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İSTANBUL TECHNICAL UNIVERSITY ★ INSTITUTE OF SCIENCE AND TECHNOLOGY

**RETROGRESSION AND RE-AGING STUDIES ON
VARIOUS ALUMINUM ALLOYS**

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**RETROGRESYON VE YENİDEN YAŞLANDIRMA İŞLEMİ
UYGULANAN BAZI ALUMİNYUM ALAŞIMLARI**

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FOREWORD

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ABBREVIATIONS

R	:Retrogression
RRA	:Retrogression and reaging
T6	:Solution treated then artificially aged
T7	:Solution heat treated and overaged
GP zones	:Guiner Preston zones
HRB	:Rockwell hardness
IGC	:Intergranular corrosion
SCC	:Stress corrosion cracking
IACS	:International Annealed Copper Standard
HCl	:Hydrochloric acid
H₂O₂	:Hydrogen peroxide
HNO₃	:Nitric acid
HF	:Hydrogen fluorur
NaOH	:Sodium hydroxide
pH	:Potential of hydrogen
ISO	:International Organization for Standardization
ASTM	:American Society of Testing and Materials
CEC	:Cavitation erosion corrosion
RWR	:Relative wear resistance

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RETROGRESYON VE YENİDEN YAŞLANDIRMA İŞLEMİ UYGULANAN BAZI ALÜMİNYUM ALAŞIMLARI

ÖZET

Yüksek mukavemete sahip alüminyum alaşımlarının teknolojik gelişmedeki yeri büyüktür. Günümüzde, hava araçlarının gövde yapılarının büyük bir kısmı alüminyum alaşımları kullanılarak imal edilmektedir. Alüminyum alaşımlarını pek çok uygulama için uygun ve ekonomik kılan özelliklerin başında; görünümü, hafifliği, fiziksel ve mekanik özellikleri ve yüksek korozyon dayanımı gelir.

Uçak sanayinde en çok kullanılan alüminyum alaşımlarından biri çökeltme sertleşmesiyle yüksek dayanım kazandırılabilen 7075 alaşımıdır. Bu alaşıma en yüksek dayanımı kazandıran ısıtıl işlem T6 temperi olarak bilinir. Ancak bu durumda alaşımın gerilmeli korozyon direnci düşmektedir. Gerilmeli korozyon direncini arttıran T73 ısıtıl işlemi ise dayanımda % 10-15'e varan bir düşmeye sebep olduğundan, bu tür alaşımlarda mekanik özelliklerin korunarak korozyon özelliklerinin geliştirilmesi önem kazanmaktadır. Retrogresyon ve yeniden yaşlandırma (RRA) olarak bilinen ısıtıl işlemle hem gerilmeli korozyon direnci hem de yüksek dayanım özelliklerini aynı alaşıma kazandırmak mümkündür. Bu işlemde, T6 durumdaki malzemeye 200-280⁰C aralığında kısa süreli bir ısıtıl işlem uygulanır ve hemen ardından yeniden yaşlandırılır. Yeniden yaşlandırma işlemi T6 temperi için önerilen koşullarda uygulanmaktadır. Son otuz yıldır 7XXX serisi alüminyum alaşımlarına ait çeşitli RRA çalışmaları yapılmasına karşın, 6XXX serisine ait yeterli çalışma bulunmamaktadır.

Bu çalışma kapsamında, RRA işleminin 7075, 7175 ve 6061 alüminyum alaşımlarının sertlik, elektrik iletkenliği, korozyon ve aşınma özellikleri üzerindeki etkisi incelenmiş; optimum özellikleri sağlayan ısıtıl işlem koşulları belirlenmiştir.

RETROGRESSION AND RE-AGING STUDIES ON VARIOUS ALUMINUM ALLOYS

SUMMARY

Aluminum alloys are important engineering materials for technological development due to their improved combination of mechanical properties and low density. They are predominantly used in construction of aircraft airframes. The main issues which make them attractive and economical for most of the applications are their appearance, lightness, physical and mechanical properties.

Among the aluminum alloys, 7075 Al alloy is widely used in aerospace industry due to its improved strength by precipitation hardening mechanism. The precipitation hardening process of this alloy is known as T6 temper. However T6 temper may result in loss in stress corrosion cracking resistance. Although T73 temper increases the stress corrosion cracking resistance, it decreases the strength of alloy by 10-15%. Therefore it is an important issue to preserve the strength which improves the corrosion resistance for aluminum alloys. The process known as retrogression and reaging (RRA) has been applied to 7075 alloys to gain the combination of both the strength of T6 temper and stress corrosion cracking resistance of T73 temper. This heat treatment involves a short period annealing of T6 temper alloy at 200-280°C. Afterwards reaging is applied in condition similar to that of T6 temper. In last thirty years, although many RRA studies have been conducted on 7XXX series aluminum alloys, a satisfactory result has not been reported about RRA treatment of 6XXX series aluminum alloys.

In this study, the influence of RRA on the hardness, electrical conductivity, corrosion and wear properties have been systematically examined to determine the optimum RRA treatment conditions for 7075, 7175 and 6061 alloys. The stipulations of heat treatment are determined supplying the optimal properties.

1. INTRODUCTION

Aluminum and aluminum alloys are among the most widely used metals, because of their perfect combination of light weight, high strength, improved corrosion resistance and affordable price [1].

The attractive combination of higher strength to weight ratio and higher fracture toughness of 7XXX aluminum alloys has rendered them as candidate materials for aerospace applications. The highest strength for 7XXX aluminum alloys is achieved by T6 temper, but in this case low corrosion resistance can limit the applicability of the alloy. The susceptibility of these alloys to corrosion can be improved with T73 temper (overaging), which is mostly accompanied by a loss of strength [3]. Since T73 temper considerably reduces strength (10-15% of peak strength), it has been a serious problem concerning 7XXX series alloys.

A two-step heat treatment, known as Retrogression and Re-aging (RRA), was performed over thirty years ago by Cina and his colleagues in Israel, in an attempt to give high corrosion resistance in 7XXX aluminum alloys equivalent to the T73 temper, together with the T6 strength levels [4]. This treatment is applied to the material in T6 condition. The retrogression and Re-aging process is followed by two steps. In the first step the alloy is subjected to a retrogression step. This is a short period of immersion at a temperature above that of the ageing but below that of the solution treatment temperature. In the second step, the alloy is re-aged at the T6 ageing temperature [5].

Extensive studies have been carried out on the kinetics, microstructure and structure-property relationships over a wide range of processing conditions and alloy compositions of 7XXX aluminum alloys [1-5]. However satisfactory studies have not been performed on Al-Mg-Si (6XXX) alloys which have a widespread usage in various engineering applications.

In the extend of our study, the influence of RRA on hardness (yield strength), electrical conductivity, corrosion and wear properties were evaluated for 7075, 7175, 6061 alloys.



2. ALUMINUM AND ALUMINUM ALLOYS

2.1 Classification of Aluminum and Aluminum Alloys

The mechanical, physical and chemical properties of aluminum alloys change with the composition of the alloying metal. Generally one or more additions are used to get proper combination. Major alloying elements are copper, manganese, silicon, magnesium and zinc. Total amount of these additions can not be more than 10% of total weight. Aluminum alloys are divided into two major groups as a wrought composition and cast composition [2].

The alloys and temper designation systems, which are used for wrought aluminum, were adopted by aluminum industry around 1955 and the current system for the cast aluminum system was established some later. The aluminum industry itself manages the creation and continuing maintenance of these systems through its industry organization, the Aluminum Association. The standards of the Aluminum Association are used widely by classifying the aluminium alloys.

2.1.1 Wrought aluminum alloys designation system

The Aluminum Association Wrought Aluminum Alloy Designation System (Table 2.1.) uses a four digit system, where digits are sometimes followed by alphabetical prefixes showing the production method.

- The first digit shows the major alloying element,
- The second digit defines variations in the original basic alloy: zero is used for the original composition, one is used for the first variation, two is used for the second variation, and so forth,
- The third and fourth digits designate the specific alloy within the series.

Table 2.1: Wrought Aluminum Alloy Designation System [1].

Alloy	Main Alloying Element	Usage
1XXX	Pure aluminum; no major alloying addition	Electrical and chemical industries
2XXX	Copper	Aircraft
3XXX	Manganese	Architectural Applications
4XXX	Silicon	Rod welding and sheet brazing
5XXX	Magnesium	Marine Environments
6XXX	Magnesium and Silicon	Architectural Applications
7XXX	Zinc	Aircraft Structural Components
8XXX	Other elements	
9XXX	Unassigned	

2.1.2 Cast aluminum alloy designation system

The cast alloying designation system (Table 2.2.) also uses four digit system like Wrought Aluminum Designation System, but there is a decimal point between the third and fourth digits to identify that the alloy is in the form of casting or foundry ingot. The second and third digits indicate the purity for the 1XX.X series and for rest of the series they identify the specific aluminum alloy [1].

Table 2.2: Cast Aluminum Alloy Designation System [1].

Main Alloying Element	Usage
1XX.X	Pure aluminum, 99.00% max.
2XX.X	Copper
3XX.X	Silicon, with added copper and/or magnesium
4XX.X	Silicon
5XX.X	Magnesium
6XX.X	Unused series
7XX.X	Zinc
8XX.X	Tin
9XX.X	Other elements

2.2 Aluminum Alloy Temper Designation System

2.2.1 Basic temper designation system

The alloy designation is separated with a hyphen to show the temper designation. The first character is a capital letter in order to indicate the general class of treatment in the temper designation.

F; as fabricated

Applies to wrought or cast products in which no special control is used over thermal conditions or strain hardening process. There are no mechanical property limits for wrought alloys concerning with this temper, but for cast alloys there generally are.

O; annealed

Applies to wrought products which are usually annealed to obtain the lower strength in order to increase subsequent workability. The O applies to cast products that are annealed to improve ductility and dimensional stability. It may be followed by a digit other than zero.

H; strain hardened

Applies to products which are strengthened by strain hardening process. They may or may not have supplementary thermal treatments to reduce the strength levels. The H is always followed by two or more digits.

W; solution heat treated

Applies to only alloys that are aged spontaneously after solution heat treating. This designation is specific only when digits are used in combination with W to indicate the period of natural aging.

T; thermally treated to produce stable tempers other than F, O or H

Applies to products, that are thermally treated, with or without supplementary strain hardening, to produce stable tempers. The T is always followed by one or more digits.

2.2.2 Subdivision of the basic tempers

The temper designation system is based on sequence of basic treatments used to produce different tempers and their variations. Subdivisions are indicated by one or more digits following the letter.

i. Subdivision of the Basic H tempers

H1, strain hardened only

Applies to products, which are strain hardened to achieve the desired level of strength without extra thermal treatment. The number of following H1 shows strain hardening degree.

H2, strain hardened and partially annealed

Applies to products that have been strain hardened more than the desired amount, and their strength is dropped to the desired level by partial annealing. The number appended to H2 determines the degree of strain hardening remaining after partial annealing.

H3, strain hardened and stabilized

Applies to products, that have been strain hardened and then stabilized either by a low temperature thermal treatment, or as a result of heat introduced during fabrication of the product. Stabilization usually improves ductility. The H3 temper is used only for those that will gradually age soften at room temperature if they are not stabilized. The number added to H3 indicates the degree of strain hardening remaining after stabilization.

H4, strain hardened and lacquered or painted

Applies to products that have been strain hardened and that have been subjected to heat during subsequent painting or lacquering operations. The number added to H4 indicates the amount of strain hardening left after painting or lacquering.

A digit following the H1, H2, H3 and H4 define strain hardening degree.

ii. Subdivision of the Basic T tempers

T1, cooled from elevated temperature shaping process and naturally aged to a substantially stable conditions

Applies to products for which the rate of cooling from an elevated temperature shaping process, such as casting and extrusion, is such that their strength is by room-temperature aging.

T2 cooled from elevated temperature shaping process, cold worked, and naturally aged to a substantially stable condition

Applies to products that are cold worked to improve strength after cooling from an elevated temperature shaping process or for which the effect of cold work may not be recognized in mechanical property limits.

T3, solution heat treated cold worked, and naturally aged to a substantially stable condition

Applies to products which are cold worked to improve strength or in which the effect of cold work in flattening or straightening is recognized in mechanical property limits.

T4, solution heat treated and naturally aged to a substantially stable condition

Applies to products that are not cold worked after solution heat treatment or which the effect of cold work in flattening or straightening may not be recognized in mechanical property limits.

T5, cooled from an elevated temperature shaping process, then artificially aged

Applies to products that are not cold worked after cooling from elevated temperature shaping process such as casting and extrusion and then artificially aged to improve the mechanical properties or dimensional stability or both.

T6 solution treated, then artificially aged

Applies to products that are not cold worked after solution treatment or in which the effect of cold work flattening or straightening may not be recognized in mechanical property limits.

T7, solution heat treated and over aged/stabilized

Applies to products which are stabilized to carry them beyond the point of maximum strength to provide control of some special characteristics.

T8, solution heat treated, cold worked, then artificially aged

Applies to products that are cold worked to improve strength or for which the effect of cold work in flattening and straightening is recognized in mechanical property limits.

T9, solution heat treated, artificially aged, then cold worked

Applies to products, that are cold worked to improve strength.

T10, cooled from an elevated temperature shaping process, cold worked, then artificially aged

Applies to products that are cold worked to improve strength or for which the effect of cold work in flattening or straightening is recognized in mechanical property limits.

In all of the T-type temper definitions just described, solution heat treatment is achieved by:

- Heating cast or wrought shaped products to a suitable temperature
- Holding them at that temperature long enough to allow constituents to enter into solid solution
- Cooling them rapidly enough to hold the constituents in solution to take advantage of subsequent precipitation and the associated strengthening i.e., precipitation hardening [1].

2.3 Properties of Aluminum Alloys

Non-heat-treatable wrought aluminum alloys are the materials which get their strength from solid-solution or dispersion hardening or by strain hardening methods. They include 1XXX, 3XXX, 4XXX, 5XXX, and some 8XXX (Al-Li-Cu-Mg) alloys. Strength ranges with various classes of wrought alloys are given in Table 2.3.

Table 2.3: Strength Ranges of Various Wrought Aluminum Alloys [2].

Aluminum Association Series	Temper of alloy composition	Strengthening method	Tensile strength range (MPa)
1XXX	Al	Cold work	70-175
2XXX	Al-Cu-Mg (1-2.5%Cu)	Heat treat	170-310
2XXX	Al-Cu-Mg-Si (3-6% Cu)	Heat treat	380-520
3XXX	Al-Mn-Mg	Cold work	140-280
4XXX	Al-Si	Cold work (Alloy 4032 is heat treatable)	105-350
5XXX	Al-Mg (1-2.5% Mg)	Cold work	140-280
5XXX	Al-Mg-Mn (3-6% Mg)	Cold work	280-380
6XXX	Al-Mg-Si	Heat treat	150-380
7XXX	Al-Zn-Mg	Heat treat	380-520
7XXX	Al-Zn-Mg-Cu	Heat treat	520-620
8XXX	Al-Li-Cu-Mg	Heat treat	280-560

2.3.1 Mechanical properties

Although pure aluminum can be substantially strain hardened, a mere 1% alloying addition produces comparable tensile strength to that of fully hardened pure aluminum with much greater ductility in the alloy. The alloys can be substantially strain hardened to produce even greater strengths.

While strain hardening increases both tensile and yield strengths, the effect is more pronounced for the yield strength so that it approaches the tensile strength, and they are nearly equal in the fully hard temper. Ductility and workability are reduced as the material is strain hardened, and most alloys have limited formability in the fully hard tempers.

2.3.2 Mechanical properties at low temperature

Aluminum alloys represent a very important class of structural metals for subzero temperature applications and are used for structural parts operating at temperatures as low as -270°C . Below zero, most aluminum alloys show little change in properties. Yield and tensile strengths can increase, and elongation can decrease slightly. Impact strength remains approximately constant. Consequently, aluminum is a useful

material for many low temperature applications. The wrought alloys most often considered for low temperature service are alloys 1100, 2014, 2024, 2090, 2219, 3003, 5083, 5456, 6061, 7005, 7039, and 7075.

2.3.3 Mechanical properties at elevated temperatures

A limitation to the use of aluminum is its loss of strength at elevated temperatures. Figure 2.1. demonstrates this clearly for both heat treatable and non heat treatable alloys. The strength of the age hardenable alloys declines rapidly if they are exposed to elevated temperatures, due mainly to coarsening of the fine precipitates on which the alloys depend for their strength. Strength at temperatures above 150°C is improved mainly by solid solution strengthening or second phase hardening.

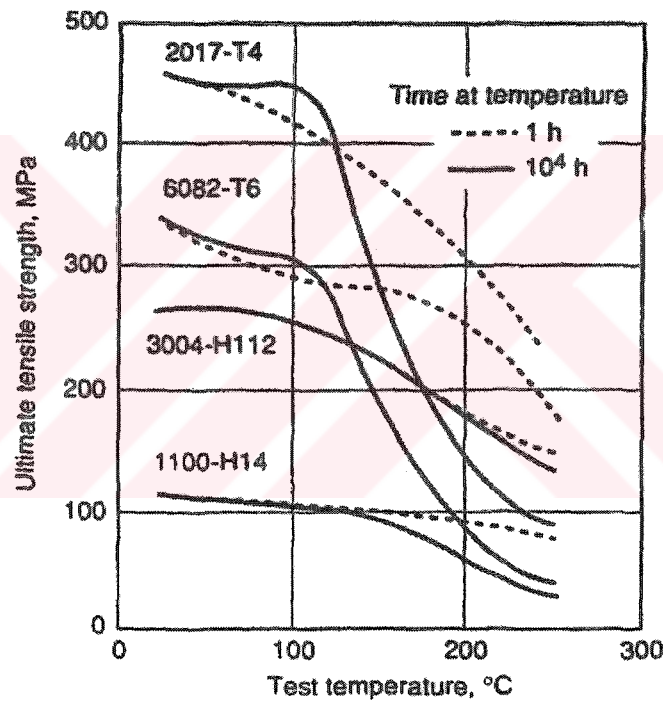


Figure 2.1: Elevated Temperature Properties of Various Aluminum Alloys [2].

2.3.4 Fracture toughness

Aluminum alloys chosen for fracture critical applications are based on Al-Cu (2XXX series, e.g., 2024 and 2124), Al-Mg-Si (6XXX series, e.g. 6061), Al-Zn-Mg (7XXX series, e.g., 7075, 7150, and 7475), and more recently, lithium containing alloys such as 8090 and 2091 series. High strength alloys with improved fracture toughness have

evolved through microstructure control obtained by increased purity, modified composition, and better fabrication and heat treatment practice.

2.4 Various Applications of Aluminum and Aluminum Alloys

Due to this unique combination of properties, the variety of applications of aluminum continues to increase. It is essential in our daily lives. We cannot fly, go by high speed train, high performance car or fast ferry without it. We cannot get heat and light into our homes and offices without it. We depend on it to preserve our food, our medicine and to provide electronic components for our computers.

i. Various Applications of 1XXX Series

1XXX, Pure Aluminum. The major characteristics of the 1XXX series are:

- Strain hardened
- High corrosion resistance, excellent formability, high electrical conductivity
- Easily joined by all methods
- Typical ultimate tensile strength range: 70 to 185 MPa

The primary uses of 1XXX series would be applications in which the combination of extremely high corrosion resistance and formability are required (e.g., foil and strip for packaging, chemical equipment, tank car or truck bodies, spun hollowware, and elaborate sheet metal work).

ii. Various Applications of 6XXX Series

6XXX, Aluminum-Magnesium-Silicon Alloys. The major characteristics of the 6XXX series are:

- Heat treatable
- Good extrudability, strength, corrosion resistance, machinability, weldability; formability
- Typical ultimate strength range: 125 to 400 MPa.

A unique feature is their great extrudability, making it possible to produce in single shapes that put the majority in single shapes that put the majority of the metal where

it will most efficiently carry the highest tensile and compressive stresses. This feature is a particularly important advantage for architectural and structural members where stiffness is critically important. Among specialty alloys in the series higher-strength alloy 6061 extrusions and plate forms find broad use in welded structural members such as truck and marine frames, railroad cars, and pipelines.

iii. Various Applications of 7XXX Series

7XXX, Aluminum-Zinc Alloys. The major characteristics of the 7XXX series are:

- Heat treatable
- Very high strength; good machinability
- Typical ultimate tensile strength range: 220 to 610 MPa

The 7XXX alloys are heat treatable and the widest application of the 7XXX alloys historically has been in the aircraft industry, where fracture-critical design concepts have provided the impetus for the high-toughness alloy development. There are several alloys in the series, notably 7150, 7075, 7175 and 7475; for these alloys, controlled impurity levels, particularly of iron and silicon, maximize the combination of strength and fracture toughness. As special applications, 7075 is used in stringers, keel beams, floor beams, in the ribs of wing construction, in horizontal and vertical stabilizers in aircrafts. 7175 is similar to alloy 7075 having superior toughness and tensile strength. It is widely used in landing gear beam and machined fittings in the aircraft industry.

3. STRENGTHENING OF ALUMINUM ALLOYS BY HEAT TREATMENT

3.1 Precipitation Hardening Mechanism

The phenomenon of precipitation hardening metals for increased strength has been occurring for many years. Applications of such metals have varied to military and industrial aircraft. Precipitation hardened metals offer high strength and high ductility. Unfortunately the wide variety of alloys and aging times has proven to make decisions difficult. Optimal temperatures and aging times are hard to pinpoint, as well as the right alloy to use. Due to their high strength and ductility, precipitation-hardened metals, especially aluminum alloys, dominate the parts industry for both aircraft and automobiles.

Heat treatment to increase strength of aluminum alloys is a three step process also shown in Figures 3.1. and 3.2.

- Solution heat treatment: Dissolution of soluble phases
- Quenching: Development of supersaturation
- Age hardening: Precipitation of solute atoms either at room temperature or elevated temperature

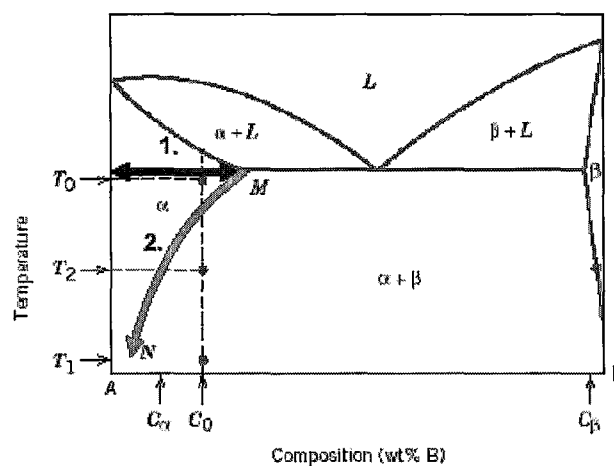


Figure 3.1: Appreciable Solid Solubility of One Alloying Alloy in the Other [6].

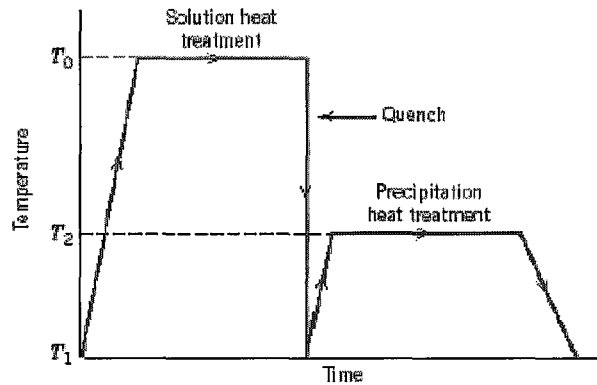


Figure 3.2: A Solubility Limit That Rapidly Decreases in Concentration of One Major Component with Temperature Reduction [6].

3.1.1 Solution heat treating

To take advantage of the precipitation hardening reaction, it is necessary first to produce a solid solution. The process by which this is accomplished is called solution heat treating, and its objective is to make into solid solution the maximum practical amounts of the soluble hardening elements in the alloy. The processes consist of soaking the alloy at a temperature sufficiently high and for a time long enough to achieve a nearly homogeneous solid solution [7].

Nominal commercial solution heat-treating temperature is determined by the composition limits of the alloy and an allowance for unintentional temperature variations. Although ranges normally listed allow variations of $\pm 6^\circ\text{C}$ ($\pm 10^\circ\text{F}$) from the nominal, some highly alloyed, controlled-toughness, high-strength alloys require that temperature be controlled within more restrictive limits. Broader ranges may be allowable for alloys with greater intervals of temperature between their solvus and eutectic melting points [7].

Care must be exercised to avoid exceeding the initial eutectic melting temperature. If appreciable eutectic melting occurs as a result of overheating, properties such as tensile strength, ductility, and fracture toughness may be degraded. Although maximum temperature must be restricted to avoid melting, the lower limit should, when possible, be above the temperature at which complete solution occurs (solvus). On the other side, when the temperatures attained by the parts or pieces being heat treated are appreciably below the normal range, solution incomplete, and strength somewhat lower than normal is expected. When heating conditions optimized,

solution heat treating temperatures would be about 490°C, 470°C and 530°C for 7075, 7175 and 6061 aluminum alloys respectively [7].

Solution-treating time depends on the thickness of the material shown in Fig. 3.1.

Table 3.1: Solution Treatment Times for Wrought Aluminum Alloys [7].

Thickness, mm	Soak time, minutes			
	Air furnace		Salt bath	
	minimum	maximum	minimum	maximum
≤0.41	20	25	10	15
0.51	20	30	10	20
0.64	25	35	15	25
0.81	25	35	15	30
1.02	30	40	20	30
1.27	30	40	20	30
1.35	30	40	20	35
1.80	35	45	25	35
2.03	35	45	25	35
2.29	35	45	25	45
2.54	40	55	30	45
3.18	40	55	30	45
4.06	50	60	35	45
4.57	50	60	35	45
6.35	55	65	35	45
6.35-12.7	65	75	45	55
For each additional 12.7 mm	+30	+30	+20	+20

3.1.2 Quenching

Quenching in many ways the most critical step in the sequence of heat-treating operations. The objective of quenching is to preserve the solid solution formed at the solution heat-treating temperature, by rapidly cooling to some lower temperature, usually near room temperature [7].

The quench rate must be fast in order to retain the solute atoms in the solution as well as to maintain a minimum number of vacant lattice sites to assist in promoting the low temperature diffusion required for zone formation.

The rapid quenching also improves resistance to corrosion and stress-corrosion cracking, creating the highest strengths attainable and also the best combinations of strength and toughness in the alloys. During quenching, the sample is rapidly cooled, usually in water at room temperature, which gives through to a new structure. The structure that is formed from the water quenching is a supersaturated solid solution

that is not in equilibrium. This happens because the atoms do not have time to diffuse to potential nucleation sites, so the precipitates do not form [8].

The resistance to stress corrosion cracking of certain copper free aluminum-zinc-magnesium alloys, however, is improved by slow quenching. Most frequently, parts are quenched by immersion in cold water or, in continuous heat treating of sheet, plate, or extrusions in primary fabricating mills, by progressive flooding or high velocity spraying with cold water [7].

The main requirement for avoidance of appreciable precipitation during quenching is that the volume, heat-absorption capacity, and the rate of flow of the quenching medium be such that little or no precipitation occurs during cooling. Any interruption of the quench that might allow reheating into a temperature range where rapid precipitation can occur must be prohibited [7]. For maximum dimensional stability, some forgings and castings are fan cooled or still-air cooled. As a broad generalization, the highest strengths attainable and the best combinations of strength and toughness are those associated with the most rapid quenching rates.

3.1.3 Age hardening

After solution treatment, the next step is age hardening process. There are two types of aging, natural and artificial. Natural aging can be accomplished by allowing the alloy to sit and cool on its own after the quenching. Artificial aging requires that the alloy be reheated again to quicken the precipitation process. The purpose of aging is to produce the finely dispersed precipitates, which is also the main objective of the precipitation hardening process. The fine precipitates in the alloy impede the movement of the dislocations during the deformation by forcing the dislocations to either cut through the precipitates or go around them.

In some alloys, sufficient precipitation occurs in a few days at room temperature to yield stable products with properties that are adequate for many applications. These alloys sometimes are precipitation heat treated to provide increased strength and hardness in wrought or cast products. Other alloys with slow precipitation reactions at room temperature are always precipitation heat treated before being used [7].

i. Natural Aging: The more highly alloyed members of the 6xxx wrought series, the copper containing alloys of the 7xxx group are almost always solution heat treated

and quenched. In contrast to the aluminum alloys reaching the stable condition in a few days, the 6xxx and 7xxx alloys are considerably less stable at room temperature and continue to exhibit significant changes in mechanical properties for many years.

Aging characteristics vary from alloy to alloy with respect to both time to initial change in mechanical properties and rate of change, but aging effects are always lessened by reductions in aging temperature.

ii. Artificial Aging: Artificial aging generally are low-temperature, long-term processes. Temperatures range from 115 to 190°C; times vary from 5 to 48 h. For example, the artificial aging temperatures times are 120°C and 24h for 7075 and 7175, 160°C and 18h for 6061 respectively [7].

Precipitation heat treatment following solution heat treatment and quenching produces T6- and T7-type tempers. Alloys in T6-tempers generally have the highest strengths practical without sacrifice of the minimum levels of other properties and characteristics found by experience to be satisfactory and useful for engineering applications. Alloys in T7 tempers are overaged, which means that some degree of strength has been sacrificed to improve one or more other characteristics [7].

3.1.4 Precipitation behaviour during age hardening

The first step in the process of aging is the formation of GP Zones. GP Zones are solute atoms that have diffused into coherent clusters. Coherent clusters are clusters of the solute atoms that distort the crystal structure, but are still connected to the rest of the crystal structure [8]. This causes a great deal of strain because of mismatch in size between the solvent and solute atoms [7]. As more solute diffuses to the clusters, eventually the matrix can no longer accommodate the matrix mismatch. A semi-coherent precipitate forms. Finally, after the semi-coherent precipitate grows to a large enough size, the matrix can no longer support crystallographic mismatch, and the equilibrium precipitate forms as incoherent clusters [9]. Incoherent structures actually weaken the alloys because they do not impede dislocation movement the way coherent clusters do, instead they allow dislocations to slide by. The GP Zones contain these solute clusters that stop the procession of dislocations, thus strengthening the material [8]. The presence of the precipitate particles, and even more importantly the strain fields in the matrix surrounding the coherent particles,

provide higher strength by obstructing and retarding the movement of dislocations [7].

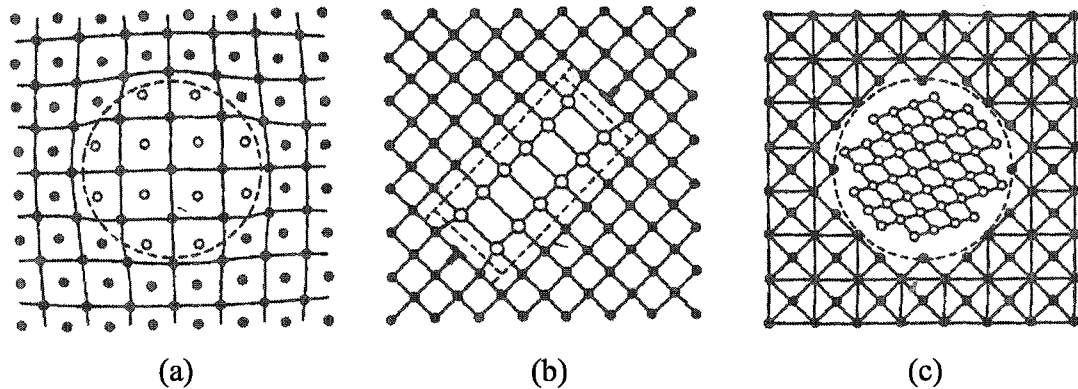


Figure 3.3: (a) Fully Coherent Precipitate Showing Coherency Strains in the Matrix. (b) Partially Coherent Precipitate Showing Interface Dislocations. (c) Non-coherent Precipitate [10].

i. 6xxx Series: The general sequence of precipitation in the Al-Mg-Si system is thus represented by:

Supersaturated solid solution $\rightarrow$ GP zones (needles) $\rightarrow \beta'$ (Mg₂Si) $\rightarrow \beta$ (Mg₂Si)

If an Al-Mg-Si alloy containing 1.3 wt% Mg₂Si is solution-heat treated at 565 °C, water –quenched, and aged at 160°C, GP zones form which are believed to have needle like shape. When the maximum strength is reached during aging at 160 °C for 24h, a high density of β' precipitate is formed, with some short needles being observed. Reheating the fully hardened Al-Mg-Si alloy 15 min at 275°C causes a coarsening of the β' needles [11].

ii. 7xxx Series: The precipitation sequence upon aging the supersaturated solid solution of Al-Zn-Mg-Cu alloys is generally recognized to be

Supersaturated solid solution $\rightarrow$ GP zones $\rightarrow \eta'$ (MgZn₂) $\rightarrow \eta$ (MgZn₂)

The GP zones are coherent with the matrix and have a spherical shape. During zone formation, the copper in Al-Zn-Mg-Cu alloys appears to be uniformly distributed. Copper in the GP zones does, however, increase their stability, as it enables the zones to exist at higher temperatures. Microstructures of 7075 alloy is the fully age-hardened T651 condition, the GP zones are $\leq 5\text{\AA}$ with some η' (~150Å) also present. Large, dark particles are chromium rich precipitates which are found in many Al-Zn-Mg-Cu alloys. After overaging the T651 material at 170 °C for 9h to produce the T7351 temper, the microstructure consists of η' (100 to 300 Å) and η (400 to 800Å).

Overaging and coarsening of the precipitates result in lower strengths. For example, the 7075-T651 material has an ultimate tensile strength of 76.7 ksi and a yield of 66.4 ksi, while the 7075-T7351 with $\eta + \eta'$ precipitates has an ultimate tensile strength of 63.7 ksi and a yield strength of 54.3 ksi [11].

3.2 Retrogression and Reaging Process

The concept of retrogression and re-aging (RRA) was first developed by Cina and his colleagues at the Israel Aircraft Industries in 1974 [12] which consists of two steps.

- Retrogression of the 7xxx-T6 material at an intermediate temperature between the aging temperature and the solutioning temperature for a short time
- Re-aging of the retrogressed alloy with that of original T6 temper.

They experimented on 7075-T6, using a silicone oil bath, and they revealed that during retrogression the material's hardness/strength would first fall to a minimum before increasing again to a secondary peak, while continued treatment would cause a further decrease in strength. Thus there appeared to be three distinct stages to the retrogression process as illustrated in Figure 3.4. Cina et al. claimed that the optimum RRA treatment was to process the T6 material to the minimum strength, Figure 3.4., during retrogression, followed by re-aging using the original T6 aging treatment.

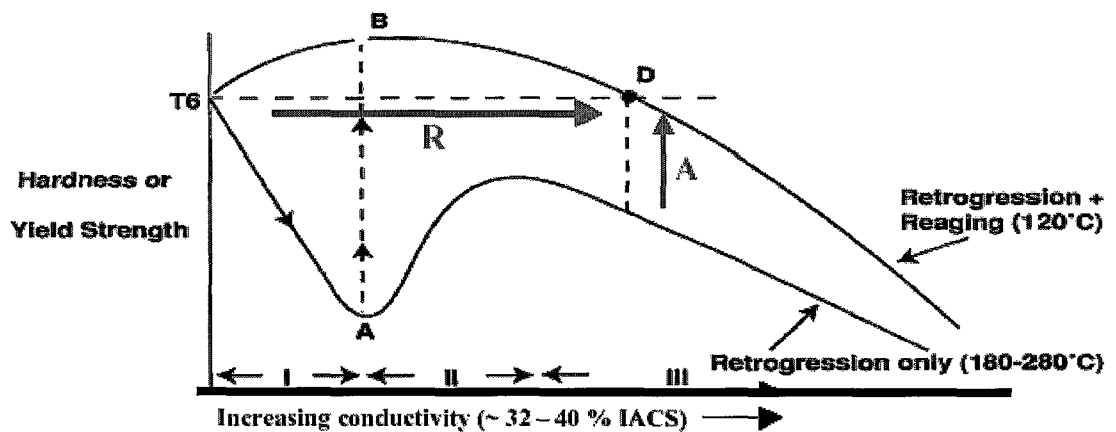


Figure 3.4: Schematic Representation of the Change in Strength and Yield Stress During Retrogression and Re-aging [16].

The retrogression and re-aging behaviour of various 7xxx series aluminum alloys, such as 7010, 7050, 7150, and 7475 depend on physical, mechanical, and corrosion properties. The studies that were performed recently proved that the time and temperature of retrogression and re-aging processes had a significant influence on the properties. [17-24].

At the beginning, the RRA process involved such short retrogression times, typically 1 to 30 sec. at temperatures of 220-280°C, that its application would only be possible for thin sections. In the following studies, low retrogression temperatures ranging from 220°C to 160°C, allowed improved properties achieved through thicker sections. Retrogression at these lower temperatures typically took 6 minutes to 3 hours. Alcoa filed a series of patents on processes similar to RRA, which were used primarily for 7x50 aluminum alloys [13-15]. In the studies, the retrogression temperatures were 170 °C and 182°C followed by processing times 7.3 h and 5 h respectively.

The microstructural changes during RRA treatment, focusing on alloy's mechanical properties such as hardness and strength related with electrical conductivity were examined by DTA (Differential Thermal Analysis) [25], DSC (Differential Scanning Calorimetry) [26, 27], SAED (Selected Area Electron Diffraction) [26], XRD (X-ray Diffraction) [28-29], TEM (Transmission Electron Microscopy) [17-18, 26-32], and SAXS (Small Angle X-Ray Scattering) [33], investigations.

Many extensive studies have been carried out on the kinetics, microstructure and structure-property relationships over a wide range processing conditions and alloy compositions.

3.2.1 Precipitation behaviour during retrogression

The precipitation behaviour of the retrogressed materials depends on many factors, especially retrogression time and temperature, the microstructure of the alloy, the manufacturing history of the alloy and so on.

Numerous η' precipitates and GP zones were observed after aging by TEM studies. Kanno retrogressed the 7075 alloy (~2 min.) at temperatures 200 to 250°C. In the first stage of the retrogression, dissolution of fine η' precipitates occur as well as GP zones. And a number of the η' precipitates remain in the matrix to grow [32]. During

the GP zone dissolution, the growth of the pre-existing η' and the nucleation of new η' particles on the remaining GP zones could occur simultaneously. However, the rapid decrease in strength would suggest that these reactions do not occur at the same rate. The TEM investigation of the first stage of retrogression showed there was no noticeable change in size and distribution of η' precipitates from the T6 condition until the minimum of the retrogression curve was reached at 200°C. This is an agreement with the suggestion that GP zone reversion and not η' growth dominates during this stage of the process. In addition the dissolution of η' do not dissolve below 250°C, which implies the dissolution of the GP zones is responsible for the initial loss of strength [18]. In the extent of other studies [25,26,32], it has been indicated that retrogression is responsible for the progressive dissolution of GP zones and η' phase. Undissolved η' precipitates remain to grow during the process. The retrogression temperature is the main property controlling factor since higher retrogression temperature means increasing the dissolution degree.

When Park and Ardell [30] retrogressed the 7075-T651 alloy at 240 °C, they detected that in the early stage of the retrogression, a significant fraction, $\sim 1/3$ of the total particles, either partially or completely dissolves and the dissolving particles are mainly η' precipitates instead of GP zones. In TEM analysis, they also observed the phase transformation of η' to η precipitates. According to their TEM results, the concentration of η particles significantly increases while η' the concentration of η' particles decreases. These observations were interpreted as an indication of more stable large η' particles transform to η particles. They also supported their findings from the idea that the particles of an intermediate phase transform to a more stable phase if their sizes exceed a critical size at higher temperatures above their solvus temperature. Also the results of the structure indicate that some coarsening of the η particles has already occurred after only a few seconds of the retrogression treatment. It is concluded that η' particles below critical size become unstable on up-quenching to the retrogression temperature. When solid solution resulting from dissolution of these particles exceeds the solubility limit of the stable phase at the retrogression temperature, the solid solution can become supersaturated with respect to the stable phase driving the nucleation of the equilibrium phase of η' at a reasonable rate. The reaction, dissolution of η' and coarsening of the η precipitates, is thought to be the

main mechanism during retrogression. The effect of both total concentration and reaction rate of particles on time is represented in Figure 3.5.

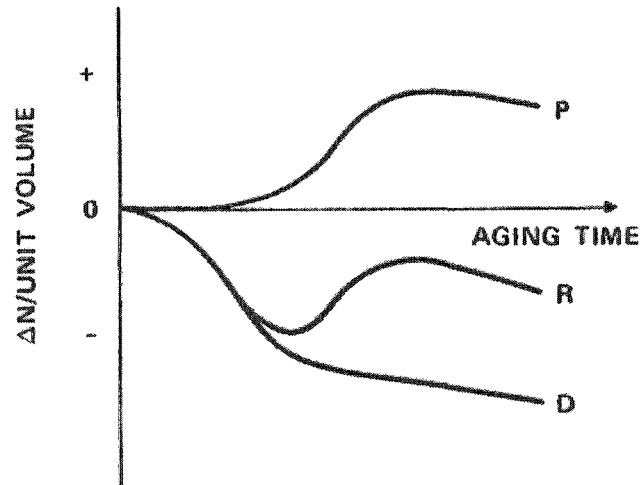


Figure 3.5: The Variation of the Concentration Particles ($\delta N/\text{Unit Volume}$) as a Function of Aging Time During Retrogression Process [30]. P: Precipitation of Particles, D: Dissolution of Particles, R: Overall Precipitates (P+D)

The time corresponding to the minimum hardness in the retrogression curve was originally thought to be an optimum time for the retrogression treatment to get the optimum combination of strength and SCC resistance [12]. But Wallace has demonstrated that the minimum has no significance. Nevertheless, the minimum happens to correspond to the time at which maximum dissolution of small η' particles occurs [34]. At this point the second stage of retrogression process is taken into consideration. It can be seen in Figure 3.5 that, as the retrogression time increase the precipitation rate increases and the dissolution rate decreases, which results in an increase in the overall particle concentration in the second stage of the process [30]. In the extent of other studies it has been told that an increase in volume fraction of η' and η precipitates from the minimum of the retrogression curve to the maximum. And the recovery of strength was attributed to this increase in volume fraction of precipitates mostly an increase in volume fraction of η' . The coarsening of the particles is also observed during the increase in strength of retrogression process [18,33].

In the third stage of the retrogression, the drop in hardness with increasing retrogression time is attributed to the coarsening of the particles; overaging

[18,30,33]. According to TEM analysis, it is observed that the retrogression process increased the size of η [17] and the distance between grain boundary precipitates [21] when it is compared to the T6 temper situation of the alloy. The distance between the grain boundary precipitates become greater because of the intake of solute of the fixed total solute concentration required for the precipitate to grow. As retrogression proceeds, the precipitate size increases. Although precipitate-free zone widths increased about 50% according to T6 temper, it did not seem to change with time at the retrogression stage [21].

According to the studies of Hall and Martin [21], the electrical conductivity increases with increasing retrogression times since there is quite a substantial decrease in the matrix solute content for the precipitation of particles.

To sum up, there is an absolute result arising from the studies explained above. That's the coarsening of the grain boundary precipitates during retrogression. Because of this, some investigators take the RRA process as grain boundary precipitate coarsening treatment [17].

3.2.2 Precipitation behaviour during reaging

Re-aging promotes a very dense precipitation inside the grains whilst the grain boundary microstructure do not change so much.

Dahn and the others [18] observed that both GP zones and η' precipitates nucleate and grow but the nucleation and the growth of η' precipitates is dominant over the nucleation and growth of GP zones. Some other researchers [26,30] declared that re-aging promotes the re-precipitation of η' while its pre-existent particles grow and transform to η or just coarsen without converting to the stable phase. It is believed that the high strength of RRA structure, which is comparable to that of the T6 structure, is partly due to the presence of many fine particles. According to the results, the concentration of η and η' precipitates are higher than the concentration of the precipitates in retrogressed and T6 conditions. The particle sizes do not exhibit a significant change after retrogression. Thus, RRA process heightens the η' concentration without affecting the particle size. As it is evident, the particles' coarsening occurs during RRA. The particle size of the grain boundary precipitates depend on the immersion time during retrogression process. When 7075 alloy was

retrogressed at 240°C, it was seen that the grain boundary size is significantly larger than that in T6 structure but comparable with T7 structure. Also it was noticed that the size increases rapidly during the initial period of the retrogression treatment [30]. The RRA microstructure is similar to that for the T6 temper, but it is a little coarser and denser inside the grains: contrarily, the grain boundary precipitation is quite different, and very similar to that of the overaged T7 temper [26]. But, among the materials, both retrogressed for a short time [17] and heat treated for a long time (T7) [35], the η size is much smaller than those of the T73 temper. However, the η' and the overall precipitate concentration in the RRA structure is significantly higher than the structure of T73 condition [30].

It is mentioned that, higher retrogression temperatures increase the dissolution degree and promote the formation of more stable precipitates on the re-aging [26].

It is noted that RRA causes heterogeneous precipitation on suitable nucleation sites which are E ($\text{Al}_{18}\text{Cr}_2\text{Mg}_3$) phase inside the grain of 7075-Cr alloy [32].

After re-aging, conductivity increases related to the retrogression time, being higher than the retrogressed values (Figure 3.6). The separation between the curves is due to the loss of solute in order to form precipitates during re-aging. At the higher temperatures there is a greater difference between the equilibrium matrix solute concentrations, hence a greater separation of the curves [21].

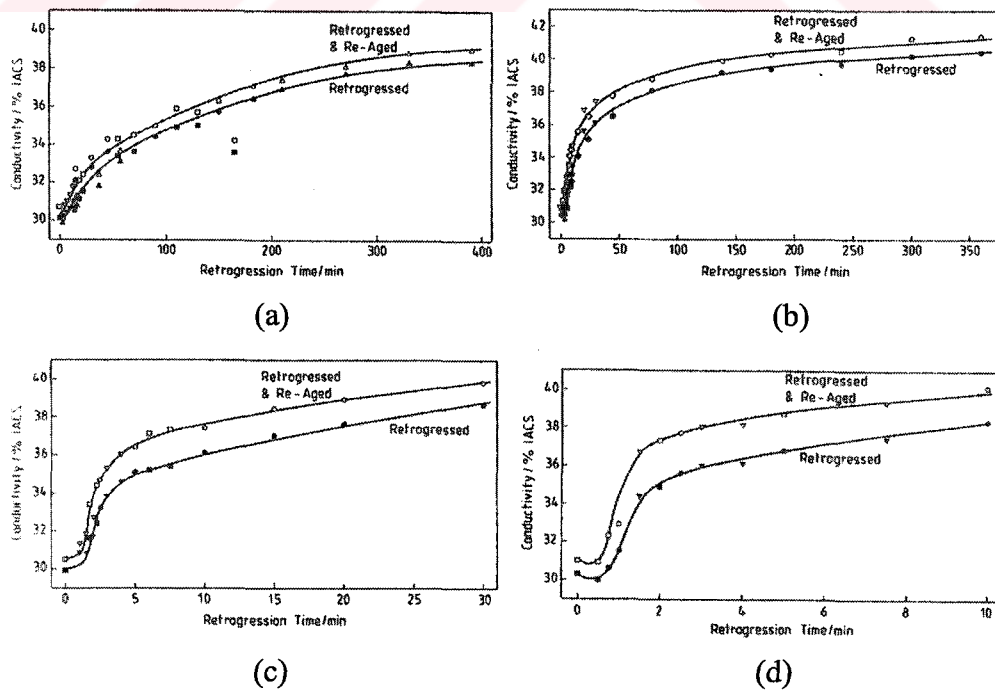


Figure 3.6: Conductivity Results During Retrogression and Re-aging (a) 180 °C (b) 200 °C (c) 220 °C (d) 240 °C [21].

4. CORROSION BEHAVIOUR OF ALUMINUM ALLOYS

Corrosion is the destructive result of chemical reaction between a metal or metal alloy and its environment. Metal atoms in nature are present in chemical compounds. The same amounts of energy needed to extract metals from their minerals are emitted during the chemical reactions that produce corrosion. Corrosion returns the metal to its combined state in chemical compounds that are similar or even identical to the minerals from which the metals extracted [36].

Aluminum has excellent resistance to corrosion and gives years of maintenance-free service in natural atmospheres, fresh waters, seawater, many soils and chemicals, and most foods. The good performance of aluminium in corrosive environments is due to the passivity produced by a protective oxide film. Unless exposed to some substance or condition that destroys the protective oxide coating, the metal remains fully protected against corrosion [2].

The chief corrosion mechanisms which occur in heat treatable aluminium alloys (6xxx and 7xxx series) are; intergranular corrosion, stress corrosion cracking and exfoliation corrosion [2,37].

4.1 Types of Corrosion

4.1.1 Intergranular corrosion

In intergranular corrosion (IGC), the grain boundaries of the corroded metal become anodic. The bulk material between the grain boundaries is not affected and therefore is cathodic. Corrosion products and, occasionally, cracking are produced on the surface of materials that corrode intergranularly. Intergranular corrosion (IGC), also referred to as intercrystalline corrosion, is selective corrosion of grain boundaries or closely adjacent regions without appreciable attack of the grains or crystals themselves. Intergranular corrosion occurs to some extent in most heat treatable,

high-strength products (7xxx alloys) and is often related to zinc depleted regions or to anodic precipitates at the grain boundary region.

Because corrosion is limited to the intermediate grain boundary region, IGC is difficult to detect without the aid of a microscope. Intergranular corrosion penetrates more quickly than pitting corrosion but reaches a self-limiting depth due to limited transport oxygen and corroding species down to the narrow corrosion path. When the depth of penetration ceases, IGC spreads laterally over the entire surface [2,37]. Various cross-sectional shapes of pits of the intergranularly corroded aluminium alloys is shown in figure 4.1.

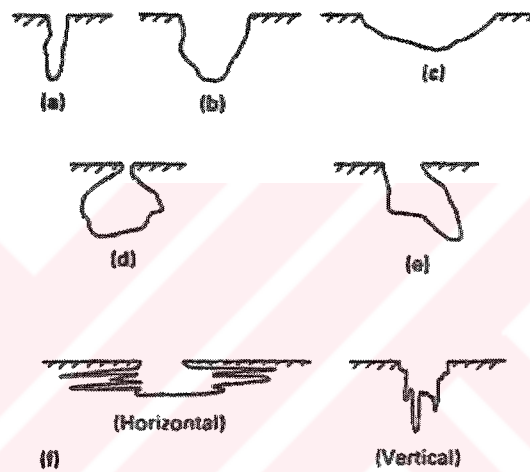


Figure 4.1: Variations in the Cross-Sectional Shapes of Pits [2]. (a) Narrow and Deep (b) Elliptical (c) Wide and Shallow (d) Subsurface (e) Undercutting (f) Shapes Determined by Microstructural Orientation.

i. IGC of 6xxx (Al-Mg-Si) Alloys: The 6xxx series alloys usually exhibit some susceptibility to IGC. With a balanced magnesium-silicon composition that results in the formation of a Mg_2Si constituent, intergranular attack is minor and less than that observed with aluminium-zinc –magnesium-copper (7xxx) alloys. When the 6xxx alloy contains an excessive amount of silicon (more than that needed to form Mg_2Si), intergranular corrosion increases because of the strong cathodic nature of the insoluble silicon constituent. Intergranular corrosion is most prevalent in the peak strength T6 tempers, especially 6xxx-T6 products exposed to atmospheres or harsh chemical environments [36].

ii. IGC of 7xxx (Al-Zn-Mg-Cu) Alloys : Precipitation occurs first at grain boundaries and causes a depleted zone, which creates a galvanic cell between the narrow

depleted zone and zinc/magnesium-rich grains. Addition of zinc and dissolution of the MgZn_2 phase both shift the potential of these alloys in the anodic direction, so that a potential difference of as much as 0.24 V could exist pure aluminum and alloys [2].

4.1.2 Exfoliation corrosion

Exfoliation corrosion, sometimes referred to as layer, stratified, or lamellar corrosion, is a form of corrosion resulting from a relatively rapid lateral attack along electrochemically anodic strata parallel to the metal surface. In the more familiar occurrences of exfoliation corrosion, attack progresses along grain boundaries. Exfoliation is characterized by leafing or splitting off alternate layers of thin, relatively uncorroded metal and thicker layers of corrosion product that are more bulky than the metal from which they came. The layers of corrosion products cause the metal to swell. Exfoliation usually proceeds inward laterally from a shared edge, rather than a rolled surface. In mild cases, it takes the form of blisters that resemble volcanoes, with corrosion product welling up in the center. In this case, pits occur first and proceed inward until the susceptible layer is encountered. The attack then changes to lateral penetration with generation of bulky corrosion products that cause the blisters to develop. Exfoliation is not accelerated by stress and does not lead to SCC. Exfoliation is a very deleterious form of corrosion because the splitting off of uncorroded metal rapidly reduces load-carrying ability. The splitting action continually exposes fresh free metal, so the rate of the corrosion is not self-limiting. Exfoliation generally proceeds at a nearly linear rate [2].

4.1.3 Erosion corrosion

Erosion-corrosion is a general term that refers to any conjoint action between corrosion and erosion in the presence of a corrosive substance. Erosion-corrosion can also be subdivided into abrasive erosion-corrosion and liquid erosion-corrosion. Abrasive erosion-corrosion takes place when abrasive solid particles entrained in a liquid impinge on a metal surface, causing the removal of the oxide film. Abrasive erosion-corrosion can also take place when hot gases with entrained solid particles impinge on metal surfaces. Virtually anything that is exposed to a moving liquid, with or without abrasive particles, is susceptible to erosion corrosion. Affected metal

surfaces often contain grooves or wavelike marks that indicate a pattern of directional attack. Soft metals, such as aluminium alloys, are often prone to erosion-corrosion and are not usually selected for applications where erosion-corrosion is a significant factor [2].

4.1.4 Cavitation erosion

Cavitation erosion occurs on metal surfaces in contact with a liquid. Pressure differentials in the fluid generate gas or vapour bubbles (cavities) in the fluid. When these bubbles or cavities encounter a high pressure zone, they collapse and cause explosive shocks to the surface. These surface shocks cause localized deformation and pitting. Cavitation pits eventually link up and cause a general roughening of the surface and material removal (i.e. erosion). It should be noted that cavitation erosion is primarily a mechanical process. The mechanical loading of the surface is caused by the violent collapse of cavities at or near the surface. These collapses produce liquid micro jets that are directed toward the surface. The repeated loading results in erosion. Because cavitation always takes place in a liquid medium, there is always the possibility of an interaction between mechanical and corrosion processes, which can produce diverse and complex effects on the materials. The interaction can be synergistic and can lead to increased damage [2].

4.1.5 Pitting corrosion

Pitting corrosion is the most common corrosion attack on aluminium alloy products. Pits form at localized discontinuities in the oxide film on aluminium exposed to atmosphere, fresh or salt water, or other neutral electrolytes. Since, in highly acidic or alkaline solutions, the oxide film is usually unstable, pitting occurs only in a pH range of about 4.5 to 9.0. The pits can be minute and concentrated, or they can be widely scattered varied in size depending upon alloy composition, oxide film quality, and the nature of the corrodent. Pitting can be locally accelerated by crevices and contact with dissimilar metals. While the shape of the pits in aluminium can vary from very shallow, saucer-like depressions to cylindrical holes, the mouth is usually more or less round, and the pit cavity is roughly hemispherical. This distinguishes pitting from intergranular corrosion, in which attack is confined to subsurface tunnels along grain boundaries, usually visible only on metallographic examination of cross

section. Intergranular corrosion can occur along with pitting, in which case intergranular fissures advance into the metal laterally and inwardly from the pit cavity.

The resistance of aluminum to pitting depends significantly on its purity; the purest metal is the most resistant with the following alloys in decreasing order of resistance:

- 1xxx pure aluminum grades
- 5xxx alloys
- 3xxx alloys
- 6xxx alloys
- 7xxx alloys
- 2xxx alloys

Although the 6xxx series alloys are susceptible to pitting corrosion, resistance to pitting decreases as the copper and iron content increases, and the effect is synergistic. The 7xxx series alloys (particularly those containing higher copper contents) are almost the least resistant to pitting corrosion. In sheet form, these high-strength alloys are normally clad to protect against pitting. The copper-free 7xxx alloys, with or without manganese, exhibit the best resistance to pitting of the high-strength alloys [2].

4.1.6 Stress corrosion cracking

Stress corrosion (SCC) is an interaction of sustained tension stress and corrosive attack causing cracking that can result in premature brittle failure of a ductile material. It is generally agreed that SCC requires three conditions. These are:

- The alloy must be susceptible
- There must be a corrosive environment
- There must be a tensile stress

In aluminum alloys, stress corrosion can be promoted by a film of moisture on the surface of metal exposed to the atmosphere or by contaminants such as chlorides. Only a prolonged surface tension stress will cause SCC, and sustained compression stresses actually prevent it. The tensile stresses required to cause SCC are small,

usually below the macroscopic yield stress. These stresses can be externally applied, but residual stresses often cause SCC failures [2,38].

There are three main theories about SCC phenomenon. These are:

- Anodic dissolution
- Hydrogen induced cracking
- Passive film

as illustrated in figure 4.2.

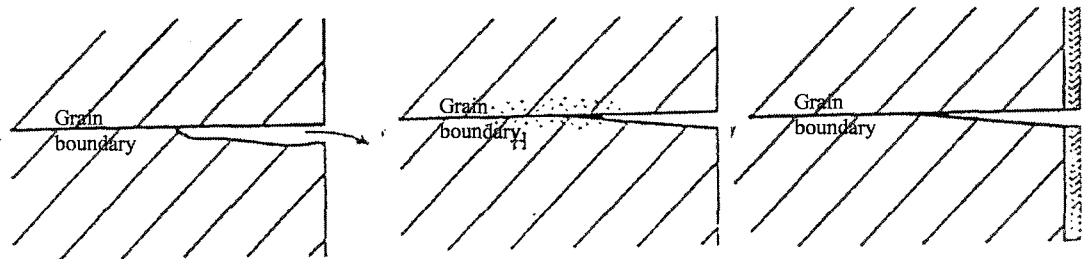


Figure 4.2: Schematic Representation of the Theories of SCC for Aluminum alloys. (a) Anodic dissolution (b) Hydrogen Induced Cracking (c) Passive film [38].

According to anodic dissolution theory, this requires a condition along grain boundaries that makes them anodic to the rest of the microstructure so that corrosion propagates selectively along them. Such a condition is produced by localized decomposition of solid solution, with a high degree of continuity of decomposition products, along the grain boundaries. The most anodic regions can be either the boundaries themselves (most commonly, the precipitate formed in them) or regions adjoining the boundaries that have been depleted of solute. In many cases, susceptibility to SCC of an aluminum alloy cannot be predicted reliably examining its microstructure. Many observations have been made of the progressive changes in dislocation network, precipitation pattern, and other microstructural features that occur as an alloy is treated to improve its resistance to SCC, but these changes have not been correlated quantitatively with susceptibility [2].

The tendency to brittle fracture and the sensitivity to SCC are explained by the diffusion of hydrogen into the grain boundaries under tensile stress and reducing the cohesive strength of the grain boundaries. But SCC of aluminium alloys will not occur in dry hydrogen but rather only in moist hydrogen or in water vapour. According to the hydrogen induced cracking theory, the critical factor is the

solubility of hydrogen in the grain boundaries. Hydrogen solubility depends on the tensile stress normal to the grain boundaries, and the concentration of zinc in solid solution. The tensile stress normal to the grain boundary effectively opens up the lattice so as to allow more hydrogen to diffuse inward. There is also a linear relation between the concentration of Zn in solid solution and the time to failure (the lower the Zn content, the longer is the lifetime). The hydrogen embrittlement resistance is related to the ability of the microstructure to trap hydrogen in the form of gas bubbles or to prevent hydrogen entry [38].

According to passive film theory, the brittle rupture occurs because the passive film breaks intergranularly [38].

4.2 The Influence of RRA on Corrosion Behaviour

In the 1980s, Alcoa developed a patented aging process designed to overcome the commercial production impracticalities of RRA. The new temper became known as T77 and the details of the process are proprietary [13-15]. Fig. 4.3 demonstrates the higher combination of strength and corrosion resistance made possible via implementation with 7xxx series alloys, like 7050, 7150 and 7055.

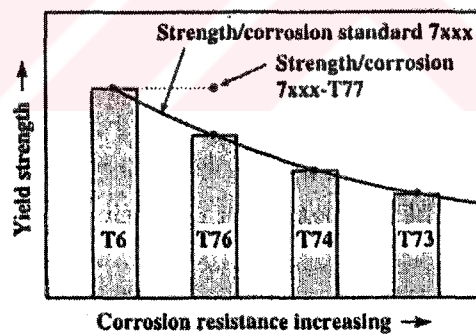


Figure 4.3: Improvement in Strength/Corrosion Combination Due to the T77 Tempers [37].

Hall and Martin [21] retrogressed and re-aged 7150 for a temperature range 180-240°C, they achieved a transition region from poor to high SCC resistance. At each retrogression temperatures, when the RRA material exhibits acceptable conductivity (conductivity > 36 to 38% IACS) for SCC resistance, they observed that the hardness lie above that of conventionally heat treated material. The retrogression time, in order to obtain the sufficient conductivity interval for SCC resistance decreases for

increasing temperature. As an example, at 180°C the conductivity of 36-38% IACS is reached within 140-240 minutes although the time varies only 1-3 minutes at 240°C.

Ural [20] retrogressed 7075-T6 at 200°C until the longest retrogression period by which its strength level is not allowed to decrease below the T6 level and then re-aged. According to the experimental results, he saw that the stress-corrosion cracking resistance is equivalent to the T73 temper together with T6 strength level. He claimed that stress corrosion cracking resistance is improved by increasing retrogression time. He reported the optimum retrogression time as 75 min for 200°C.

Aran [19] examined 7075 alloy at 200°C for retrogression times in between 30s and 12 min. He observed that intergranular corrosion susceptibility varies with retrogression time and dramatically decreases at longer retrogression times. After reaging the maximum hardness was obtained at the minimum point of the retrogression curve which yielded the worst intergranular corrosion resistance. It is concluded that retrogression time has significant importance to achieve the optimum combination of strength and corrosion which is 7 min for RRA process at 200°C for 7075 alloy. This RRA condition provides corrosion resistance similar to that of T73.

Tsai and Chuang [19] examined the influence of the matrix and grain boundary precipitate sizes on the atmospheric SCC resistance of the 7475 alloy. When grain boundary precipitates are larger than a critical size the matrix precipitates become important to affect the SCC resistance. On the other hand, if grain boundary precipitates are smaller than a critical size, matrix precipitates lose their importance. After RRA tempered treatment, the atmospheric SCC resistance of the 7475 aluminum alloy is comparable to that T73 condition while not sacrificing the strength of the T6 temper. This observation can be attributed to the larger sizes of both matrix and grain boundary precipitates formed during RRA temper.

Puiggali and others [40] investigated the susceptibility to stress corrosion cracking of 7010 alloy of different tempers (T351, T651 and T7451). They observed that the prolonged ageing results in a decrease in susceptibility to SCC. The prolonged ageing on susceptibility to SCC may be attributed to the change in the volume and the distribution of MgZn₂ precipitates which are small in T351 underaged alloy and the big in T7451 overaged alloy.

5. WEAR BEHAVIOUR OF ALUMINUM ALLOYS

5.1 Definition of Friction

The force known as friction may be defined as the resistance encountered by one body in moving over another. This broad definition embraces two important classes of relative motion: sliding and rolling. The distinction between rolling and sliding friction is useful, but the two are not mutually exclusive, and even apparently 'pure' rolling nearly always involves some sliding [41].

In both ideal rolling and sliding, as illustrated in fig. 5.1., a tangential force F is needed to move the upper body over the stationary counterface.

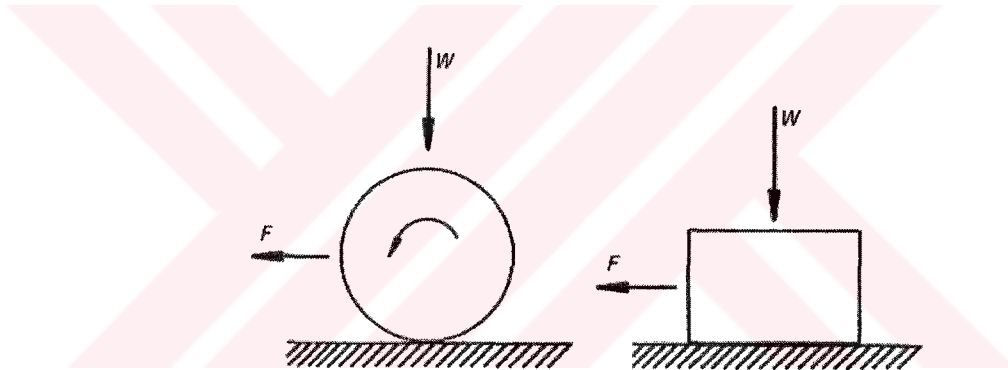


Figure 5.1: A Frictional Force, F , is Needed to Cause Motion by (a) Rolling or (b) Sliding [41].

The ratio between this frictional force and the normal load W is known as the coefficient of friction, and is usually denoted by the symbol μ :

$$\mu = F/W \quad (5.1)$$

The magnitude of frictional force is conveniently described by the value of coefficient of friction, which can vary over a wide range from 0.001 in a slightly loaded rolling bearing greater than 10 for clean metals sliding against themselves in vacuum. For most common materials sliding in air, however, the value of μ lies in narrower range from about 0.1 to 1 [41].

5.2.1 The laws of friction

Under some conditions of sliding, μ for a given pair of materials and fixed conditions (or absence) of lubrication may be almost constant. This observation led to the formulation of two empirical Laws of Sliding Friction, often called after Amontons who discovered them in 1699; Leonardo da Vinci, however, had been the first to describe them 200 years earlier. The laws of friction may be stated as follows:

- (1) the friction force is proportional to the normal loads;
- (2) the friction force is independent of the apparent areas of contact
- (3) the friction force is independent of the sliding velocity.

These three Laws of Friction are varying reliability, but except in some important cases they do provide useful summaries of empirical observations. The first Law indicates that the coefficient of friction, μ , is independent of the normal load.

The Second Law of Friction has not been so widely explored as the first, but it nevertheless well attested for most materials, with the exception of polymers.

The third Law of Friction is rather less well founded than the first two. It is a matter of common observation that the frictional force needed to initiate sliding is usually greater than that necessary to maintain it, and hence that the coefficient of static friction (μ_s) is greater than the coefficient of dynamic friction (μ_d). But once sliding is established, μ_d is found for many systems to be nearly independent of sliding velocity over quite a wide range, although at high speeds, of the orders of tens or hundreds of metres per second for metals, μ_d falls with increasing velocity [41].

5.2 Sliding Wear

The wear that occurs when two solid surfaces slide over each other is called sliding wear. In most practical applications sliding surfaces are lubricated in some way, and the wear that occurs is then termed lubricated sliding wear. In some engineering applications, however, and in many laboratory investigations, surfaces slide in air without a lubricant. The resulting wear is then often called dry sliding wear, although it usually takes place in ambient air of appreciable humidity [41].

A simple model for wear suggests that the amount of material removed from a sliding body should depend on the distance slid and on the nominal pressure (normal

load divided by the nominal contact area) over the contact region. It is wise to measure and record the friction force continuously during a wear test. A continuous friction record not only provides numerical values for μ , but also allows changes in sliding behaviour to be monitored. Wear under sliding conditions depends on the distance slid, but also to some extent on both the sliding velocity and the duration of the test, independently. The sliding velocity affects the rate of frictional force energy dissipation, and hence the temperature at the interface [41].

Contact stresses, thermal conditions, sliding speeds and chemical environment are all vital ingredients in any wear test. It is also sensible to ensure, by measurement of friction and by close examination of the worn surfaces and wear debris after test, that the mechanism of wear is the same in the test as in the service application.

5.3 Corrosive Wear

Corrosive wear is a general term relating to any form of wear dependent on a chemical or corrosive process. If a metal is corroded to produce a film on its surface while it is simultaneously subjected to a sliding contact then one of the four following processes may occur [42]:

- a durable lubricating film which inhibits both corrosion and wear may be formed;
- a weak film which has a short life-time under sliding contact may be produced and a high rate of wear may occur due to regular formation and destruction of the films. The friction coefficient may or may not be low in this instance;
- the protective surface films may be worn and a galvanic coupling between the remaining films and the underlying substrate may result in rapid corrosion of the worn area on the surface;
- the corrosive and wear processes may act independently to cause a material loss which is simply the sum of these two processes added together.

The hypothetical models of corrosive wear are illustrated schematically in Figure 5.2.

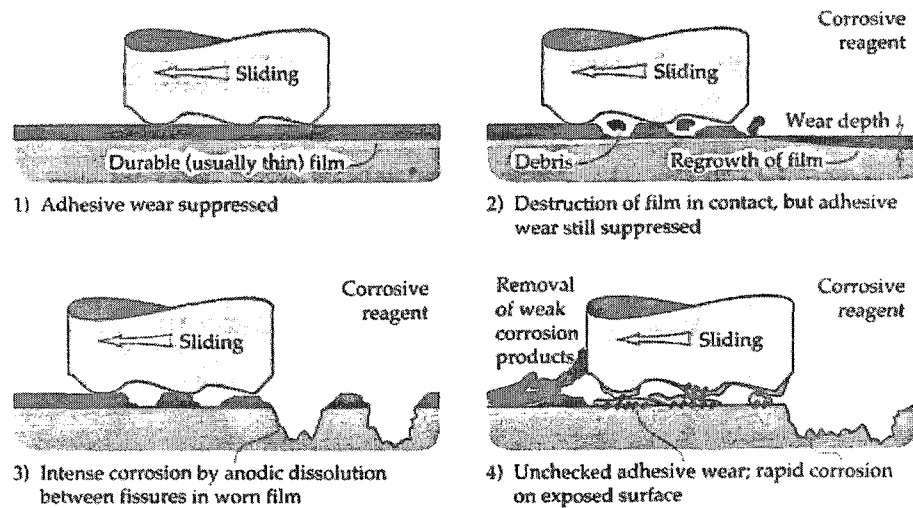


Figure 5.2: Models of Interaction between a Corrosive Agent and a Worn Surface [42].

The first process is dominated by the formation of durable lubricating films. If such films prevail then the worn contacts are well lubricated and corrosive wear does not occur. Unfortunately very few corrosion product films are durable so that this category of film formation is rarely seen in practice. The second process is related to the formation of a sacrificial or short life-time corrosion product film under sliding contacts. This is the most common form of corrosive wear since most corrosion films consist of brittle oxides or other ionic compounds. The third process relates to wear in highly corrosive media while the fourth process is effectively limited to extremely corrosive media where the corrosion products are very weak and probably soluble in the liquid media. It is very unlikely that wear and corrosion, if occurring in the same system, can proceed entirely independently since the heat and mechanical agitation of a sliding contact would almost inevitably accelerate corrosion [42].

Whenever the two independent processes of corrosion and wear occur simultaneously, it is almost certain that there will be a strong mutual interaction. Except in cases where a limited degree of corrosion or more exactly, surface film forming reaction, is essential to prevent corrosive wear.

5.4 The Influence of RRA on Wear Behaviour

Previous works have mostly focused on the effect of RRA treatment on the intergranular corrosion (IGC) resistance, stress corrosion cracking resistance, as well as microstructure, for 7xxx series alloys [3,18-19,21,30]. However, there is no

sufficient data concerning the effect of RRA treatment on the corrosive wear resistance of these alloys.

Baydogan, Cimenoglu and Kayali [43] examined dry sliding and corrosive wear rates of the T6-tempered and RRA-treated 7075 aluminum alloys. They retrogressed the samples at different temperatures (170, 220, 240 and 380°C) for various times from 15s to 60 min. Wear tests were conducted on a ball-on-disc-type wear tester by rubbing Al_2O_3 balls against the surfaces of the alloys. Corrosive wear tests were carried out in a 30g/l NaCl+10g/l HCl solution. They reported that retrogression at 220 and 240°C improved the dry sliding wear resistance when compared to the T6 temper. However, RRA-treated alloys exhibited higher corrosive wear rate than that of the T6-tempered alloy probably due to the limited lubricating effect of the corrosion products. Thus, relatively high corrosive wear resistance of the T6-tempered alloy than that of RRA treated alloy is due to presence of high amount of corrosion products at the interface of the ball and the alloy. It was suggested that the presence of relatively thick layer on the contact interface inhibits direct contact between the alloy and the Al_2O_3 ball [43].

Dry sliding and corrosion wear resistances and cavitation erosion-corrosion (CEC) behaviour of T6 and RRA treated 7039 alloys were compared by Mindivan et al [44]. Wear performance of the alloy was examined similar to Ref. [43]. CEC and corrosion wear tests were conducted in a “30g/l NaCl+10ml/l HCl” solution. The experimental results revealed that under dry sliding conditions, both RRA and T6 treated alloys resisted more than twice when compared to corrosion wear. Under dry sliding conditions the RRA treatment yielded better (about 20%) wear resistance than the T6 treatment. However, this was not the case for the corrosion wear. The corrosion wear resistance of the T6 treated alloy was about 40% higher than that of the RRA treated alloy. RRA treated alloy exhibited three fold higher resistance than T6 tempered alloy during CEC tests.

6. EXPERIMENTAL

This research was aimed to investigate the corrosion and wear performance of T6 and RRA treated aluminum alloys. Experimental procedure was composed of three main sections;

- Materials and heat treatment
- Corrosion tests
- Wear tests

6.1 Materials and Heat Treatment

Chemical compositions of the commercial 7075, 7175 and 6061 aluminum alloy billets utilized in this study are given in Table 6.1. Experiments were conducted on 3 mm thick samples machined from the billets. The width and the length of the samples were 15 mm and 20 mm, respectively. The longest length of the samples were along the deformation direction of the billets.

Table 6.1: Chemical Compositions of the Investigated Alloys.

Alloy	Cu (%)	Mn (%)	Zn (%)	Si (%)
7075	1.52	0.029	4.72	0.09
7175	1.58	0.026	5.05	0.072
6061	0.26	0.044	0.013	0.292

The examined aluminum alloys were heat treated according to T6 temper procedure as depicted in Table 6.2. Solutionising and aging stages of T6 temper were conducted in Nabertherm L3/C6 type atmospheric electric furnace. In the present work, alloys were retrogressed at various temperatures in between 220 and 280°C for various times (15 s to 12 min) after T6 temper. The retrogression process was carried out in a commercial AS 135 annealing salt bath (trademark of Petrofer Co.) in Nabertherm HO 60/E type furnace. Retrogressed samples were then reaged in Binder

electrical furnace as proposed for the aging stage of T6 temper. The samples were cooled in iced-water (7 °C) after each heat treatment step.

During the heat treatment processes hardness and the electrical conductivity of the samples were measured. Hardness measurements were made by Brooks MAT 24 hardness tester using Rockwell indenter (in HRB scale) under a normal load of 100 kg. The results of the hardness tests were evaluated by taking the average of five measurements. Electrical conductivities of the samples were measured with Hocking Autosigma 3000DL in a unit of International Annealed Copper Standard (% IACS). The probe diameter was 13 mm.

Table 6.2: T6 Temper Procedure of the Examined Aluminum Alloys [7].

Alloy	Solutionizing Temperature and Time	Aging Temperature and
7075	490°C, 40 min	120°C, 24h
7175	470°C, 40 min	120°C, 24h
6061	530°C, 40 min	180°C, 18h

6.2 Corrosion Tests

Corrosion behaviours of the investigated alloys were examined by intergranular and cavitation erosion tests.

6.2.1 Intergranular corrosion tests

Intergranular corrosion (IGC) tests were performed according to ISO 11846 [45] and ASTM G 110 (military specification MIL-H6088D) standards [46]. The procedures of the IGC tests are presented briefly in Table 6.3. pH and conductivity of the solutions were measured through out the IGC tests. A WTW pH/Cond 340i pHmeter having Sen Tix 41 pH and TetraCon 325 conductivity probe was used.

IGC tests were conducted by holding the samples in two different solutions namely HCl and H₂O₂ (Table 6.3) having amounts of 0.3 ml/mm² depending on the surface area of the samples. Before the tests, samples were immersed in pre-corrosion solutions and then their weights were measured with a balance having accuracy of 0.1 mg. Progress of corrosion was followed according to the weight loss method by measuring the weights of the samples at certain periods. Weights of the corroded samples were measured after cleaning their surfaces by immersing in HNO₃ solution.

Weight loss of the samples were then converted into weight loss per unit area (W/A) according to equation of

$$W / A = \frac{W_i - W_f}{A_t} (gr / cm^2) \quad (6.1)$$

where, W_i is the initial weight of the sample before the corrosion test, W_f is the weight of the corroded sample and A_t is the total surface area of the sample.

Table 6.3: IGC Test Procedures Applied to the Examined Alloys.

IGC test solution	Composition of Corrosion Solution	Pre-Corrosion Solution Temperature and Holding Time	Cleaning Solution and Holding Time	Relevant Standart
HCL	30g/l NaCl + 10ml/l HCl	% 10 NaOH 50-60 °C, 2.5 min	HNO ₃ , 2 min.	ISO 11846 [45]
H ₂ O ₂	57 gr/l NaCl + 10 ml/l H ₂ O ₂	945 ml water + 50 ml HNO ₃ (%70) + 5 ml HF (%48) 93 °C, 1 min	HNO ₃ , 1 min.	ASTM G 110 [46]

Total IGC testing times were 24 h and 12 h for HCl and H₂O₂ solutions, respectively. The results of the IGC tests were also evaluated by measuring the sizes of the pits on cross-sections of the samples along the longest section (20 mm) after employing conventional metallographic preparation procedure. Metallographic samples were etched with a modified Keller's solution, and then examined with Leica DM6000 M type confocal microscope. After measuring the widths of the pits, the pit concentration (PC) was quantified as [19]:

$$\%PC = \frac{\text{Sum of the pit widths}}{\text{Length of the section (20 mm)}} \times 100 \quad (6.2)$$

6.2.2 Cavitation - erosion corrosion tests

The cavitation-erosion corrosion (CEC) behaviour of T6 and RRA treated alloys was examined by immersing the samples in vibrating corrosive solutions utilised in IGC tests. The amount of solution was estimated by taking into account the surface area

of the specimens as 0.3 ml/mm^2 . Vibration was maintained by Bandelin Sonorex RK 52H ultrasonic vibration machine. The temperature of the solution was kept less than 40°C throughout the tests. The evaluation of the CEC tests were made according to weight loss and pit concentration as mentioned in Section 6.2.1.

6.3 Wear Tests

The sliding wear behaviour of the T6 and RRA treated alloys examined with a reciprocating wear tester in normal atmospheric conditions (room temperature and 40-50 % humidity) and in HCl and H_2O_2 corrosive solutions (Table 6.3), which were termed “dry sliding wear” and “corrosion wear”, respectively. Wear tests were conducted by applying a normal load of 100 g to the surfaces of the samples with a 10 mm diameter Al_2O_3 ball. The stroke of the ball was 12 mm. During the tests, the sliding speed was 26 mm/s for a total sliding distance of 120 m. Wear tests were performed for 90 min. After the tests, samples were cleaned with alcohol and profiles of the wear tracks were recorded by a Perthern S8P profilometer. The worn surfaces of the samples were examined by Leica MZ 16 FA stereo microscope.

7. RESULTS AND DISCUSSION

The effect of retrogression and re-aging on the hardness and electrical conductivity of 7075, 7175 and 6061 alloys are evaluated. On the basis of IGC and CEC test results, optimum RRA parameters are determined for each alloy. Finally tribological performances of the alloys are examined under dry sliding and corrosive wear testing conditions.

7.1 Effect of Heat Treatment on Hardness and Electrical Conductivity

The effect of retrogression and re-aging on the hardness of 7075 alloy is shown in Figure 7.1 in comparison with that of the T6 temper. The retrogression and re-aging curves are similar to those reported in the literature for 7XXX series aluminium alloys [12, 19, 21, 30].

When the samples are re-aged at 120 °C for 24h after retrogression, significant changes in hardness occur as presented in Figure 7.1.

The retrogression curves show that the hardness decreases with retrogression time, which can be seen more obviously in Figure 7.1.c. The hardness curves for specimens subjected to the RRA treatment indicate that within 15 seconds the retrogressed specimens recover their original T6 temper. After that, the hardness decreases with retrogression time. The decrease in hardness at 220°C after re-aging takes place at extended retrogression times (more than 120 s). The hardness decreases below T6 temper at shorter retrogression times with increasing retrogression temperatures (less than 120 s at 240°C and less than 60 s at 260°C). As it can be seen in Figure 7.1, the hardness sharply drops in shorter retrogression times at 260°C when compared to the retrogression temperatures of 220 and 240°C.

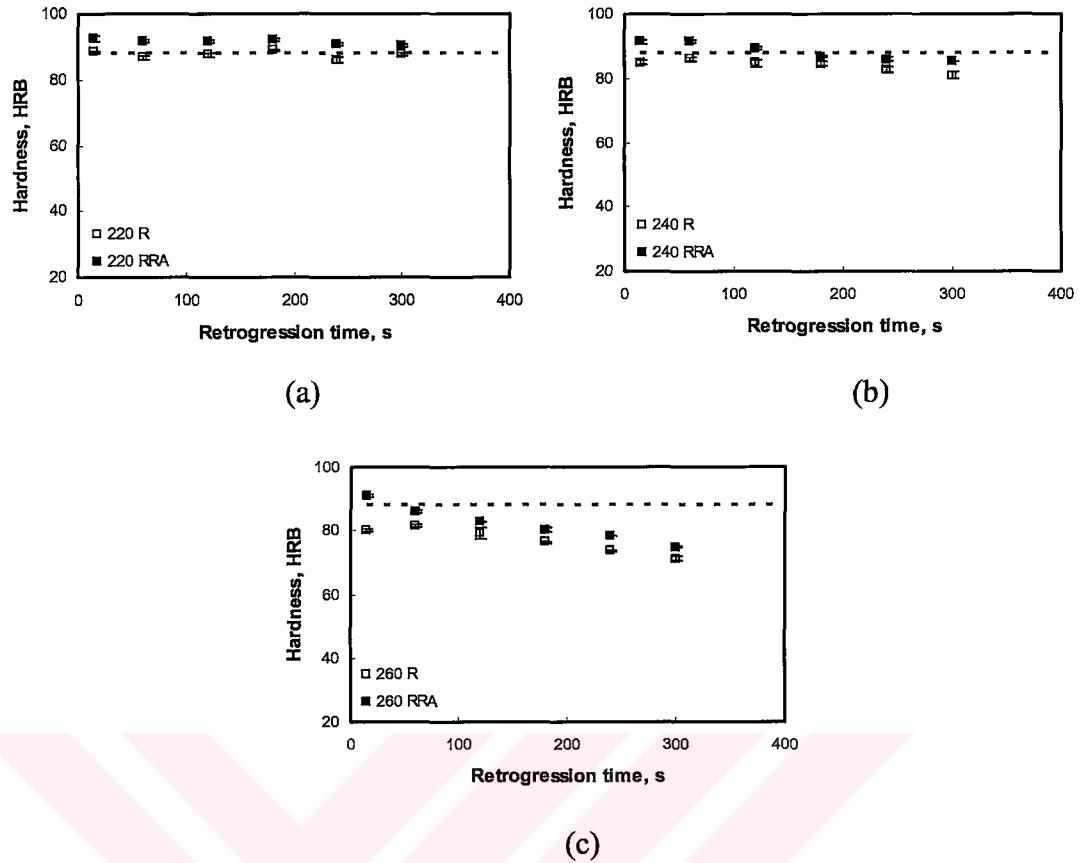


Figure 7.1: The Effect of Retrogression Time on the Hardness of 7075 Alloy at Retrogression Temperatures of (a) 220°C (b) 240°C and (c) 260°C. The Dashed Line Represents the Hardness After T6 Temper.

In Figure 7.2, the relationship between hardness and RRA treatment is shown for 7175 alloy. Similar to 7075 alloy, hardness values are about T6 temper up to a certain time of retrogression. Hardness decreases below T6 temper at longer retrogression times at lower retrogression temperatures (less than 180 s at 220°C, less than 60 s at 240°C and 260°C). The softening of the alloy is more evident at 260°C which indicates that temperature is the main controlling parameter of the RRA process. Higher the retrogression temperature, more the dissolution degree is achieved. For 7xxx series Al alloys Viana et al. [26] attributed hardness increase in the re-aging stage to the precipitation and growth of η' and transformation of η . In a retrogression process the initial decrease in hardness is attributed to the partial dissolution of Guiner-Preston zones [18] or precipitation of η' particles [30]. Re-aging of this partially dissolved microstructure leads to formation of fresh precipitates and solute depletion from the matrix which accompanied by an increase in hardness. Extended retrogression times cause coarsening of precipitates which accompanied by an abrupt reduction in hardness.

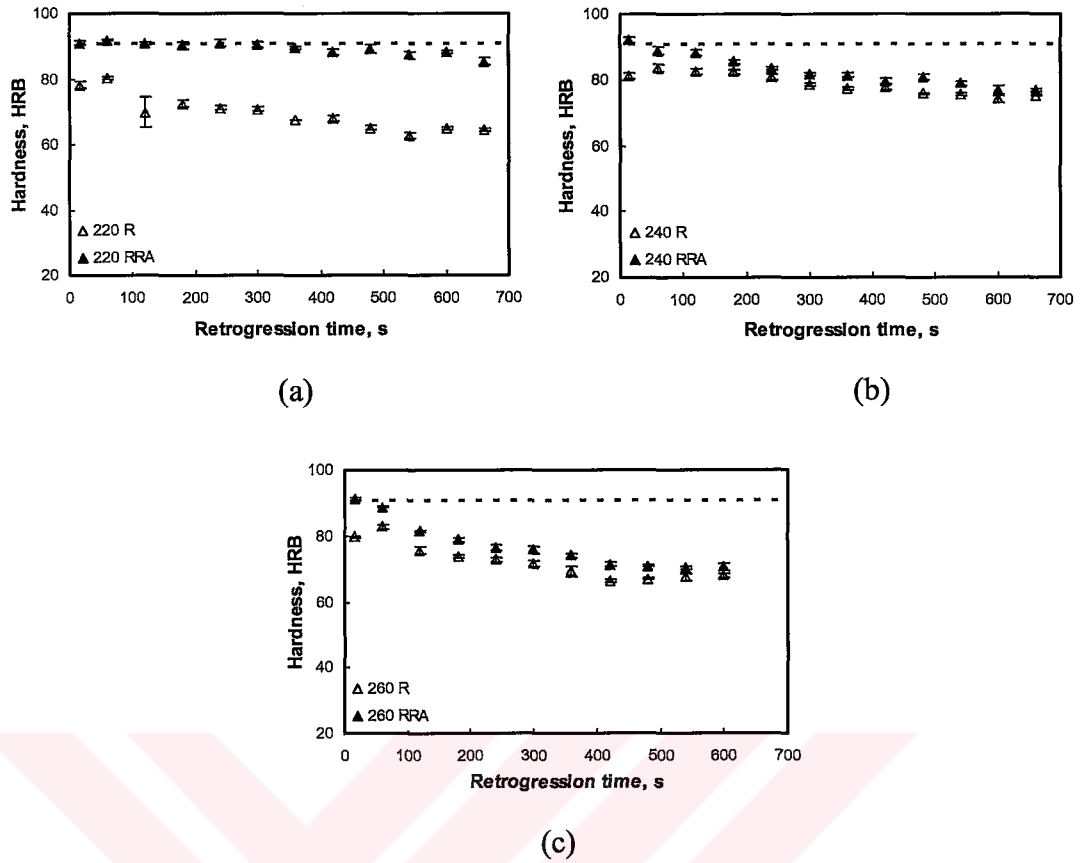


Figure 7.2: The Effect of Retrogression tTime on the Hardness of 7175 Alloy at the Retrogression Temperatures of (a) 220°C (b) 240°C and (c) 260°C. The Dashed Line Represents the Hardness After T6 Temper.

The hardness of the 6061 alloys retrogressed at 220, 240, 260 and 280°C are shown in Figure 7.3. In the literature there is no report related to RRA treatment for 6061 alloys. Figure 7.3 shows that at retrogression temperatures of 220, 240 and 260°C, 6061 alloy exhibit different behaviour than 7075 and 7175 alloys. At these retrogression temperatures, the hardness curve of 6061 alloy do not show a characteristic trend of RRA process. In Figure 7.3.a, the T6 hardness value is firstly reached within 300 s and then decreases which is followed by an increment above the hardness of T6 temper after retrogression for 480 s. Similar trends are also evident for 240 and 260°C. Retrogression temperature of 280°C yields the characteristic RRA curve for 6061 alloy. At 280°C, the peak hardness is reached at 15 s followed by a significant loss in hardness with increasing retrogression time.

Electrical conductivity measurements are widely used to evaluate the microstructural changes during precipitation hardening treatment besides hardness measurements. Increase of electrical conductivity implies purification of the matrix due to solute

depletion, which arises formation of new precipitates and/or coarsening of preexisted precipitates [21].

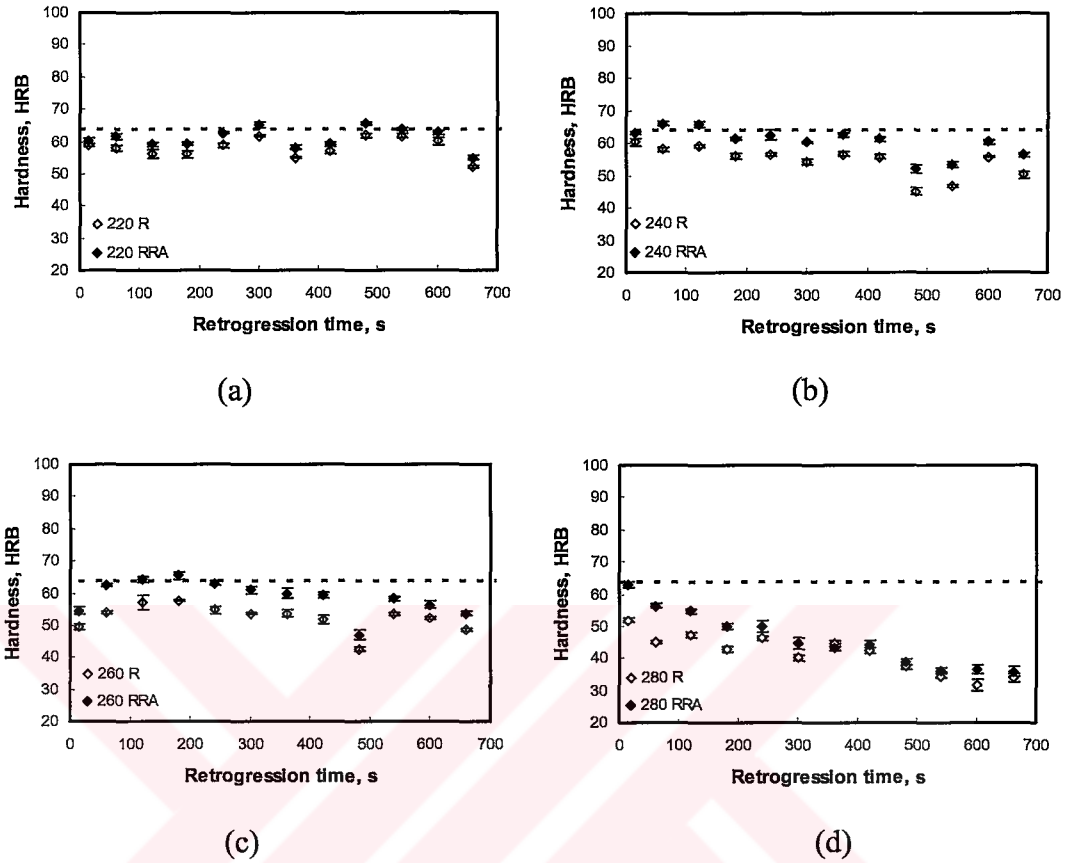


Figure 7.3: The Effect of Retrogression Time On the Hardness of 6061 Alloy at The Retrogression Temperatures of (a) 220°C (b) 240°C (c) 260°C and (d) 280°C. The Dashed Line Represents the Hardness After T6 Temper.

Figure 7.4 and 7.5 presents the electrical conductivity values for 7175 and 6061 alloys respectively. The electrical conductivity of 7175 alloy is significantly higher than that of the T6 temper at all temperatures and increase with increasing retrogression time. It can be seen that the minimum T73 conductivity of 38% IACS (percent of International Annealed Copper Standards) is obtained in these specimens. The minimum retrogression time required reach at least 38% IACS is ~7 min at 220 °C, ~3 min at 240 °C, or ~2 min at 260 °C.

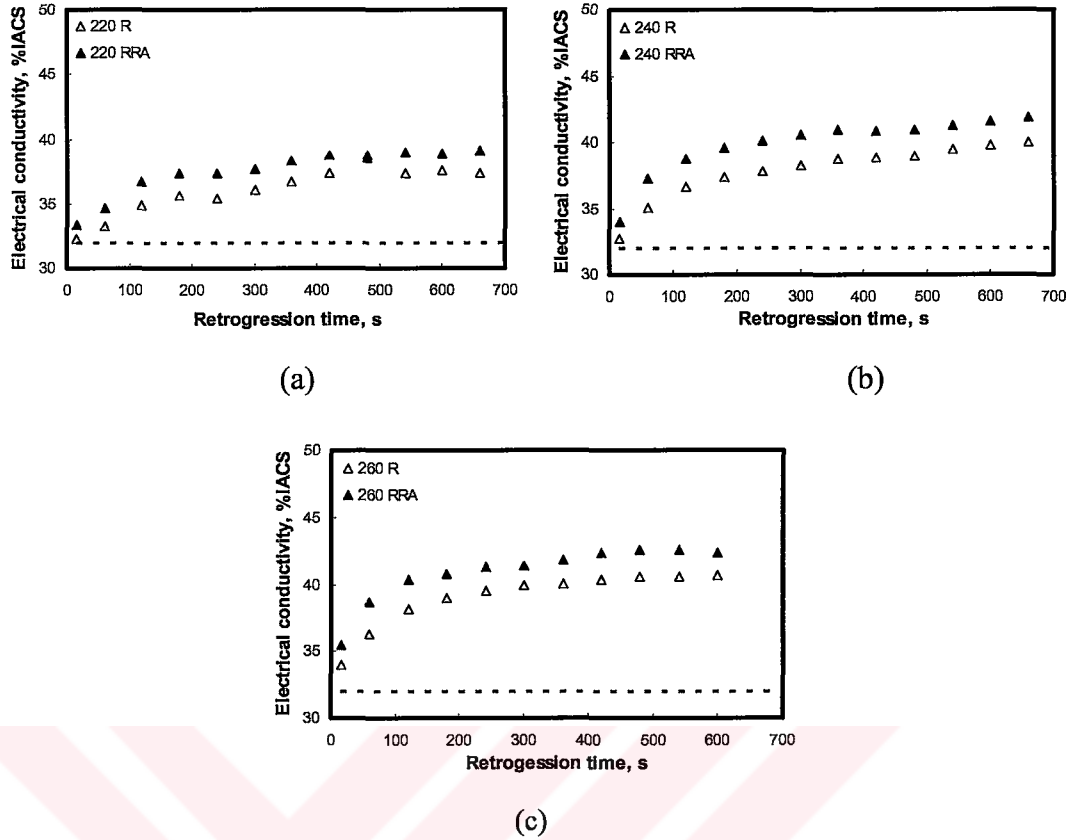


Figure 7.4: The Effect of Retrogression Time on the Electrical Conductivity of 7175 Alloy at the Retrogression Temperatures of (a) 220°C (b) 240°C and (c) 260°C. The Dashed Line Represents the Electrical Conductivity After T6 Temper.

Figure 7.5 shows the variation of electrical conductivity of 6061 alloy upon RRA treatment for retrogression temperatures of 220, 240 and 260°C, retrogression, retrogression and re-aging and T6 temper conditions yield almost similar electrical conductivity values. The observations indicate that at retrogression temperatures below 260°C no detectable change in the microstructure takes place during RRA process. Upon RRA treatment at 280°C, electrical conductivity increase with increasing retrogression times reaching higher values than T6 temper conditions.

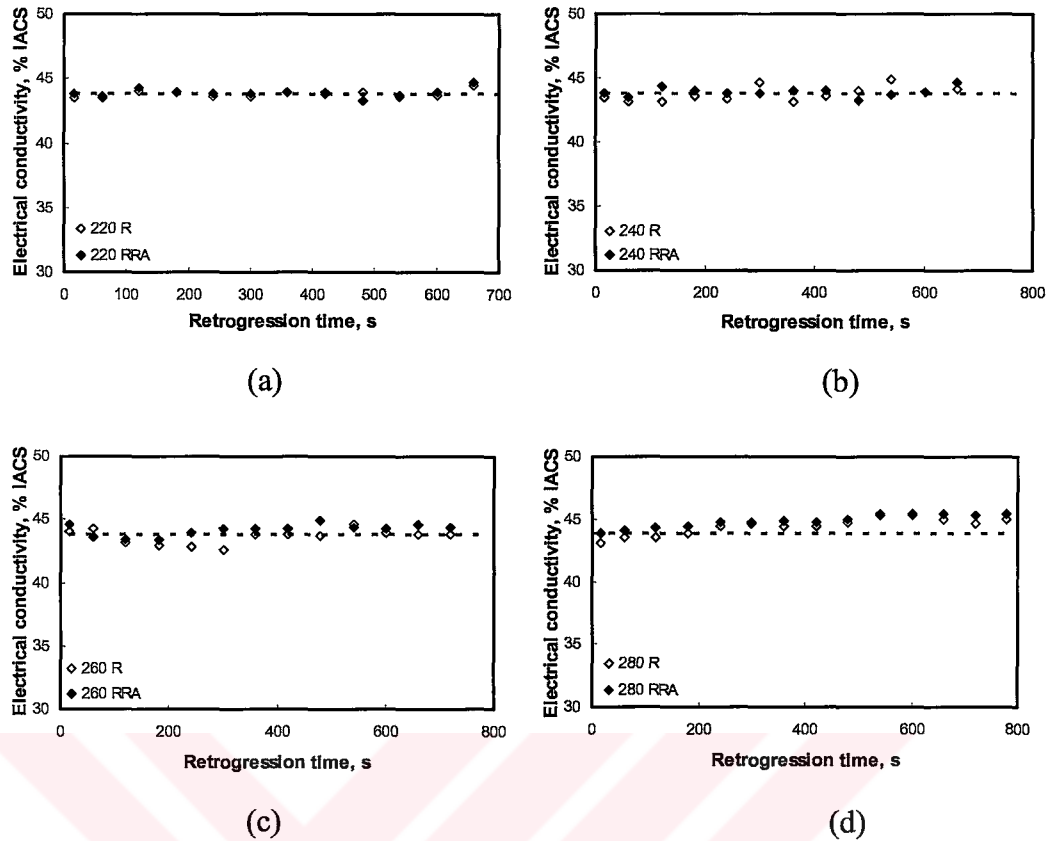


Figure 7.5: The Effect of Retrogression Time on the Electrical Conductivity of 6061 Alloy at the Retrogression Temperatures of (a) 220°C (b) 240°C (c) 260°C and (d) 280°C. The Dashed Line Represents the Electrical Conductivity After T6 Temper.

Table 7.1 list the RRA hardness values corresponding to the shortest retrogression time which gives hardness values close to T6 temper. In this study, further studies are conducted on the alloys retrogressed and re-aged as listed in Table 7.1.

Table 7.1: Hardness of T6 and RRA Treated Alloys. The Times Given in the Parentheses Are the Shortest Retrogression Times Giving Hardness Values Close to T6 Temper.

Alloy	Hardness (HRB)				
	T6	220 RRA	240 RRA	260 RRA	280 RRA
7075	88	92 (15 s)	91 (15 s)	91 (15 s)	-
7175	91	91 (15 s)	92 (15 s)	91 (15 s)	-
6061	63,8	65 (5 min)	66 (1 min)	65 (4 min)	63 (15 s)

7.2 Results of Corrosion Tests

IGC and CEC behaviours of the examined alloys are followed by weight loss and pitting concentration measurements of the samples along with pH and liquid conductivity measurements of the solutions. After the tests, pit concentrations of the samples are determined by linear intercept method.

7.2.1 IGC test results

Figures 7.6, 7.7 and 7.8 depict the weight loss of T6-tempered and RRA-treated alloys in HCl and in H₂O₂ solutions. As a general trend the increase of holding time in corrosion solution increase the weight loss of the samples. When compared to H₂O₂ solution, HCl is very aggressive since the weight loss dramatically increases with increasing testing time.

As shown in Figure 7.6, RRA treatments improve corrosion resistance of 7075 alloy both in HCl and H₂O₂ solutions. In HCl solution samples retrogressed at 220°C exhibit the best corrosion resistance due to its lowest weight loss. However in H₂O₂ solution, both T6 tempered and RRA treated alloy exhibit similar behaviour. Detailed analysis of Figure 7.6.a reveals that retrogression at 260°C promotes better corrosion resistance.

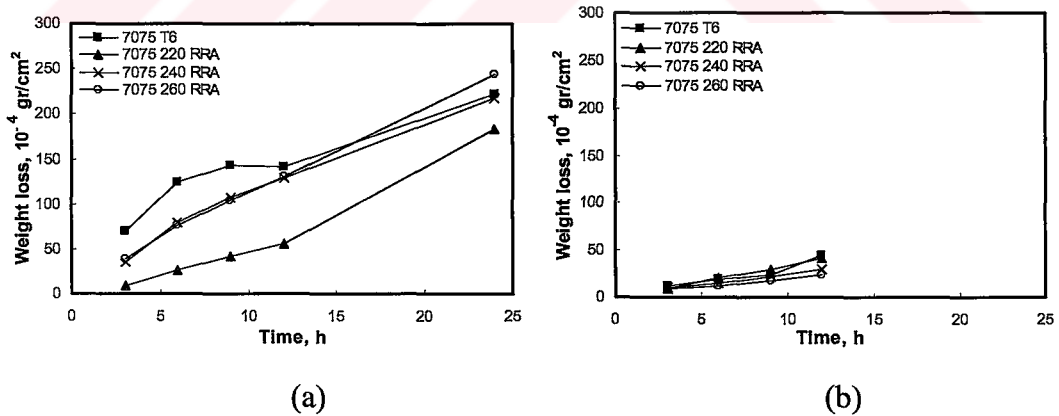


Figure 7.6: The Weight Loss Values of 7075 Alloys Corroded (a) in HCl Solution and (b) in H₂O₂ Solutions for 24 and 12 Hours Respectively.

Weight loss of T6 and RRA treated 7175 alloys are given in Figure 7.7. RRA treated and T6 tempered 7175 alloys do not exhibit different behaviour in H₂O₂ solution. Thus, their weight loss values are very close to each other. However in HCl solution

T6 tempered alloy exhibits lower weight loss than RRA treated alloys. Among the RRA treated alloys the lowest weight loss is obtained upon retrogression at 240°C.

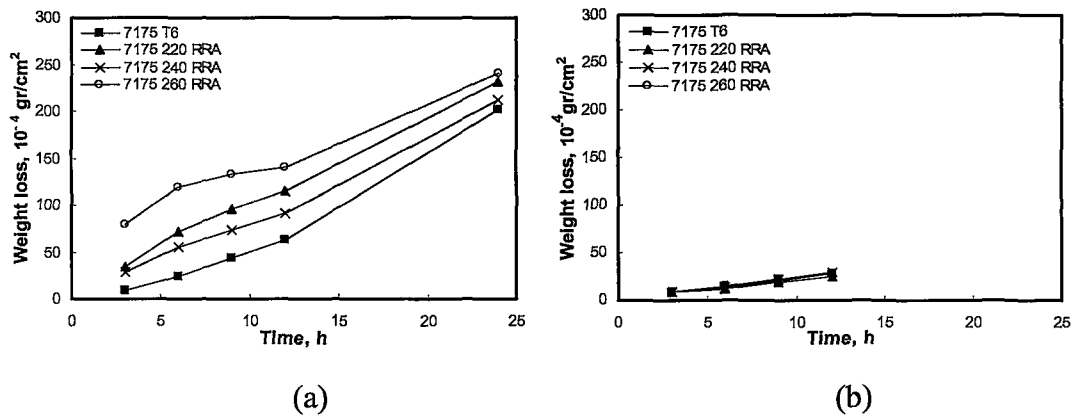


Figure 7.7: The Weight Loss Values of 7175 Alloys Corroded (a) in HCl Solution and (b) in H₂O₂ Solutions For 24 and 12 Hours Respectively.

For 6061 alloy, no considerable difference between T6 tempered and RRA treated alloys are evident both in HCl and H₂O₂ solutions as seen in Figure 7.8. At extended testing times, weight of RRA treated sample is slightly lower than that of T6 tempered alloy.

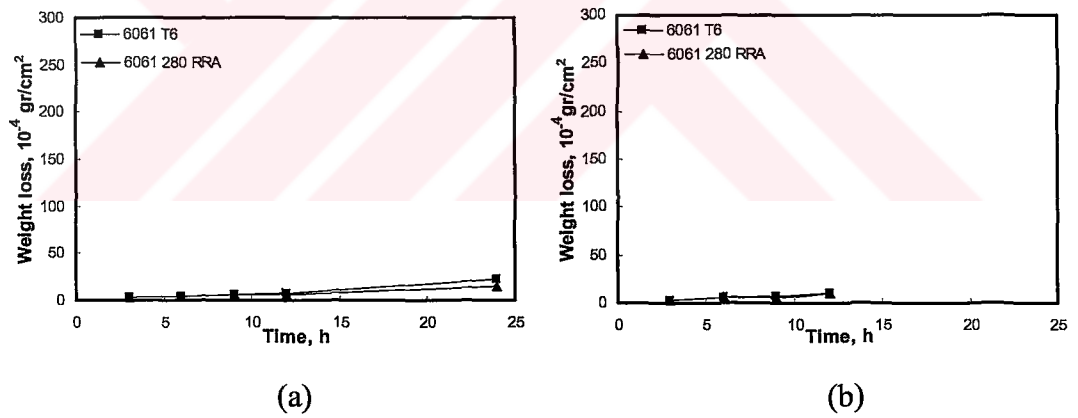


Figure 7.8: The Weight Loss Values of 6061 Alloys Corroded (a) in HCl Solution and (b) in H₂O₂ Solutions for 24 and 12 Hours Respectively.

Figure 7.9 illustrates the pH (acidity) variation of the HCl and H₂O₂ solutions during corrosion testing of 7075 alloy. PH values increase during corrosion testing. While the PH values slightly increase in HCl solution the pH value of H₂O₂ solution gets stable nearly after testing time of 6 hours.

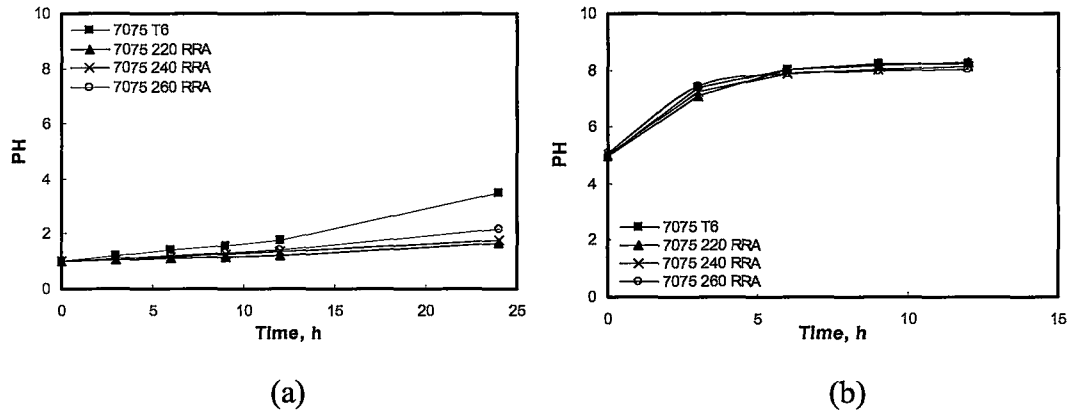


Figure 7.9: The pH Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 7075 Alloy.

Figure 7.10 denotes the pH variation of corrosive solutions utilized in testing of 7175 alloy. There is not a strong distinction in the pH values of HCl and H₂O₂ solutions depending on the heat treatment condition of the 7175 alloy.

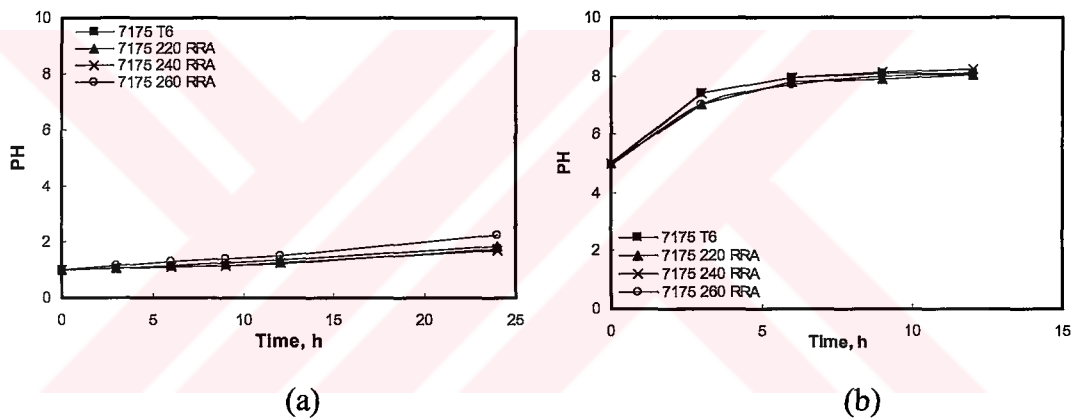


Figure 7.10: The pH Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 7175 Alloy.

Figure 7.11 compares the pH values of the corrosion solution during testing of T6-tempered and at 280°C retrogressed 6061 aluminum alloys. The heat treatment condition of the alloys being tested does not have a considerable influence on the pH values of both HCl and H₂O₂ solutions.

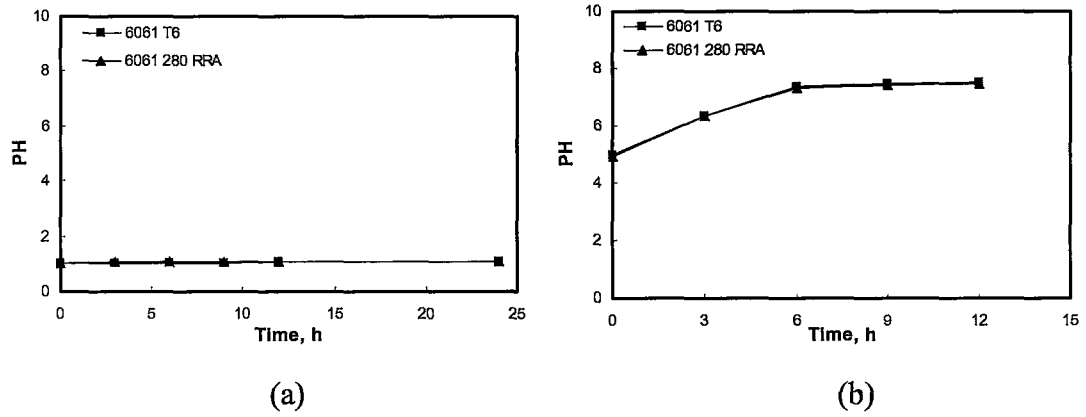


Figure 7.11: The pH Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 6061 Alloy.

The liquid conductivity values of the corrosion solutions utilized in testing as 7075 alloys are shown in Figure 7.12. While the liquid conductivity of HCl solution decreases with increasing testing time (7.12.a), no considerable change observed for H₂O₂ solution (7.12.b).

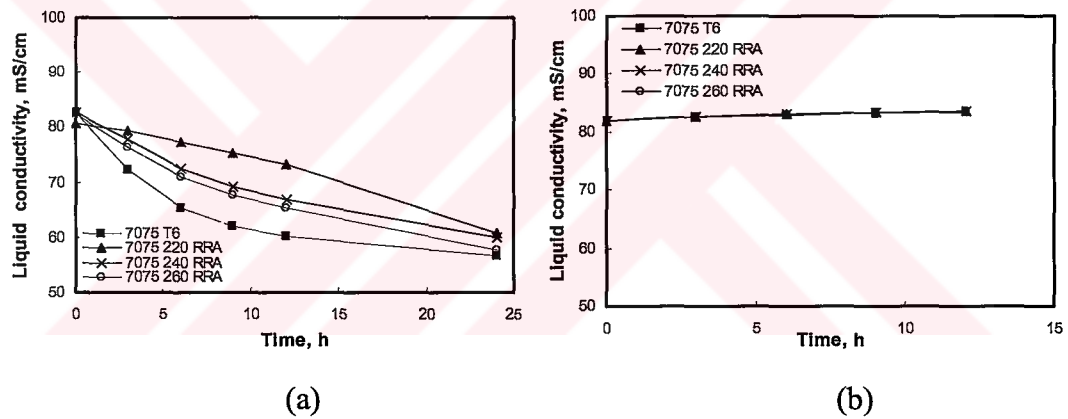


Figure 7.12: The Liquid Conductivity Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 7075 Alloy.

Figure 7.13 shows the relationship between liquid conductivity of corrosion solution with respect to testing time of 7175 alloys. H₂O₂ solution yields a constant conductivity through out testing time. The conductivity of HCl solution continuously decreases with increasing testing time.

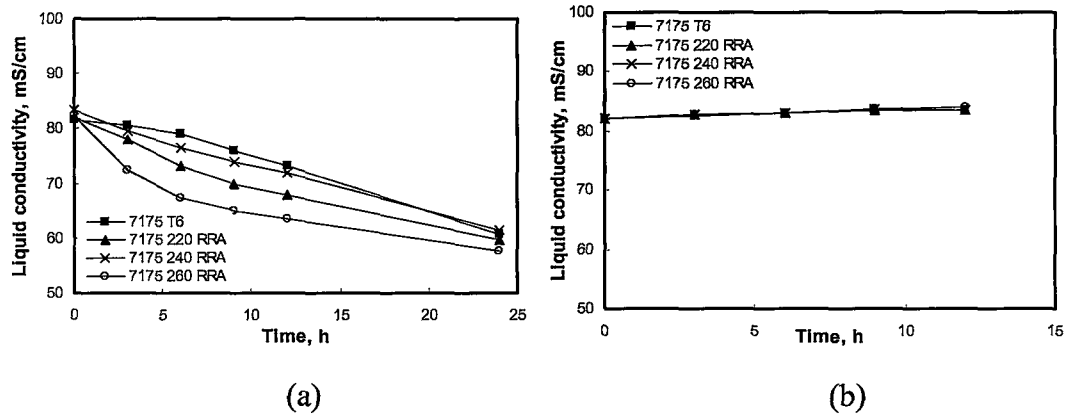


Figure 7.13: The Liquid Conductivity Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 7175 Alloy.

Liquid conductivities of corrosion solutions utilized in testing of 6061 alloys are presented in 7.14. The conductivity of both HCl and H₂O₂ solutions are almost constant through out the IGC tests of 6061 alloy.

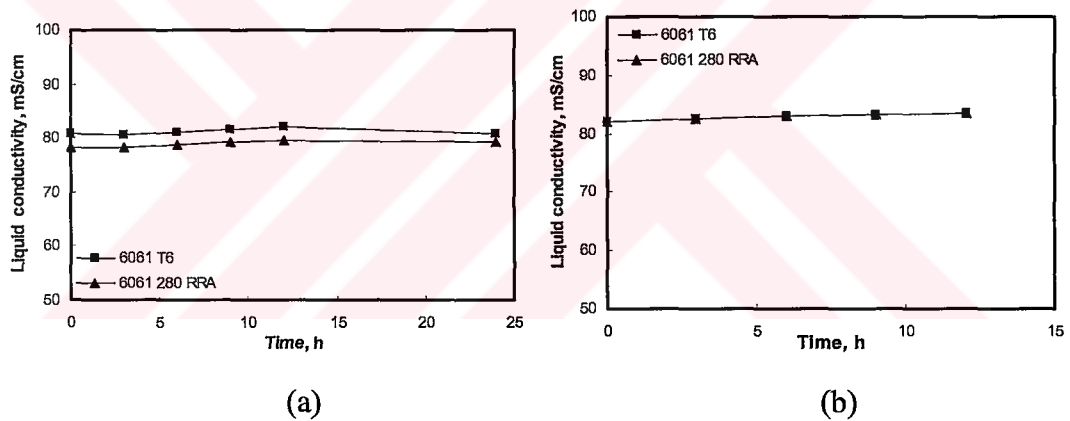


Figure 7.14: The Liquid Conductivity Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 6061 Alloy.

The results of microscopic examinations conducted on the alloys hold in HCl and H₂O₂ solutions for 24h and 12h respectively given in Table 7.2 in terms of pit concentration and depth of deepest pit.

Table 7.2 shows that RRA process increases the corrosion resistance decreasing the pit concentration and pit depth. Among 7075 alloys, retrogression at 260°C improves the corrosion resistance in both of the HCl and H₂O₂ solutions when compared to T6 temper. 7175 alloy has the best corrosion resistance after retrogression 260°C similar to 7075 alloy. RRA treatment also provides higher corrosion resistance to 6061 alloy.

The improvement in the IGC resistance of the examined Al alloys upon RRA treatment can be attributed to the formation precipitates within the grains rather than grain boundaries. At grain boundaries precipitation and dissolution of GP zones within the grains cause a high difference in the electrochemical potentials of these regions which result in a high intergranular corrosion susceptibility [19]. Superior combination of hardness and IGC resistance of RRA-treated alloys can be attributed to the unique microstructure which consist of η' precipitates inside the grains and η precipitates in the grain boundaries [3].

Table 7.2: Pit Concentration and Maximum Pit Depth of the Investigated Alloys in HCl and H₂O₂ Solutions During IGC Tests.

Heat Treatment	Pit concentration %		Deepest pit (μm)	
	HCl	H ₂ O ₂	HCl	H ₂ O ₂
7075 Alloy				
T6	60	24	85	90
220 RRA	43	27	226	114
240 RRA	29	26	90	97
260 RRA	27	17	76	50
7175 Alloy				
T6	35	20	185	94
220 RRA	36	22	317	76
240 RRA	36	13	218	105
260 RRA	22	12	227	109
6061 Alloy				
T6	16	4	114	68
280 RRA	10	4	52	71

The morphologies of the pits formed on the surface of samples in HCl solution are shown Figure A1 - A3 as an example. Microscopic examinations conducted along the longest axis of the samples reveal that corrosion attack is intergranular in nature. All the alloys exhibit horizontal morphology probably due to microstructural orientation.

7.2.2 CEC test results

The CEC tests are conducted on T6 tempered and RRA treated (at 260°C) 7175 alloys. When weight loss results are considered, it is seen in Figure 7.15 that the weight loss increases with increasing holding time in vibrating corrosion solutions.

No considerable difference is measured between the weight loss of RRA treated and T6 tempered alloys.

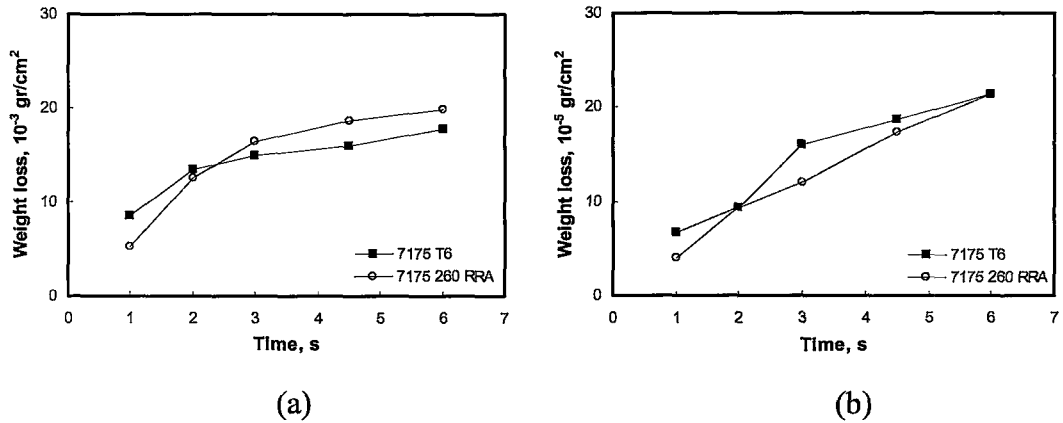


Figure 7.15: The Weight Loss Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 7175 Alloy.

In Figure 7.16 the pH values of the corrosion solution through out testing of 7175 alloys are presented. PH values of the HCl solution increased with increasing testing time. However, the pH value of the H₂O₂ solution stays almost constant at certain testing time (2 h).

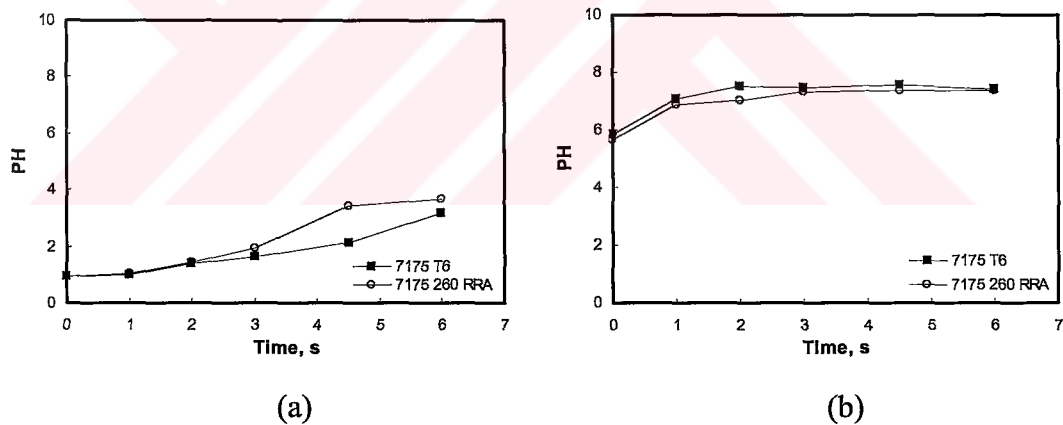


Figure 7.16: The pH Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 7175 Alloy.

Figure 7.17 depicts the liquid conductivity of the corrosion solution utilized in testing of 7175 alloy. The liquid conductivity of HCl solution sharply decreases while the conductivity of H₂O₂ solution tends a slight increment.

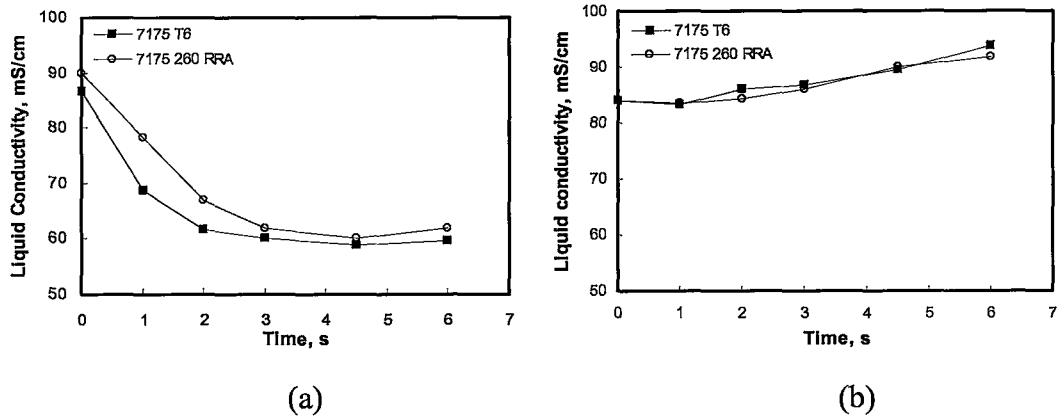


Figure 7.17: The Liquid Conductivity Values of (a) HCl Solution and (b) H₂O₂ Solutions During Corrosion Testing of 7175 Alloy.

The results of microscopic examinations conducted on the 7175 alloy hold in vibrating HCl and H₂O₂ solutions for 6h are given in Table 7.3.

Table 7.3: Pit Concentration and Maximum Pit Depth of the Investigated Alloys in HCl and H₂O₂ Solutions During CEC Tests.

Heat Treatment	Pit concentration %		Deepest pit (μm)	
	HCl	H ₂ O ₂	HCl	H ₂ O ₂
7175 Alloy				
T6	16	1	210	42
260 RRA	25	1	143	45

When compared to H₂O₂ solution, HCl solution results in progress of deep and intense pits on the surface of the 7175 alloys. T6 tempered alloys exhibit the lower pit concentration but deeper pit depth than RRA treated alloy. Finally it is concluded that, testing time of 6 h is almost insufficient to identify the heat treatment condition on CEC resistance of the examined alloy exactly. Thus extended times are needed to clarify the difference in CEC resistance depending on the heat treatment condition.

7.3 Results of Wear Tests

Wear tests are carried out to compare the dry sliding and corrosive wear resistances of T6 tempered alloys with those of the RRA treated alloys. Wear tests are performed under normal atmospheric condition and also in HCl and H₂O₂ solutions. In this study the former is named as dry sliding wear and the latter is named as corrosion wear. Figure A4-A6 display the 3-D profiles of the wear tracks developed on the surfaces of T6 and RRA treated alloys during reciprocating wear tests. Corrosion

wear tests developed wider and deeper wear tracks on the surfaces of the samples than dry sliding wear tests. This observation clearly defines the synergistic effect of corrosion and wear during corrosion wear tests.

Table 7.4 lists the relative wear resistance (RWR) of the T6 and RRA treated alloys. Since smallest wear track area is obtained from the 260°C RRA treated 7075 alloy under dry sliding condition, this condition is taken as the reference point. The RWR of each alloy is calculated by dividing the wear track area of the 260°C RRA treated 7075 alloy to that of each alloy.

Table 7.4: RWR Values of the Investigated Alloys.

Heat Treatment	RWR (%)		
	Dry Sliding	HCl	H ₂ O ₂
7075 Alloy			
T6	35	13	6
260 RRA	100	13	6
7175 Alloy			
T6	78	16	5
260 RRA	96	16	8
6061 Alloy			
T6	37	4	4
280 RRA	36	4	5

Table 7.4 indicates that for 7xxx series RRA treatment improves the dry sliding wear resistance when compared to T6 temper condition. However almost similar dry sliding wear resistance is obtained from T6 and RRA treated 6061 alloy. When compared to 7xxx series alloys, the lowest wear resistance is obtained from 6061 alloy due to its low hardness (Table 7.1). In corrosive solution, wear resistance of the examined alloy dramatically decreases. When compared to HCl solution, lower RWR value obtained in H₂O₂ solution. It is interesting to notice that both in H₂O₂ and HCl solutions, considerable difference is not evident depending on heat treatment condition (T6 temper and RRA treatment). Tse and Suh [47], Baydogan et al [43] declared lubricating effect of corrosion products during sliding wear tests. Therefore, relatively high corrosive wear resistance of the alloys in HCl solution can be attributed to formation of increased amounts of corrosion products in the wear tracks than in H₂O₂ solution. IGC tests also reveal more aggressive attack of HCl solution than H₂O₂ solution (Table 7.2). It is suggested that the presence of relatively thick

layer on the contact interface inhibits direct contact between the alloy and the Al_2O_3 ball.

The worn surfaces of the T6 and RRA treated alloys are given in Figure 7.18 and Figure 7.19. When compared the dry sliding condition, wider wear tracks develop in corrosive solutions as shown in Figure 7.18. However, relatively narrower wear tracks are formed on the surface of RRA treated 7xxx alloys than T6 temper under dry sliding wear conditions. No considerable difference in the width of wear is evaluated during corrosion wear tests is evident for T6 and RRA treated alloys. These observations are compatible with RWR data of investigated alloys (Table 7.4).

High magnification microscopic examination of the wear tracks done under dry sliding wear conditions reveal that, wear progressed by ploughing action of the Al_2O_3 ball leading to heavy plastic deformation at the contact surfaces. The topography of the wear tracks developed during corrosion wear tests is quite different and within the wear tracks corrosion pits are present. Therefore, the formation of a wider and deeper wear track area during corrosive wear testing than that of dry sliding wear (Figure A4-A6) can be attributed to the progress of corrosion on the bare contact surfaces of the alloys that appeared after removal of the protective oxide layer from the surface by the grooving action of the Al_2O_3 ball.

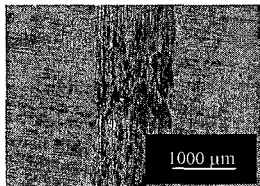
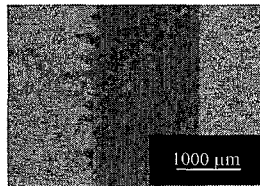
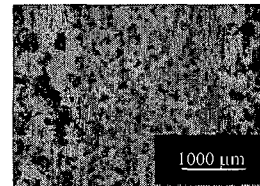
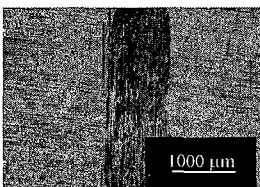
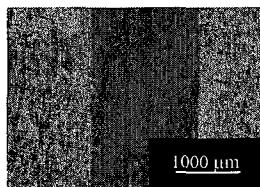
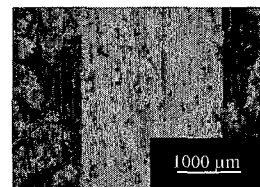
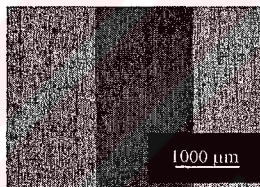
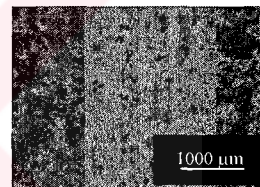
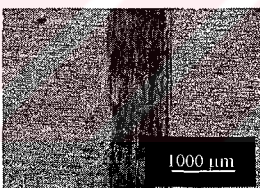
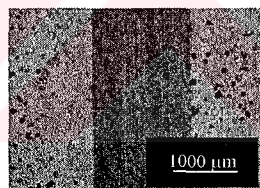
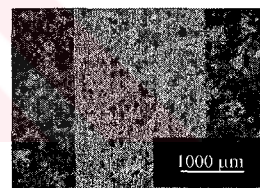
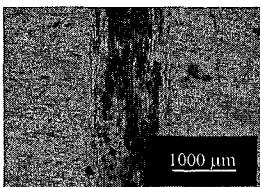
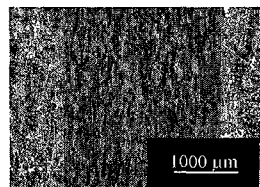
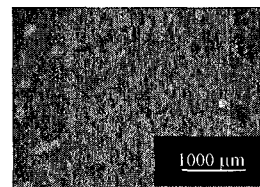
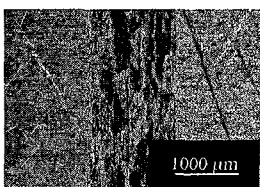
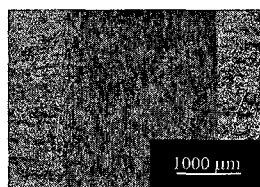
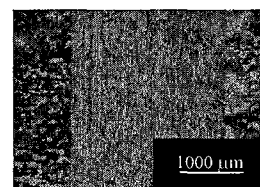
Heat Treatment	RWR (%)		
	Dry Sliding	HCl	H ₂ O ₂
7075 Alloy			
T6			
260 RRA			
7175 Alloy			
T6			
260 RRA			
6061 Alloy			
T6			
280 RRA			

Figure 7.18: Low Magnification Optical Micrographs of the Wear Tracks Developed on the Surfaces of the Examined Alloys.

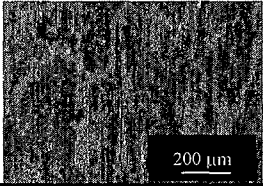
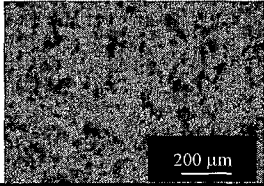
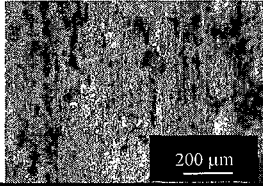
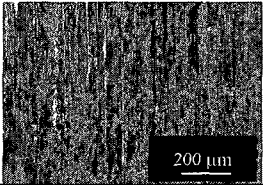
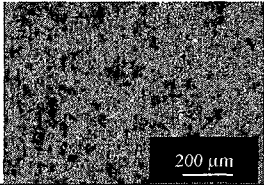
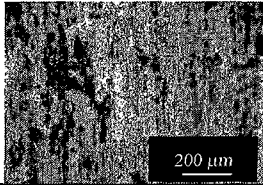
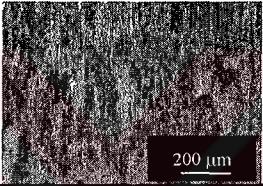
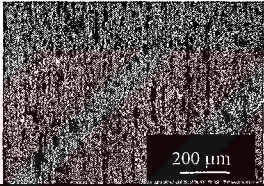
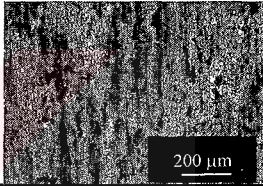
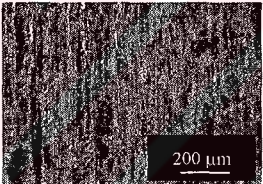
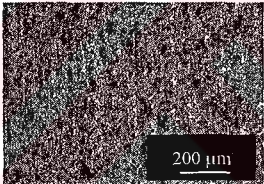
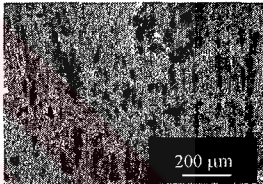
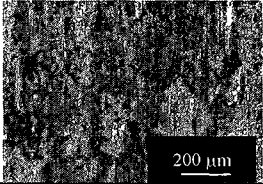
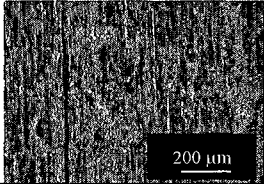
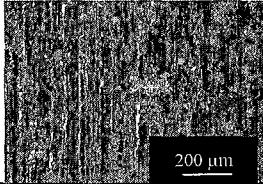
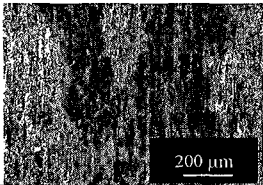
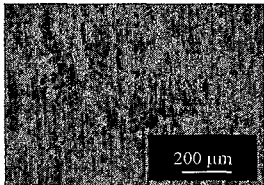
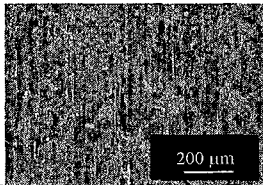
Heat Treatment	RWR (%)		
	Dry Sliding	HCl	H ₂ O ₂
7075 Alloy			
T6			
260 RRA			
7175 Alloy			
T6			
260 RRA			
6061 Alloy			
T6			
280 RRA			

Figure 7.19: High Magnification Optical Micrographs of the Wear Tracks Developed on the Surfaces of the Examined Alloys.

8. CONCLUSION

Following conclusions can be made depending on the experiment results conducted on T6 tempered and RRA treated aluminum alloys (7075, 7175 and 6061).

1 – RRA treatment increases the hardness of 7075 alloy. However no considerable hardening is observed upon RRA treatment for 7175 and 6061 alloys when compared to T6 temper.

2 – RRA treatment improves the IGC resistance of the examined alloys in HCl (30g/l NaCl+10ml/l HCL) and H₂O₂ (57g/l NaCl+10 ml/l H₂O₂) solutions. HCl solution causes heavier corrosion attack than H₂O₂ solution.

3 – RRA treatment improves the dry sliding wear resistance of 7075 and 7175 alloys but do not achieve any considerable improvement for 6061 alloy when compared to T6 temper condition.

4 – Wear resistance of the examined alloys dramatically decrease in HCl and H₂O₂ solutions. Alloys exhibit higher corrosion wear rate in H₂O₂ solution than in HCl solution. In both solutions, no considerable difference in wear rates is evident depending on the heat treatment condition (T6 temper and RRA treatment) for each alloy.

5 – Optimum RRA treatment conditions of the examined alloys are as follows:

Alloy	Retrogression temperature (°C)	Retrogression time (s)	Hardness (HRB)
7075	260	15	91
7175	260	15	91
6061	280	15	63

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APPENDIX A

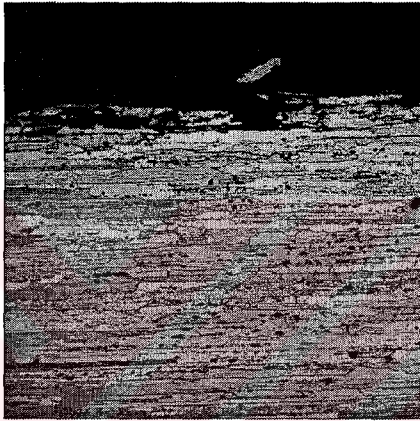
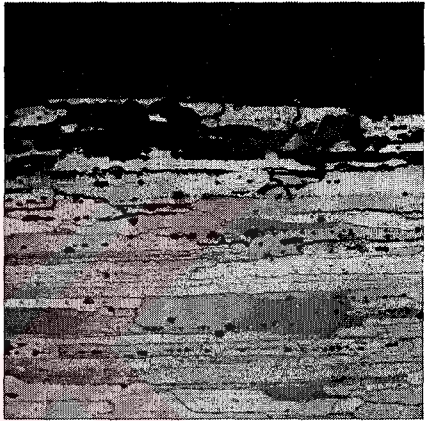
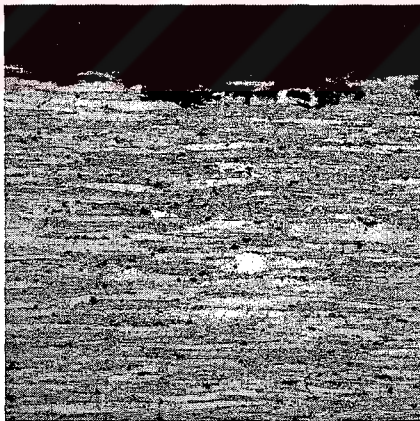
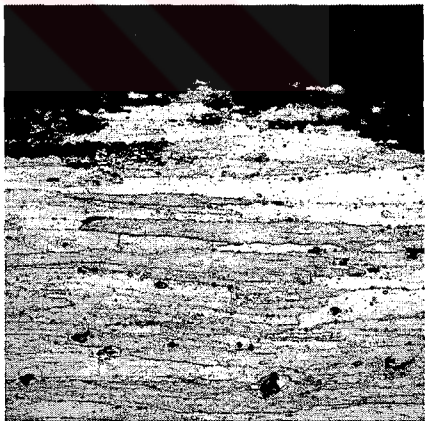
Heat Treatment	200x	500x
7075 Alloy		
T6		
260 RRA		

Figure A.1 Cross-Sectional Confocal Micrographs of the 7075 Aluminum Alloy after IGC Tests in HCl Solution.

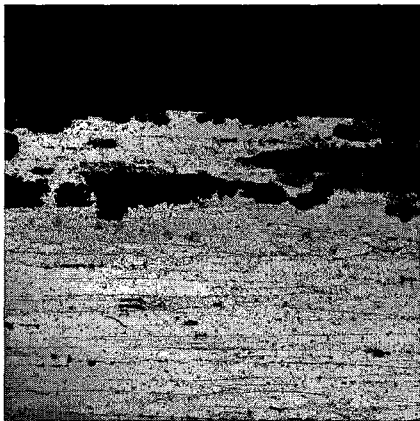
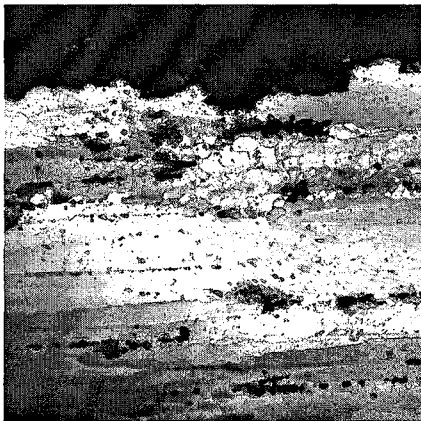
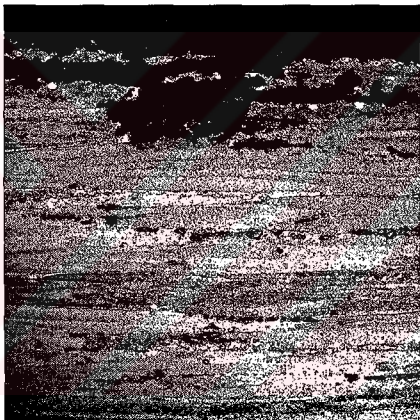
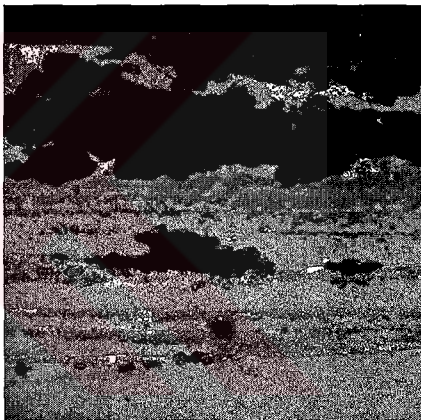
Heat Treatment	200x	500x
7175 Alloy		
T6		
260 RRA		

Figure A.2 Cross-Sectional Confocal Micrographs of the 7175 Aluminum Alloy after IGC Tests in HCl Solution.

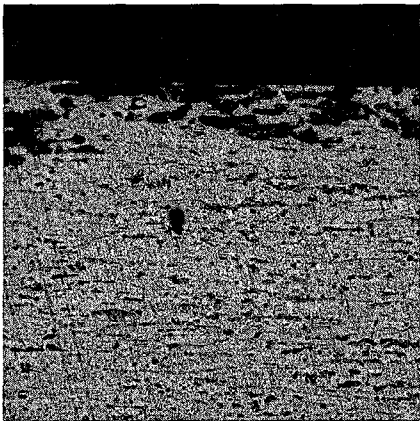
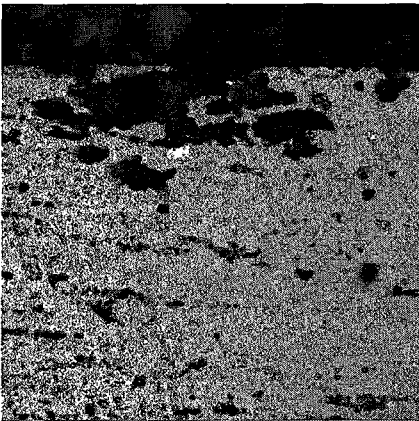
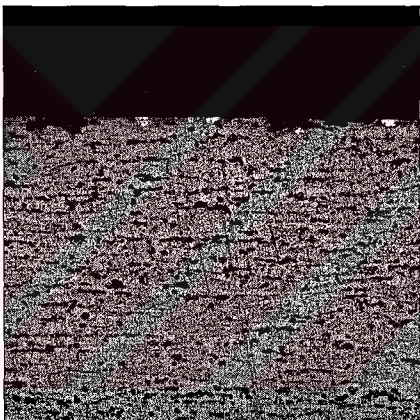
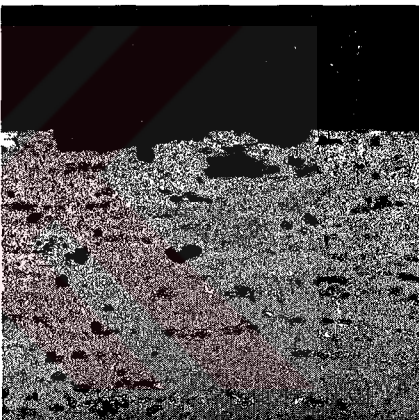
Heat Treatment	200x	500x
6061 Alloy		
T6		
260 RRA		

Figure A.3 Cross-Sectional Confocal Micrographs of the 6061 Aluminum Alloy After IGC Tests in HCl Solution.

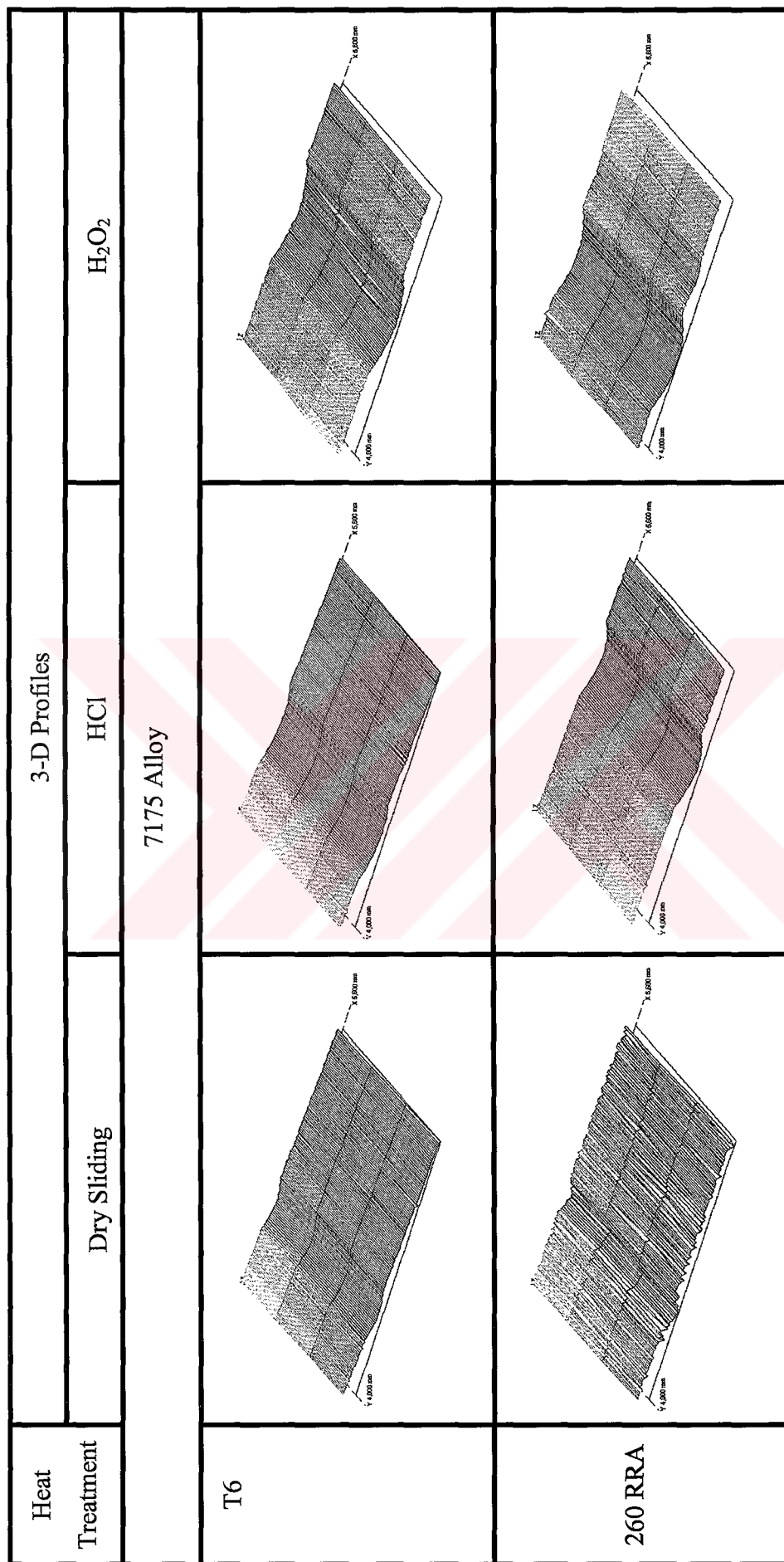


Figure A.4 3-D Profiles of the Wear Tracks of Investigated 7075 Alloys.

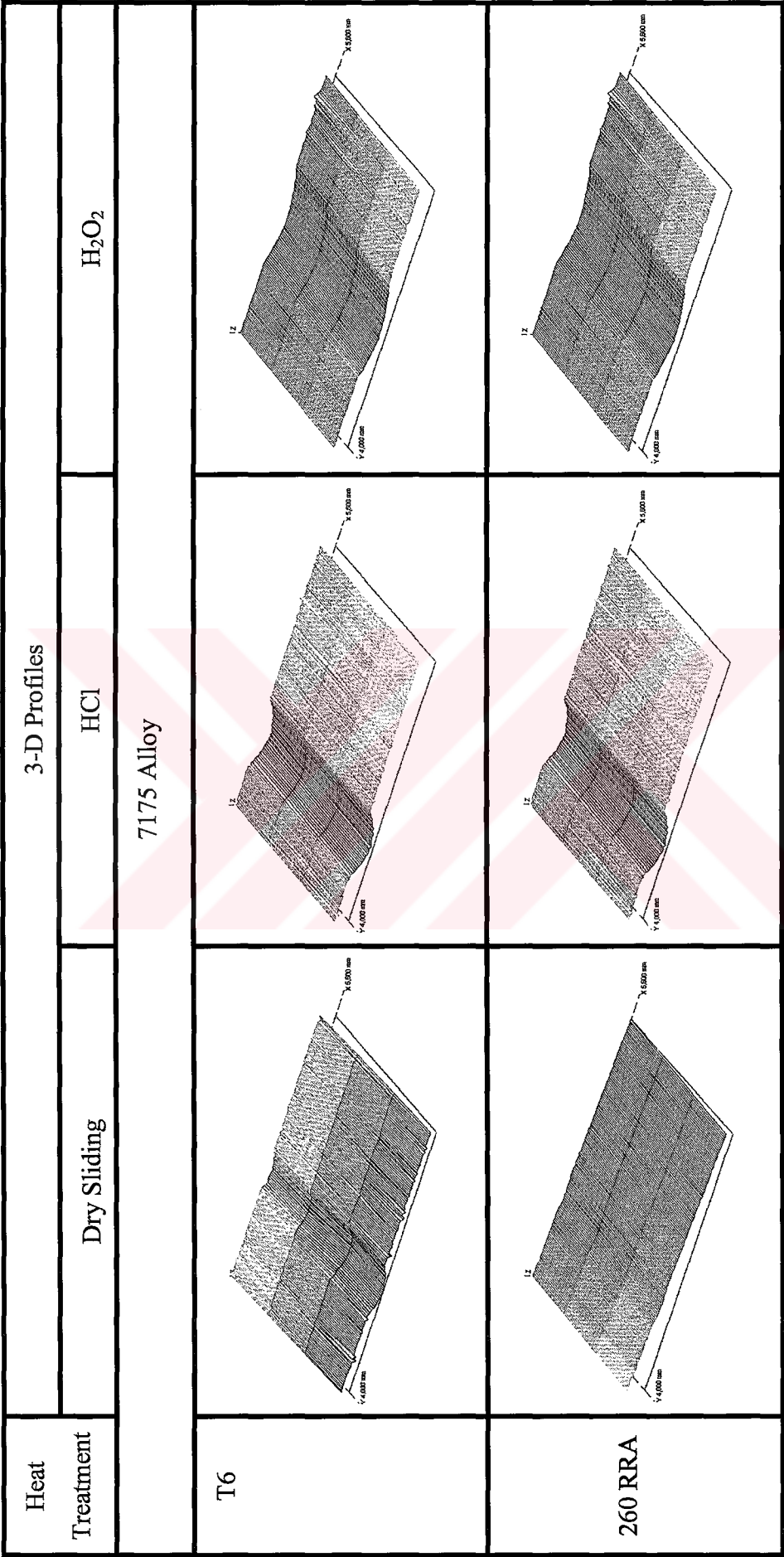


Figure A.5 3-D Profiles of the Wear Tracks of Investigated 7175 Alloys.

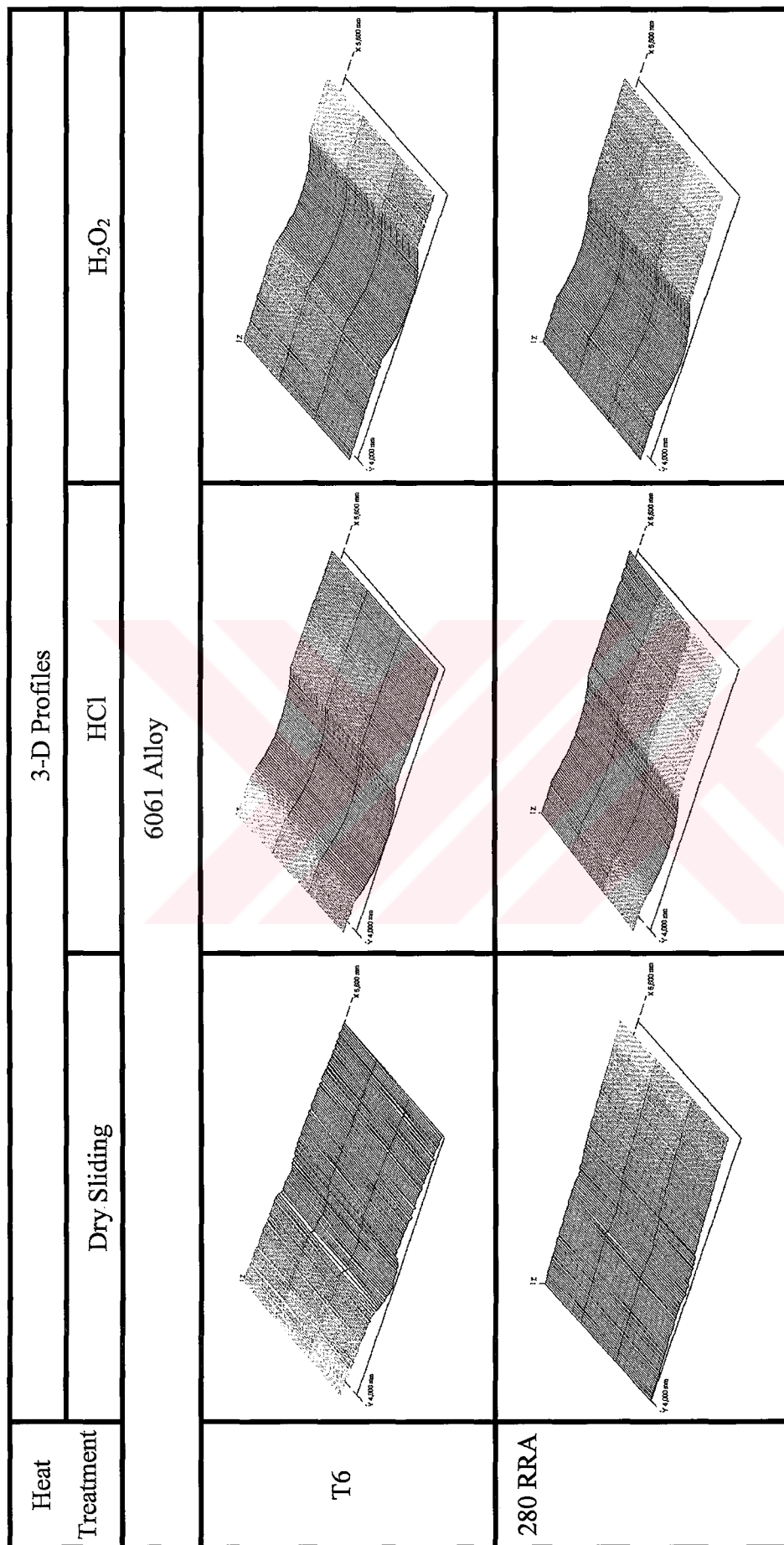


Figure A.6 3-D Profiles of the Wear tracks of Investigated 6061 Alloys.

BIOGRAPHY

İlke DAĞLI was born in Balıkesir Turkey in 1980. She graduated from Sırrı Yırcalı Anatolian High School in 1998 and in the same year attended to Aerospace Engineering Department of İstanbul Technical University. She was honored the degree of B.Sc. in 2002. She started her graduate study at İstanbul Technical University Mechanical Engineering in Materials and Manufacturing Programme.

