# EFFECT OF RETROGRESSION AND REAGING HEAT TREATMENT ON CORROSION FATIGUE CRACK GROWTH BEHAVIOR OF AA7050 ALLOY

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## ABSTRACT

# EFFECT OF RETROGRESSION AND REAGING HEAT TREATMENT ON CORROSION FATIGUE CRACK GROWTH BEHAVIOR OF AA7050 ALLOY

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The effect of retrogression and reaging heat treatment on corrosion fatigue crack growth behavior on AA7050 T73651 aluminum alloy is investigated. CT (Compact Tension) specimens are prepared in LS direction for fatigue crack growth tests . Samples are solution heat treated at 477 °C and aged at 120 °C for 24 h (T6 condition). After that, samples are retrogressed at 200 °C for times of 1, 5, 30, 55 and 80 minutes in a circulating oil bath. Then, samples are reaged at 120 °C for 24 h (T6 condition). Hardness measurements are taken at different retrogression times and at the end of the heat treatment.

Fatigue crack growth tests are performed at as received condition and at different retrogression times with sinusoidal loading of R=0.1 and f=1 in both laboratory air and corrosive environment of 3.5% NaCl solution. The highest fatigue crack growth resistance is observed for 30 min. and 5 min. retrogression for laboratory air and corrosive environment respectively. It is concluded that RRA can successfully be used to improve fatigue performance of this alloy.

**Keywords:** Fatigue crack growth rate, corrosion fatigue, retrogression and reaging heat treatment, crack length, stress intensity range, salt water solution.

# AA 7050 ALUMİNYUM ALAŞIMINDA RETROGRASYON VE YENİDEN YAŞLANDIRMA (RRA) ISIL İŞLEMİNİN KOROZYONLU YORULMA ÇATLAK İLERLEME DAVRANIŞINA ETKİSİ

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Bu çalışmada, retrograsyon ve yeniden yaşlandırma (RRA) ısıl işleminin AA 7050 T73651 aluminyum alaşımındaki korozyonlu çatlak ilerleme davranışına etkisi incelenmiştir. Yorulma çatlak ilerleme testleri için LS örnekleme yönünde kompakt çekme (KÇ) numuneleri hazırlanmıştır. Numuneler 477 °C'de çözündürülmüş ve 120 °C'de 24 saat yaşlandırılmıştır (T6 ısıl işlemi). Daha sonra, numunelere 200 °C'de karıştırmalı yağ banyosunda 1, 5, 30, 55 ve 80 dakika sürelerince retrograsyon uygulanmıştır. Ardından numuneler 120 °C'de 24 saat yeniden yaşlandırılmıştır (T6 ısıl işlemi). Değişen retrograsyon zamanlarında ve ısıl işlem sonunda sertlik değerleri alınmıştır.

Yorulma çatlak ilerleme testleri işlem görmemiş ve değişik retrograsyon zamanlı ısıl işlem görmüş numunelerde, R= 0.1 ve f=1 Hz yükleme durumunda sinüzoidal yük dalgası uygulanarak, hem laboratuvar hem de 3.5% NaCl çözeltili korozyonlu çevre şartlarında gerçekleştirilmiştir. En yüksek yorulma çatlak ilerleme dayancı laboratuvar ve korozyonlu çevre şartları için sırasıyla 30 dakika ve 5 dakika retrograsyon zamanlı numunelerde gözlemlenmiştir. Bu alaşımda yorulma performansının iyileştirilmesinde RRA ısıl işleminin başarıyla uygulanabileceği gözlemlenmiştir.

Anahtar Kelimeler: Çatlak büyüme hızı, korozyonlu yorulma, retrograsyon ve yeniden yaşlandırma ısıl işlemi, çatlak uzunluğu, gerilim şiddet aralığı, tuzlu su çözeltisi.

to my family....

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# **TABLE OF CONTENTS**

PLAGIARISM	iii
ABSTRACT	iv
ÖZ	vi
DEDICATION	viii
ACKNOWLEDGEMENTS	ix
TABLE OF CONTENTS	X

# CHAPTERS

1	INTRODUCTION	1		
2	THEORY			
	2.1 Mechanism of Fatigue Crack Growth	3		
	2.2 Fatigue Crack Growth Rate	7		
	2.2.1 Crack Propagation Laws	9		
	2.2.2 Growth Rate Diagram	11		
	2.3 Corrosion Fatigue Crack Propagation	12		
	2.3.1 Frequency and Waveshape Effects	13		
	2.3.2 Stress Ratio Effect	17		
	2.3.3 Environmental Effects	18		
	2.4 Mechanism of Corrosion Fatigue of Al Alloy	21		
	2.4.1 Strain Enhanced Anodic Disslotion	22		

		2.4.2 Surface Energy Reductions	24
		2.4.3 Hydrogen Embrittlement	25
	2.5	High Strength Al-Zn-Mg-Cu Alloys	27
	2.6	Heat Treatment of Aluminum Alloys	29
		2.6.1 Aging of High Strength Aluminum Alloys	29
		2.6.2 Retrogression and Reaging (RRA) Heat Treatment	31
3	EXF	PERIMENTAL PROCEDURE	36
	3.1	Testing Material	36
	3.2	Compact Tension (CT) Specimen	39
	3.3	Heat Treatment	41
	3.4	Test Equipment	43
	3.5	Testing Procedure and Program	46
4	RES	SULTS AND DISCUSSION	48
	4.1	Retrogression and Reaging Heat Treatment of CT Specimens	48
	4.2	Determination of Stress Intensity Range	50
	4.3	Determination of Crack Growth Rate	52
	4.4	Crack Growth Rate vs Stress Intensity Range Plot.	58
	4.5	Application of Fatigue Crack Growth Laws	65
	4.6	Effect of Heat Treatment on Crack Growth Rate	78
	4.7	Macro Examination of the Fracture Surfaces	84
	4.8	SEM Examination of the Fracture Surfaces	87
5	CON	NCLUSIONS	101
REFE	RENC	CES	102

## **CHAPTER 1**

#### **INTRODUCTION**

Constant demands in weight reduction have become one of the largest driving forces for the improvement of the properties of metallic materials in the transport industry. The properties, which need to be optimized in order to minimize the weight of airplanes or cars, are numerous; such as yield strength, toughness or corrosion resistance. The high strength Al-Zn-Mg-Cu alloys, based on the ternary Al-Zn-Mg system (7000 series), applied in the airplane and machine industry, are produced in cast and wrought states alike.

When the 7000 series alloys were first utilized for strength of critical structural components, it was understandable that aircraft manufacturers would try to exploit the useful strength advantage of these alloys offered compared with the 2000 series. Alloys were heat treated to the maximum strength (T6 temper) condition and many unfortunate stress-corrosion cracking (SCC) failures occurred. Over-aging to T7 temper can increase the corrosion resistance of this alloy but a strength loss of 10-15% is inevitable.

Corrosion fatigue properties of 7050 aluminum alloy, therefore, gained great importance due to their extensive use in structures of aircrafts flying in marine atmosphere. Especially military aircrafts are exposed to seawater due to wind or salt water spray from the sea during low altitude flights over sea. In addition to this, seawater which is condensed on the aircraft structures increases the corrosive effect.

Heat treatment studies have introduced combination of improved strength and corrosion resistance by retrogression and reaging (RRA) heat treatment which involves a shorter high temperature aging between two T6 heat treatments.

In this study, the effect of RRA heat treatment on fatigue crack behavior of 7050 aluminum alloy was investigated in laboratory air and salt solution. A pre-exposure in 3.5 % NaCl solution was selected to simulate the severe effect of sea water condensed on the aircraft structures. Corrosive environment of 3.5 % NaCl solution was used in determining the corrosion fatigue crack behavior. Another aim of this study was to determine the best retrogression time for fatigue properties in laboratory air and corrosive environment.

## **CHAPTER 2**

#### THEORY

# 2.1. Mechanism of Fatigue Crack Growth

Fatigue cracks can initiate under the action of cyclic loads as a result of cyclic plastic deformation. Even if the nominal stresses are well below the elastic limit, locally the stresses may be above the yield due to stress concentrations at inclusions or mechanical notches [1].

In fatigue fractures the loading level is usually macroscopically below that required to initiate gross or macroscopic plastic deformation. However, due to surface irregularities, microstructural features, and so on, the local microscopic stress exceeds the yield strength and plastic deformation occurs due to repeated fatigue loading. Fatigue failures are simply divide into three stages:

- i) Crack initiation
- ii) Crack propagation
- iii) Final fracture

Stages of fatigue crack formation is illustrated in Figure 2.1. The planes under high shear stress at the surface give rise to crack initiation at stage I. The crack

growth rate in this stage is on the order of Angstroms per cycle. Stage II starts after the initial crack extends a few grains in length. The crack growth rate in Stage II is relatively higher, which is on the order of microns per cycle. In this stage, fatigue crack growth occurs in the direction normal to the loading axis. Each loading cycle creates a characteristic fracture surface called a striation [2].



**Figure 2.1.** Schematic illustration of stage-I and stage-II fatigue crack formation [2].

The mechanism of crack initiation is described by Wood's model which is depicted in Figure 2.2. The figure illustrates the formation of slip-band extrusions and intrusions.



Figure 2.2. Woods model for fatigue crack initiation [1].

Slip occurs on a favorably oriented slip plane as the load rises in a cycle. Due to the fact that slip on the first plane is inhibited by strain hardening and by oxidation of the newly created free surface, slip takes place in the reverse direction on a parallel slip plane while the applied load decreases. Figure 2.2 shows that first cyclic slip forms an extrusion or an intrusion in the metal surface. The increase in the stress intensity at the intrusion site can grow into a crack by continuing plastic flow in the following cycles. This type of mechanism still exists even if the fatigue loading is cyclic tension-tension since the plastic deformation formed at the load increase will give rise to residual compressive stresses during load release [1]. Several stages of fatigue crack growth by slip cyle are shown step by step in Figure 2.3.



Figure 2.3. Possible model for fatigue crack growth [1].

A fatigue crack, once started, can also grow by a mechanism of reversed slip. Slip occurs quite easily at the crack tip where a large stress concentration exists due to a sharp crack in the tension field. The material above crack (stage1 and 2 in Figure 2.3) may slip along a favourable slip plane in the direction of maximum shear stress. The slip results in an opening of the crack and also increase in the crack length (stage3). Finally other slip planes are activated by work hardening and increasing stress, resulting in a blunt crack tip (stage 4). The crack is extended by an amount of  $\Delta a$  until the load reaches its maximum point.

Plastic deformation is formed in a small region at the crack tip covered by elastic surroundings. The elastic surroundings will contact during load release and the plastically deformed region becomes large enough to loose fit with its surroundings. In order to achieve the fit, compressive stresses are exerted on the plastic region by the elastic region as the load decreases. The compressive stresses formed by this attraction will locally be above the yield point again. These reversed reversed plastic deformation will close and resharpen the crack tip (stage 5) [1].

### 2.2. Fatigue Crack Growth Rate

The fatigue crack growth rate is simply the crack extension,  $\Delta a$ , within a number of cycles,  $\Delta N$ , and expressed as the ratio  $\Delta a / \Delta N$ . This crack growth rate can also be written as da/dN by taking the slope of crack growth curve ( a vs N curve).

When a component or a specimen containing a crack is subjected to cyclic loading, the crack length (a) increases with the number of fatigue cycles, N, if the load amplitude ( $\Delta P$ ), loading ratio (R) and the frequency of the cyclic loading (f) are kept constant. The crack growth rate, da/dN increases as the crack length increases during the test. In addition to these, da/dN will also be higher at higher load amplitudes. Thus, the following expression is derived to describe the combined effect.

$$\left(\frac{da}{dN}\right)_{R,f} = f(\Delta \mathbf{P}, a)$$

where the function "f" is dependent on the geometry of the specimen, the crack length, the loading configuration and the cyclic load range. This general relation is simplified by the use of the stress intensity range parameter,  $\Delta K$ . The magnitude of the load range ( $\Delta P$ ) as well as the crack length and the geometry is defined by  $\Delta K$  as follows:

$$\Delta K = K_{max} - K_{min}$$
  
 $K_{max} = f(\sigma_{max}, a) \text{ and } K_{min} = f(\sigma_{min}, a)$ 

Stress intensity range can also be expressed in terms of stress ratio by the following relationships:

$$\Delta K = (1 - R) K_{max}$$
 for  $R \ge 0$ , and  
 $\Delta K = K_{max}$  for  $R \le 0$ 

As it is seen from the expression above,  $\Delta K$  is equal to  $K_{max}$  when the minimum stess is compressive or zero. The stress ratio, R, can also be written as the  $K_{min}$  to  $K_{max}$  ratio [3].

#### 2.2.1. Crack Propagation Laws

The rate of fatigue crack growth is dependant on the stress intensity range,  $\Delta K$ . The increase in stress intensity range,  $\Delta K$ , directly corresponds to an increase in the crack growth rate, da/dN. Therefore, the crack growth rate can be expressed as a function of  $\Delta K$  as follows:

$$\frac{da}{dN} = f(\Delta K)$$

In practice,  $f(\Delta K)$  is determined empirically by the data obtained from the crack growth rate test [4]. But many workers attempted to describe the crack growth rate curve in semi- or wholly empirical manner. The first and most widely applied model is published by Paris and coworkers [5]. The model can conveniently represent the data for a wide range of mode-I crack growth rates, from  $10^{-9}$  to  $10^{-6}$  m/cycle.

$$\frac{da}{dN} = C(\Delta K)^m$$

where C and m are experimentally determined constants and generally regarded as material properties. For aluminum alloys m is proposed to be 4. It is seen from the model that the effect of mean stress,  $K_{th}$  and  $K_c$  are not considered. Many further attempts were made to express the influence of the stress ratio R= $\sigma_{min}/\sigma_{max}$  on da/dN, as published by Forman in 1967:

$$\frac{da}{dN} = \frac{C(\Delta K)^m}{(1-R)K_c - \Delta K}$$

where  $K_c$  is the critical stress intensity factor. It is often found that, the growth rate is virtually independent of R for steel when R is positive. However, the crack growth rate increases with increasing R for aluminum alloys [4].

Pearson et al. modified this expression to describe the fatigue crack growth behavior of thick specimens made of aluminum alloys for both high and low fracture toughness:

$$\frac{da}{dN} = \frac{C(\Delta K)^m}{\left[(1-R)K_c - \Delta K\right]^{1/2}}$$

Ewalds and Wanhill describes the complete sigmoidal curve by the following formula:

$$\frac{da}{dN} = C(\Delta K)^m \left[ \frac{1 - \left(\frac{\Delta K_{th}}{\Delta K}\right)^{n_1}}{1 - \left(\frac{\Delta K_{max}}{K_c}\right)^{n_2}} \right]^{n_3}$$

where  $\Delta K_{th}$  is the threshold intensity factor and  $n_1$ ,  $n_2$  and  $n_3$  are empirically determined exponents [6].

#### 2.2.2. Growth Rate Diagram

The growth rate diagram is drawn by plotting the data of da/dN with respect to  $\Delta K$  on a double logarithmic chart. The characteristic sigmoidal shape of fatigue crack growth rate curve for both inert and corrosive environment are shown in Figure 2.4.



**Figure 2.4.** Fatigue crack growth rate curve for inert and corrosive environments [7].

The crack growth curve is divided into three regions according to the curve shape, the mechanisms of crack extension and various influences on the curve.

In region I, there is a threshold value,  $\Delta K_{th}$ , below which cracks do not propagate or crack growth is too slow to be significant. Above this value the crack growth rate increases relatively rapidly with increasing  $\Delta K$ .

In region II, there is often a linear log-log relation between da/dN and  $\Delta K$ . This linear relation is often referred to as Paris regime of crack growth and can be described by the equation:

$$\frac{da}{dN} = C(\Delta K)^m$$

where c and m are experimentally determined constants.

Finally, in region III the crack growth rate curve rises to an asymptote where the maximum stress intensity factor,  $K_{max}$ , in the fatigue stress cycle becomes equal to the critical stress intensity factor,  $K_{IC}$ . This period of the crack growth is known as stage-3 or the terminal stage of the crack growth [7, 8].

#### 2.3. Corrosion Fatigue Crack Propagation

Corrosion fatigue in metals is the result of combined effect of cyclic stress and corrosive environment. For a given material, fatigue life decreases in the presence of an aggressive environment. The corrosion fatigue crack propagation is mainly affected by three factors; frequency and waveshape, stress ratio and the environment. Corrosion fatigue behavior depends on the interactions of these variables [3].

### 2.3.1. Frequency and Waveshape Effects

Cyclic load frequency is the most important variable that influences the corrosion fatigue for most material, environment and stress intensity conditions. Corrosion fatigue can be eliminated at excessively high frequencies. The dominance of the frequency is related directly to the time dependence of the mass transport and chemical reaction steps required for brittle cracking. In short, insufficient time for chemical attacks will decrease the corrosion effect and thus the crack propagation will be purely mechanical. The effect of frequency on the crack propagation is shown in the Figure 2.5.

N. J. H. Holroyd and D. Hardie [9] have also shown the differences in fracture appearance in terms of loading frequency, crack growth rate and  $\Delta K$  in Figure 2.6. Transition A and Transition B represent the transition from flat transgranular to frequency-independent corrosion fatigue and the transition from intergranular to flat transgranular cracking, respectively.



**Figure 2.5.** Crack growth rates during fatigue of 7017-T651 aluminum alloy in dry air at 4 Hz and in seawater at frequencies from 0.1 to 70 Hz, as a function of  $\Delta K$  [9].

In addition to the effect of loading frequency, the applied waveform also significantly affects the fatigue crack growth data. J. M. Barsom [10] studied the corrosion fatigue crack growth behavior of 12Ni-5Cr-3Mo maraging steel in 3 % NaCl solution under various cyclic stress fluctuations which is shown in Figure 2.7.



**Figure 2.6.** Corrosion fatigue crack growth of 7017-T651 aluminum alloy in seawater.

a) Combinations of loading frequency and  $\Delta K$  responsible for various modes of failure.

b) Dependence of critical velocity for fracture mode transitions A and B on loading frequency [9].

The data shows that the corrosion fatigue crack growth rates determined with the negative sawtooth wave and with the square sawtooth wave are essentially the same as the fatigue crack growth rate determined in air. These results confirm the fact that corrosive effect depends on the applied waveform.



**Figure 2.7.** Corrosion fatigue crack growth rates in 12Ni-5Cr-3Mo steel in 3 % NaCl solution under various cyclic stress fluctuations [10].

Corrosive effect is not observed in the case of negative sawtooth and the square waveform. A similar study was carried out by R. J. Selines and R. M. Pelloux [11] for 7075-T6 aluminum alloy. Fatigue crack growth data for this alloy tested in salt solution at 0.1 Hz under sinusoidal, square, positive and negative sawtooth loading form showed a behavior very similar to that presented for the 12Ni-5Cr-3Mo maraging steel in salt water.

#### 2.3.2. Stress Ratio Effect

The corrosion fatigue crack growth rate is generally enhanced by increased stress ratio, R, which is the ratio of the applied minimum stress to maximum stress. The effect of increased stress ratio is illustrated in Figure 2.7 for carbon steel stressed cyclically in pressurized nuclear reactor water at 288 °C.

The extent of the corrosion fatigue effect relative to dry air increased from about a factor of 4 at low R (0.11 to 0.24) to as much as 20 to 30 times at high R (0.61 to 0.71).

Corrosion fatigue probably proceeds by repeated passive film formation and rupture. Increased stress ratio at constant  $\Delta K$  results in increased crack tip strain and strain rate, increased film rupture and therefore enhanced corrosion fatigue crack propagation [3].



**Figure 2.7.** Effect of stress ratio on corrosion fatigue crack propagation in ASTM A 533 B and A508 carbon steels exposed to pressurized high-purity water at 288 °C. Frequency: 0.017 Hz [3].

### 2.3.3. Environmental Effects

The fatigue strength or fatigue life at a given maximum stress, generally decreases in the presence of an aggressive environment. This effect varies widely, depending primarily on the characteristics of the material / environment combination. The environment affects the probability of fatigue crack initiation, crack growth rate or both. For many materials, the stress range

required to cause failure diminishes progressively with increasing time with the number of cycles of applied stress.

A corrosive environment can clearly reduce the crack initiation time by any form of localized attack to form a stress concentration. Corrosion attack would clearly be favored at new unfilmed metal surfaces formed at a persistent slip band. A clear example for the importance of stress concentration due to corrosion is illustrated in Figure 2.8, which shows fatigue endurance results for plain and notched specimens of a 13% Cr ferritic steel in distilled water and salt water.

The increase in the chemical activity of the environment by lowering the PH of the solution, by increasing the concentration of the corrosive species or by increasing the pressure of the gaseous environment generally yield a decrease in the resistance of the material to corrosion fatigue. Corrosion fatigue behavior is related to the relative humidity or partial pressure of water vapor in the air for aluminum alloys and high strength steels. Corrosion fatigue crack growth rates of these alloys generally increase with increase in water vapor pressure until a saturation condition is achieved [3].



**Figure 2.8.** Corrosion fatigue endurance data for specimens of 13% Cr steel. Rotating bending test applied at 50 Hz and at a temperature of 23 °C [3].

The aqueous solutions that accelerate the stress corrosion cracking, such as solutions containing Cl<sup>-</sup>, Br<sup>-</sup> and  $\Gamma$  ions, also accelerate the corrosion fatigue crack growth in high strength aluminum alloys (e.g. for the 7079-T651 aluminum alloy). Similar to the case discussed above, the solutions containing  $SO_4^{=}$  ions do not affect stress corrosion cracking or corrosion fatigue crack growth in the same alloy [12]. The effect of different type of aqueous solutions on the corrosion fatigue crack growth behavior can be seen in Figure 2.9 below.



**Figure 2.9.** Effect of cyclic stress intensity range on the corrosion fatigue crack growth rate in a high strength aluminum alloy exposed to various environments [12].

# 2.4. Mechanism of Corrosion Fatigue of Al Alloys

There are three candidate mechanisms to describe environmentally assisted crack propagation (stress corrosion and corrosion fatigue). These are;

- i) Strain enhanced anodic dissolution
- ii) Surface energy reductions
- iii) Hydrogen embrittlement

#### 2.4.1. Strain Enhanced Anodic Dissolution

The strain enhanced anodic dissolution mechanism suggests that the strain induced rupture of the protective film on the crack surface is followed by rupture by metal dissolution. According to this mechanism the rate of film rupture, the rates of solution renewal and removal at the crack tip and the rate of passivation determine the rate of propagation.

In this model, crack growth is performed by the extremely localized anodic dissolution. The protective film at the crack surface, which is usually an oxide, is ruptured by the plastic strain at the crack tip, while the film at the remained crack surface and on the undeformed crack walls is kept unruptured. Due to this fact, an electrolytic cell is formed at this region, by the unbroken surface film acting as the cathode and the surface of the bare metal, which was formed as a result of the film rupture, acting as the anode. A rapid anodic dissolution occurs at the bare metal exposed to aggressive environment.

Cyclic process of the rupture of the protective film, dissolution and the film repair results in the crack growth. Figure 2.10 describes the crack growth by the strain enhanced anodic dissolution mechanism.



**Figure 2.10.** Schematic drawing of crack initiation and growth by enhanced anodic dissolution mechanism.

The rate of repassivation of the bare metal surface exposed to the corrosive environment is the main factor in determining the susceptibility to stress corrosion cracking or corrosion fatigue, since the protective film is considered to be passive. If the repassivation rate is too low, crack blunting may occur due to excessive dissolution at the crack tip. On the contrary, if this rate is excessively high, the anodic dissolution at the crack tip is minimized. For this reason, the corrosive effect is maximized at the intermediate passivation rates [7, 13].

#### 2.4.2. Surface Energy Reductions

This model suggests that the cohesive bonds at the surface of the metal are weakened as a result of the adsorption of damaging components in the aggressive environment. The decrease in local bond energies leads to brittle fracture at lower stresses and also to an increase in crack propagation rate. The metal-metal bond energy is reduced as a result of the energy used in binding the aggressor to the metal atoms, thus allowing an easier mechanical separation to occur. The possible mechanism of crack propagation by surface energy reduction model is illustrated in Figure 2.11.



**Figure 2.11.** Adsorption of aggressive species at the crack tip, causing a reduction in metal-metal bond strength [7].

Surface energy reduction model is widely accepted when the liquid metal embrittlement or stress corrosion cracking of plastics are considered. On the other hand, in the case of aqueous solutions, this model faces up with some problems. One problem comes up with the very rapid reformation of the protective film on the metal surface. The direct interaction between the anions in the aqueous solution and the bare metal surface may be prevented by these films. The damage caused by the anion in the adsorption process may decrease or disappear. Another problem may occur due to the formation of crack blunting at the crack tip resulting in a loss of atomic sharpness [7].

### 2.4.3. Hydrogen Embrittlement

The hydrogen embrittlement mechanism is based on the formation of the hydrogen atoms as a result of the reduction of hydrogen ions at the crack tip. The adsorption of the hydrogen atoms by the bare metal are thought to weaken or embrittle the metal-metal bonds just beneath the crack tip. When the case of acidification of the local environment by cation hydrolysis is concerned, discharge and absorption of hydrogen is favoured, especially if the passive film at the crack site is damaged or destroyed by this acidification. Once hydrogen is absorbed by the metal, this may lead to cleavage, intergranular separation or highly localized plastic fracture. Formation of brittle hydrides can be a part of fracture mechanism especially in hydride forming metals.

The possible reasons for the hydrogen embrittlement mechanism are shown in Figure 2.12. One of them is the formation of metal hydrides which are well known for their brittle nature. This has probably been the most favored
mechanism to describe the general phenomenon of the hydrogen embrittlement in the past. On the other hand, the possibility of the decrease in bond strength by adsorption process should have been taken into account. A third possible reason is the formation of hydrogen in tiny amounts. The thermodynamic tendency for the diffusion of hydrogen into the bare metal and hydrogen gas formation is very great. Once the hydrogen gas is formed at the cavities of the bare metal, it is unable to diffuse through the metal lattices, leading to pressure increase in the cavities. This extreme pressure increase can give rise to crack propagation in the material [7, 13].



Figure 2.12. Possible of hydrogen embrittlement mechanisms [7].

The sequence of events that occur during subcritical crack propagation due to hydrogen embrittlement can be summarized as follows:

- i) Diffusion of a reducible hydrogen containing species [e.g. H3O<sup>+</sup>] to the crack tip region.
- ii) Reduction of the ions that contain hydrogen and giving adsorbed hydrogen atoms.
- iii) Interstitial diffusion of these hydrogen atoms to the process zone in front of the crack tip.
- iv) Once the critical level of hydrogen concentration over a volume is achieved, localized crack initiation in this zone is followed by rapid crack propagation back to main crack tip [13].

Beside all these described above, the most important factor to be taken into account is the mechanism that hydrogen enters the metal. The permeation of the hydrogen to the bare metal is very low due the characteristic of the naturally formed film on the crack surface. Therefore, the crack initiation and propagation processes will highly be affected by the mechanical or chemical events which weaken or damage this protective film. The exposure of hydrogen to emerging dislocations will be achieved by this chemical or mechanical damage to the protective film, resulting in separation of precipitate matrix interfaces by the pumping of hydrogen into the crack process zone [14].

## 2.5. High Strength Al-Zn-Mg-Cu Alloys

The high strength Al-Zn-Mg-Cu aluminum alloys are well canditates for aircraft constructions due to their high modulus and low density. However, the

use of these alloys are limited especially in marine environments because of their susceptibility to stress corrosion cracking.

The main alloying elements in 7000 series aluminum alloys are Al, Zn, Mg and Cu. The amount of Zn is generally higher than Mg. Although they can be used as both castings and wrought products, wrought products are preferred because of the poor castibility of these alloys.

The main alloying elements in 7000 series aluminum alloys are Zn and Mg. The best strength and response to heat treatment is obtained by high Zn/Mg ratio. But the susceptibility to SCC and grain boundary corrosion increases with increasing Zn/Mg ratio. On the other hand, lower Zn/Mg ratio produces better weldability and lower quench sensivity. The best fatigue resistance is also obtained at lower Zn/Mg ratios.

Cu has a limited strenghening effect but provides corrosion resistance under stress conditions.

The total amount of Zn, Mg and Cu determines the properties in use. When the total amount is higher than 9 %, high strength is achieved as well as corrosion resistance, formability and weldability. Total amount between 6 to 8 % still provides high strength, but with a better formability and weldability. Below 5-6 % total amount, susceptibility to SCC dissappear and fabricability becomes paramount [15].

Aluminum and aluminum intermetallic compounds are very active, yielding high susceptibility to intergranular corrosion when precipitates segregate at the grain boundaries. In Al-Mg-Zn (e.g. 7030) alloys, MgZn<sub>2</sub> is attacked anodically. In copper containing alloys such as Al-Cu (2024) and Al-Zn-Mg-Cu (7075), the copper depleted zone adjacent to grain boundary precipitates is again attacked anodically [16].

#### 2.6. Heat Treatment of Aluminum Alloys

Heat treatment of aluminum alloys has been a subject for investigations to benefit its high strength with low density. Improved corrosion resistance can be achieved by overaging heat treatments, but with a loss in strength.

## 2.6.1. Aging of High Strength Aluminum Alloys

The heat treatment applied to aluminum alloys of 7000 series, for strengthening the matrix by means of precipitation from the supersaturated solid solution (SSS), is composed of two main, consecutive stages:

• Homogenization, affecting the morphology and phase composition of constituents of the alloy;

• Supersaturation and ageing, affecting the state of the solid solution

In the ternary Al-Zn-Mg system, the sequence of decomposition as the aging time increases is as follows:

Supersaturated Solid Solution 
$$\rightarrow$$
 Guiner-Preston Zones  $\rightarrow \eta$   
 $\rightarrow \eta(MgZn_2) \rightarrow T[(AlZn)_{49}Mg_{32}]$ 

Addition of copper facilitates and accelerates nucleation of GP zones and  $\eta$  phase, without changing the decomposition of the solid solution [17].

Corrosion and stress corrosion cracking in aluminum alloys is almost transgranular. Therefore particular attention should be given to the microstructure and chemistry of the grain boundary. The distribution and presence of the grain boundary phase are very sensitive to the heat treatment. In 7000 series aluminum alloys, the boundary most likely contains  $\eta(MgZn_2)$ or T phase [(AlZn)<sub>49</sub>Mg<sub>32</sub>] depending on the aging time [18].

Unless the sample is rapidly quenched into water, in many cases the precipitates will nucleate during quenching. As the cooling rate decreases, the precipitates becomes coarser and separated. If the precipitates nucleate during aging, they will initially be very fine and closely spaced [18].

In 7000 series aluminum alloys, there is a depletion of Mg and Zn occurs around  $MgZn_2$  and  $(AlZn)_{49}Mg_{32}$ ) precipitates. As the aging time increases, these zones usually become more and more depleted. The precipitates at the

grain boundary are surrounded by a precipitate free zone. The first reason for this zone is the solute depletion discussed above. The driving force for precipitation decreases due to depletion in this region. The second is the decrease in vacancy concentration. Vacancies retained from quenching will annihilate themselves by moving towards the grain boundaries [18].

#### 2.6.2. Retrogression and Reaging (RRA) Heat Treatment

7000 series aluminum alloys are prone to SCC if aged by using tempers where strengthening is provided mainly by Guiner-Preston (GP) zones and transition precipitate of  $\eta$ . The mechanism of SCC in 7000 alloys is still subject to investigation but most authors seem to favor anodic dissolution in combination of with a hydrogen embrittlement mechanism [19, 20].

It is well known that when overageing heat treatment (T7x duplex aging treatment which produces a matrix strengthened by  $\dot{\eta}$  and  $\eta$ ) was applied to the high strength 7000 series aluminum alloys, reduced susceptibility to stress corrosion can be achieved at the expense of their strength. Recently, new heat treatment designated retrogression and reaging (RRA) treatment, which is effective in improving the stress corrosion resistance of Al-Zn-Mg-Cu alloys without a significant loss of strength, has been developed [21, 22].

The discovery of the phenomenon of retrogression and reaging (RRA) by Cina [23] has resulted in the experimental application of these types of thermal treatments to many high strength 7000 series alloys introducing improved combinations of strength and SCC resistance. Initial RRA investigations were mainly based on 7075, the most widely used alloy at the time [21, 24, 25].

The RRA process is fundamentally a three-step aging process: the first step is low temperature aging (~120  $^{\circ}$ C), usually under the T6; the second step is retrogression at higher temperatures (180-260  $^{\circ}$ C); and the third step is reaging at low temperatures (~120  $^{\circ}$ C), usually under the T6 condition once again. The general overview of the hardness versus ageing time characteristics of retrogression stage is given in Figure 2.13.



**Figure 2.13.** Schematic representation of the change in hardness during retrogression and reageing [23].

The evaluation of strength during retrogression is particularly interesting. The strength first decreases at a pace which steeply increases with temperature, showing a minimum at a soak time, also strongly affected by the temperature. Longer treatments produce a gradual reduction in the strength. Park and Ardell, who conducted transmission electron microscopy observations, suggested that this behavior could be understood in terms of the following phase transformations:

(1) The initial decrease in the strength should be caused by the dissolution of the precipitate phases formed during T6 treatment. There is a lively controversy on whether the T6 microstructure consists of primarily  $\eta$  particles, as suggested by Park and Ardell [26].

(2) Then, new particles precipitate concurrently with the dissolution of the phases formed at lower temperatures; this process would be responsible for the minimum showed by the strength.

(3) The final decrease of the strength should be caused by general coarsening of particles [21, 27].

Many microstructural investigations have been conducted to determining the reason for enhanced SCC resistance in RRA treated 7000 series alloys. Two theories have been put forward. The first and most widely quoted is that similar to the process occurring during overageing, RRA promotes coarse precipitation

of the equilibrium phase  $\eta$  (MgZn<sub>2</sub>) in the grain and sub-grain boundaries while maintaining a fine distribution of  $\dot{\eta}$  in the grain interiors. These coarse precipitates then act as hydrogen trapping sites, locally reducing the atomic hydrogen concentration in the matrix around the grain boundary [25, 28]. The other suggestion by Cina is that the dislocations introduced during quenching are given the opportunity to anneal out during the RRA treatment reducing the susceptibility to hydrogen embrittlement at the grain boundaries [29].

The high mechanical strength developed by the alloy in the T6 temper is associated with a high density metastable precipitation, distributed homogeneously in the aluminum matrix; whilst a lower density of thicker precipitates, more stable in the nature, is the microstructure responsible for the inferior strength of the overaged T7 temper.

Whilst maintaining the T6 temper microstructure inside the grains, RRA treatments promote coarsening of the grain boundary precipitation; this microstructure becomes similar to that of the T7 temper; the overall microstructure being more stable than that for the T6 temper. The high density of fine precipitates is responsible for the high mechanical strength achieved with these treatments [30].

The stress corrosion resistance of the alloy is generally thought to be controlled by the microstructure near to grain boundaries (the size and spacing of precipitates, free precipitation zones, and solute concentration gradients): the effect of these parameters is however not fully understood, but it is well known that the susceptibility of these alloys to stress corrosion cracking decreases with ageing time [31, 32].

## **CHAPTER 3**

## **EXPERIMENTAL PROCEDURE**

# 3.1. Testing Material

The material used in this study was a cold rolled plate of 7050 T73651 aluminum alloy. The chemical composition in weight percentages of this alloy is given in Table 3.1.

**Table 3.1.** The chemical composition of 7050 T73651 aluminum alloy inweight percentages [33].

	Al	Zn	Mg	Cu	Zr	Ti	Cr	Mn	Si	Fe
Min.	87.48	5.7	1.9	2.0	0.08	-	-	-	-	-
Max.	89.85	6.7	2.6	2.6	0.15	0.06	0.04	0.1	0.12	0.15

The T73651 heat treatment, which was applied to the material by the supplier, was solution heat treatment at 477°C, followed by double aging at 121°C for 24 hours and at 163°C for 24 hours. The microstructure of this plate is shown in Figure 3.1.



**Figure 3.1.** The microstructure of 7050 T73651 cold rolled aluminium plate in 3 directions (X200).

As it is seen from the micrograph, about one third of the microstructure is recrystallized and the rest is polygonized grains. Evenly distributed very fine precipitates were observed throughout the structure and larger intermetallic compounds were found among the recrystallized grains. It should be noted that flattening of recrystallized grains in the short transverse (S) direction and elongation of them in the longitudinal (L) direction were observed as a result of the cold rolling process.

The directionality in the microstructure also results in a variation in mechanical properties due to orientation. The mechanical properties of the plate reported by Industrieanlegen Betriebsgesellschaft mbH, IABG, Germany are given in Table 3.2 [33].

 Table 3.2. Mechanical properties of cold rolled 7050-T73651 aluminum alloy

 in 3 directions [33].

Direction	UTS (MPa)	σ <sub>y</sub> (MPa)	E (MPa)	Elongation (%)
L	510	447	72200	10
LT	519	444	73300	9
ST	502	423	72800	7

The plain strain fracture toughness,  $K_{IC}$ , values of 7050-T73651 aluminum alloy determined by IABG in 4 orientations is given in Table 3.3.

**Table 3.3.** The plain strain fracture toughness,  $K_{IC}$ , values of 7050-T73651 plate in 4 orientations [33].

Orientation	LT	TL	ST	SL
$K_{IC}, MPa\sqrt{m}$	38.42	30.55	30.26	27.35

## **3.2.** Compact Tension (CT) Specimen

In this study, compact tension (CT) specimens were used to investigate the fatigue crack growth behaviour of 7050 aluminum alloy. The CT specimens were prepared from the cold rolled plate in LS direction. The designation L and S indicates the loading and the fatigue crack growth direction respectively. Possible directions for loading and crack growth are shown in Figure 3.2.

The shape and dimension requirements of the CT specimen were determined according to ASTM E647-00 standard. The relative dimensions are shown in Figure 3.3.



Figure 3.2. Crack plane orientation code for rectangular sections [34].



Figure 3.3. Dimension of the CT specimen according to the ASTM E 647 [35].

In this study, CT specimens with a width of W=36 mm was used. The thickness was selected as B=7 mm, which was proposed within the intervals as  $W/20 \le B \le W/4$  in ASTM E647. Chevron notch was preferred as a fatigue crack starter notch, which was described in ASTM E647. The shape and the details of chevron notch are given in Figure 3.4.



Figure 3.4. The shape and the dimensions of chevron notch [35].

## 3.3. Heat Treatment

In this study, the effect of retrogression and reaging (RRA) heat treatment on fatigue and corrosion fatigue crack growth behaviour was investigated. RRA heat treatment was a three step process followed by solutionizing: the first step was a low temperature aging at 120 °C, usually under the T6; the second step was a retrogression at 200 °C; and third step was a reaging at low temperature at 120 °C, usually under the T6 condition once again.

In order to determine the aging characteristics of 7050 aluminum alloy, 7 mm thick specimens were solution treated at 477 °C for 65 minutes in muffle furnace and water quenched as described in ASM Speciality Handbook [36]. Then, the specimens were aged at 120 °C up to 44 hours in oil bath, which was circulated in order to achieve effective heat conduction to the specimen from the furnace and homogeneous temperature distribution within the medium. The photographs of the oil bath are given in Figure 3.5.

The temperature of the oil bath was kept in  $\pm 1$  °C. Specimens were water quenched immediately after aging in order to avoid further precipitation and grain growth. Vickers hardness tests at 10 kg (HV-10) were applied and at least 5 measurements were taken to minimize the experimental error. The hardness profile of 7050 aluminum alloy is given in Figure 3.6.



Figure 3.5. Photographs of circulating oil bath.



Figure 3.6. Hardness profile of 7050 aluminum alloy aged at 120°C.

In order to investigate the effect of RRA heat treatment on hardness, 7 mm thick specimens were solutionized at 477 °C for 65 minutes in muffle furnace and water quenched. The samples were aged at 120 °C for 24 hours, followed by retrogression at 200 °C for 1, 2, 5, 10, 20, 30, 40, 60 and 80 minutes. Then the samples were reaged 120 °C for 24 hours. At least 5 hardness measurements were taken after solutionizing, retrogression and reaging steps.

#### 3.4. Test Equipment

All fatigue pre-crack and crack growth tests were conducted on a closed-loop, servo- hydraulically activated MTS universal testing machine. A corrosion cell

made of Plexiglas was adopted on the specimen. The schematic view of the corrosion cell is given in Figure 3.7.



Figure 3.7. Schematic view of the corrosion cell in 3D.

The corrosion cell was designed in such a way that the specimen loading pins were prevented from contacting with the corrosive medium to avoid a galvanic action. The design of the corrosion cell also provided a complete contact of corrosive medium through the crack tip up to the unstable failure of specimen. The photograph of the corrosion cell attached to the specimen is given in Figure 3.8.



Figure 3.8. Photograph of corrosion cell attached to the CT specimen.

Crack lengths on both sides of the CT specimen were measured optically by travelling microscope with a resolution of  $\pm$  0.01 mm. Corrosive medium was sent to the corrosion cell at room temperature under free potential by a closed-loop circulation system.



**Figure 3.9**. Fatigue crack growth tests. a) Fatigue crack growth measurement in laboratory air. b) CT specimen after failure in corrosive environment.

#### **3.5. Testing Procedure and Program**

The aim of this study was to study the effect of RRA heat treatment on the fatigue and corrosion fatigue crack growth behaviour of 7050 aluminum alloy in LS orientation. Therefore, 12 CT specimens were machined. As received and heat treated specimens were tested in both laboratory air and in corrosive environment. Selected retrogression times for fatigue and corrosion fatigue crack growth tests were 1, 5, 30, 55 and 80 minutes.

All CT specimens were first pre-cracked by subjecting them to constant amplitude sinusoidal load cycles with a frequency of 10 Hz to achieve an infinitely sharp fatigue pre-crack which satisfies the requirements suggested in ASTM E647 standard. The crack lengths achieved during fatigue pre-cracking were measured by travelling microscope with a resolution of  $\pm 0.01$  mm and the difference between crack lengths from the both sides of the specimen were kept in the limits suggested in ASTM E647 standard.

Fatigue crack growth tests (FCGT) in laboratory air and corrosive environment were conducted by constant amplitude sinusoidal load waves with a frequency of 1 Hz. The stress ratio, R, was selected as 0.1. All fatigue and corrosion fatigue crack growth tests were conducted until the failure of the specimen.

The specimens tested in corrosive environment were first immersed into 3.5% NaCl solution for 72 hours to simulate the flight deck environments of an

aircraft carrier. The stress ratio and the frequency were same as used in laboratory air tests. All corrosion fatigue crack growth (CFCG) tests were conducted in 3.5% NaCl solution under free corrosion potentials. Corrosive medium was prepared with distilled water and pure MERCK salt. The velocity of NaCl solution through the corrosion cell was 0.5 lt/min.

The specimen-pin-clevis assembly and the alignment quality of the testing machine were checked carefully throughout the experiments.

The fracture surfaces were cleaned with alcohol after the experiments. Then these surfaces were macro-examined and photographed. Samples were cut from the fracture surfaces and cleaned by an ultrasonic technique in alcohol bath for scanning electron microscope studies. Microphotographs were taken from the fracture surfaces.

#### **CHAPTER 4**

#### **RESULTS AND DISCUSSION**

## 4.1. Retrogression and Reaging Heat Treatment of CT Specimens

In order to investigate the effect of RRA heat treatment on hardness, 7 mm thick specimens were solutionized at 477 °C for 65 minutes in muffle furnace and water quenched. The samples were aged at 120 °C for 24 hours, followed by retrogression at 200 °C for 1, 2, 5, 10, 20, 30, 40, 60 and 80 minutes. Then the samples were reaged 120 °C for 24 hours. Hardness measurements were taken after solutionizing, retrogression and reaging steps. Hardness profile is shown in Figure 4.1.

As it is seen from the figure, there was a significant decrease in the hardness at 1 and 2 min. retrogression times after retrogression stage. It was stated by Park and Ardell that the initial decrease in hardness is caused by dissolution of precipitate phases formed at initial T6 heat treatment. The minimum hardness at retrogression stage is explained by the precipitation of new particles concurrently dissolution of phases at lower temperatures [26]. The peak hardness was achieved after 10 minutes of retrogression. As the retrogression time increases, a decrease in hardness was observed. This can be caused by general coarsening of particles [21, 27].

After the reaging heat treatment, the minimum hardness was observed again at 1 minute and the peak hardness was achieved at 5 minutes retrogression time. It should also be noted that there was a gradual decrease in the hardness after the peak hardness. Retrogression and reaging heat treatment was applied to the CT specimens at the same heat treatment conditions described above. Retrogression times selected for fatigue and corrosion fatigue crack growth tests were 1, 5, 30, 55 and 80 minutes and shown as "•" on Figure 4.1. CT specimens were kept at –30 °C to avoid the possibility of natural aging.



**Figure 4.1.** Hardness profile of 7050 aluminum alloy with respect to retrogression time, for both retrogression and reaging heat treatment steps. Selected retrogression times for fatigue and corrosion fatigue crack growth tests are shown as " $\bigcirc$ " on the figure.

The hardness values measured on CT specimens are given in Table 4.1 for all heat treatments.

 Table 4.1. Hardness values of CT specimens used for fatigue crack growth tests.

Heat Treatment	Hardness Values		
	(HV-10)		
As Received (T 73651)	165±2		
1 min. retrogression	188±2		
5 min. retrogression	200±2		
30 min. retrogression	187±2		
55 min. retrogression	181±2		
80 min. retrogression	167±2		

# 4.2. Determination of Stress Intensity Range

The stress intensity range,  $\Delta K$ , has been calculated by the corresponding crack length for CT specimens using the following formula suggested in ASTM E647-00 standard [35].

$$\Delta K = \frac{\Delta P}{B\sqrt{W}} \frac{(2+\alpha)}{(1-\alpha)^{3/2}} (0.886 + 4.64\alpha - 13.32\alpha^2 + 14.72\alpha^3 - 5.6\alpha^4)$$

where  $\alpha = a/W$  and the  $\Delta P = P_{max} - P_{min}$ 

This expression for  $\Delta K$  is considered to be valid for  $a/W \ge 0.2$ . The crack length, a, were calculated by taking the average of crack lengths measured optically from both surfaces of the CT specimen.

Maximum stress intensity,  $K_{max}$ , was determined by using critical crack length, a<sub>c</sub>. Critical crack length, which is the maximum value of the crack just before the unstable fracture, was measured on the fracture surfaces. The crack front curvature was taken into account during the crack length measurements. Therefore, five equally spaced points were selected on the final crack front, which was clearly apparent on the fracture surface. Crack lengths at each point were measured with a sensitivity of 0.01 mm by a travelling microscope and the average of these 5 values were used to calculate the a<sub>c</sub>.  $K_{max}$  values calculated for each specimen tested in laboratory air and corrosive environment are given in Table 4.2.

Heat Treatment	$K_{max}$ , MPa.m <sup>1/2</sup>			
	Lab. Air	Salt Solution		
As Received (T 73651)	54.66	36.30		
1 min. retrogression	62.26	40.33		
5 min. retrogression	40.91	48.05		
30 min. retrogression	101.42	34.64		
55 min. retrogression	52.79	42.45		
80 min. retrogression	52.08	45.87		

**Table 4.2.** Maximum Stress Intensity Range,  $K_{max}$ , in air and salt solution.

# 4.3. Determination of Crack Growth Rate

The average crack length measured on both surfaces of the CT specimen versus the elapsed stress cycles data were collected during the experiments. Crack length measurements were made at intervals such that da/dN data were nearly evenly distributed with respect to  $\Delta K$ . For CT specimen, the suggested intervals in ASTM E647 standard were:

$$\begin{split} \Delta a &\leq 0.04 \text{ W for } 0.25 \leq a/W \leq 0.40 \\ \Delta a &\leq 0.02 \text{ W for } 0.40 \leq a/W \leq 0.60 \\ \Delta a &\leq 0.01 \text{ W for } a/W \geq 0.40 \end{split}$$

Crack length, a, versus number of cycles, N, curves of the specimens at as received and heat treated conditions obtained from laboratory air and corrosive environment are given in Figures from 4.2 to 4.7. The comparative a vs N curves for all laboratory air tests and corrosive environments are shown in Figure 4.8 and Figure 4.9, respectively.

The average value of crack lengths at the two surfaces of CT specimens was used in all  $\Delta K$  and crack growth rate calculations. The crack growth rate, da/dN, data was evaluated by seven-point incremental polynomial technique. The basic computer program, which was recommended in ASTM E647, was used in determining the da/dN data.



**Figure 4.2.** Crack length vs number of cycles curves for specimens at as-received condition.



**Figure 4.3.** Crack length vs number of cycles curves for 1 min. retrogressed specimens.



**Figure 4.4.** Crack length vs number of cycles curves for 5 min. retrogressed specimens.



**Figure 4.5.** Crack length vs number of cycles curves for 30 min. retrogressed specimens.



**Figure 4.6.** Crack length vs number of cycles curves for 55 min. retrogressed specimens.



**Figure 4.7.** Crack length vs number of cycles curves for 80 min. retrogressed specimens.



**Figure 4.8.** Comparative a vs N curves of laboratory air fatigue tests of asreceived and RRA heat treated specimens.



**Figure 4.9.** Comparative a vs N curves of corrosion fatigue tests of as-received and RRA heat treated specimens.

## 4.4. Crack Growth Rate vs Stress Intensity Range Plots

Logarithmic plots of da/dN vs  $\Delta K$  for as received and different retrogression times are given in Figures from 4.10 to 4.15 with two-slope Paris representation. Laboratory air and corrosion fatigue crack growth data are given on the same plot for comparison. The increase in fatigue crack growth rates due to salt solution can be easily seen on the figures.



**Figure 4.10.** da/dN vs  $\Delta K$  plot of as received specimens with two-slope representation of Paris equation.



**Figure 4.11.** da/dN vs  $\Delta$ K plot of 1 min. RRA heat treated specimens with two-slope representation of Paris equation.



**Figure 4.12.** da/dN vs  $\Delta K$  plot of 5 min. RRA heat treated specimens with two-slope representation of Paris equation.


**Figure 4.13.** da/dN vs  $\Delta K$  plot of 30 min. RRA heat treated specimens with two-slope representation of Paris equation.



**Figure 4.14.** da/dN vs  $\Delta$ K plot of 55 min. RRA heat treated specimens with two-slope representation of Paris equation.



**Figure 4.15.** da/dN vs  $\Delta K$  plot of 80 min. RRA heat treated specimens with two-slope representation of Paris equation.

## 4.5. Application of Fatigue Crack Growth Laws

Two-Slope Paris equation was used to represent the data while predicting the crack growth rate in air and corrosive environment.

For slow fatigue crack growth rates, less than or equal to  $2 \times 10^{-7}$  m/cycle, Paris equation was used in the following form:

$$\frac{da}{dN} = A_1 (\Delta K)^m$$

For higher crack growth rates (greater than  $2 \ge 10^{-7}$  m/cycle) Paris equation was applied in the following form:

$$\frac{da}{dN} = A_2 (\Delta K)^{m_2}$$

A<sub>1</sub>, n<sub>1</sub>, A<sub>2</sub> and n<sub>2</sub>, constants were computed separately for laboratory air and corrosive environment data. The coefficients of the fits determined by linear regression analysis are tabulated in Table 4.3. The fitted curves were also represented on da/dN vs  $\Delta$ K plots in Figures from 4.10 to 4.15 as two-slope straight lines.

Heat Treatment	Environment	A <sub>1</sub>	<b>m</b> 1	$r_1^2$	$\mathbf{A}_2$	<b>m</b> <sub>2</sub>	$r_2^2$
As Received (T 73651)	Air	-	-	-	2 x 10 <sup>-9</sup>	1.85	0.97
	Salt	-	-	-	4 x 10 <sup>-9</sup>	2.21	0.93
1 min. Retrogression	Air	3 x 10 <sup>-19</sup>	11.04	0.78	7 x 10 <sup>-10</sup>	2.45	0.97
	Salt	1 x 10 <sup>-14</sup>	8.58	0.58	2 x 10 <sup>-9</sup>	2.48	0.99
5 min. Retrogression	Air	7 x 10 <sup>-18</sup>	8.70	0.74	1 x 10 <sup>-11</sup>	3.48	0.98
	Salt	3 x 10 <sup>-16</sup>	3.76	0.80	8 x 10 <sup>-10</sup>	2.66	0.97
30 min. Retrogression	Air	2 x 10 <sup>-24</sup>	15.14	0.95	2 x 10 <sup>-10</sup>	2.45	0.99
	Salt	1 x 10 <sup>-26</sup>	20.52	0.88	9 x 10 <sup>-10</sup>	2.83	0.95
55 min. Retrogression	Air	2 x 10 <sup>-15</sup>	7.42	0.93	3 x 10 <sup>-10</sup>	2.49	0.99
	Salt	9 x 10 <sup>-20</sup>	13.26	0.85	9 x 10 <sup>-10</sup>	2.84	0.99
80 min. Retrogression	Air	1 x 10 <sup>-14</sup>	6.60	0.84	7 x 10 <sup>-10</sup>	2.36	0.98
	Salt	1 x 10 <sup>-57</sup>	53.33	0.97	6 x 10 <sup>-10</sup>	3.29	0.97

 Table 4.3. Constants of two-slope Paris equation.

A third degree polynomial fit was applied to each da/dN vs  $\Delta K$  data, for both laboratory air and corrosion fatigue crack growth tests in the following form:

$$\log (da/dN) = C(1) + C(2) \log(\Delta K) + C(3) \log^{2}(\Delta K) + C(4) \log^{3}(\Delta K)$$

The coefficients for third degree polynomial fits and corelation factors computed by least square data fitting technique are given in Table 4.4.

The fitted polynomials for all heat treatments are given in Figures 4.16 to 4.21. The fits are shown on laboratory air and salt solution data in the same figure for comparison. In order to see the effect of heat treatment, all air and corrosion fatigue curves were shown side by side in Figures 4.22 and 4.23, respectively.



**Figure 4.16.**  $3^{rd}$  degree polynomials fitted to da/dN vs  $\Delta K$  plot of as received specimens tested in air and salt solution.



**Figure 4.17.**  $3^{rd}$  degree polynomials fitted da/dN vs  $\Delta K$  plot of 1 min. RRA heat treated specimens tested in air and salt solution.



**Figure 4.18**  $3^{rd}$  degree polynomials fitted da/dN vs  $\Delta K$  plot of 5min. RRA heat treated specimens tested in air and salt solution.



**Figure 4.19.**  $3^{rd}$  degree polynomials fitted da/dN vs  $\Delta K$  plot of 30 min. RRA heat treated specimens tested in air and salt solution..



**Figure 4.20.**  $3^{rd}$  degree polynomials fitted da/dN vs  $\Delta$ K plot of 55 min. RRA heat treated specimens tested in air and salt solution.



**Figure 4.21.**  $3^{rd}$  degree polynomials fitted da/dN vs  $\Delta K$  plot of 80 min. RRA heat treated specimens tested in air and salt solution.

**Table 4.4.** The coefficients of best fit  $3^{rd}$  degree polynomials computed by processing  $\log(da/dN)$  vs  $\Delta K$  data in the form of  $\log(da/dN) = C(1) + C(2)\log(\Delta K) + C(3)\log^2(\Delta K) + C(4)\log^3(\Delta K)$ .

Heat Treatment	Environment	C(1)	C(2)	C(3)	C(4)	$\mathbf{R}^2$
As Received	Air	-30.322	53.787	-41.351	10.911	0.987
(T 73651)	Salt	-13.807	14.015	-8.505	2.0133	0.974
1 min.	Air	-35.497	59.9	-41.47	9.9019	0.980
retrogression	Salt	-9.296	4.377	-1.797	0.5465	0.986
5 min.	Air	-112.2	225.7	-161.85	39.167	0.980
retrogression	Salt	-32.288	57.244	-42.502	10.941	0.992
30 min.	Air	-73.772	145.01	-104.94	25.588	0.995
retrogression	Salt	-24.152	39.167	-28.954	7.632	0.969
55 min.	Air	-26.397	41.869	-30.293	7.7025	0.996
retrogression	Salt	-26.937	47.21	-36.298	9.792	0.989
80 min.	Air	-55.535	106.51	-77.493	19.101	0.988
retrogress ion	Salt	-36.27	66.075	-48.679	12.422	0.987



**Figure 4.22.** Effect of retrogression time on fatigue crack growth behavior in laboratory air.



**Figure 4.23.** Effect of retrogression time on fatigue crack growth behavior in salt solution.

Application of two-slope Paris equation gives acceptable results for most laboratory air data. The accuracy of the fits was better for medium and high crack growth rates (  $da/dN \ge 2 \times 10^{-7}$  m/cycle ). It can also be seen from  $r_2^2$  data in Table 4.3 that two out of six fit values are 0.99. The rest of the fits with a lower  $r_2^2$  values of 0.98 and 0.97.

The accuracy in the fits of corrosion fatigue data for the same fatigue crack growth rates were realtively low. Although two out of six fit values were 0.99, the  $r_2^2$  values 0.95 and 0.93 of the two fits can not be acceptable. The deviation from linearity can be seen from Figures 4.10 and 4.13, respectively.

Two-slope Paris equation applied to the laboratory air and corrosion fatigue data at realtively lower crack growth rates (  $da/dN < 2 \times 10^{-7}$  m/cycle ) gave unacceptable fit values. The highest  $r_1^2$  value was 0.97 and the rest data fitting values varied 0.58 to 0.95.

It can be concluded that this equation can be applied succesfully to laboratory air and with a lower accuracy to corrosion fatigue data at medium and high crack growth rates. On the other hand, the same equation applied to lower crack growth rates ( below  $2 \times 10^{-7}$  m/cycle ) gives unacceptable fit values.

Data fitting by  $3^{rd}$  degree polynomials to whole da/dN vs  $\Delta K$  curves give succesfull results for both laboratory air and corrosion fatigue data. The lowest accuracy of the fit was achieved at 30 min. RRA treated specimen tested in salt

solution with a  $R^2$  value of 0.969. It can be concluded from the data in Table 4.4 that  $3^{rd}$  degree polynomials can be applied succesfully to both laboratory air and corrosion fatigue data.

## 4.6. Effect of Heat Treatment on Crack Growth Rate

It can easily be seen from da/dN vs  $\Delta K$  plots that fatigue crack growth rate increases in varying amounts with the existence of salt solution. Even for relatively low  $\Delta K$  levels at the start of Paris regime, the difference between the crack growth rates in salt solution and laboratory air is significant in Figures from 4.16 to 4.21.

Many scientists have made investigations on the effect of environment on fatigue threshold,  $\Delta K_{th}$ , for different materials and environments. It was observed in some studies on Al alloys that  $\Delta K_{th}$  is reduced by corrosive environments [37, 38, 39]. On the other hand, an increase in  $\Delta K_{th}$  was observed in some other studies on steels [12, 40].

In this study, it is difficult to discuss the effect of environment on  $\Delta K_{th}$  because of the lack of data below  $10^{-8}$  m/cycle. It should be noted that fatigue crack growth rate should be  $10^{-8}$  m/cycle or lower to determine the  $\Delta K_{th}$ . Nevertheless, it can be stated from our data that corrosive environment of salt solution has a slight influence on  $\Delta K$  at crack growth rates very close to the near-threshold region for all heat treatments. In region II fatigue crack growth, the effect of salt solution becomes more significant. The dependence of heat treatment on the extent of this effect can be summarized in Table 4.5. Fatigue crack growth rates at the stress intensity range of 20 MPa.m<sup>1/2</sup> was selected, since this  $\Delta K$  value passes through region II of all da/dN vs  $\Delta K$  plots obtained in laboratory air and salt solution tests (Figures from 4.16 to 4.21). The effect of salt solution on fatigue crack growth rate is shown by FCGR<sub>Salt/Air</sub> ratio in Figure 4.24.



Figure 4.24. Variation FCGR<sub>Salt/Air</sub> ratio with respect to heat treatment.

Heat Treatment	Environment	FCGR, (m/cycle)	FCGR <sub>Salt/Air</sub>	FCGR <sub>Air/As Rec</sub> .	FCGR <sub>Salt/As Rec</sub> .	
As Rec. (T 73651)	Air	5.175 x 10 <sup>-7</sup>	5.28	1.00	1.00	
	Salt	2.731 x 10 <sup>-6</sup>	5.20			
1 min.	Air	1.181 x 10 <sup>-6</sup>	2.02	2.28	1.26	
Retrogression	Salt	3.450 x 10 <sup>-6</sup>	2.92			
5 min. Retrogression	Air	4.649 x 10 <sup>-7</sup>	4.05	0.90	0.84	
	Salt	2.301 x 10 <sup>-6</sup>	4.95			
30 min.	Air	3.472 x 10 <sup>-7</sup>	12.19	0.67	1 55	
Retrogression	Salt	4.228 x 10 <sup>-6</sup>	12.10	0.07	1.55	
55 min.	Air	5.802 x 10 <sup>-7</sup>	7.42	1.12	1 59	
Retrogression	Salt	4.303 x 10 <sup>-6</sup>	7.42		1.50	
80 min.	Air	8.181 x 10 <sup>-7</sup>	5.00	1 50	1.50	
Retrogression	Salt	4.093 x 10 <sup>-6</sup>	5.00	1.38	1.50	

**Table 4.5.** Fatigue crack growth rate data at 20 MPa.m $^{1/2}$ .

From the FCGR<sub>Salt/Air</sub> ratio given in Table 4.5, the highest effect of salt solution is observed at 30 and 55 min. RRA heat treatment with a ratio of 12.18 and 7.42. This effect decreases for the heat treatments T73651, 80 min., 5 min. and 1 min. retrogression times, respectively. The fatigue crack growth rate in salt solution is about 5 times faster compared to air specimens for as received (T73651), 5 min. and 80 min. RRA heat treatments. The minimum effect of salt solution was observed at 1 min. retrogressed specimen with the ratio of 2.92.

It should be also noted that the effect of salt solution is not same if the  $m_2$  values are considered. The slope of the da/dN vs  $\Delta K$  curve increases in region II by the effect of salt solution, except in 5 min. RRA heat treatment. The  $m_2$  values were 3.48 and 2.66 for laboratory air and corrosive environment, respectively. Therefore, it will not be true to state that there is an increase in the slopes of region II of da/dN vs  $\Delta K$  curve by salt solution.

Fatigue crack growth rate data at 20 MPa.m<sup>1/2</sup> also gives valuable information about the effect of heat treatment on fatigue crack growth behavior. The variation of fatigue crack growth rates due to heat treatment is given in Figure 4.25. The minimum value on this curve indicates the best heat treatment for improved fatigue performance in laboratory air and salt solution.



**Figure 4.25.** Fatigue crack growth rates at 20 MPa.m $^{1/2}$  for different heat treatments.

The ratio of fatigue crack growth rates compared to as received condition are also listed in Table 4.5 for both laboratory air (FCGR<sub>Air/As Rec.</sub>) and salt solution (FCGR<sub>Air/As Rec.</sub>). In laboratory air experiments, it was observed that 30 min. and 5 min. RRA heat treatment results in a decrease in fatigue crack growth rate and thus leading to a better fatigue resistance than as received condition. It can be concluded that the best fatigue performance can be achieved by 30 min. RRA heat treatment with a FCGR<sub>Air/As Rec.</sub> ratio of 0.67 in laboratory air.

The FCGR<sub>Air/As Rec.</sub> ratio for 55 and 80 min. RRA heat treatment were 1.12 and 1.58, respectively. The worst fatigue performance was observed at 1 min. RRA

heat treatment with a 2.28 times faster fatigue crack growth compared to as received condition.



Figure 4.26. Variation FCGR<sub>Air/As Rec.</sub> ratio with respect to heat treatment.

For salt solution experiments, improved corrosion fatigue resistance was achieved at 5 min. RRA heat treatment with a  $FCGR_{Salt/As Rec.}$  ratio of 0.84. This ratio was about 1.5 for 30, 55 and 80 min. retrogression times. It is also significant from Figure 4.27 that 5 min. RRA heat treatment results in improved corrosion fatigue properties among all heat treatments.



Figure 4.27. Variation FCGR<sub>Salt/As Rec.</sub> ratio with respect to heat treatment.

It can be concluded from Figure 4.26 that 30 min. RRA treatment has the best fatigue performance in laboratory air. For corrosion fatigue tests, 5 min. RRA treatment shows the highest corrosion fatigue resistance among all other heat treatments (Figure 4.27).

## 4.7. Macro Examination of the Fracture Surfaces

The close-up macro photographs of the fracure surfaces for both laboratory air and corrosive envronment are given in Figures 4.28 to 4.31.



**Figure 4.28.** Fracture surfaces of as received, 1 and 5 min. RRA heat treated specimens (from left to right) tested in laboratory air.



**Figure 4.29.** Fracture surfaces of 30, 55 and 80 min. RRA heat treated specimens (from left to right) tested in laboratory air.



**Figure 4.30.** Fracture surfaces of as received, 1 and 5 min. RRA heat treated specimens (from left to right) tested in salt solution.



**Figure 4.31.** Fracture surfaces of 30, 55 and 80 min. RRA heat treated specimens (from left to right) tested in salt solution.

## 4.8 SEM Examination of the Fracture Surfaces

The results of examination of the fracture surfaces by scannning electron microscope, SEM, are given as micrographs in Figures from 4.32 to 4.42. The direction of the crack growth is downwards.

Fracture surface examinations by scanning electron microscope, SEM, shows transgranular type of crack propagation in laboratory air and salt solution environments. Transgranular planes containing fatigue striations join by tear ridges, which are parallel to the crack growth direction in all specimens.

The tear ridges and furrows indicate that crack propagation occurs on different planes with several local fronts. These local cracks then join with the neighboring crack fronts as the macro fatigue crack propagates. The coalescence of individual crack fronts occurs with a ductile tearing between the local crack planes, forming tear ridges or furrows (Figures 4.35a, 4.36b and 4.40b).





Figure 4.32. SEM photographs of as received specimen tested in air.
a) Fatigue striations, c.g.r. ≈ 9.1 x 10<sup>-7</sup> m/cycle, X1500
b) A large secondary crack at relatively high crack growth rate, X300.





(b)

- Figure 4.33. SEM photographs of 1 min. RRA treated specimen tested in air. a) Typical indications of fatigue; tear ridges with striations on them, X400.

  - b) Fatigue striations on tear ridges, c.g.r.  $\approx 1.5 \times 10^{-6}$  m/cycle, X1000.









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Figure 4.35. SEM photographs of 30 min. RRA treated specimen tested in air.
a) Crack coalescence of different crack growth planes at low crack growth rate, c.g.r. < 2 x 10<sup>-7</sup> m/cycle, X400.
b) Fracture surface just before fast fracture, c.g.r. ≈ 1.4 x 10<sup>-6</sup> m/cycle, X1000.











Figure 4.37. SEM photographs of 80 min. RRA treated specimen tested in air.
a) General view of fracture surface, c.g.r. ≈ 4 x 10<sup>-7</sup> m/cycle, X400.
b) Fracture surface at the vicinity of fast fracture, c.g.r. ≈ 2.4 x 10<sup>-6</sup> m/cycle, X400.





Figure 4.38. SEM photographs of as received specimen tested in salt solution.
a) Fatigue striations with brittle nature, c.g.r. ≈ 1.6 x 10<sup>-6</sup> m/cycle, X3000.
b) Fatigue striations, c.g.r. ≈ 1.2 x 10<sup>-6</sup> m/cycle, X1000.





Figure 4.39. SEM photographs of 1 min. RRA treated specimen tested in salt solution.

- a) Fatigue striations, c.g.r.  $\approx 2.5 \times 10^{-6}$  m/cycle, X1500 b) A large secondary crack at relatively high crack growth rate, X400.





Figure 4.40. SEM photographs of 5 min. RRA treated specimen tested in salt solution.

- a) Fatigue striations covered by corrosion product, c.g.r. ≈ 1.3 x 10<sup>-6</sup> m/cycle, X3000
  b) General view of fracture surface at relatively high crack growth rate, c.g.r. ≈ 3.5 x 10<sup>-6</sup> m/cycle, X400.





Figure 4.41. SEM photographs of 30 min. RRA treated specimen tested in salt solution.

- a) Fracture surface at relatively higher crack growth rate, X1000.
  b) Closer look at fatigue striations covered by corrosion products in the same region, c.g.r. ≈ 2.8 x 10<sup>-6</sup> m/cycle, X3000.


(b)

Figure 4.42. SEM photographs of 80 min. RRA treated specimen tested in salt solution.

- a) General appearance of fracture surface, X400.b) Fracture surface at relatively high crack growth rate, X400.

Fatigue striations are observed on transgranular planes, which are separated by tear ridges (Figures 4.34a, 4.36b and 4.41b). The convex curvature of striations indicates the position of the propagating local crack front.

The fatigue crack growth rates measured by SEM observations showed good correlation with the experimental data.

It was also observed that the density of fatigue striations decreases as the crack growth rate increases. At relatively high  $\Delta K$  levels, i.e. at the vicinity of fast fracture region, dimples and voids begin to appear together with striations (Figure 4.38b).

Fracture surfaces of the specimens tested in laboratory air showed ductile type fatigue striations. On the other hand, striations observed on corrosion fatigue fracture surfaces are sometimes brittle type (Figure 4.38a). Striations covered by corrosion products can be seen in Figure 4.41b and they also seem to be brittle type.

Secondary cracks perpendicular to the main crack growth direction are frequently observed in all specimens (Figures 4.40b and 4.41a). The coalescence of secondary cracks with each other and forming longer cracks can be seen in Figures 4.32b and 4.34a. It is also significant from Figures 4.34b,

4.35b and 4.36b that large number of secondary cracks is formed between striations at the valley of these striations.

Intermetallic particles cut by crack propagation are observed in some fracture surfaces. On the other hand, particles in striated regions are not cut by crack growth. Instead of these, crack growth occurs around the particles as seen from Figures 4.32a and 4.41b. This phenomenon may be due to the increase in the stress concentration around the intermetallic particles.

Fast fracture regions showed ductile type failure with dimples in all specimens.

## **CHAPTER 5**

## CONCLUSIONS

- 1. RRA heat treatment can be successfully applied to 7050 aluminum alloy to improve fatigue performance in both laboratory air and salt solution.
- The highest fatigue crack growth resistance is obtained at 30 and 5 min.
  RRA heat treatment for laboratory air and salt solution, respectively.
- 3. The fatigue crack growth behavior of 7050 aluminum alloy is affected seriously by salt solution in all heat treatments. The crack growth rates are a few times faster than those tested in laboratory air. The adverse affect of salt solution was the highest at 30 min. RRA heat treated specimen.
- 4. The application of two-slope Paris equation to describe the crack growth rate behavior gives acceptable results at medium and high crack growth rates for both laboratory air and corrosion fatigue tests.
- Based on the fractographic analysis, crack propagation is transgranular type, which occurs with several local fronts on different growth planes. Fatigue striations and secondary cracks are observed on crack propagation regions of all fracture surfaces.

## REFERENCES

- D. Broek, "Elementary Engineering Fracture Mechanics", Mechanisms of Crack Growth, 1986, pp. 48
- [2] C. R. Brooks, "Failure Analysis of Engineering Materials", Fatigue Fracture Topography, 2002, pp. 157.
- [3] ASM Handbook, Fatigue and Fracture, Vol 19, pp. 61-210.
- [4] W. Schütz, "Fatigue Crack Growth", Fracture Design Methodology, AGARD Lecture Series No 97, 1978.
- [5] P. C. Paris, M. P. Gomez and W. E. Anderson, "A Rational Analytic Theory of Fatigue", The Trend in Engineering, University of Washington, Vol 13, No 1, 1961.
- [6] S.A. Meguid, "Fatigue Crack Growth", Engineering Fracture Mechanics, 1989, pp. 243
- [7] K. R. Trethewey, J. Chamberlain, "Environment Sensitive Cracking", Corrosion for Science and Engineering, 1995, pp. 214.
- [8] H. L. Edwalds and R. J. H. Wanhill, "Fatigue Crack Growth", Fracture Mechanics, 1987, pp. 171.
- [9] N. J. H. Holroyd, and D. Hardie, "Corrosion Fatigue of 7000 Series Aluminum Alloys", Environment-Sensitive Fracture: Evaluation of Test Methods: a Symposium sponsored by ASTM Committee G-1 on Corrosion of Metals and National Bureau of standards, 1982, pp. 534.

- [10] J. M. Barsom, "Effect of Cyclic Stress Form on Corrosion Fatigue Propagation Below K<sub>ISCC</sub> in a High Yield Strength Steel", International Conference Series, Vol NACE-2, National Association of Corrosion Engineers, Houston, 1972.
- [11] R. J. Salines and R. M. Pelloux, "Effect of Cyclis Stress Wave Form on Corrosion Fatigue Propagation in Al-Zn-Mg Alloys", Department of Metallurgy and Materials Science Report, M.I.T, Cambridge, Mass., 1972.
- [12] J. M. Barsom, "Corrosion Fatigue Crack Propagation", Fracture and Fatigue Control in Structures, 1987, pp. 392
- [13] P. Marcus and J. Oudor, Stress Corrosion Mechanisms, Corrosion Mechanisms in Theory and Practice, 1995, pp. 311.
- [14] D. J. Duquette, "Mechanism of Corrosion Fatigue of Aluminum Alloys", AGARD Conference Proceedings No 316, Corrosion Fatigue, April 1982, 1-1.
- [15] L. F. Mondolfo, "Al-Alloys Structure and Properties", London: Boston: Buterworths, 1976, pp. 842.
- [16] D. A. Jones, Principles and Prevention of Corrosion, 1996, pp. 307.
- [17] A. Gazda, M. Malgorzata and W. Wierzchowski, "DTA investigations of the retrogression and re-aging in some AlZnMgCu alloys", Thermochimica Acta, 303, 1997, pp. 197-202.
- [18] C. L. Briand, Mechanical Aspects of Environmental Failures, 1985, pp. 181.
- [19] T.D. Burleigh, Corrosion, Vol. 47, 1991, pp. 89-98.

- [20] D. Najjar, T. Magnin and T. J. Warner, "Influence of Critical Surface Defects and Localized Competition Between Anodic Dissolution and Hydrogen Effects During Stress Corrosion Cracking of a 7050 Aluminum Alloy", Mat. Sci. Eng., A238, 1997, pp. 293-302.
- [21] J. K. Park and A. J. Ardell, "Effect of Retrogression and Reaging Treatments on the Microstructure of Al-7075-T651", Met. Trans., 15A, 1984, pp. 1531.
- [22] M. Kanno and I. Araki, "Precipitation Behavior of 7000 Alloys During Retrogression and Reaging Treatment", Mat. Sci. Tech., Vol. 10, 1994, pp. 599.
- [23] B. Cina, US Patent, 3856584, 1974.
- [24] J. Lankford, D. L. Davidson, W. L. Morris and R.P. Wei, Eds., ASTM, 1983, pp. 283.
- [25] R. S. Kaneko, "Retrogression and Reaging: Solution for Stress Corrosion Problems with T6 Temper Aluminum", Metal Progress, Vol.118, 1980, pp. 41-43
- [26] J. K. Park and A. J. Ardell, "Precipitate Microstructure of Peak-Aged 7075 Al", Scripta Metall., 22, 1988, pp. 1115.
- [27] J. K. Park, "Influence of Retrogression and Reaging Treatments on the Strength and Stress Corrosion Resistance of Aluminum Alloy 7075-T6", Mat. Sci. Eng., A103, 1988, pp. 223-231.
- [28] W. Rajan, W. Wallace and J. C. Beddoes, "Microstructural Study of a High-Strength Stress-Corrosion Resistant 7075 Aluminum Alloy", Journal of Mat. Sci., Vol. 17, 1982, pp. 2817-2824.

- [29] M. Talianker and B. Cina, "Retrogression and Reaging and the Role of Dislocations in the Stress Corrosion of 7000-Type Aluminum Alloys", Met. Trans. 20A, 1989, pp. 2087.
- [30] F. Viana and A. M. P. Pinto, "Retrogression and Reaging of 7075 Aluminum Alloy: Microstructural Characterization", Journal of Mat. Proc. Tech., 1999, pp. 54-59.
- [31] W. Hepples, M. R. Jarret, J. S. Crompton and N. J. Holroyd, In: Proc. Environment-Induced Cracking of Metals, 1989, pp. 383.
- [32] N. J. H. Holroyd, In: Proc. Environment-Induced Cracking of Metals, 1989, pp. 311.
- [33] Bericht Nr. TF-621.3 Bauteilspezifische Wekstoffuntersuchungen, Untersuchungen an Halbzeug 7050-T73651, Bruchzahigkeit and Ermudungsverhalten, IABG,1976.
- [34] ASTM E 399-90, Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials.
- [35] ASTM E 647-00, Standard Test Method for Measurement of Fatigue Crack Growth Rates.
- [36] ASM Speciality Handbook, "Heat Treating of Aluminum Alloys", Aluminum and Aluminum Alloys, pp. 675-718.
- [37] J. Albrecht, J. W. R. Martin, G. Lütjering and J. W. Martin, "Influence of Micromechanisms on Fatigue Crack Propagation Rate of Al-Alloys", Proceedings of the 4<sup>th</sup> Int. Conference on the Strength of Metals and Alloys, France, Vol. 2, 1976, pp. 463.

- [38] J. Petit, "Some Aspects of Near-Threshold Crack Growth: Microstructural and Environmental Effects", Fatigue Crack Growth Threshold Concepts, Edited by D. Davidson and S. Suresh, 1984, pp. 3.
- [39] S. Suresh, A. K. Vasudevan and P. E. Bretz, "Mechanisms of Slow Fatigue Crack Growth in High Strength Aluminum Alloys: Role of Microstructure and Environment", Met. Trans. A, 1984, pp. 369-379.
- [40] A. Cigada, M. P. D. Ambrosio, T. Pastore and P. Pedeferri, "Fatigue Behavior in Seawater of an Offshore Steel in the Presence of Sharp Defects", Proceedings of 5<sup>th</sup> Int. Offshore Mechanics and Arctic Eng. Symposium, Vol. 2, ASME, 1986, pp. 262.