, whereby it has a high tendency for engaging in reactions and presence in intermetallic compositions. Further, it has solubility in Al. corrosion behavior of alloys (7000) has a direction relationship with corrosion behavior of Al-Cu-Mg and other Mg compositions. Fuzzy diagram of Al-Mg has been represented in figure 10.

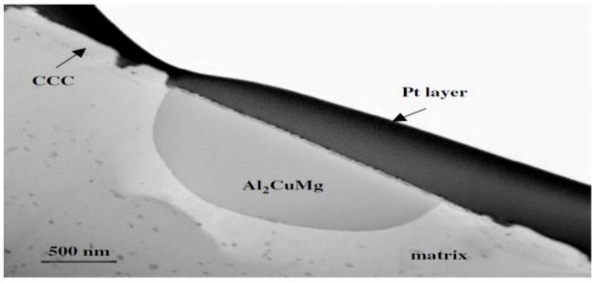

Figure 10: Fuzzy diagram of Al-Mg system (Dieter, 1986)

Amount of Mg in all these deposits indicate high importance of this metal in deposition process. Further, Mg alone can enter into the reaction with Al, and develops compositions such as Al_8Mg_5 and Al_3Mg_7, forming mainly in grain boundary. These compositions have been anodic rather than inside grain, and strengths tendency of alloy in corrosion of grain boundary. Then, it can say that holes can occur in grain boundaries such as Al_2CuMg, where on the cracks will be mainly of grain boundaries. Refer to figure 11.

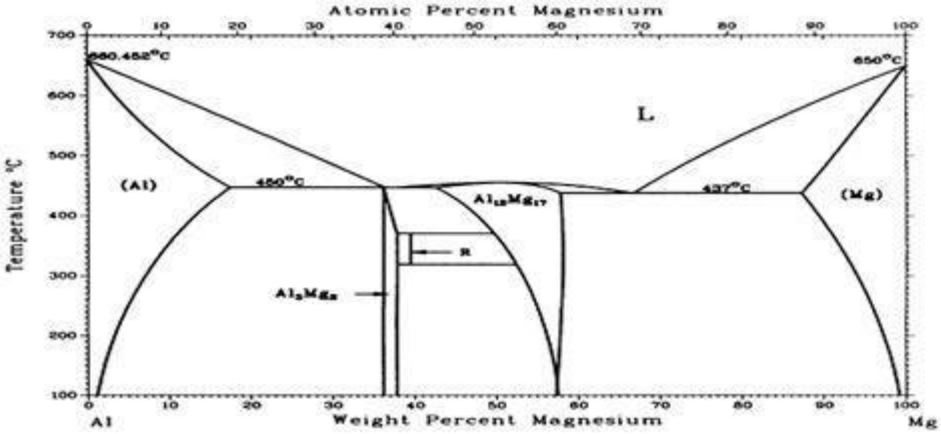

Figure 11: Al_2CuMg impurity and occurrence of the first crack (Vargel, 2004)

Stress corrosion cracking for AA7075-T6 aluminum alloy
Stress corrosion cracking for AA7075-T6 aluminum alloy (in macro perspective)
Stress corrosion cracking can be characterized as follows:
-firstly, anodic and cathodic reactions can occur inside crack as well as out of crack.
-finally, due to oxygen depletion at area of crack, only anodic reaction will occur at crack area, and cathodic reaction will no longer occur at crack area.
-to neutralize charge at gap area, immigration of chloride ions from the main solution to the gap area in which metal ions exist occur.
- Hydrolysis of metal chloride solution at crack area causes fall of PH at this area.
- Chloride ions and hydrogen ions at gap area help for anodic reaction, resulting in acceleration of stress corrosion regarding fixed stress.
The reactions for AA7075-T6 aluminum alloy can be characterized as follows:
anodic reaction(oxidation, loss of electron)
(dissolution of metal): Metal - e → Metal 3e + (1) Al → Al^{+3}
cathodic reaction(donation of electron)
donation of hydrogen and oxygen ions

(2) $O_2 + 2H_2O + 4e \rightarrow 4OH\ (pH\uparrow)$ (3) $2H^+ + 2e \rightarrow H_2\uparrow$

Mechanisms of Dissolution

-amplification of dissolution due to stress: the crack extends with local anodic dissolution. Amplification of dissolution is the major role of changing plastic form.

-formation of film at the wall of cracks comes to realize based on mechanisms of dislocation of the planes. The cracks start at the area where sliding stairs reach to surface. Growth of cracks as the result of metallic dissolution develops. By growing crack, the shell on the wall of crack develops and serves as cathode.

- Enrichment rather than noble element

-cracking as the result of stress corrosion progresses with tearing inactive films.

-immigration of Chloride ion

Generally, presence of holes due to tendency of alloy to pitting corrosion occurs at the environments containing chloride. Chloride has relatively small ionic radium, and enjoys high mobility in aqueous solutions, thus it penetrates on surface in oxide layer, causing formation of pit.

-further, Cl^- can substitute with OH^- in $Al(OH)_3$, and produce $AlCl_3$, causing the protective oxide film loses its characteristic.

Conclusion

Thickness of coated layer ranges from 0.1 to 10 micron, and this difference turns back to the role of the compositions such as Al2CuMg, so that coating in high thicknesses represents better strength of stress and stress corrosion.

According to standards ASTM G-47 and ASTM G-38, to end test and start evaluation of microscopic analysis, macrocracks must be seen, thus macrocracks were seen in simple samples during 670 hours, whereby this period has been less due to formation of oxide shell and reduction of cracks.

In the samples under Chromate conversion coating, macrocracks were seen during 1150 hours.

Increase of strength to corrosion of metal or Chromate conversion coating resulted in delay at emergence of the first effects of corrosion on basic metal and coating metal.

According to examination of stress corrosion under a fixed stress in simple samples and samples coated with Chromate, corrosion mechanisms between two analysis samples and duration of corrosions and emergence of microcracks were examined.

According to microscopic examination of samples, effects of Al2CuMg in corrosive environment were understood, where it was perceived what destructive effects can be on surface features of AA7075-T6 aluminum alloy with and without chromate coating.

By emergence of the first microcracks and penetration of invasive ions inside crack gap, acidic environment emerges in crack gap, through which acceleration of corrosion and release of aluminum ions will be followed.

As aluminum oxide is not an effective barrier to penetration of chloride ions and water to surface of alloy, thus penetration of chloride ions to the surface of alloy does not determine speed of corrosion. But, breakage of top layer of Chromate and formation of a solution is the determinant stage in film-metal interface, through which an active dissolution of metal will come to realize.

Suggestions

-Investigate Dacromet coatings on alloys in aerospace industry

-Investigate specific local corrosions on alloys in aerospace industry which result on irreparable losses

-Access to specific conversion Nano- coatings to indicate higher strength with fixed of cyclic stresses

-Access to growth rate data for fatigue in gap, as well as effect of crack microscopic in alloys in aerospace industry

-Measurement with high accuracy and modeling element limited from stress and pressure and penetration of hydrogen to tip of crack and stress area

-Recognize fundamental mechanism of hydrogen and chloride in which fatigue loss occurs in low growth rate with development of failure standards, e.g. observations from deformation of morphology in 0.5-5 mm on crack tip, and examination of crystallographic feature and a surface of environmental cracking

-Mechanism of chemical-mechanical model of growth rate of crack in stress corrosion
-Examination of environmental effects on crack growth rate under loading range of variable as well as being subjected to chemical environmental in aerial industries.

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